Effect of Bottle Storage on Colour, Phenolics and Volatile Composition of Malvasia and Moscato White Wines

A. Del Caro[1*], P. Piombino[2], A. Genovese[2], L. Moio[2], C. Fanara[1], A. Piga[1]

(1) Dipartimento di Agraria, Università degli Studi di Sassari, Viale Italia 39/A, 07100 Sassari, Italy
(2) Dipartimento di Agraria, Università degli Studi di Napoli Federico II, via Università 100, 80055 Portici (NA), Italy

The authors contributed equally to this study

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The effect of bottle storage on the colour, phenolics and volatile composition of Malvasia and Muscat wines obtained from grapes grown in Sardinia was evaluated. Colour was evaluated by UV-VIS spectrophotometry and by tristimulus colorimetry. Polyphenols were analysed by UV-VIS spectrophotometry and HPLC-DAD. GC/MS was used to identify and quantify the content of free and bound volatile compounds. As expected, the absorbance values at 420 nm increased significantly for both wines during storage, due to oxidative browning, while difference in colour (DE*) from the beginning of storage and after 18 months was more intense in the Muscat wine than in the Malvasia wine. A significant decrease was observed in different phenolic compounds over time, especially in the Malvasia wine. In-bottle storage for 18 months at 20°C in the dark resulted in a significant decrease in all the classes of free and bound volatiles. These finding enhance knowledge regarding the effects of bottle storage on Muscat and Malvasia wines. This is of interest because, rather surprisingly, this topic has been poorly investigated in relation to these two varieties.

INTRODUCTION

The continuous changes in phenolic composition, colour and volatile compounds occurring during the bottle storage of white wine have been studied extensively (González-Viñas *et al*., 1996; De la Presa-Owens & Noble, 1997; Cejudo-Bastante *et al*., 2011; Zafrilla *et al*., 2003; Recamales *et al*., 2011). It is well known that the oxidation of phenols to quinones and their polymerisation results in yellow-brown macromolecules that are responsible for wine browning reactions (Singleton, 1987; Li *et al*., 2008). Another reaction that potentially contributes to the browning of white wine is the conversion of flavonols into yellow xanthylium pigments that increase the absorption in the 400 to 500 nm spectra region (Es-Safi *et al*., 2000). In particular, oxidative browning of white wines seems to be related more to the flavanols to which it is subjected during storage than to oxidation and polymerisation reactions (Hernanz *et al*., 2009; Kallithraka *et al*., 2009). In fact, many studies report significant losses in phenolic content during bottle storage, in particular of flavan-3-ols and flavonols (Pérez-Magariño & Gonzales-San José, 2001; Zafrilla *et al*., 2003; Cejudo-Bastante *et al*., 2013). A study by Cheynier *et al*. (1990) showed that other compounds undergoing oxidation and browning during storage are the hydroxycinnamic acid derivatives, but controversial results were published after this study. In fact, Recamales *et al*. (2006) reported a decrease in the concentration of the tartaric acid esters and an increase in their respective acids, while Mayen *et al*. (1997) and Cejudo-Bastante *et al*. (2011) had opposite results.

Flavonol concentration decreases during storage due to oxidative degradation (De Beer *et al*., 2005), while its aglycons increase due to hydrolysis reactions (Zafrilla *et al*., 2003; Fang *et al*., 2007). White wine colour changes during storage are the result of a concomitant increase in the chroma (C*) and decrease of the hue (h*) (Recamales *et al*., 2006; Hernanz *et al*., 2009). Moreover, a shift from pale yellow to yellow-brown as a result of an increase in a* and b* values has been reported (Cejudo-Bastante *et al*., 2011).

