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SHORT COMMUNICATION

Sensory attributes and volatile composition of a dry white wine under different packing configurations

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Abstract The aim of this work was to study the effect of different configurations of packaging on the volatile composition and sensory properties of a white wine. Certain oenological parameters were also evaluated. Bag-in-box (BIB) and glass bottles sealed with two different cork stoppers, natural and Neutrocork (technical), were used in the experiments. Analysis were carried out before packaging and after 3, 6 and 12 months of storage. Results showed that wines packaged in BIB presented higher levels of brown color than wines in bottles sealed with corks. In all packaging configurations, the content of free SO_2 decreased with storage time; however, BIB wines showed a lower content of free SO_2 than bottle wines during 12 months. Moreover, wines under BIB presented a significant lower amount of 2-phenylethanol, 2-phenylethyl acetate, isoamyl acetate, ethyl butanoate, ethyl hexanoate, ethyl octanoate, linalool and β-damascenone than bottled wines.

Keywords White wine · Cork stopper · Bag-in-box · Volatile composition

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Introduction

Flavor stability is one of the most important quality criterion for dry white wines. During storage, white wine quality is gradually decreased and the production of new compounds and browning can occur. From a sensory point of view, white wine loses the freshness and fruitiness and develops an unpleasant oxidized character. Browning, is characterized by a brown–yellow color that progressively replaces the initial pale-yellow color though the influence of oxygen (Karbowiak et al. 2010). During aging, the volatile composition of wines will depend on (1) chemical wine composition, pH and levels of oxygen, antioxidants and precursors, and on (2) storage conditions (packaging, temperature, light exposure, within others). Consequently, winemakers must continuously direct efforts toward the production of a wine with a balanced flavor and ensure that the conditions of wine bottle maturation and storage favor the preservation of wine quality rather than wine development towards oxidation (Skouroumounis et al. 2005).

The International Organization of Vine and Wine (OIV) has estimated that wine production should have reached in 2016, 259.5 m hectolitres (m hL), where 40–45% of which are white wines. Wine packaging also remains rather concentrated in format, with still light grape wine and the ubiquitous 750 mL glass bottle with a cork representing the bulk of volumes (21 billions of units). Red, white and rosé wine together hold a share of 70% of global packaging volume (83% for glass packaging); characteristics which come to a great extent from the traditions of wine production and consumption in the Old World (http://www.thedrinksreport.com/news/2013/15046-special-report-wine-packaging.html#). Nevertheless, alternative packaging such as bag in box (BIB) is expanding rapidly among some of the largest wine drinking countries in North America and also Europe (0.5 billion units). The US is the biggest world market for BIB sales. The economy factor offered by the BIB format in comparison to the glass bottle has been taken on positively by American consumers. Convenience is the second driver behind the success of BIB in the US, with the 3-L format largely benefiting from a growing pattern of wine consumption outdoors as its size and light weight nature enables it to be easily.

The choice of wine closure type will have a considerable impact on the extent of wine preservation. According to several studies (Blake et al. 2009), glass containers with cork stoppers are preferred to bottle wine. However, in recent years, a new package of polyethylene terephthalate material has been used and some works are reported in literature (Blake et al. 2009; Chatonnet et al. 2000; Dombre et al. 2015; Etievant 1991).

The main objective of this work is to assess the effect of prolonged storage at different packaging on the volatile fraction and sensorial properties of a white wine. Experiments were performed using wines packaged in BIB containers and in glass bottles closed with natural corks and Neutrocork® (manufactured from microagglomerate cork granules). The changes in sensory characteristics and volatile profile of wines were monitored during 12 months. Color intensity, measured by spectrophotometry, free and total SO_2 levels were also assessed.

Materials and methods

Wine

White wines from the 2012 vintage were produced from Rabo de Ovelha, Roupeiro and Tamarez grape varieties grown on the Borba Region (Alentejo, Portugal). The grapes were harvested during the month of September and transported to Adega de Borba Cellar's where they were destemmed and crushed. Fermentation was performed in stainless steel tanks under 15 °C during 20 days until a sugar content lower than 1 g/L. After alcoholic fermentation (sugar content < 1 g/L), tartaric precipitation of wine was carried out in isotherm tanks under constant temperature of 3 ± 1 °C during 7 days. Before packaging in glass bottles sealed with cork stoppers and in BIB containers, wine was filtered through diatomaceous earth and plate filters system (0.6 µm plates).

