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Changes in soil water availability in vineyards can be traced by the carbon and nitrogen isotope composition of dried wines

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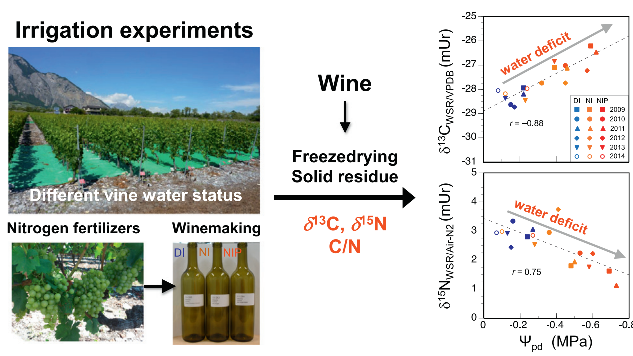
HIGHLIGHTS

- Field experiment simulated soil water restriction in vineyards under climate change.
- ^{13}C and ^{15}N were analyzed in dried wines from diverse vine water status and vintages.
- ^{13}C-enrichment highly correlates with vine water-stress and dryness of vintage summer.
- N content and $\delta^{15}N$ decrease with soil water deficit due to limited nutrient flow.
- Vine in dried soils uses the internal organic N reserves.

GRAPHICAL ABSTRACT



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ABSTRACT

The grapevine is one of the most important edible fruit plants cultivated worldwide, and it is highly sensitive to changes in the soil water content. We studied the total carbon and nitrogen contents and stable isotope compositions (C/N_{WSR}, $\delta^{13}C_{WSR}$ and $\delta^{15}N_{WSR}$ values) of the solid residues obtained by freeze-drying wines produced from two white grapevine cultivars (*Vitis vinifera* L. cv Chasselas and Petite Arvine) field grown under different soil water regimes while maintaining other climatic and ecopedological conditions identical. These experiments simulated the more frequent and extended climate change-induced periods of soil water shortage. The wines were from the 2009–2014 vintages, produced using the same vinification procedure. The plant water status, reflecting soil water availability, was assessed by the predawn leaf water potential (Ψ_{pd}), monitored in the field during the growing seasons. For both wine varieties, the $\delta^{13}C_{WSR}$ values are highly correlated with Ψ_{pd} values and record the soil water availability set by soil water holding capacity, rainfall and irrigation water supply. These relationships were the same as those observed for the carbon isotope composition of fruit sugars (i.e., must sugars) and plant water status. In Chasselas wines, the nitrogen content and $\delta^{15}N_{WSR}$ values decreased with soil water deficit, indicating control of the flux of soil-water soluble nutrients into plants by soil water availability. Such a correlation was not found for Petite Arvine, probably due to different N-metabolism processes in this genetically atypical cultivar. The results presented in this study confirm and generalize what was previously found for red wine (Pinot noir); the carbon isotope composition of wine solid residues is a reliable indicator of the soil and the plant water status and thus can be used to trace back local climatic conditions in the vineyard's region. In most wines (except Petite Arvine) the C/N_{WSR} and $\delta^{15}N_{WSR}$ values are indicators of the origin of the nitrogen supplied to the plant's fruit (grape) that can be used to assess the N dynamics in the soil-water-plant system.