With regard to colour changes, a loss of sensory attributes of young wines during storage, such as fresh, floral, citrus and fruity, and the development of new ones, like biscuit, honey, toffee, toast, nutty and others, have been reported (Skouroumounis *et al*., 2005; Bueno *et al*., 2010). This sensory evolution has been correlated with the change in several volatile compounds and it has been pointed out that chemical hydrolysis reactions result in concentration fluctuations in various fermentation-derived esters, thus resulting in the loss of the fresh fruity characters (Leino *et al*., 1993; González-Viñas *et al*., 1996; De la Presa-Owens

**Corresponding author: E-mail address: delcaro@uniss.it [Tel.: +39 079 229346; Fax: +39 079 229320]*
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& Noble, 1997; Recamales *et al*., 2011). More particularly, González-Viñas *et al*. (1996) provided evidence for the loss of fruity attributes after 18 months of storage under commercial conditions, while high storage temperatures result in wine aroma changes after only five days (De la Presa-Owens & Noble, 1997). Moreover, the increase in concentration of long-chain esters and the decrease of acetates, such as isoamyl acetate, isopentyl acetate and 2-phenylethyl acetate, have been demonstrated (González-Viñas *et al*., 1996; Cejudo-Bastante *et al*., 2011). In general, however, the concentration of most of the wine volatile compounds decreases after one year of bottle storage (Cejudo-Bastante *et al*., 2011), and the major parameters influencing the rate of volatile change in concentration during ageing are temperature (Leino *et al*., 1993; De la Presa-Owens & Noble, 1997; Bueno *et al*., 2010; Robinson *et al*., 2010; Recamales *et al*., 2011; Butzke *et al*., 2012; Makhotkina & Kilmartin, 2012) and wine packaging materials (Fu *et al*., 2009; Mentana *et al*., 2009; Ghidossi *et al*., 2012; Hopfer *et al*., 2012). Research done on aroma evolution has been directed mainly at non-aromatic grapes (Airen, Chardonnay, Riesling, Sauvignon blanc), while little information is present on wine produced from aromatic grapes such as Muscat and Malvasia (Pérez-Magariño *et al*., 2013).

This study thus was carried out with the aim to evaluate the effect of 18 months of bottle ageing on colour, phenolic content and free and glycosylated volatiles of two white wines obtained from two important aromatic grapes of Sardinia, namely Malvasia of Bosa, which is a synonym for Malvasia of Sardegna (number 7266 in the Vitis International Variety Catalogue), and Muscat of Sorso-Sennori, which is a synonym for Moscato Bianco (number 8193 in the Vitis International Variety Catalogue). To the best of our knowledge, no data on the bottle ageing of these wines are available in the literature.

MATERIALS AND METHODS

Wine samples

The Malvasia (MV) was produced by an important Sardinian winery, while the Muscat (MS) was obtained from a north Sardinian cooperative wine growers' association. Grape harvesting took place in the second half of October 2008 at 25.5 (MV) and 28°Brix (MS). MV grapes (around 3 600 kg) were crushed, 5 g/100 kg of SO_2 were added and the must was left overnight for skin contact (12°C), after which it was drained and inoculated with selected yeast (US-01, Unistrains SrL, Sassari, Italy) at 30 g/hL. Fermentation was carried out in stainless steel tanks of 1 000 L capacity at 18 to 20°C, and racking was performed at a residual sugar level of 3°Brix. Fermentation was stopped by chilling at -3°C, followed by stabilisation.

After crushing, MS grapes (around 4 000 kg) were given 5 g/100 kg of SO_2, 3 g/100 kg of pectic enzyme and 15 g/100 kg of tannins to protect the must from oxidation (Cejudo-Bastante *et al*., 2010), and 6 g/100 kg of ascorbic acid. After cooling at 19°C, inoculation took place at 30 g/hL, following yeast rehydration (Uva ferm Ghm, Lallemand, Montreal, Canada) in warm water for 30 min, as suggested by the manufacturer. Fermentation was carried out with skin contact in stainless steel tanks of 1 000 L capacity at 19 to 20°C, until the initial sugar content had been halved. Racking was carried out when a residual sugar level of 8°Brix was attained, while fermentation was stopped by chilling at -3°C, followed by stabilisation.

The MS and MV wines were bottled six months after winemaking. Bottles were transparent, had a capacity of 750 mL and were sealed with cork stoppers. The bottles were stored in an upright position in the dark at a controlled temperature of 20°C and sampled at the beginning of the experiment and after 18 months of bottling. We decided to store the bottles for 18 months as we think this is a reasonable period, due to the fact that the maximum storage life for the optimal quality of young white wines stored in glass bottles has been suggested to be 24 months (Gonzalez-Vinas *et al*., 1996).

