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Characterization and suitability of polyphenols-based formulas to replace sulfur dioxide for storage of sparkling white wine

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ABSTRACT

The sparkling wine protection against air is of interest for maintaining its sensorial profile and it is achieved through the use of antioxidants while disgorging. Sulfur dioxide (SO_2) is commonly added, but its amount should be limited due to human health problems. The suitability of three polyphenols-based commercial formulas containing plant gallic and ellagic acids extracted from grape (*Vitis vinifera* L.) (AO1), plant ellagic acid and gum arabic (AO2), and plant gallic, ellagic acids and *Saccharomyces cerevisiae* cell-wall fractions (AO3) was evaluated after 7 months storage (at 15 °C and 25 °C) of disgorged sparkling white wine. The phenolic composition of these formulas was investigated through spectrophotometric measurements. Moreover, the phenols were characterized and quantified by HPLC-MS analyses. The sotolon concentration and the absorbance values at 420 nm were determined in wines. The HPLC-MS analysis showed that the formula AO1 mainly contained gallotannins, ellagic tannins and flavan-3-ols, while AO2 had high levels of flavan-3-ols and gallotannins. Flavan-3-ols were the only phenols found in AO3. The addition of these formulas increased the yellow hue. Sotolon was higher than the perception threshold in the samples with AO2 and at trace amount in the samples with both AO1 and AO3 only stored at 25 °C. The tested antioxidant formulas seemed to be less effective of SO_2 for the storage of sparkling white wine. However, the investigation of phenolics in antioxidant formulas could be helpful for the proper choice of a potential substitute of SO_2 due to increase interest in sulfur-free wine production.

1. Introduction

Disgorging and corking are critical steps in sparkling wine production because the wine can be easily exposed to the air which leads to oxygen dissolution. Oxygen can worsen the sensorial properties of sparkling wine and shorten the shelf life because it can degrade some aromatic esters and terpenes (Roussis, Lambropoulos, & Tzimas, 2007) and it can speed up the formation of compounds with oxidized off-odor such as sotolon (4,5-dimethyl-3-hydroxy-2,5-dihydrofuran-2-one) (Lavigne, Pons, Darriet, & Dubourdieu, 2008).

Sotolon odor is perceived as a defect in young dry white wine since it decreases the intensity of the fruity and flowery notes as well as the expected freshness character (Silva Ferreira, Barbe, & Bertrand, 2003). Sotolon can arise from the aldol condensation of 2-ketobutyric acid and ethanal (Cutzach, Chatonnet, & Dubourdieu, 1999; Kobayashi, 1989; König et al. 1999), as well as from the Maillard reaction (Pons, Lavigne, Landais, Darriet, & Dubourdieu, 2010) and the oxidative degradation of ascorbic acid in a hydro-alcoholic solution (König et al. 1999). These pathways are quantitatively favored as the concentrations of oxygen and reducing sugars increase (Camara, Marques, Alves, & Silva Ferreira, 2004; Cutzach et al. 1999; Lavigne et al. 2008). Its perception threshold in white wine was reported to be 7–8 μg/l (Guichard, Pham, & Etievant, 1993) and sotolon might be adopted as a chemical marker of oxidative aging.

In order to avoid oxidation of aromatic compounds and the formation of oxidized off-flavors, sulfur dioxide (SO_2) is commonly added to sparkling white wine while disgorging since this compound is rapidly oxidized to sulfate by an oxidation/reduction cycle of hydroxycinnamoyl tartaric acids (Danilewicz, 2003). As a consequence, the dissolved oxygen can be consumed quicker in

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presence of this antioxidant (Danilewicz, 2011). Though SO_2 is useful to limit the oxidative damage of white wine, its amount should be limited because of the detrimental effect on human health and the intolerance shown by a number of wine consumers, mainly asthmatics (Lester, 1995; Pozo-Bayón, Monagas, Bartolomé, & Moreno-Arribas, 2012; Vally & Thompson, 2001). Therefore, other antioxidant compounds safer to human health should be considered and tested in winemaking. Ascorbic acid could be effective to this aim (Marks & Morris, 1993) due to its low redox potential (Danilewicz, 2003), but its oxidation gives rise to both hydrogen peroxide (Riberau-Gayon, Glories, Maujean, & Dubourdieu, 2006, chap. 5) and 2-ketobutyric acid (Pons et al., 2010). Glutathione (GSH) showed to be effective in decreasing sotolon formation in the oxidative aging of barreled white wine (Lavigne & Dubourdieu, 2004). Nevertheless, high concentrations of GSH might need to be effective, but its average amounts in wine hardly exceed few milligrams per liter (Cassol & Adams, 1995; Du Toit, Lisjak, Stander, & Prevoo, 2007; Fracassetti & Tirelli, 2015). Oxygen in wine can also be consumed by polyphenols due to their low redox potential. Polyphenols containing trihydroxyphenyl groups (i.e. galloylated phenols) have a lower redox potential than polyphenols containing dihydroxyphenyl groups and they can completely deplete oxygen from wine (Danilewicz, 2011, 2012). White wine usually contains negligible amounts of trihydroxyl substituted phenyl compounds and the addition of mixtures containing phenols into the wine might limit the oxidative reactions in sparkling white wine during shelf life. Recently, the use of plant phenolics extract was shown to be effective as an alternative to SO_2 in white wine aged in barrels (Gonzáles-Rompinelli et al., 2013). The addition of gallotannins showed to play a positive role in the maintenance of esters in white wine after 1 year storage (Sonni, Chinnici, Natali, & Riponi, 2011). However, it is known that astringency and bitterness are affected to high concentration of tannins, but their perception is strictly dependent to the phenols concentration (Robichaud & Noble, 1990). The effectiveness of polyphenols-based preparation needs to be elucidate since no data are available related to their phenolic content and the nature of the single phenols. The knowledge of the phenols composition can be helpful for better comprehend the effect of these antioxidant preparation in wine. The investigation of the consequences on oxidative damage of sparkling white wine in comparison to SO_2 is also required.

