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Effects of irrigation over three years on the amino acid composition of Treixadura (*Vitis vinifera* L.) musts and wines, and on the aromatic composition and sensory profiles of its wines

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ABSTRACT

Amino acids and volatile compounds play an important role in wine aroma and sensory characteristics. The concentrations of these compounds might be altered by climate interanual variability and by management practices such as irrigation. The aim of the current study was to assess the amino acid profile of musts and wines, volatile composition and sensory profile of wines from *Vitis vinifera* (L.) cultivar 'Treixadura' obtained from vines under rain-fed and irrigation conditions over three consecutive vintages (2012–2014). Musts and wines from the irrigation treatment tended to be higher in acidity than those from rain-fed. However, amino acid and aromatic profiles were mostly affected by climate conditions of each year; although irrigation exerted a significant effect on several compounds (proline, cysteine, tryptophan, phenylalanine, α-terpineol and geraniol). Wines from both treatments received similar marks in the sensory tests, suggesting that irrigation did not greatly modify wine quality under the conditions of this study.

1. Introduction

Amino acids in musts play a role of paramount importance during alcoholic fermentation (Bell & Henschke, 2005). They constitute a nitrogen source for yeasts and their concentration depends on a great number of factors including grapevine variety, fertilization, soil and climate conditions, vineyard management and enological practices (Garde Cerdán et al., 2014). Moreover, the amino acid profile of the musts will influence final wine quality due to the transformations that amino acids undergo during the fermentation process, producing volatile compounds such as higher alcohols, volatile fatty acids and ethyl esters (Swiegers, Bartowsky, Henschke, & Pretorius, 2005) that define wine aroma.

Higher alcohols are the most abundant volatile compounds produced during alcoholic fermentation. When the total concentration of these compounds is below 300 $mg\,L^{-1}$ they intensify floral notes in wines, whereas they are negative to wine quality when their concentration surpasses that threshold (Hirst & Ritcher, 2016). Ethyl esters are responsible for floral and fruity nuances in young wines (Falqué, Darriet, Fernández, & Dubourdieu, 2008); whereas volatile fatty acids might be negative to wine quality when they are found over their perception threshold, providing cheesy and buttery notes to wines.

Volatile compounds accumulate in grapes during ripening depending on temperature and water availability (Robinson et al., 2014). Therefore, irrigation management in the vineyard is a fundamental tool for controlling berry growth and quality (Jackson & Lombard, 1993). In fact, concerns about climate change altering the temporal distribution of rainfall and increasing the drought events have raised a great concern in viticultural regions worldwide (Fraga, Malheiro, Moutinho-Pereira, & Santos, 2013) and irrigation is increasingly being used to cope with this and to minimize interannual variability in yields even in cool humid regions such as Galicia, northwest Spain (Cancela et al., 2016). Nevertheless, the effects that irrigation practices might have on berry composition and wine aromatic profile have seldom been studied,

Abbreviations: ALB, Albariño; AOC, Appellation d'Origine Controllée; DEEMM, diethyl ethoxymethylenemalonate; AOC, Appellation of Origin; EFC, electronic flow control; ET_c, crop evapotranspiration; ET_0, potential evapotranspiration; GC, Gas Chromatography; HPLC, High-Performance Liquid Chromatography; IS, Internal Standard; LOD, Limit of Detection; LOQ, Limit of Quantification; MS, mass spectrophotometer; OAV, Odour Activity Value; PCA, Principal Component Analysis; PC, Principal Component; UV, Ultraviolet. Amino acids; Arg, arginine; GABA, aminobutyric acid; Thr, threonine; Ala, alanine; Ser, serine; Glu, glutamine; Glu acid, glutamic acid; Val, valine; Phe, phenylalanine; Leu, leucine; His, histidine; Asp acid, aspartic acid; Ile, isoleucine; Try, tryptophan; Tyr, tyrosine; Asp, asparagine; Lys, lysine; Cys, cysteine; Pro, proline; Gly, glycine; Orn, ornithine; Met, methionine

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and contrasting results have emerged, depending on the cultivar and colour of the grapes (Deluc et al., 2009) and on the intensity of water stress, which is usually low in cool-humid areas (Balint & Reynolds, 2017). In this sense, Matthews, Ishii, Anderson, and O'Mahony (1990) concluded that irrigation can modulate wine sensory characteristics, but Talaverano et al. (2017) observed that irrigation only exerted a significant influence on alcohols and C6 compounds. Studies on white varieties are scarcer; Ortega Heras et al. (2014) reported that irrigation increased the amino acid concentrations in berries of the cultivar Verdejo, which could have altered the wine aromatic composition. Furthermore, water stress affected amino acids metabolism of the red cultivar Cabernet Sauvignon but not that of the white cultivar Chardonnay (Deluc et al., 2009); basically, proline concentrations increased with water deficit.

In the Northwest of the Iberian Peninsula (Galicia and North of Portugal), white grapevine cultivars are predominantly grown and their organoleptic characteristics might be altered by increasing temperatures and the occurrence of drought periods during the growing cycle. Among the cultivars that are traditionally grown in these regions, Treixadura is one of the most important since it is used to obtain balanced wines with a high aromatic potential (Falqué, Fernández, & Dubourdieu, 2002). Volatile composition of wines produced with this cultivar has already been described (Blanco, Mirás-Avalos, Suárez, & Orriols, 2012), but the amino acid profile of the musts and wines, as well as the effect that irrigation exerts on them has not been previously assessed. Therefore, the aim of the current study was to assess the effects of irrigation on the amino acid composition of musts and wines, and the aromatic and sensory profiles of wines from the white grapevine (*Vitis vinifera* L.) cultivar Treixadura, traditionally grown in Galicia, during three consecutive years (2012, 2013 and 2014) in Ribeiro.