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1. Introduction

Agriculture and food security are some of the sectors most affected by climate and environmental change (Schmidhuber and Tubiello, 2007; Lobell et al., 2008; Bonamente et al., 2016). Among all cultivated crops, the grapevine (*Vitis vinifera L.*) emerges as one of the most important worldwide and the most hydro-climatic sensitive and expensive. Since the past decade, this has motivated various studies focusing on the effect of the plant (vine) water status on fruit maturation and grape and wine quality and quantity (Koundouras et al., 2006; Lund and Bohlmann, 2006; van Leeuwen et al., 2009; Bonamente et al., 2016; Pagay et al., 2016; Rayne and Forest, 2016; van Leeuwen and Darriet, 2016). The chemistry and sensory characteristics of the wine, unlike other crop commodities, are mostly determined by the geographical location of the vineyard, with relatively minor input from the viticultural and winemaking practices (Bisson et al., 2002). This geographical link between the quality and typicity of the wine with the location of grape and wine production, originally referred to in French wines by the term *terroir*, is a concept integrating soil (pedology, geology), climate, and topography (Seguin, 1986; Jackson and Lombard, 1993; van Leeuwen et al., 2004). More recent studies have shown that at least for some vine varieties, most of a wine quality — particularly the development of flavors — is closely correlated with the vine water status (van Leeuwen et al., 2009; Reynard et al., 2011; Picard et al., 2017). Accurate quantification of current water stress on plants and their products, the possibility to trace back its evolution in time, and the associated changes in soil water availability, even at a local scale, are of prime importance in areas under rapidly changing climatic conditions.

The vine water status is closely correlated to the stable carbon isotope composition ($\delta^{13}C$ values) of berry sugar (Gaudillère et al., 2002). This is explained by different isotopic fractionations in plants using the same photosynthetic pathway (i.e., the Calvin-Benson cycle for grapevines) for fixation of atmospheric carbon dioxide but with decreased stomatal conductance for low water availability (O'Leary, 1988; Farquhar et al., 1989; Flexa and Medrano, 2002). The newly formed photosynthate (the carbohydrates glucose and pentose) in the photosynthetic leaf cell form sucrose, which is transported in the vascular system (mainly in the phloem by sucrose translocation through phloem tissue), dislocated, partitioned and unloaded in different plant parts to synthesize all the organic compounds (e.g., proteins, carbohydrates, lipids, chlorophyll) in the different plant organs and tissues. In grapevines, part of the sucrose accumulates in the fruits (berries) during ripening, and the part remaining after the biosynthesis of plant organic compounds is stored as starch in the roots for reserve energy to be used for the development of primary leaves in the next growing season (Zapata et al., 2004; Zufferey et al., 2015). Therefore, the $\delta^{13}C$ values of sugars from berries at harvest or separated from the must before fermentation give an integrative measure of the photosynthetic fractionation during berry ripening with some potential contribution from sugars from the previous growing season (vintage). These $\delta^{13}C_{sugar}$ values have the potential to record the water availability in vineyards during grape ripening (Gaudillère et al., 2002). The main carbon-containing compound in wine is ethanol (8 to >15 vol%, 11.2 vol% on average; Sumby et al., 2010), which is produced by fermentation of glucose. A carbon isotope fractionation of 2 mUr is associated with the fermentation of a C_3-derived glucose to ethanol ($\Delta = \delta^{13}C_{glucose}$ - $\delta^{13}C_{ethanol}$; Hobbie and Werner, 2004). Thus, the carbon isotope composition of wine ethanol, and most probably the total organic carbon in bulk wine will reflect that of the must sugars. Recently, Guyon et al. (2015) reported a good correlation between the $\delta^{13}C$ values of bulk wine and vine water status for different cultivars (Merlot, Cabernet Sauvignon and Cabernet Franc) and vintages (1998–2000). Chemical and physical deterioration of wine may occur during storage (Waterhouse and Elias, 2010). In particular, oxidative spoilage reactions in bottled wine will convert a fraction of the ethanol into acetaldehyde, acetic acid, and its acetals (Escudero et al., 2000; Skouroumounis et al., 2005; Bartowsky and Henschke, 2008). These biochemical reactions are most likely to induce an isotopic fractionation. There are also problems with the bottle closure (cork) that may cause evaporative losses of ethanol. These depend on the temperature of storage and will cause a ^{13}C-enrichment in the wine ethanol. Therefore oxidative spoilage reactions and evaporative losses may cause changes in the ethanol $\delta^{13}C$ values, and thus a difference between the original (after winemaking) and that measured in old wine bottles.