Packaging

The glass bottles were of Antique green color and 750 mL of capacity with the following specifications: diameter of 18–19 mm at a depth of 3 mm and diameter of 19–21 mm at a depth of 45 mm from the bottle entrance.

The cork closures were of average quality with 44 mm length and 24 mm diameter. The natural corks were previously washed with a mixture of water and hydrogen peroxide, steamed and dried up 8% of moisture, and coated with a mixture of silicone and paraffin. The Neutrocork were submitted to the same process; however, they were coated with a food grade silicone elastomer.

The bottling line comprised a filler and a multiple headed corker (Bertolazo, Zimella, Verona, Italy). All bottles were filled to 63 ± 2 mm from the top, and then they were sealed with a 44 mm of natural, resulting in a headspace 19 ± 2 mm (~ 5.7 mL). The cork stoppers were compressed to a diameter of 16 mm before insertion under vacuum into bottles. A total of 15 bottles were filled and sealed with each type of cork.

The BIB pouches of 5 L in capacity were composed by a laminated metalized polyester layer with 72 µm of thickness and an inner layer (45 µm) of low-density polyethylene (LDPE). The containers were provided by Conotainer (Madrid, Spain). According to BIB supplier, the oxygen permeability is lower than 1.0 cc/m^2/24 h (measured at 23 °C and 75% of relative humidity). Pouches were filled under vacuum on the winery bottling line filling the pouch headspace with inert gas (nitrogen) and placed inside paperboard cartons so as to provide access to the plastic valve. A total of 15 BIB containers were filled with white wine.

After filling step, BIB and bottles which were left upright for 1 h and then stored horizontally and kept at room temperature. Bottles were stored in 6 bottles paperboard boxes at the same room storage conditions. Samples from BIB packaged and bottled wine were tested over 3, 6 and 12 months. On day 0, approximately 200 mL of wine was immediately analyzed.

Chemicals

All chemicals were purchased from Merck (Darmstadt, Germany), Sigma-Aldrich (Madrid, Spain) and Fluka (Madrid, Spain), with the highest purity available. SPME fibers were purchased from Supelco (Madrid, Spain).

Enological parameters

Just before the filling and bottling of the samples, wines presented the following chemical composition: pH of 3.30, ethanol content of 13%, total acidity of 5.50 g/L and 27 and 119 mg/L of free and total SO_2, respectively. During storage time, the following parameters were evaluated: free and total SO_2, and color intensity. Free and total SO_2 was determined by amperometric titration corrected with acetaldehyde. Color intensity was determined by measuring the absorbance of wines at 420 nm, using a UV–Vis Varian, Cary 50 scan spectrophotometer (Palo Alto, CA, USA).

Esters, terpenes, norisoprenoids and 2-phenylethanol analysis

Esters (ethyl butanoate, ethyl hexanoate, ethyl octanoate 2-phenylethyl acetate, hexyl acetate), terpenes (linalool, α-terpineol), norisoprenoids (β-damascenone) and 2-phenylethanol were quantified according to the method described by Barros et al. (2012). The method combined automated headspace solid-phase microextraction (HS-SPME) with gas chromatography-ion trap/mass spectrometry (GC-IT/MS). The volatile compounds were extracted using a divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) fiber, 50/30 mm. After incubation of 5 mL of sample with 2 g NaCl at 45 °C during 5 min, the extraction was performed during 20 min at the same temperature, under continuous stirring (250 rpm). A desorption time into GC injector was 2 min at the 220 °C, in splitless mode.

GC-IT/MS analysis were performed on a Varian CP-3800 gas chromatograph (USA) equipped with a Varian Saturn 4000 ion trap mass detector (USA) and a Saturn GC-IT/MS workstation software version 6.8. Chromatographic separation was achieved using a capillary column VF-5 ms (30 m × 0.25 mm × 0.25 μm) from Varian and a high purity helium C-60 (Gasin, Portugal) as carrier gas at a constant flow of 1.0 mL/min. An initial oven temperature of 40 °C was held for 1 min, then increasing 5 °C/min to 250 °C (5 min) followed to increase 5 °C/min to 300 °C (0 min). The ion trap detector was set as follow: the transfer line, manifold, and trap temperatures were 280, 50 and 180 °C, respectively. All mass spectra were acquired in the electron impact (EI). The mass range was 35–600 m/z, with a scan rate of 6 scan/s. The emission current was 50 μA, and the electron multiplier was set in relative mode to auto-tune procedure. The maximum ionization time was 25.000 μs, with an ionization storage level of 35 m/z. The analysis was performed in full scan mode. Quantitative analysis was assessed by selected ion current mode. All wines were analyzed in triplicate.