Colour measurement

Wine colour was assessed using the CIELAB space colour system with a tristimulus colorimeter Minolta CR-300. The following parameters were evaluated: L*, an approximate measure of lightness, a* and b* coordinates, which represent the red-green colour and yellow-blue colour respectively, C* (Chroma), which is considered the quantitative attribute of colourfulness, and h*, which represents the qualitative attribute of the colour. To evaluate differences in colour between the beginning of storage and after 18 months, the DE^*_{ab} was calculated using the Euclidean distance, as reported in Gamasa *et al*. (2009). The absorbances at 420 and 520 nm were determined spectrophotometrically (Hewlett Packard mod. 8453, Palo Alto, CA) (Iland *et al*., 2004). A total of 10 readings were taken for each sample.

Analyses of polyphenols by spectrophotometry

Spectrophotometric analyses of polyphenols were carried out according to Di Stefano *et al*. (1989). These methods are able to eliminate interferences due to salts, sugars and proteins by isolating the phenolic compounds on a Sep-Pak C18 cartridge. A UV-VIS spectrophotometer was used (HP 8453 Palo Alto, CA). The following parameters were determined: total polyphenols (mg/L of catechin), vanillin index (mg/L of catechin) and proanthocyanidins (mg/L cyanidin). Analyses were done in triplicate.

Extraction and HPLC-DAD analyses of polyphenols

Wine samples were filtered through 0.22 µm cellulose acetate filters according to La Torre *et al*. (2008), and injected directly into a liquid chromatograph (Hewlett-Packard Series 1050, Palo Alto, CA) coupled with an HP 1050 diode array detector. The column used was a LiChrosphere C18, 4 mm x 250 mm, 5 µm; 20 µL loop; 0.5 mL/min flow; mobile phase: A) 50 mM $NH_4H_2PO_4$ solution brought to pH 2.6 with H_3PO_4, B) 80% CH_3CN and 20% phase A, C) 200 mM H_3PO_4. The phenols were monitored at three different wavelengths: 280 nm for catechins, 316 nm for hydroxycinnamic acids and 365 nm for flavonols. The compounds were quantified by calibration with the following pure standards: gallic acid, catechin, epicatechin, protocatechuic acid, caffeic acid, *p*-coumaric acid, ferulic acid, tyrosol and trans-caftaric acid, all provided by Sigma Chemical Co. (St. Louis, MO). The quercetin 3-glucuronide, for which there is not a standard,

was quantified as rutin equivalent and the ethyl ester of caffeic acid as caffeic acid. Every sample was analysed twice.

Extraction and gas chromatography-mass spectrometry (GC-MS) analysis of volatiles

Free and glycosylated volatile compounds were extracted from the wines according to the solid phase extraction methods proposed by Di Stefano (Di Stefano, 1991; Mateo *et al*., 1997) and subsequently modified (Moio *et al*., 2004; Piombino *et al*., 2010; Del Caro *et al*., 2012). Twenty-five mL of wine were diluted with the same amount of water and 2-octanol was added as internal standard (125 mL of a 200 mg/L methanol solution), after which it was loaded on an activated 1 g C-18 cartridge (Phenomenex, Torrence, CA) and passed through at 3 mL/min. The cartridge was then washed with 10 mL of water. The free volatile compounds were eluted with 5 mL of dichloromethane, and then 10 mL of methanol were added for the recovery of the glycoconjugated fraction (bound volatiles). The dichloromethane fraction was concentrated to dryness with Na_2SO_4 and then reduced to a small volume (ca. 100 µL) with nitrogen flushing.

The methanol fraction was dried with a rotary evaporator and dissolved in 5 mL of phosphate-citrate buffer containing 40 mg of Novarom^{tm} Blanc ß-glucosydase enzyme at pH 5.0 (Novozymes, Bagsvaerd, Denmark). After 16 h of incubation at 40 ± 2°C, 125 mL of an alcoholic solution of 2-octanol was added as internal standard, and the mixture containing free aglycons was loaded on a C-18 SPE cartridge. The volatiles were extracted with 5 mL of dichloromethane. The extract was dried over Na_2SO_4 and concentrated under N_2 (1.5 L/min) for GC-MS analysis. Each extraction was carried out in triplicate.

