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Evaluation of extractable polyphenols released to wine from cooperage byproduct by near infrared hyperspectral imaging

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ABSTRACT

Extractable total phenolic content of American non-toasted oak (*Quercus alba* L.) shavings has been determined using near infrared hyperspectral imaging. A like-wine model solution was used for the simulated maceration procedure. Calibrations were performed by partial least squares regression (MPLS) using a number of spectral pre-treatments. The coefficient of determination of wood for extractable total phenolic content was 0.89, and the standard error of prediction was 6.3 mg g^{-1}. Thus, near infrared hyperspectral imaging arises as an attractive strategy for predicting extractable total phenolic content in the range of 0–65 mg g^{-1}, of great relevance from the point of view of quality assurance regarding wood used in the wine sector. Near infrared hyperspectral imaging arises as an attractive strategy for the feasibility of enhancing the value of cooperage byproduct through the fast determination of extractable bioactive molecules, such as polyphenols.

1. Introduction

Wood is the second great natural source of phenols for wine. In addition to aromatic compounds, wood releases to wine phenolic acids and hydrolysable tannins, which are not naturally found in grape. These compounds are involved in the stabilization of wine color by means of co-pigmentation processes and protection against oxidation and they play an important role in the astringency of wines (Ribérau-Gayon, Glories, Maujean, & Dubourdieu, 2000; Zamora, 2003).

Oak wood has been used for centuries for the construction of barrels where the wine is stored during aging. Although the original function of the barrels was the mere storage and transport of wine, it has been shown that during barrel aging, wine undergoes a series of transformations leading to important changes in aroma, color, taste, and astringency (Glabasnia & Hofmann, 2006; Puech, Feuillat, & Mosedale, 1999; Zamora, 2003). Since the European Community approved the addition of wood chips to produce accelerated aging (CE 2165/2005 and CE 1507/2006), it has become an extended practice (Bautista-Ortín et al., 2008; Del Álamo, Nevares, Gallego, Martin, & Merino, 2008; Frangipane, Santis, & Ceccarelli, 2007; Guchu, Díaz-Maroto, Pérez-Coello, González-Viñas, & Ibáñez, 2006; Rodríguez-Bencomo, Ortega-Heras, Pérez-Magarino, & González-Huerta, 2009).

Quercus genus is widely distributed all over the world. However, the main producing areas of the three oak species used for the production of barrels are France and the United States of America. In fact, in cooperated the terms French oak and American oak are widely used. Alternative sources of wood for barrel making have been assayed, including mainly chestnut, acacia and cherry, in an effort to give a particular personality to aged wines (Alañón, Castro-Vázquez, Díaz-Maroto, & Pérez-Coello, 2012; De Rosso, Panighel, Vedova, Stella, & Flamini, 2009; Fernández de Simón, Martínez, et al., 2014; Fernández de Simón, Sanz, et al., 2014; Rodríguez-Bencomo et al., 2009). Even so, most of the oak barrels that are marketed at this time continue to be oak wood and, in particular, American white oak (*Quercus alba* L.) and European oak (*Quercus robur* L.).

Winemakers are continuously looking for high quality wines. In geographical areas with typical climatological conditions of warm climate, such as Andalucía, the stressful climate increases the difficulty of obtaining high quality red wines because of color instability over time (Gordillo et al., 2012). In these warm regions, the phenolic maturity does not coincide with the technological maturity of grapes, therefore, at the moment of harvesting, different levels of both phenolic and sugar maturity exist (Mira de Orduña, 2010; Mori, Sugaya, & Gemma, 2005). Thus, copigmentation phenomena, which contribute to color stabilization, are hampered by the shortfall of pigments and copigments (Boulton, 2001). Taking that into account, an extra contribution of phenolic compounds might be necessary to partially solve the problem. The external addition of phenolic compounds from natural sources to the musts has been reported for the purpose of color stabilization (Canals, Llaudy, Canals, & Zamora, 2008; Cejudo-Bastante et al., 2016;

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Gordillo, Cejudo-Bastante, Rodriguez-Pulido, Gonzalez-Miret, & Heredia, 2013; Gordillo et al., 2014; Pedroza, Carmona, Alonso, Salinas, & Zalacain, 2013). However, the use of raw wood shavings, cooperage byproduct, to get high quality red wines is not so frequent.

Wood processing for the elaboration of barrels generates byproducts which could be burned to meet energy needs in the wood industries but in most cases they are directly disposed as waste products. It is known that cooperage byproduct contain many compounds whose extraction could create new business opportunities. In this context, oak barrel-shoot wastes seem to be an interesting product due to its potential to release high added value compounds for wine.

Factors such as oak species, geographic origin, seasoning and toasted degree have been shown to affect the chemical composition of the oak wood used (Del Alamo Sanza, Nevares Domínguez, Cárcel Cárcel, & Navas Gracia, 2004; Del Álamo Sanza & Nevares Domínguez, 2006; Fernández de Simón, Cadahía, del Álamo, & Nevares, 2010; Frangipane et al., 2007) and even various anatomical parts of wood may differ in terms of concentration and composition (Colares et al., 2016). However, the characteristics of the final wines depend not only on the composition of the oak wood used but also on the amount of phenolic compounds that may be extracted from wood to the must. This transfer processes could be affected by contact surface between wine and wood, alcoholic degree, duration time etc. The contact surface depends on the size of the oak fragment and on the amount used (Arapitsas, Antonopoulos, Stefanou, & Dourtoglou, 2004; Bautista-Ortín et al., 2008).

