



Oak wood extracts applied to the grapevine. An alternative to obtain quality Garnacha wines

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ABSTRACT

Stomata in leaves regulate gas interchange and transpiration in the grapevine and through these pores both the penetration of aqueous solutions with nutrients as well as the excretion of products take place. The aim of this work was to study the influence of spraying the vineyard with toasted and untoasted oak extracts on the volatile composition and on the organoleptic quality of wine made from Garnacha grapes. The results were compared with a Garnacha control wine obtained with grapes sprayed with distilled water. The absorption of the compounds from the oak wood extracts was irregular and some of these compounds were modified by the yeast during fermentation so as to prevent fermentation problems. This was observed particularly in the case of furfural which were transformed into furfuryl alcohol by yeast in order to avoid irreversible cellular damage. On applying a discriminant analysis to the concentration of volatile compounds in the wines, the three treatments (control, toasted and untoasted oak extracts) were differentiate. The wine obtained from grapes treated with toasted oak extract showed a more intense wood aroma after 18 months of bottle aging than the other wines. Likewise, spicy aromas were found to be more intense in the samples treated with some type of oak wood extracts.

1. Introduction

In the elaboration of wine, the barrel is not a mere container but rather it gives volatile substances to the product, providing notes of toasted bread, spices and brushwood... as well as tannins which influence on the taste and mouth sensation (Garde-Cerdán & Ancín-Azpilicueta, 2006; Jiménez-Moreno, Torrea-Goñi, & Ancín-Azpilicueta, 2003). The barrel also allows a complex interchange phenomena of wood-liquid-atmosphere which helps the wine to achieve an adequate quality over time (Peyron, Boukharta, & Feuillat, 1994). For all these reasons, the oak wood barrel is a sure guarantee of quality in the best wines, but it requires a large amount of wood with the consequent problem for forests preservation. The trees used in barrel-making should be minimum 100–150 years old with diameters between 45 and 60 cm, and only heartwood from the lower part of the trunk is used. In the case of French oak, which is more porous, the staves are obtained by splitting to ensure suitable tightness of the barrel. Splitting has a disadvantage compared to the sawing of the American oak, and is the low

yield of the wood. The use of oak wood barrels has other problems such as high costs, loss of wine and risk of wine contamination if the barrels are not properly conditioned (Jackson, 2014). On the other hand, some grape varieties, such as Garnacha do not age in barrel as well as other ones do, such as Tempranillo or Cabernet Sauvignon. The Garnacha variety is one of the red wine grapes most widely used around the world. This grape ripens rather late and so it needs dry warm conditions to fully develop as it can properly do in the Spanish climate, where this variety is likely to have originated. It also grows in Sardinia, in the south of France, in Australia and in San Joaquin Valley in California. This variety is very fruity, with a dark intense color, but with a moderate to low acidity. Garnacha grapes give very aromatic, fresh and fruity wines, but they are extremely sensitive to oxidation and they age poorly in barrel if the necessary precautions are not taken. For these above-mentioned reasons, it seems interesting to provide wood aromas to the Garnacha wine in order to obtain a high quality wine, without the use of wooden barrels. To do so, there are different approaches, each with its advantages and disadvantages. Adding shaving, cubes, chips or

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powder to the wine is cheaper than using barrels and it avoids contamination problems. However, there are several inconveniences difficult to control such as surface area-to-volume contact and shape of the chips, which could have an important influence on the final wine quality (Cabrita, Dias, & Freitas, 2011; Noguerol-Pato, González-Álvarez, González-Barreiro, Cancho-Grande, & Simal-Gándara, 2013). Another economic alternative is the immersion of oak wood strips attached to a stainless steel structure and leave them in contact with wine for several weeks (Rieger, 1996). Another simple process to provide oak flavors to the wine would be the spraying of oak extracts to the grapevine leaves with the aim of obtaining wine with wood aroma but without losing the typical freshness of a young wine (Martínez-Gil, Garde-Cerdán, Zalacáin, Pardo-García, & Salinas, 2012). In this way, the waste discarded in cooperage take advantage to obtain these oak wood extracts, and therefore the use of the wood is optimized.

Although leaves are not specialized organs in the absorption of substances such as roots, several studies have shown that they are capable of absorbing compounds through the cuticle and the stomata (Schreiber, 2005). It has been observed that in these areas of the leaves, both the penetration of aqueous solutions as nutrients and the excretion of products take place (Xia & Cheng, 2004). Thus, the application of foliar urea as a complement to traditional fertilization of the soil, increases the concentration of amino nitrogen and yeast assimilable nitrogen in must of *Vitis vinifera* var. Tempranillo. This means that wines coming from grapes that have been treated with foliar urea display a higher aroma intensity and a more fruity character than the wines made from untreated grapes, as well as also showing a higher concentration of amines (Ancín-Azpilicueta, Nieto-Rojo, & Gómez-Cordón, 2011; Ancín-Azpilicueta, Nieto-Rojo, & Gómez-Cordón, 2013). It has also been observed that some fungicide treatments, applied to the grapevine through its leaves, have an influence on the wine aroma. Noguerol-Pato, Sieiro-Sampedro, González-Barreiro, Cancho-Grande, and Simal-Gándara (2015) found that the concentration of volatile compounds in Tempranillo and Graciano wines showed different changes depending on the type of fungicide applied. Vitalini et al. (2014) found that the application of chitosan and benzothiadiazole in vineyard of *Vitis vinifera* L. cv Gropello Gentile, changed the aroma profile and sensory attributes of wine. Over the past few years, wine producers from Australia, Canada, Africa, Greece and California have observed a significant increase in cases of wine contamination by smoke after the exposition of vineyards to controlled burns, forest fires, and wildfires (Chong & Cleary, 2012; Høj, Pretorius, & Blair, 2003; Sheppard, Dhesi, & Eggers, 2009). The aroma from wines affected in this way has been described as undesirable, smoky, dirty and burnt, as well as leaving an unsavory and persistent taste of ash in the mouth (Høj et al., 2003). In all the wines coming from smoked grapevines, high levels of guaiacol, 4-methylguaiacol and 4-ethylphenol have been found by GC-MS and it is well-known that repeated exposure to smoke produces an accumulative effect in the concentration of these compounds (Kennison, Wilkinson, Pollnitz, Williams, & Gibberd, 2009).