GC-MS analysis was done with a GC/MS-QP2010 mass spectrometer (Shimadzu, Shimadzu Corp., Kyoto, Japan) in split/splitless mode and with a DB-WAX column (60m x 0.250 i.d., 0.25 µm film thickness; J&W Scientific Inc., Folsom, CA 95360, USA). The oven temperature was set at 40°C for 5 min and then raised at 2°C/min to 220°C, and it was held at maximum temperature for 20 min. Carrier gas (He) flow was 1.02 mL/min. Injections of 1 µL were performed and the injector port and ion source were maintained at 250°C and 230°C respectively. Positive electron impact spectra were recorded at 70 eV in the range m/z 33 to 350. The identification of compounds was confirmed by injection of pure standards and comparison of their retention indices and MS data reported in the literature, and the mass spectra stored in the NIST database were compared with those obtained for each compound. Compounds for which pure reference standards were not available were identified only on the basis of their retention times and MS spectra.

Odour threshold values (OTV) reported in literature were used to calculate the odour activity value (OAV) of the most relevant volatiles detected, by dividing the concentration detected by the OTV (Guth, 1997; Ferreira *et al*., 2000; Vilanova & Sieiro, 2006).

Statistical analysis

Data on colour, phenolic compounds and volatile compounds were evaluated by one-way ANOVA (Statistica), with storage time being used as the group variable. Means, when significant, were separated using LSD Fisher's test ($p \leq 0.05$).

RESULTS AND DISCUSSION

Colour changes during storage

One-way ANOVA applied to the MS and MV data showed that the absorbance at 520 nm did not present significant changes during storage in both wines, while the absorbance values at 420 increased significantly (Fig. 1). This last value is widely used as a marker of white wine browning, and in fact represents the true estimation of yellow-brown pigments formed in wines during storage. Both wines showed evidence of an increase in this index due to oxidative browning (Zoecklein *et al*., 1995; Iland *et al*., 2004).

Regarding the CIELAB parameters, a not significant decrease of h* was observed for both wines, as already reported (Recamales *et al*., 2006). There was a significant increase of a* coordinate in the MS wine but not in the MV wine, probably due to the higher content of flavan-3-ols that could react with the glyoxylic acid that arose from tartaric

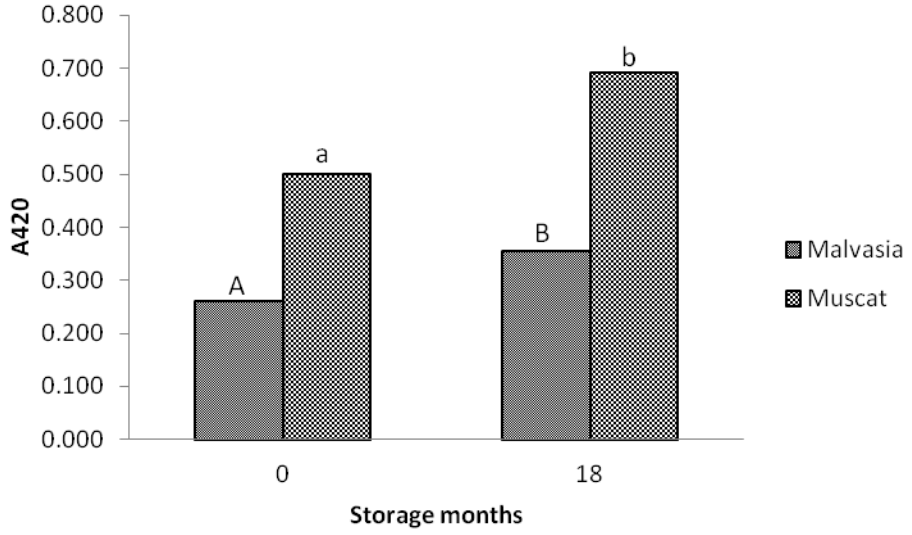

FIGURE 1

A420 values of Malvasia and Muscat during storage. Different letters for the same wine mean significant differences ($p \leq 0.05$).

acid oxidation to form more xanthylium salts (yellow-orange pigments) (Es-Safi *et al*., 2000). The difference in colour (DE*) from the beginning of storage to after 18 months was more intense in the MS wine than in the MV wine (6.30 and 2.98 respectively). It seems that, in general, differences in colour greater than 3 units permit the human eye to discriminate the changes in wine colour (Martinez *et al*., 2001; Gamasa *et al*., 2009).

