APPLICATION OF FTIR-ATR SPECTROSCOPY FOR THE QUALITY CONTROL OF WINE SPIRITS, BRANDIES AND GRAPE MARC SPIRITS

Ofélia Anjos[1],Ilda Caldeira[2]

[1] Polytechnique Instituto of Castelo Branco (IPCB), Castelo Branco, Portugal,
ofelia@ipcb.pt
[2]Instituto Nacional de Investigação Agrária e Veterinária I.P (INIAV), Dois Portos,
Portugal, ilda.caldeira@iniav.pt

This study attempted to apply a rapid method for the determination of methanol, acetaldehyde and ethyl acetate in wine spirits, brandies and grape marc spirits, using Fourier Transform Infrared – Attenuated Total Reflectance (ATR) Spectroscopy.

The current quality control of the brandies includes the use of several analytical methods that are time consuming and sometimes they employ very expensive chemicals. In order to solve this problem it is possible use some different analytical techniques, namely the Fourier transform infrared (FTIR) spectroscopy with ATR.

76 samples of wine spirits, brandies and grape marc spirits were analyzed by CG-FID according to the method validated by Luis et al (2011). GC-FID analysis was carried out using an Focus GC gas chromatograph (Thermo Scientific, USA) equipped with a flame ionization detector-FID (250ºC) and a fused silica capillary column of polyethylene glycol (DB-WAX, JW Scientific, Folsom, CA, USA), 60 m length, 0.32 mm i.d., 0.25 μm film thickness with the conditions described by Luis et al (2011).

Duplicate spectra per sample were obtained with 32 scans per spectrum at a spectral resolution of 4 cm^{-1}. The calibration model were developed by PLS-R using the Bruker OPUS Quant software by comparing the analytical results of methanol, acetaldehyde and ethyl acetate with the FTIR–ATR spectra, first in all spectral region (4000 cm^{-1} to 400^{-1}) and after by selection of the better corresponding spectral zone and the better preprocessing method. The model was calculated to a maximum rank of 5 and the results of cross-validation were evaluated by the coefficient of determination (r^2), root mean square error of cross-validation (RMSECV) mean square error of prediction (RMSEP) and residual prediction deviation (RPD).

The average ATR-FTIR spectrum of the all wine spirits, brandies and grape marc spirits show representative peaks at 2968 cm^{-1} and 2906 cm^{-1} which is due to C–H stretch. Absorption peak at 1632 cm^{-1} is associated to –OH deformation and C–OH peak at 1452 cm^{-1}. The peaks, present at 1084 cm^{-1} and 1045 cm^{-1}, are attributed to the C–O stretch.

Since the major components absorption analyzed the partial least squares regression was done in the selected region of 3000 to 2800 cm^{-1} and 2000 to 1000 cm^{-1} and the selected models were first derivative + multiplicative scatter correction and second derivative.

The calibration statistic obtained for methanol, acetaldehyde and ethyl acetate were obtained by PLS-R modelling with high coefficients of determination of the cross-validation (R^2= 0.9659 to R^2=0, 7957; RPD=6,9 to RPD=2,5). The external validation was made also with promising results. The values of RMSECV and RMSEP was similar of the obtained for the laboratorial analytical methods develop with GC-FID.

The results show that FTIR-ATR could be a good technique to screening the methanol, acetaldehyde and ethyl acetate concentration in brandies sample. The more robust calibration model was found for methanol concentration.

Keywords: methanol, acetaldehyde, ethyl acetate; brandies, FTIR-ATR spectroscopy

Luís A, Mota D, Anjos O, Caldeira I, 2011. Single-Laboratory Validation of Determination of Acetaldehyde, Ethyl Acetate, Methanol and Fusel Alcohols in Wine Spirits, Brandies and Grape Marc Spirits Using GC-FID. Ciência e técnica vitivinícola/journal of viticulture and enology. 26(2): 69-76