Sensory analysis

Triangle sensory analyses were performed at 3, 6 and 12 months post-bottling by a panel of 6–7 wine specialists recruited from the staff of the Comissão de Viticultura da Região dos Vinhos Verdes and Amorim & Irmãos, S.A. (Portugal). 70% of the panelists, with extensive experience in wine tasting, participated at all sensory sessions.

All sensory assessments were performed at the tasting room of Comissão de Viticultura da Região dos Vinhos Verdes (Porto, Portugal) in individual booths under a room temperature of 18±1 °C and daylight lighting. Samples were assessed by the judges independently in blind tasting conditions using standardized procedures. Fifty mL of wine were presented in standard ISO 3591 'XL5-type' tasting glasses with glass covers identified by three digit random codes and assessed within 1 h of pouring.

The sensory differences between bottled and BIB wines were measured using 5 replicate series of triangle tests at 3, 6 and 12 months post-bottling. At each time point, 5 sets of 3 glasses samples were presented to each panelist per type of wine. The panelists were asked to identify the different sample and to provide a short comment on the perceived differences among samples. Panelists were instructed to assess first the appearance/color, then aroma and finally the palate of wines. The tasting was prepared in order to compare, migroagglomerate and natural cork sealed wines against wine packaged in BIB.

Statistical analysis

An analysis of variance (ANOVA) was applied to the experimental data in order to evaluate differences between wines over time and Tukey tests were applied for paired mean comparisons. Results were considered significant if the associated p value < 0.05. A principal component analysis was also applied to the generated data. All statistical analysis were performed using the software SPSS® 17.0 for Windows® (SPSS Inc., Chicago, USA).

Results and discussion

Color intensity, total and free SO_2

The color intensity of the wine was measured on the different packing configurations over a period of 12 months. The resulting data are presented in Table 1. The color intensity is a measure of the degree of yellow (or brown) color in white wines, and can be used as an indicator of the browning of wine due to oxidation (Godden et al. 2001). This oxidative process involves sugars, lipids, amino acids and phenols (Ghidossi et al. 2012). In general, the absorbance values of wines increased for all packaging materials with storage. However, after 3 months of storage, the wine packaged in BIB presented higher levels of brown color than wines in bottles sealed with corks, showing the same

Table 1 Colour intensity, free and total SO_2, and volatile compounds profile of white wines packed in bottles with two different cork stoppers (natural and Neutrocork) and in bag-in-box (BIB), before filling (Control) and after a storage period of 3, 6 and 12 months