Polyphenol changes during storage

Data on the spectrophotometric analyses of polyphenols are reported in Table 1. MS had a higher content of all polyphenol classes than the MV wine, particularly of proanthocyanidins, which, due to their high oxidability (Hernanz *et al*., 2009; Kallithraka *et al*., 2009), conferred a more intense brown colour on the MS wines (Fig. 1). This content could be related both to the tannin addition after grape crushing and to the proanthocyanidin content of MS grapes. Total polyphenols did not change significantly during storage, in contrast to what has been observed in Chardonnay and Chenin blanc white wines, even if the storage temperature of these wines was different (0 °C, 15 °C and 30 °C versus 20°C) (De Beer *et al*., 2005). The only significant decrease was for procyanidins in the MV wine, even though their value was very low at the beginning of bottling. The decrease is generally ascribed to polymerisation, oxidation and polysaccharide interaction reactions occurring during storage (Cheynier *et al*., 1990; Gómez-Plaza *et al*., 2002; Kallithraka *et al*., 2009). The vanillin index remained constant in both wines, thus confirming the stability of flavanol during 12 months of storage reported by de Beer *et al*. (2005). HPLC analysis showed a significant decrease in different phenolic compounds over time, while a limited number of them increased (Figs 2 and 3). In particular, a significant decrease in trans-caftaric acid and a increase in caffeic and p-coumaric acid were registered for the MS wine (Fig. 2), in accordance with data in the literature (Recamales *et al*., 2006; Hernanz *et al*., 2009; Kallithraka *et al*., 2009; Cejudo-Bastante *et al*., 2011). The decrease in the trans-caftaric acid content could be due to degradation reactions such as hydrolysation of the esters to their corresponding acids (Ivanova *et al*., 2011), oxidations and complexation (Zafrilla *et al*., 2003). The MV wine showed a significant decrease in trans-caftaric acid and caffeic acid content and a significant increase in the caffeic ethyl ester (Fig. 3) (Hernanz *et al*., 2009).

MS wine underwent a significant decrease in catechin and epicatechin over time, with the latter also decreasing significantly in MV, thus confirming the data in the literature (Recamales *et al*., 2006; Hernanz *et al*., 2009; Kallithraka *et al*., 2009). Moreover, the decrease in flavan-3-ols during storage was due to their strong influence on the susceptibility of white wines to oxidative browning, leading to oxidation

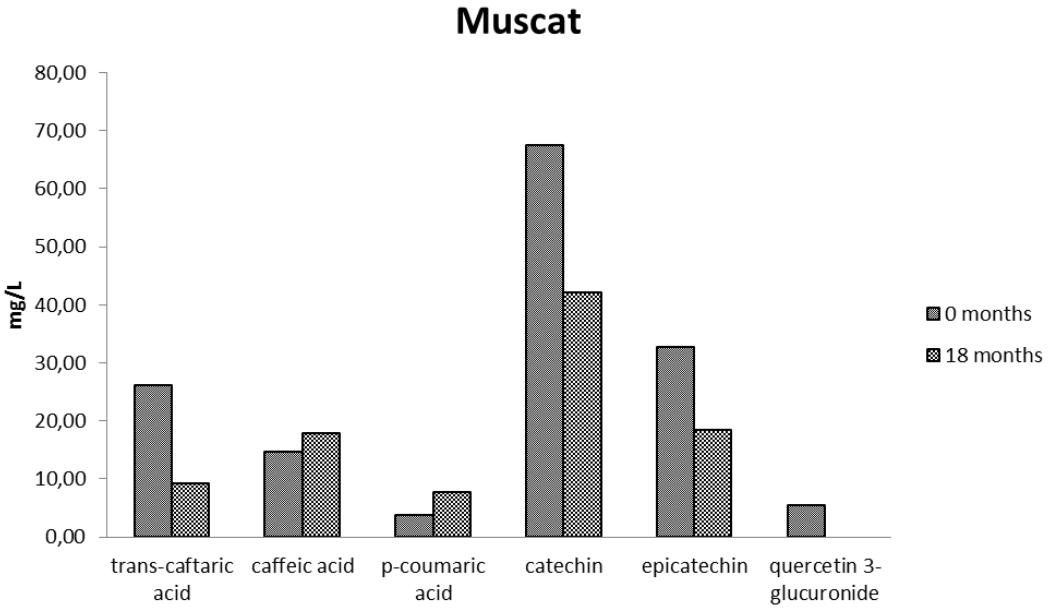

FIGURE 2

Evolution of polyphenolic compounds of Muscat of Sorso-Sennori during storage. Reported here are all the compounds that were significantly different after 18 months of storage ($p \leq 0.05$).

