LWT - Food Science and Technology 91 (2018) 330–338

Contents lists available at ScienceDirect

LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt

Oak barrel tannin and toasting temperature: Effects on red wine condensed tannin chemistry

Aude A. Watrelot[a,*], Marie-Laure Badet-Murat[b], Andrew L. Waterhouse[a]

[a] Department of Viticulture and Enology, University of California Davis, One Shields Ave., Davis, CA 95616-5270, USA
[b] Oenologie by MLM, 25 rue Chazeau, 33160 Saint-Médard-en-Jalles, France

ARTICLE INFO

Keywords:
Ellagitannins
Oak wood
Flavan-3-ol polymers
Tannin activity
Molecular mass

ABSTRACT

Red wine is known to derive specific aromas and to have its color enhanced by oak wood compounds, but the effect of oak barrels on wine tannin chemistry is not well known. Cabernet Sauvignon wines were aged from 8 to 12 months in oak barrels made with three different ellagitannin levels (Tannin Potential, TP) and two toasting levels. Condensed tannin composition, activity and ellagitannin contents have been characterized using HPLC-DAD and protein precipitation. Red wines aged in high TP barrels contained more ellagitannins, condensed tannins with lower mass and lower pigmented tannins percentage than in low TP barrels. Red wines aged in high-temperature toasted barrels contained less ellagitannins, condensed tannins with higher mass and higher pigmented tannins percentage than in low-temperature toasted barrels. Combined effects of ellagitannin, toasting levels and aging have been identified. The selection of barrels for ellagitannin content and toasting temperature can be used to manage red wine development.

1. Introduction

Red wine quality is usually defined as the sensory perception of finished wine in the mouth. Condensed tannins are the most important macromolecules which influence the bitterness/mouthfeel perception of red wine (Vidal et al., 2003). They are oligomeric and polymeric forms of flavan-3-ols ((−)-epicatechin, (+)-catechin, (−)-epigallocatechin and (−)-epicatechin-3-*O*-gallate), linked mainly by C4−C8 linkages. Depending on the degree of polymerization, average number of constitutive units, condensed tannins can interact more or less with salivary proteins and induce an astringent mouthfeel (Poncet-Legrand et al., 2010). This tannin perception can vary by the tannin structure or their behavior in solution, caused by exterior parameters such as oxidation reactions (Poncet-Legrand et al., 2010) and interactions with other macromolecules (Carvalho et al., 2006).

For thirty years, research work devoted to the study of wood molecules giving nuances and flavors to wine has identified a number of impact compounds. Among aromatic compounds released by wood, the *trans*- and *cis*-whisky lactone, vanillin, eugenol and isoeugenol (Prida & Chatonnet, 2010; Spillman, Sefton, & Gawel, 2004); 5-hydroxymethylfurfural, which contributes to wine's toasty character, and furfural and 5-methylfurfural, involved in the reduction of the fruity character (Prida & Chatonnet, 2010). However, ellagitannins, the major non-volatile extractives from oak, also play a major role with regard to astringency and mouthfeel (Glabasnia & Hofmann, 2006, 2007; Michel, Jourdes, Giordanengo, Mourey, & Teissedre, 2012; Mosedale, Puech, & Feuillat, 1999). These are hydrolyzable tannins, characterized by one or more hexahydroxydiphenoyl (HHDP) moieties esterified with a sugar, typically glucose (Michel et al., 2011). Ellagitannins are easily extractable in red wine during aging as they are soluble in hydroalcoholic solution. Due to their structure, such as the presence of the galloyl functional groups, it has been proposed that they are involved in the oxidation process by being the first compound in red wine to be oxidized. Indeed, the oxidation of the pyrogalloyl ring at the glucose C1 position of the castalagin lead to a cyclopentenone moiety, forming oxidation products, whiskey tannins (Fujieda, Tanaka, Suwa, Koshimizu, & Kouno, 2008). They are also known to be highly reactive with other flavonoids by reacting with condensed tannins through condensation reactions to form flavano-ellagitannin products (Ishimaru et al., 1988; Saucier, Jourdes, Glories, & Quideau, 2006) and with anthocyanins to stabilize red wine color (Dumitriu, Lerma, Cotea, Zamfir, & Peinado, 2016), such as 1-deoxyvescalagin-malvidin. The levels of these compounds in wines after aging depend on many parameters including raw material (wood) content, and toasting. When hydrolyzed from the glucose, HHDP spontaneously lactonizes to ellagic acid in aqueous solution, and these reactions can be used to simplify the quantification of ellagitannins (Mämmelä, Savolainen, Lindroos, Kangas, & Vartiainen, 2000).