On this purpose, this study was aimed to investigate the addition of three different antioxidant formulas added to an Italian sparkling white wine (*Champenoise* method) while disgorging as potential substitutes of SO_2. The phenolic composition of these antioxidant formulas was attentively characterized by spectrophotometric and HPLC-MS analysis. The latter allowed the identification and quantification of the single phenolic compounds. The levels of sotolon and GSH, and the changes of color were also evaluated. To the best of our knowledge, the phenolic composition of industrially-produced antioxidant formulas for oenological purpose has never been investigated as well as their effect throughout sparkling wine storage.

2. Material and methods

2.1. Chemicals

All the chemicals were of analytical grade. 3-Mercaptopropionic acid (3MPA) and p-benzoquinone (pBQ) were purchased from Fluka (Switzerland). Glutathione (GSH), cysteine (Cys), sotolon, ascorbic acid (AA), dehydroascorbic acid (DHA), 1,2-phenylenediamine dihydrochloride (OPDA), dichloromethane (DCM), $FeSO_4.7H_2O$, sodium chloride (NaCl), anhydrous sodium sulfate and trifluoroacetic acid (TFA) were purchased from Sigma–Aldrich (St. Louis, MO, USA). Polyvinylpolypyrrolidone (PVPP) was purchased from Dal Cin (Sesto San Giovanni, Milan, Italy). Citric acid was purchased from J. T. Baker (Phillipsburg, NJ, US); HPLC grade methanol was from Panreac (Barcelona, Spain), and HPLC grade water was obtained by a Milli-Q system (Millipore Filter Corp., Bedford, MA, USA). The synthetic wine solution contained 5 g/l tartaric acid in 12% ethanol/water solution (v/v), adjusted to pH 3.5 with 12 M sodium hydroxide (Sigma–Aldrich). Three commercial powders containing phenolics as antioxidant purpose for the winemaking use were purchased on the market. These formulas were labeled as mixtures of plant gallic and ellagic acids extracted from grape (*Vitis vinifera* L.) (sample coded as AO1), plant ellagic acid and gum arabic (sample coded as AO2), and plant gallic, ellagic acids and *Saccharomyces cerevisiae* cell-wall fractions (samples coded as AO3).

2.2. Sparkling wine samples

The sparkling white wine was industrial-scale produced by a cellar located in the Franciacorta area (Lombardy, Italy) in the 2010 vintage from Chardonnay grape. The rational winemaking procedures usually adopted in the winery for the manufacture of *Champenoise* sparkling wine were followed and no addition of SO_2 was carried out. Base wine (10 hl) was bottled, the second fermentation was performed and the sparkling wine was maintained 12 months on the yeast lees before the disgorging.

2.3. Experimental design

Sulfur dioxide (50 mg/l) and the three antioxidant formulas (20 mg/l and 40 mg/l) were separately added to bottled sparkling white wine samples after *à la glace* disgorging. The bottles were manually filled with 10 ml of the same sparkling white wine containing the antioxidant in order to reach the final volume of 750 ml and they were closed with crown cap. Control samples were disgorged, filled with sparkling white wine antioxidant-free and capped. The chemical parameters of both base wines (control and test) are reported in Table 1 and only negligible differences were found. All the bottles were stored for 7 months in two different rooms at 15 °C and 25 °C in the dark. For each treatment and temperature investigated, the content of GSH, sotolon, AA and DHA, and the absorbance values at 420 nm were evaluated. Each trial was performed in duplicate.

2.4. Determination of sotolon

Sotolon was measured in both sparkling wines and antioxidant formulas. The wine samples preparation was carried out as described by Gabrielli, Fracassetti, and Tirelli (2014). Briefly, 3 g of NaCl were dissolved in 30 ml wine in a 100 ml bottle then 40 ml of dichloromethane (DCM) were added. The bottle was hermetically closed and shaken for 10 min with a wrist action stirrer (Griffin

Table 1
Chemical composition of the base wines produced in triplicate fermentation.

Parameter	Wine control	Wine test
Ethanol (%)	12.4 ± 0.6	12.3 ± 0.4
Sugar (g/l)	<2	<2
pH	3.3 ± 0.1	3.2 ± 0.1
Total acidity (g tartaric acid/l)	6.6 ± 0.3	7.1 ± 0.5
Volatile acidity (g acetic acid/l)	0.43 ± 0.04	0.45 ± 0.02
Free sulfur dioxide (mg/l)	<5	<5
Total sulfur dioxide (mg/l)	30 ± 4	20 ± 3