



HAL
open science

Characterization of enological oak tannin extracts by multi-analytical methods approach

Hélène Hallé, Kevin Pascotto, Aude Watrelot, Aurélie Roland, Emmanuelle Meudec, Pascale Williams, Stéphanie Carrillo, Bertrand Robillard, Nicolas Sommerer, Céline Poncet-Legrand, et al.

► To cite this version:

Hélène Hallé, Kevin Pascotto, Aude Watrelot, Aurélie Roland, Emmanuelle Meudec, et al.. Characterization of enological oak tannin extracts by multi-analytical methods approach. OENO Macrowine 2023, Jul 2023, Bordeaux, France. hal-04167983

HAL Id: hal-04167983

<https://hal.inrae.fr/hal-04167983>

Submitted on 21 Jul 2023

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution| 4.0 International License

Title :

Characterization of enological oak tannin extracts by multi-analytical methods approach

Hélène Halle^{a,b}, Kevin Pascotto^c, Aude Watrelot^{a,b,d}, Aurélie Roland^{a,b}, Emmanuelle Meudec^{a,b}, Pascale Williams^a, Stéphanie Carrillo^a, Bertand Robillard^c, Nicolas Sommerer^{a,b}, Céline Poncet-Legrand^a, Véronique Cheynier^{a,b}

^a Univ. Montpellier, SPO, INRAE, Institut Agro Montpellier Supagro, 34070 Montpellier, France

^b INRAE, PROBE research infrastructure, PFP polyphenols analysis facility, 34070 Montpellier, France

^c Institut Œnologique de Champagne, Epernay, France

^d Iowa State University, Department of Food Science and Human Nutrition, Ames, USA

Oak tannin extracts are commonly used to improve wine properties. The main polyphenols found in oak wood extracts are ellagitannins¹ that release ellagic acid upon hydrolysis and comprise numerous structures². Moreover, oak tannin extracts contain other compounds giving a complex mixture. Consequently, the official OIV method based on gravimetric analysis of the tannin fraction adsorbed on polyvinylpyrrolidone is not sufficient to describe their composition and highlight their chemical diversity.

Eight commercial oak tannins were characterized by a combination of analytical approaches. Polyphenols were analyzed using the official OIV method, UV spectrophotometry, UPLC-UV-MS analysis before and after acidic methanolysis, and HPLC-SEC-UV. Neutral sugars and polyols were determined as alditol acetates by GC-FID analysis, before and after hydrolysis. Protein content was estimated by the Kjeldahl method. Finally, samples were compared by a non-targeted metabolomic approach based on UHPLC-HRMS/MS.

Gravimetric analysis, absorbance values at 280 nm, and the quantities of ellagic acid released by methanolysis revealed some differences between samples, indicating variations in their tannin composition. This was confirmed by HPLC-SEC-UV analysis evidencing differences in tannin size distribution, particularly in larger polymer content.

All samples contained significant quantities of sugars, and in particular xylose, mostly found in the linked form, and of quercitol, a polyol marker of oak origin. These compounds contributed to up to 25% of the whole extract composition, the proportions of free and combined sugars and polyols also showing large variations between tannins. The protein content was very low, generally representing less than 1% of the mass.

Non targeted UPLC-HRMS analysis detected major ellagitannins such as vescalagin, castalagin, and roburins A-E, but also a large number of derivatives as well as other molecules such as lignans and quercotriterponosides, and highlighted large differences between samples. Tannin extracts also contained aldehydes (HMF, furfural, syringaldehyde, sinapaldehyde, vanillin) in variable quantities.

This work demonstrates the variability in the composition of commercial oak tannin extracts, likely to impact their properties, and emphasizes the need for detailed multi-method characterization in the frame of quality control and selection of tannins for specific applications.

- (1) Simón, B. F. de; Cadahía, E.; Conde, E.; García-Vallejo, M. C. Ellagitannins in Woods of Spanish, French and American Oaks. **1999**, *53* (2), 147–150.
<https://doi.org/doi:10.1515/HF.1999.024>.
- (2) Moutounet, M.; Souquet, J.-M.; MEUDEC, E.; Leaute, B.; DELBOS, C.; Doco, T. Analyse de La Composition de Tanins Oenologiques. *Rev. Fr. Oenologie* **2004**, No. 208, 22–27.