The nitrogen in must and wines appears in the form of inorganic compounds, such as ammonia, nitrate and nitrite anions, and organic compounds, such as amines, amides, free amino acids, pyrazines, nitrogen bases, pyrimidines, proteins, peptides, nucleic acids, amino esters, and vitamins (Moreno-Arribas and Polo, 2009; Sumby et al., 2010; Jackson, 2014). Most of the nitrogen-containing compounds are non-volatile and are therefore preserved in solid residue after drying of the wine. It was shown that the volatile compound concentrations in the solid residue are significantly higher when freeze-drying is used, compared to oven drying at 40–60 °C (de Torres et al., 2010). The dehydrated and dealcoholized solid obtained by freeze-drying wine contains trace amounts of residual fermentable carbohydrates or sugars (i.e., glucose and fructose), non-fermentable carbohydrates (i.e., xylose, arabinose, ribose, rhammose), C_6 higher alcohols, esters, carboxylic acids, ketones, terpenes, C_{13} norisoprenoids, phenolic and heterocyclic oxygen compounds (Moreno-Arribas and Polo, 2009; Sumby et al., 2010). The use of combined carbon and nitrogen molar and isotope ratios (C/N, $\delta^{13}C$ and $\delta^{15}N$ values) in grapevine organs and must is very limited. Two recent studies utilized these discrimination techniques for the optimization of nitrogen partitioning in grapevine organs following foliar urea application (Verdenal et al., 2015, 2016). $\delta^{15}N$ values alone (Paolini et al., 2016) or combined with δ^2H, $\delta^{13}C$, and $\delta^{18}O$ (Durante et al., 2016) from the soil-grape-wine system were explored as potential markers of the geographic origin of wines made in Modena and Trento, North Italy. One of these studies showed that neither fermentation nor aging affect the $\delta^{15}N$ of grape must (Paolini et al., 2016); thus, the $\delta^{15}N$ of wine solids would be an indicator of the origin of nitrogen used by the vine and supplied to the berries. Recently we have shown that the carbon and nitrogen stable isotope composition of the wine solid residues ($\delta^{13}C_{WSR}$ and $\delta^{15}N_{WSR}$ values) of Pinot noir red wine, obtained from grapes of field-grown grapevines exposed to controlled soil-plant water status during the 2009 to 2014 growing seasons, is highly correlated with the severity of vine water deficit (Spangenberg et al., 2017). Field experiments were performed under the same geomorphological and pedo-climatic conditions, with the soil-plant water status set up by different irrigation treatments as the only variable (Zufferey et al., 2017). This experimental setup simulates the changes in soil water availability due to global climate changes. The Pinot noir plants were grown under natural conditions with no application of fertilizers and/or amendments in the field, nor the addition of nitrogen yeast-nutrients during the winemaking processes (Spangenberg et al., 2017). It was established that the C/N_{WSR} molar ratios combined with the $\delta^{15}N_{WSR}$ values allow to trace the flow of N-nutrients in soil-grapevine-wine systems.

For the production of white wine, it is generally necessary to optimize the yeast assimilable nitrogen requirements in must (or grape juice) to enhance the fermentation activity and avoid the problems associated with sluggish and/or stuck fermentation and optimize the production of volatile organic compounds (Rapp and Versini, 1995; Alexandre and Charpentier, 1998; Bell and Henschke, 2005; Torrea et al., 2011). This is done by increasing the nitrogen content in grapes via nitrogen fertilization of the vine, and if necessary, by the addition of organic or inorganic nitrogen to the grape juice as a yeast nutrient. The aim of the present study was to investigate if the trends observed for Pinot noir red wines are also valid for white wines derived from grapevines that received nitrogen fertilization and did or did not undergo further addition of organic nitrogen as a yeast nutrient during