TABLE 1

Changes in the different classes of polyphenols in Malvasia and Muscat wines during in-bottle storage. Different letters within the column for each wine mean significant differences ($p \leq 0.05$) during storage.

Wines	Storage months	Total polyphenols (mg/L of catechin)	Vanillin Index (mg/L of catechin)	Proanthocyanidins (mg/L of cyanidin)
Malvasia	0	235.4a	98.2a	6.0
	18	264.7a	77.6a	traces
Muscat	0	457.3a	191.6a	195.2a
	18	555.6a	199.4a	158.9a

and polymerisation reactions (Simpson, 1982). During wine ageing, flavanols can react with glyoxylic acid, an oxidation product of tartaric acid, to give rise to coloured pigments such as xanthylium salts (Es-Safi *et al*., 2000). The results obtained here confirmed the change in colour for the two wines, in particular the MS wine, whereas the a* coordinate increased significantly during storage. The flavonol quercetin-3-glucuronide was found only in the MS wine and it decreased during storage, presumably due to oxidative degradation, precipitation or hydrolysis, as reported in other papers (Mayen *et al*., 1997; Zafrilla *et al*., 2003; Recamales, 2006; Ivanova *et al*., 2011). This last reaction leads to the formation of aglycons, which can precipitate more easily due to their low solubility, as reported in Zafrilla *et al*. (2003).

Volatile compounds

A total of 52 free volatile compounds were detected in the MV and MS wines, although two were present only in traces (Tables 2 and 3). Fifteen higher alcohols, three C-6 alcohols, thirteen terpenoids, thirteen esters, three acids, two lactones, one aldehyde, one volatile phenol and one other compound were identified. Alcohols, esters and acids were the main compounds in both wines, as reported in a previous paper (Del Caro *et al*., 2012).

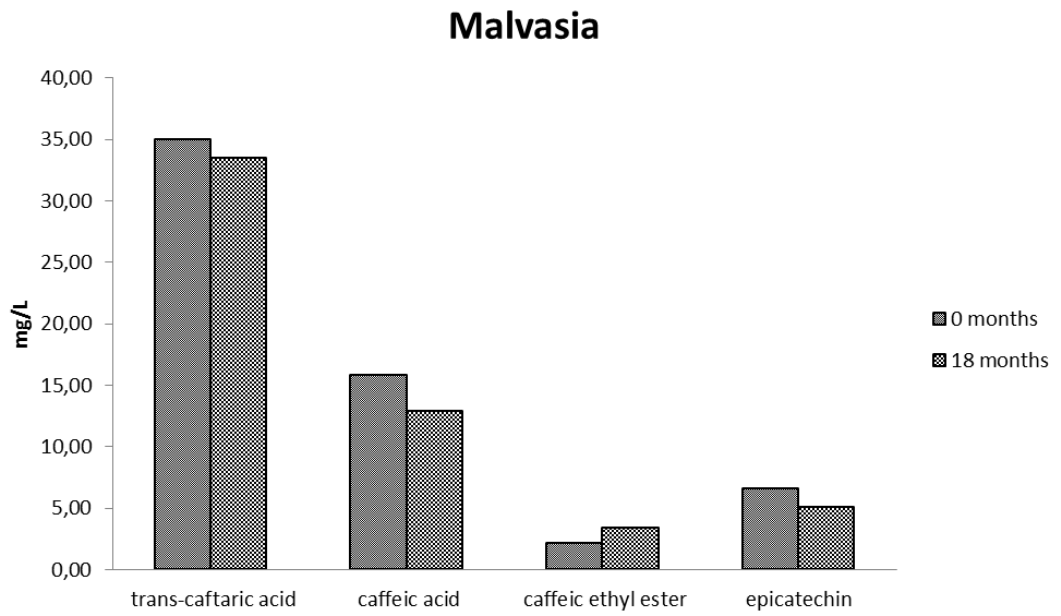

FIGURE 3

Evolution of polyphenolic compounds of Malvasia of Bosa during storage. Reported here are all the compounds that were significantly different after 18 months of storage ($p \leq 0.05$).