2. Materials and methods

2.1. Chemical reagents

A Milli-Q equipment (Millipore, Bedford, MA, USA) was used for generating ultra-pure water. Super-gradient HPLC grade acetonitrile and methanol were purchased from Scharlau (Sentmenat, Spain). Ammonium chloride was from Merck (Darmstadt, Germany). L-α amino acids: aspartic acid, glutamic acid, asparagine, serine, glutamine, histidine, glycine, threonine, arginine, alanine, γ-aminobutyric acid (GABA), proline, tyrosine, valine, methionine, cysteine, isoleucine, tryptophan, leucine, phenylalanine, ornithine hydrochloride, lysine, 2-aminoadipic acid and diethylethoxymethylenemalonate (DEEMM) were from Acros Organics (New Jersey, USA). Solutions of amino acids were prepared with HCl 0.1N.

For the extraction of free terpenes, volatile fatty acids, ethyl esters, acetates of higher alcohols and C6 alcohols, we used dichloromethane, *n*-pentane and anhydrous sodium sulfate (Scharlau, Sentmenat, Spain). Standards for volatile compounds were purchased from different suppliers: Merck (Madrid, Spain), Aldrich (Madrid, Spain), Fluka (Seelze, Germany), Alfa Aesar (Barcelona, Spain) and Sigma (Madrid, Spain). The internal standards (Merck, Madrid, Spain) used for each method were 4-methyl-2-pentanol in the case of major volatile compounds (methanol, higher alcohols, acetaldehyde, ethyl acetate, ethyl lactate, acetoine, 2-phenyl-ethanol and 2,3-butanediol); 4-decanol for terpenes and C6 alcohols; 1-heptanol for volatile fatty acids, ethyl esters and acetates of higher alcohols. All the standards were prepared in 50% hydroalcoholic solutions.

2.2. Study site and experimental design

The experiment was conducted over three consecutive growing seasons (2012, 2013, and 2014) in a 0.2 ha Treixadura vineyard, located in the experimental farm of the Estación de Viticultura e Enoloxía de Galicia (EVEGA), in Leiro (42°21.6′ N, 8°7.02′ W, elevation 115 m), Ourense, Spain, within the Appellation d'Origine Controllée (AOC) Ribeiro (Mirás-Avalos, Trigo-Córdoba, Bouzas-Cid, & Orriols-Fernández, 2016).

Treixadura vines, grafted in 1998 on 196-17C rootstock, were vertically trellised on a single cordon system (10–12 buds per vine). Rows were East-West oriented; spacings were 1.25 and 2.4 m between vines and rows, respectively. Soil was sandy textured, slightly acidic, and of medium fertility. Treatments consisted of a rain-fed and an irrigation to the 50% of crop evapotranspiration (ET_c), using a crop coefficient equal to 0.8 taken from previous reports on vineyards with developed canopies (Fandiño et al., 2012; Williams, 2012). Each treatment had three replicates (three rows of 8 vines each) in a randomized complete block design. Water was applied with two pressure-compensated emitters of $4\ L\ h^{-1}$ located 25 cm on either side of the vine. Irrigation was applied, according to the former week ET_c from end June to mid-August, approximately two weeks before harvest. Total water amounts applied were 50, 79 and 50 mm for 2012, 2013 and 2014, respectively (Trigo Córdoba, Bouzas Cid, Orriols Fernández, & Mirás Avalos, 2015), allowing for a clear differentiation on grapevine water status under both treatments. Rainfall over the growing season (April to harvest) was 313, 163 and 185 mm for 2012, 2013 and 2014, respectively. Moreover, the mean temperature for the growing season increased from year to year (Supplementary Table 1).

Physiological and production data have been reported elsewhere (Trigo Córdoba et al., 2015). In summary, rain-fed vines showed more negative midday stem water potentials than those irrigated during the three studied years; whereas yield was significantly higher in irrigated vines only in 2014 and this was caused by a greater number of clusters per vine (Supplementary Table 2).

2.3. Samples

Grapes from the different treatments were manually harvested on the same day and transported to the experimental winery. Vinifications were performed separately on samples of about 40 kg per replicate, thus three vinifications per treatment were carried out each year, as previously described for another white variety (Mirás-Avalos et al., 2016).

Briefly, grapes from each replicate were separately destemmed, crushed and pressed in a pneumatic press (yielding approximately 50% must). A replicated 250 mL sample from each treatment was collected for analysis. Pectolytic enzyme was added ($4\ g\ hL^{-1}$) to favor settling. SO_2 ($50\ mg\ L^{-1}$) was added to avoid oxidation. After 24 h, musts were racked and then fermented in 35 L stainless steel tanks. Commercial yeast (Excellence FW, Lamothe-Abiet, Bordeaux, France) was added at a rate of $20\ g\ hL^{-1}$. Density and temperature of fermentations were daily monitored. Once alcoholic fermentation finished, wines were racked and sulphited to $35\ mg\ L^{-1}$ free sulphur dioxide. A natural clarification was carried out at 4 °C for one month. Finally, wines were filtered, bottled and stored.

2.4. Analytical methods

Basic parameters of musts (such as total soluble solids, pH and titratable acidity) and wines (including alcohol content and pH among others) were determined by Fourier transform infrared spectrometry (FTIR) using a WineScan FT120 analyzer (FOSS Electric, Barcelona, Spain) calibrated according to the official methods (OIV, 2009). Analytical determinations in the wines were carried out in triplicate five months after bottling.

2.4.1. Determination of amino acids in musts and wines

The determination of the amino acids present in the Treixadura musts and wines was performed according to Gómez Alonso, Hermosín Gutiérrez, and García Romero (2007) and Garde Cerdán et al. (2009) with slight modifications. This method is based on a reaction of