Parameters	Control	Natural cork			Neutrocork			BIB		
		3 months	6 months	12 months	3 months	6 months	12 months	3 months	6 months	12 months
Colour intensity	0.070 (0.00)^{a}	0.077 (0.001)^{a,b}	0.077 (0.001)^{a,b}	0.080 (0.001)^{d,e}	0.076 (0.001)^{b}	0.076 (0.000)^{b}	0.079 (0.000)^{c,d}	0.083 (0.001)^{f}	0.083 (0.002)^{e,f}	0.091 (0.003)^{g}
Free SO_2	27 (0)^{f}	25.2 (1.1)^{e,f}	23.6 (0.5)^{d,e}	19.8 (2.3)^{b,c}	23.2 (0.8)^{b,c}	22.0 (0.0)^{d,e}	17.2 (1.1)^{b}	21.0 (2.2)^{c,d}	22.2 (0.8)^{c,d}	11.8 (2.3)^{a}
Total SO_2	119 (0)^{e}	114 (1)^{d,e}	114 (3)^{d,e}	105 (6)^{b}	115 (0)^{d,e}	112 (3)^{c,d}	106 (2)^{b,c}	110 (4)^{b,c,d}	111 (2)^{b,c,d}	92.4 (4.9)^{a}
Volatile compounds										
Alcohol (mg/L)										
2-Phenylethanol	16.6 (0.4)^{e}	16.2 (1.5)^{d,e}	13.2 (1.3)^{c}	9.13 (1.13)^{b}	16.2 (1.2)^{d,e}	13.0 (1.6)^{c}	6.57 (0.59)^{a}	14.7 (1.6)^{c,d}	10.2 (1.3)^{b}	5.54 (0.45)^{a}
Esters (mg/L)										
2-Phenylethyl acetate	0.385 (0.024)^{e}	0.230 (0.034)^{d}	0.227 (0.011)^{d}	0.213 (0.014)^{c,d}	0.228 (0.012)^{d}	0.236 (0.027)^{d}	0.170 (0.022)^{b}	0.225 (0.022)^{d}	0.189 (0.022)^{b,c}	0.086 (0.014)^{a}
Hexyl acetate	0.600 (0.007)^{d}	0.305 (0.059)^{c}	0.282 (0.065)^{c}	0.179 (0.019)^{b}	0.170 (0.024)^{b}	0.138 (0.035)^{a,b}	0.130 (0.011)^{a}	0.100 (0.032)^{a}	0.138 (0.035)^{a,b}	0.099 (0.006)^{a}
Isoamyl acetate	1.67 (0.04)^{d}	1.53 (0.14)^{d}	1.13 (0.19)^{c}	0.874 (0.083)^{b}	1.20 (0.18)^{c}	1.15 (0.21)^{c}	0.520 (0.104)^{a}	1.26 (0.06)^{c}	1.07 (0.19)^{b,c}	0.325 (0.038)^{a}
Ethyl butanoate	0.282 (0.020)^{e}	0.272 (0.046)^{d,e}	0.178 (0.024)^{b}	0.164 (0.020)^{b}	0.236 (0.032)^{c,d}	0.190 (0.032)^{b}	0.108 (0.026)^{a}	0.233 (0.014)^{c,d}	0.203 (0.035)^{b,c}	0.080 (0.015)^{a}
Ethyl hexanoate	2.63 (0.06)^{d}	1.81 (0.25)^{c}	1.16 (0.27)^{b}	0.805 (0.117)^{a}	1.33 (0.21)^{b}	1.02 (0.23)^{a,b}	0.809 (0.058)^{a}	1.20 (0.37)^{b}	1.22 (0.38)^{b}	0.702 (0.149)^{a}
Ethyl octanoate	3.57 (0.77)^{e}	1.49 (0.20)^{c}	1.51 (0.22)^{c}	1.01 (0.15)^{a,b}	1.24 (0.15)^{a,b,c}	1.52 (0.11)^{c}	1.01 (0.07)^{a,b}	1.95 (0.63)^{d}	1.32 (0.28)^{b,c}	0.877 (0.186)^{a}
Terpenes (μg/L)										
Linalool	3.91 (0.33)^{d}	2.93 (0.34)^{b}	4.03 (0.26)^{d}	3.20 (0.93)^{b,c}	3.15 (0.53)^{b,c}	3.77 (0.66)^{c,d}	1.19 (0.14)^{a}	3.17 (0.28)^{b,c}	3.16 (0.52)^{b,c}	1.19 (0.14)^{a}
α-Terpineol	0.612 (0.010)^{d}	1.17 (0.07)^{e}	0.263 (0.236)^{b,c}	0.301 (0.045)^{b,c}	1.18 (0.06)^{e}	0.344 (0.299)^{c}	0.231 (0.039)^{b,c}	1.08 (0.03)^{e}	nq^{a}	0.149 (0.033)^{a,b}
Norisoprenoid (μg/L)										
β-Damascenone	0.692 (0.046)^{f}	0.332 (0.019)^{d}	0.363 (0.030)^{d,e}	0.150 (0.016)^{c}	0.382 (0.010)^{e}	0.365 (0.032)^{d,e}	0.015 (0.004)^{b}	0.335 (0.026)^{d}	0.055 (0.045)^{a}	0.060 (0.010)^{a}

Values in parenthesis are standard deviations from four determinations; values not sharing the same superscript letter (a–g) within the horizontal line are different according to the Tukey test

nq not quantified

trend over a period of 12 months. The increase of color intensity of white wines may be related to the oxidation of wine phenolic compounds, reaction mechanisms involving acetaldehyde and glyoxylic acid, caramelization and Maillard reactions (Li et al. 2008).

The concentration of free and total SO_2 determined at different time points during storage is presented in Table 1. SO_2 is the most common preservative used in winemaking, exhibiting both antioxidant and antimicrobial properties (Ribéreau-Gayon et al. 2006). In all packaging configuration, the content of free SO_2 decreased with storage time. However, BIB wines showed a lower content of free SO_2 than bottle wines during 12 months. Comparing wines in bottle sealed with corks, results showed that over a period of 6 months, Neutrocork presented a lower level of free SO_2 than natural cork. This value is almost the same for both cork stoppers after 12 months. Which concerns the concentrations of total SO_2, a low value was obtained in wines in BIB than in bottles after 12 months of storage. Results showed that wine sealed in bottle with corks retained more SO_2, than wines sealed in BIB. Similar results were previously reported by Chatonnet et al. (2000) in white wines.