TABLE 2

Evolution of free volatiles[x] (µg/L) of Muscat of Sorso-Sennori wine after 18 months of bottle storage

Volatile compound	Storage time (months)		Volatile compound	Storage time (months)	
	0	18		0	18
C-6 Alcohols			***Terpenes***		
(Z)-3-Hexen-1-ol	13a	5.2b	(E) Linalool oxide	30a	21b
1-Hexanol	1271a	457b	(Z) Linalool oxide	56a	8b
(E)-3 Hexen-1-ol	12a	4.9b	Linalool	326a	134b
Total	**1296a**	**467.1b**	Terpinen 4-ol	11	NPb
			Ho-trienol	12a	8a
Alcohols			α-Terpineol	114a	56b
2-Methyl-1-propanol	1916a	606b	2,6-Dimethyl-3,7-octadien-2,6-diol	108a	29b
1-Butanol	99a	32b	Epoxylinalol I	NP	tr
3+2-Methyl-1-butanol	45602a	18369b	β-Citronellol	185a	50b
2-Methyl-3-buten-1-ol	14a	4.3b	Nerol	249a	143b
1-Pentanol	164a	52.3b	Geraniol	229a	NPb
4-Methyl-1-pentanol	17a	NPb*	2,6-Dimethyl-1,7-octadien-3,6-diol	49a	5b
3-Methyl-1-pentanol	33a	9.2c	Geranic acid	689a	331b
2-Octanol	17a	NPb	**Total**	**2067a**	**785b**

TABLE 2 (CONTINUED)

Volatile compound	Storage time (months)		Volatile compound	Storage time (months)	
	0	**18**		**0**	**18**
1-Octen-3-ol	79a	26.5b			
1-Heptanol	338a	105b	***Acids***		
2-Ethyl-1-hexanol	17a	6b	Hexanoic acid	614a	366b
1-Octanol	51a	11c	Octanoic acid	429a	192b
3-Methyl-1-propanol	30a	NPb	Decanoic acid	157a	44b
Benzyl alcohol	119a	47b	**Total**	**1200a**	**602b**
2-Phenylethanol	14256a	4141b			
Total	**62318a**	**23409.3b**	***Lactones***		
			γ-butyrolactone	128a	21b
Esters			5-pentildihydro-2(3H)-furanone	241a	91b
Ethyl-2-methylpropanoate	10a	2.7b	**Total**	**369a**	**112b**
Ethyl butanoate	148a	33b			
Ethyl-2-methylbutanoate	Tr	NP	***Volatile phenols***		
Ethyl-3-methylbutanoate	Tr	NP	4-Vinyl guaiacol	61a	43b
3-Methylbutyl acetate	178a	59b	**Total**	**61a**	**43b**
Ethyl hexanoate	206a	66b			
Ethyl heptanoate	16a	6b	***Aldehydes***		
Ethyl lactate	226b	264a	Benzaldehyde	240a	39b
Ethyl octanoate	161a	49b	**Total**	**240a**	**39b**
Ethyl decanoate	63a	8b			
Diethyl succinate	1192b	1375a	***Others***		
Ethyl vanillate	106a	30b	N-3-Methylbutil acetamide	510a	190b
2-Phenylethyl acetate	21a	10b	**Total**	**510a**	**190b**
Total	**2327a**	**1902.7b**			

[x] Means followed by different letters within each row are significantly different according to Tukey's test at $p \leq 0.01$.
[NP] Not present.

TABLE 3
Evolution of free volatiles[x] (μg/L) of Malvasia di Bosa wine after 18 months of bottle storage