The above results lead one to believe that the grapevine could absorb characteristic compounds of oak wood through its leaves and provide typical oak volatile compounds to the grape and thus, wine with wood aroma could be made without using oak barrels. For this reason, the aim of this work was to study the influence on the volatile composition of Garnacha wine from a vineyard sprayed both with toasted and untoasted oak wood extracts. These results were compared with a control wine obtained from the same grapes but only sprayed with distilled water, without any oak wood extract.

2. Materials and methods

2.1. Samples and vinification

In this work, *Vitis vinifera* var. Garnacha grapes (2014 vintage) from a vineyard located in Navarra region (north of Spain) were used.

Vineyard characteristics were a plantation frame of 2.80 × 1.40 m, and a bilateral cordon Guyot system of vine training. Two plots of the vineyard were sprayed with 2 different oak extracts. Each plot consisted of 72 vines, 24 vines per replica of each treatment. The oak wood extracts used in this work were provided by Protea France S. A. (Gensac LaPallue, France) and they are defined as technological auxiliaries. These extracts were obtained by infusion in hot water at 100 °C of toasted and untoasted oak chips, respectively. The wood used was French oak wood which had been naturally seasoned over 18 months. The wood extracts were applied during the veraison stage, because at this moment, the metabolic activity of the plant is greater and most of the changes are taking place in the berry. To do so, the extracts were diluted in distilled water at a concentration of 40% and the treatment was divided into four applications throughout veraison: the first was performed at the beginning and the remaining three at 7, 14 and 21 days. The treatment was applied during August 2014. During this period it did not rain in the geographical area where the vineyard is located, and temperatures ranged between 10 and 35 °C. Each vine was sprayed with 300 mL of the corresponding treatment. Another plot of the vineyard, with the same number of vines as the previous ones, was sprayed with distilled water (without any extract) and its grapes were used as control sample. The same worker always carried out the treatments during the first hours of the day, when the temperature does not exceed 20 °C and the absorption is maximum.

Grapes were harvested from the three plots: control plot (C) and plots treated with untoasted (U) and toasted (T) oak extracts. After harvest, grapes were washed, destemmed and crushed, and afterwards potassium metabisulphite (Aldrich, Gillingham, UK) was added to the must at a concentration of 60 mg/L of total SO₂. Fermentation was carried out in stainless steel tanks of 100 L at 28 °C, and with native yeast of the grape. For each tank between 85 and 100 kg of grape were used and between 50 and 70 L of wine were obtained in each case. Three different fermentations were performed for each treatment, so that nine fermentation tanks were used (3 × C, 3 × U, 3 × T). Maceration was carried out with daily pumping over during fermentation. All wines were fermented to dryness (reducing sugar concentration < 2.5 g/L) and fermentation lasted thirteen days in all cases. After alcoholic fermentation, free run wine was pumped off into tanks and skins were softly pressed in a vertical pneumatic press (Della Toffola, Signoressa di Trevignano, Italy). The press wine was blended with the free run wine, and malolactic fermentation took place in all cases after the inoculation of *Oenococcus oeni* with a dose of 0.63 g/hL (Viniflora Oenos, CHR Hansen, Denmark). After malolactic fermentation, the wine was cold stabilized (30 days at 1 °C) and the concentration of SO₂ was corrected to 30 mg/L, before being filtered (Seitz K200, Pall, New York, USA) and bottled.

Samples of the different treatments were taken at four stages of the winemaking: initial must, 50% of alcoholic fermentation, after alcoholic fermentation and after malolactic fermentation.

2.2. Analysis of volatile compounds by gas chromatography

The volatile compounds analyzed were 2-furancarboxaldehyde (furfural), 5-methyl-2-furancarboxaldehyde (5-methylfurfural), 5-hydroxymethyl-2-furancarboxaldehyde (5-hydroxymethylfurfural), 2-furanmethanol (furfuryl alcohol), *cis*-β-methyl-γ-octalactone (*cis*-oak lactone), *trans*-β-methyl-γ-octalactone (*trans*-oak lactone), 4-hydroxynonanoic acid lactone (γ-nonalactone), 4-hydroxybutyric acid lactone (γ-butyrolactone), β-ionone, 4-hydroxy-3-methoxybenzaldehyde (vanillin), 3,5-dimethoxy-4-hydroxybenzaldehyde (syringaldehyde), 4-hydroxy-3-methoxycinnamaldehyde (coniferaldehyde), 4-allyl-2-methoxyphenol (eugenol), 2-methoxyphenol (guaiacol), 4-methyl-2-methoxyphenol (4-methylguaiacol), 4-propyl-2-methoxyphenol (4-propylguaiacol), *p*-cresol, *m*-cresol, 2-phenylethanol, ethyl lactate, ethyl butyrate, ethyl hexanoate and ethyl octanoate. The extraction of these compounds was made following the method

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