Volatile composition of wines

Changes on the volatile composition of wines were observed during storage (Table 1). After 3 months of storage, wines with natural cork presented a similar content in 2-phenylethanol, isoamyl acetate, ethyl butanoate and a higher level in α-terpineol than control wine. In general, wines under BIB presented the lowest values. Over storage time and comparing the two kinds of cork stoppers, results showed that wines with natural cork showed a significant higher level of 2-phenylethanol, 2-phenylethayl acetate, ethyl octanoate, α-terpineol and β-damascenone than wines on Neutrocork packaging. After 12 months of storage, wines under BIB presented a significant lower amount of 2-phenylethanol, 2-phenylethayl acetate, isoamyl acetate, ethyl butanoate, ethyl hexanoate, ethyl octanoate, linalool and β-damascenone than bottle wines. Even present in trace amounts in wines, esters, terpenes and norisoprenoides are extremely important for the flavor profile of white wines. Esters are produced by yeasts during alcoholic fermentation and wine aging (Ribéreau-Gayon et al. 2006). The most important esters in wine are 2-phenylethyl acetate (flowery, roses, honey notes), isoamyl acetate (fruity, banana aroma), hexyl acetate (pear aroma) and ethyl hexanoate (pineapple, fruity, apple notes). Linalool and α-terpineol are the most odoriferous terpenic alcohols, which contribute to the floral aroma of wines (Maicas and Mateo 2005; Mateo and Jimenez 2000). Terpenes are present in grapes and wines, in free and glycosylated forms. The terpene contents increase in wine through enzymatic or chemical hydrolysis. Norisoprenoids compounds, such as β-damascenone, contribute to fruity and floral notes and could be produced from direct carotenoid molecules degradation (Baumes et al. 2002; Winterhalter and Rouseff 2002) and from the hydrolysis of glycoside molecules (Oliveira et al. 2006; Silva Ferreira and Guedes de Pinho 2004). The decrease in ester concentration of white wines is due to the hydrolysis of the esters (Etievant 1991). However, the reduction and loss of aroma compounds of wines under BIB is probably enhanced by their sorption by the packaging material (flavor scalping phenomena) (Hopfer et al. 2012; Revi et al. 2014). Results shows that glass proved to be the most inert packaging material for white wine.

The principal component analysis presented in Fig. 1 allowed a better interpretation of variations of volatile compounds through storage time. Three factors explained 87.6% of the total variance. All the volatile compounds were considered, as well as the free and total SO_2 content and the color intensity of wines. The variables with higher contribution to the first principal component (PC1), which explained 61.2% of total variance, were free (FS) and total SO_2 (TS) content, the color intensity (CI), 2-phenylethanol (A), ethyl butanoate (E1), ethyl butanoate (E2), ethyl octanoate (E3), 2-phenylethyl acetate (E5) and linalool (T1). Results show that wines tend to be separated according to storage time. In general, increasing the storage time, wines tend to lose esters, terpenes and β-damascenone, being placed in the negative PC1. Wines with 3 and 6 months of storage are placed closed in the projection, being similar concerning the measured variable. However, after 12 months of storage, the wines are clearly separated according to the packaging configurations used. The bottled wines with natural cork are placed in opposition to BIB wines.

Sensory analysis

Supplementary Table (Table S1) summarizes the results of the triangle test conducted at 3, 6 and 12 months of storage comparing bottled white wines sealed either with microagglomerate or natural corks with wines packaged under BIB. At 3, 6 and 12 months, the white wines under BIB were significantly different from those in bottles regardless the type of cork stopper used ($p \leq 0.05$). At 3 months, the majority of panellists considered the wines under BIB more developed (oxidised) than bottled wines; however, other panellists stated that BIB wines slightly reduced or even musty. At 6 and 12 months of storage, the sensory differences between bottled and BIB wines became more noticeable; panellists unanimously considered wines under BIB oxidised. Wines under BIB displayed yellow golden colour coupled with developed sensory characters