* Corresponding author.
E-mail address: awatrelot@ucdavis.edu (A.A. Watrelot).

https://doi.org/10.1016/j.lwt.2018.01.065
Received 27 July 2017; Received in revised form 18 January 2018; Accepted 20 January 2018

Regarding the raw material, the variability in chemical composition of oak wood is now well known; both between trees (Doussot, De Jéso, Quideau, & Pardon, 2002; Michel et al., 2012; Snakkers, Nepveu, Guilley, & Cantagrel, 2000) and within the same tree (Masson, Moutounet, & Puech, 1995; Mosedale, Charrier, Crouch, Janin, & Savill, 1996), as well as between the resulting barrel (Towey & Waterhouse, 1996). Recent studies based on the classification of cooperage oak according to its ellagitannin levels confirmed a high variability in levels of these compounds (Michel et al., 2013), and demonstrated the influence of the tannins on red wine oral sensation. Wood ellagitannins have been observed to be related to red wine astringency perception either by direct interaction with salivary proteins, or by combination with condensed tannins that lead to synergized interaction with salivary proteins (Chira & Teissedre, 2015). In addition, some major changes in wood chemical composition occur during toasting. While thermal degradation of wood macromolecules generates many of the important oak aroma compounds (furanics, phenolic aldehydes, and phenols), the ellagitannins are also affected, and are partially degraded by heavy toasting conditions (temperature, duration and level of wood moisture) (Chira & Teissedre, 2013; Fernández de Simón, Cadahía, del Álamo, & Nevares, 2010). By changing the thermal profile, it is therefore possible to modulate the aromatic and taste impact of the barrels (Chira & Teissedre, 2015).

However, the interactions between oak barrel ellagitannin composition on condensed tannins properties have not been studied yet despite the impact of ellagitannins and condensed tannin structure on red wine mouthfeel perception (Harbertson, Parpinello, Heymann, & Downey, 2012; Rinaldi, Gambuti, Moine-Ledoux, & Moio, 2010). Here, two red wines with different tannin concentration were aged for 8–12 months in oak barrels made with staves of three different ellagitannin levels and toasted at two temperature levels, and the resulting wines were evaluated for tannin concentrations, molecular weight, and activity, and the formation of wine pigments.

2. Material and methods

2.1. Reagent and chemicals

All solvents were HPLC grade. Acetonitrile, methanol, acetic acid, L-(+) ascorbic acid, hydrochloric acid, lithium chloride, *o*-phosphoric acid, *N,N*-dimethylformamide, and anhydrous sodium acetate were purchased from VWR International (Radnor, PA). Phloroglucinol, (−)-epicatechin, (+)-catechin hydrate and ellagic acid were purchased from Sigma-Aldrich (St. Louis, MO). Procyanidin B1 and (−)-epicatechin-3-*O*-gallate were purchased from Extrasynthèse (Lyon, France).

2.2. Oak barrel characteristics

The commercial barrels (225 L) were made from woods coming from French forests (90%) and neighboring countries (10% mainly Germany) that were naturally seasoned for 30 months.