Due to the importance in the different productive sectors, the qualitative and quantitative characterization of woods applying fast and efficient analytical methods has been required. Spectroscopic methods based on near-infrared spectroscopy (NIR) have shown great potential for qualitative and quantitative analyses of wood. The rapid assessment of the chemical composition using NIR in applications related to the quality has provided an increasing number of publications for industrial applications in this area (Da Silva et al., 2013; Jones, Schimleck, Peter, Daniels, & Clark, 2006; Sun, Liu, Liu, & Yang, 2011; Yao et al., 2010). In a further step, near infrared hyperspectral imaging has also been applied for analyses of wood (Lestander, Geladi, Larsson, & Thyrel, 2012; Thumm, Riddell, Nanayakkara, Harrington, & Meder, 2010). References for the use of this technique in the analysis of byproducts of oak wood for oenological purposes have not been found, although in the literature the use of conventional near infrared techniques is described for the quantification of oak wood extractives (Michel et al., 2013; Zahri et al., 2008) and for the correlations between NIR quantification and classification with extractive level in wood, (Giordanengo et al., 2009). Correlation between oak wood extractives and oxygen consumption of wine aged in contact of wood in relation of the classified wood has been already reported in some studies (Michel et al., 2016; Navarro et al., 2016). In none of these studies, the assignment of these compounds from wood to wine is estimated by the use of spectral techniques recording the spectrum of the wood. A rapid estimation of wood extractable content of phenolic compounds could assist in selecting oak wood suited for improving wine quality through the addiction of external copigments to wine. For the same purpose near-infrared hyperspectral imaging has been applied previously in our lab in order to predict different parameter in grapes (Hernández-Hierro, Nogales-Bueno, Rodríguez-Pulido, & Heredia, 2013; Nogales-Bueno, Baca-Bocanegra, Rodríguez-Pulido, Heredia, & Hernández-Hierro, 2015; Nogales-Bueno, Hernández-Hierro, Rodríguez-Pulido, & Heredia, 2014).

The aim of this study is to look at the feasibility of enhancing the value of cooperage byproduct through the extraction of bioactive molecules, such as polyphenols. To this end near infrared hyperspectral imaging has been used for the screening of the extractable content of phenolic compounds in raw wood.

2. Material and methods

2.1. Samples

American non-toasted oak (*Quercus alba* L.) shavings, cooperage byproduct, were used for this study. Samples were provided by Toneleria Salas S.L. (Bollullos Par del Condado, Huelva, Spain). Wood staves were seasoned under natural conditions in the open air during 24 months approximately before being used in the process of making barrels. In order to achieve representative sample sets, the samples were taken at 4 different points in the process of making barrels, so four samples were collected at each date (A, B, C and D). Samples belonging to A, B and C groups are generated by different sawing processes of the staves but always in the longitudinal direction of the fibers. In detail, an automatic or manual processing of the staves is the only difference between the samples A and B respectively. With respect to the group D, the samples are the result of a processing of the staves in the transversal direction of the fibers. All sampling points are pre-molded and pre-toasted of the staves. Samples were collected periodically, between June of 2015 and January of 2016. Two hundred samples were collected during the aforesaid period. Upon receipt, the samples were sieved and subjected to the hyperspectral analysis. Wood shaving samples were screened using 2 mm and 10 mm mesh sieves placed in tandem. Only shaves of each sample which were retained between them were taken into account in the assay. After that, wood shavings were placed in stoppered plastics bags and stored in a dry chamber until use.

2.2. Hyperspectral image acquisition

Hyperspectral imaging device (Infaimon S.L., Barcelona, Spain) comprised a Xenics® XEVA-USB InGaAs camera (320 × 256 pixels; Xenics Infrared Solutions, Inc., Leuven, Belgium), a spectrograph (Specim ImSpector N17E Enhanced; Spectral Imaging Ltd., Oulu, Finland) covering the spectral range between 900 and 1700 nm (spectral resolution of 3.25 nm), two 70 W tungsten iodine halogen lamps (Prilux, Barcelona, Spain) mounted as source light, a mirror scanner (Spectral Imaging Ltd.) and a computer system. The hyperspectral image of each sample on a polyethylene plastic was recorded. Equipment and procedure used to image recording are described in detail elsewhere in Hernández-Hierro et al. (2013).

After calibration and segmentation processes, the average spectral profile for each sample was saved. Noisy wavebands at both extremes of the spectra range were removed and only spectral data in the resulting effective wavelength 950–1650 nm regions were used in data analysis due to reduced efficiency outside this range in the used device.

2.3. Extractable total phenolic content

Wood shavings were immersed in a model wine hydroalcoholic solution (4 g L^{-1} tartaric acid, 12.5% ethanol, adjusted at pH 3.6 with NaOH 0.5 M) for a maceration period of 72 h. Oak wood shavings were added to the wine-like solution in a 4 g L^{-1} ratio. The supernatant was used in the analysis. Extractable total phenolic content was determined using the Folin-Ciocalteu method (Singleton & Rossi, 1965). For quantification, results were expressed as mg of gallic acid equivalents per gram of wood.

2.4. Image processing and data analysis

2.4.1. Image processing

Prior to the qualitative and quantitative analysis, a discriminant method was applied to the shaving images to isolate the shavings from other parts of the image. Firstly, a set of reflectance spectra belonging to shaving and background (regions of interest (ROIs)) were selected to develop a stepwise lineal discriminant model. The aforementioned discriminant model classified each pixel into two classes (sample or no

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