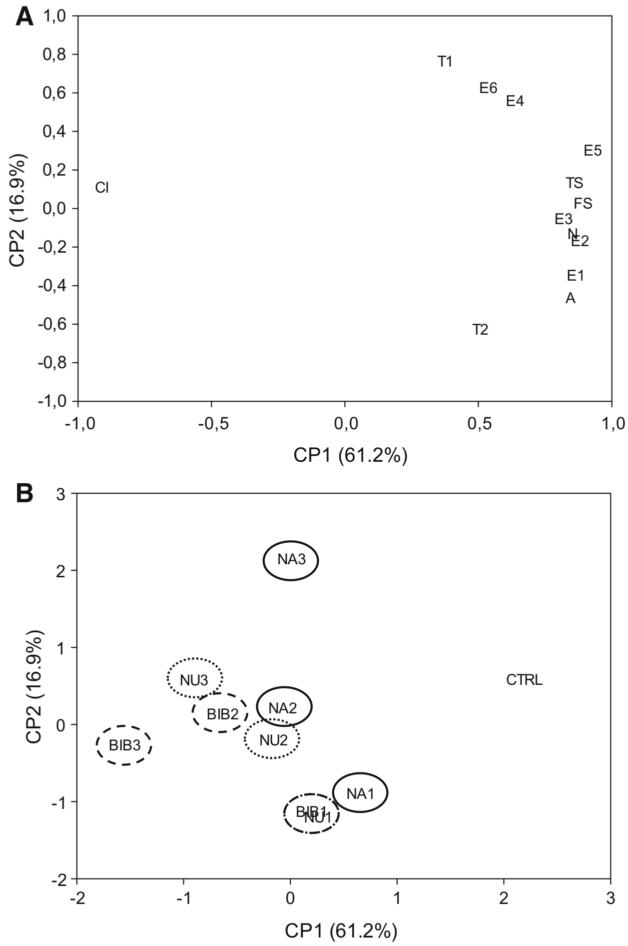

Fig. 1 Principal component analysis: projection of variables and wines in the space defined by the first and second principal components. **a** Variables: free SO_2 (FS), total SO_2 (TS), color intensity (CI), 2-phenylethanol (A), ethyl butanoate (E1), ethyl butanoate (E2), ethyl octanoate (E3), isoamyl acetate (E4), 2-phenylethyl acetate (E5), hexyl acetate (E6) and β-damascenone (N), linalool (T1), α-terpeniol (T2). **b** Wines: control (CTRL), bottled wines sealed with natural cork stoppers (NA), Neutrocork (NU) and wines in bag-in-box (BIB) followed by 1 (3 months of storage), 2 (6 months) and 3 (12 months)

such as cooked fruits, estery/confectionary and honey, ranging to oxidised characters (aldehyde, bruised apple, cardboard, wet wool). Conversely, bottled were presented the highest aroma intensity, freshness and overall fruit attributes, which is in agreement with the higher content esters, terpenes and norisoprenoids detected in bottled wines.

BIB wines promotes a fast development of wines towards oxidation than the glass bottles sealed with cork stoppers, confirming the analytical assessments. The higher color intensity and lower SO_2 observed BIB wines seems to be related with oxygen barrier properties of pack material. High oxygen transfer rates, as shown by the BIB, caused irreversible damage to the wine during storage. Due to the continuous entry of oxygen through this system, sulfur dioxide contents were largely depleted, which led to the consequent development of oxidized characters and yellowish/brown color during storage. Conversely, bottled wines sealed with cork, with low oxygen transfer rates, displayed the greatest concentrations of sulfur dioxide and lower color intensity. Sensory results clearly show that after 3 months of storage, the wine in the BIBs still had a minimum acceptable quality, although lower than that observed in the bottle. The quality of the wines differed over time, and at 6 months, the wines in BIB were clearly inferior to bottle wines, which maintained quality throughout the 12 months of the trial. These results suggest that BIB wine should be consumed in a short space of time, up to 3 months after its packaging, which put great pressure on the logistics chain of the wine industry, especially when it is exported. The present results are in excellent agreement with those of Hopfer et al. (2012, 2013) who reported pronounced color intensity and ‘‘oxidized’’ flavors in Chardonnay and Cabernet Sauvignon wines stored in BIB when compared with bottled wines. The same trends were also observed by Fu et al. (2009), Blake et al. (2009), Dombre et al. 2015, Ghidossi et al. (2012) and Revi et al. (2014).

Conclusion

The results showed that the choice of wine packaging had major impact on the evolution of chemical and sensory properties of white wines. Under the conditions tested, the microaglomerate cork, Neutrocork, resulted in wines with similar degree of oxidation as wines sealed with natural cork. The wine packed in BIB containers were considered less aromatic and presented higher levels of oxidized aromas.

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