2.2.1. Oak wood sorting methodology according to its tannin potential (TP)

Wood classification according to its ellagitannin content was performed by near-infrared spectroscopy (NIRS) using a technique based on the use of an acousto-optic tunable filter (AOTF, Brimrose, USA) (Michel et al., 2011). After machining, the untoasted staves were first being intended to gather spectral data and then ellagitannins total level analyzed through ellagic acid dosage in HPLC-DAD after extraction and hydrolysis in acidic medium (method described in 2.3.1.). The calibration was performed from a partial least squares (PLS) regression after selecting the most discriminant spectral zones. The correlation coefficient between spectral measurement and HPLC dosage of total ellagitannins (0.89) shows the performance of the model used. The classification from the NIRS method was used to sort the staves into three groups of tannin potential (TP) i.e. the ellagitannin content in untoasted wood: Low or LTP < 4000, Medium or MTP from 4001 to 6000 and High or HTP from 6001 to 8000 μg of ellagic acid equivalent/g of dry wood. The thickness of staves was of 22 mm and the porosity of staves was fine grain (visual classification: < 2 mm) for LTP and MTP barrels and medium grain (visual classification: 2–3 mm) for HTP barrels. The bottoms of the barrels contained the same tannin potential but were not toasted.

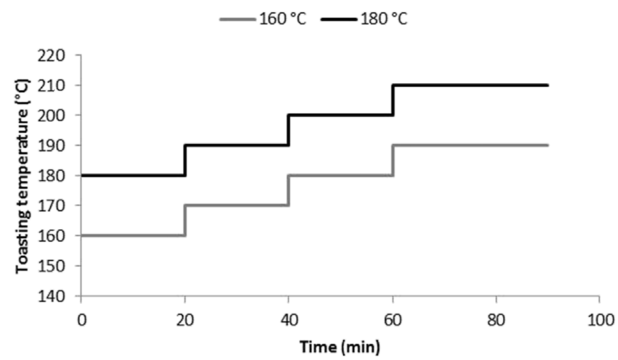

Fig. 1. Toasting temperature versus time of oak barrel toasted at 160 and 180 °C as initial temperature.

2.2.2. Wood toasting methodology

Toasting was made after a 4 min automated steam bending, which allows obtaining a neutral white barrel inside. Toasting, controlled by computer, was performed using radiant heat rather than direct contact with flame. The toasting pot was fed regularly with fuel (100% oak pellets) by an auger. The barrel, placed on a turntable, rotates around a double cone that covers the fire and channels the heat source, during the entire toasting phase. An infrared sensor, performing measurements on the internal surface of the shell, provides temperature control, with a heating accuracy of ± 3 °C. In our study, all barrels (LTP, MTP and HTP) underwent a gradual toasting (G) with a temperature increase of 10 °C every 20 min (Fig. 1), and two initial temperatures were compared (160 and 180 °C).

2.3. Red wine winemaking and characterization

Red wines were made by two Californian wineries (B and CR) from 2015 Cabernet Sauvignon grapes of Napa Valley. The alcoholic fermentation was made using Zymaflore F15 and D254 yeast strains in B and CR wines, respectively. Spontaneous malolactic fermentations occurred after yeast fermentation was complete. Sulfur dioxide, 60 mg/L, was added to the wines prior to barreling and three wine samples of each winery were analyzed. The wine from each winery was barreled in 18 barrels (225 L) (triplicate of 6 modalities). Barrels were topped every month (with the same wine coming from another barrel), racked every three months and samples were analyzed each time. B wines were aged for 12 months in 18 barrels (triplicate of 6 modalities) and CR wines were aged for 8 months in 18 barrels (triplicate of 6 modalities) (Fig. 2). The cellar humidity was around 80%. pH, ethanol content and titratable acidity were determined using a Winescan FT120 (FOSS, Eden Prairie, Minnesota, USA) for each modality and replicate, and the average and standard deviation of the eighteen wines (from 18 barrels: triplicates of 6 barrel modalities) was used to compare these parameters between wineries.

Prior to barreling, B and CR wines contained 15.4 and 14.8% (v/v) of ethanol, had a pH of 3.9 and 3.7, and a titratable acidity of 5.3 and 4.9 g/L, respectively. Surprisingly, after 12 and 8 months of aging of B and CR wines, the pH values had significantly decreased down to 3.7 and 3.6 respectively. The ethanol content had slightly decreased in B wines to 15.2% v/v and increased in CR wines (14.9% v/v). Titratable acidity increased with aging up to 6.0 and 5.9 in B and CR wines