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

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Title :

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

Oak tannin extracts are commonly used to improve wine properties. The main polyphenols found in oak wood extracts are ellagitannins that release ellagic acid upon hydrolysis and comprise numerous structures. 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. 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 SEC 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.

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 quercotriterpenosides, and highlighted large differences between samples.

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.

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