Volatile compound	Storage time (months)		Volatile compound	Storage time (months)	
	0	**18**		**0**	**18**
C-6 Alcohols			***Terpenes***		
(Z)-3-Hexen-1-ol	45a	16b	(E) Linalool oxide	NPb	4a
1-Hexanol	1098	388b	(Z) Linalool oxide	NPb	3a
(E)-3 Hexen-1-ol	170a	55b	Linalool	42b	169a
Total	**1313a**	**449b**	Terpinen 4-ol	599a	9b
			Ho-trienol	26a	18b
			α-Terpineol	52b	149a
Alcohols			2,6-Dimethyl-3,7-octadien-2,6-diol	366a	14b
2-Methyl-1-propanol	2652a	835b	Epoxylinalol I	57a	NPb
1-Butanol	50a	16b	β-Citronellol	NPb	4a
3+2-Methyl-1-butanol	52966a	24080b	Nerol	25a	5b
2-Methyl-3-buten-1-ol	6a	NPb*	Geraniol	25a	NPb
1-Pentanol	47a	12b	2,6-Dimethyl-1,7-octadien-3,6-diol	tr	tr
4-Methyl-1-pentanol	47a	NPb	Geranic acid	23a	3b
3-Methyl-1-pentanol	45a	13b	**Total**	**1227a**	**378a**
2-Octanol	17a	NPb			

TABLE 3 (CONTINUED)

Volatile compound	Storage time (months)		Volatile compound	Storage time (months)	
	0	**18**		**0**	**18**
1-Octen-3-ol	7a	3b	***Acids***		
1-Heptanol	40a	12b	Hexanoic acid	1349a	222b
2-Ethyl-1-hexanol	17a	4b	Octanoic acid	2273a	754b
1-Octanol	23a	5b	Decanoic acid	1121a	378b
3-Methyl-1-propanol	154a	37b	**Total**	**4743a**	**1354a**
Benzyl alcohol	91a	22b			
2-Phenylethanol	22696a	8743b	***Lactones***		
Total	**78858a**	**33782a**	γ-butyrolactone	42a	12b
			5-pentildihydro-2(3H)-furanone	36a	NPb
			Total	**78a**	**12a**
Esters					
Ethyl-2-methylpropanoate	NPb	20a	***Volatile phenols***		
Ethyl butanoate	NPb	44a	4-Vinyl guaiacol	101a	24b
Ethyl-2-methylbutanoate	NPa	1a	**Total**	**101a**	**24a**
Ethyl-3-methylbutanoate	NPa	5a			
3-Methylbutyl acetate	522a	140b	***Others***		
Ethyl hexanoate	420a	133b	N-3-Methylbutyl acetamide	83a	NPb
Ethyl heptanoate	Tr	Tr	**Total**	**83a**	**NPb**
Ethyl lactate	600a	171b			
Ethyl octanoate	727a	220b			
Ethyl decanoate	389a	96b			
Diethyl succinate	2149a	1032b			
Ethyl vanillate	34a	11b			
2-Phenylethyl acetate	78a	20b			
Total	**5099a**	**1893a**			

[x] Means followed by different letters within each row are significantly different according to Tukey's test at $p \leq 0.01$.
[NP] Not present.

In-bottle storage for 18 months at 20°C in the dark resulted in a significant decrease in all the classes of free volatiles (Tables 2 and 3). Only ethyl lactate and ethyl succinate increased a little, likely due to a spontaneous malolactic fermentation that occurred in the MS wine. An interesting increase in linalool and α-terpineol was detected in the MV wine. C-6 alcohols and higher alcohols underwent a significant decrease in both wines, as already observed by other authors (Recamales *et al*., 2011), even if an increase in C-6 alcohols has been reported by some authors and explained as the product of ester hydrolysis (Pérez-Coello *et al*., 2003; Makhotkina *et al*., 2012). The decrease in alcohols, as already reported by Oliveira *et al*. (2008) in relation to Alvarinho wines, may be attributed to the fact that this class of compounds is involved in different reactions, such as oxidation and esterification, resulting in a modification of the base wine aroma.

Ethyl and acetate esters decreased in both wines. The hydrolysis mechanism has been shown to explain the reduction in the ethyl esters of fatty acids and acetate esters during wine storage (Ferreira *et al*., 1997; Pérez-Coello *et al*., 2003; Roussis *et al*., 2005; Garde-Cerdán *et al*., 2008; Papadopoulou & Roussis 2008; Hernanz *et al*., 2009; Robinson *et al*., 2010; Makhotkina *et al*., 2012), while esterification can result in the formation of ethyl esters of branched acids during bottle storage (Ferreira *et al*., 1997; Pérez-Coello *et al*., 2003; Garde-Cerdán *et al*., 2008; Hernanz *et al*., 2009; Robinson *et al*., 2010; Makhotkina *et al*., 2012). This behaviour has been related to the pH, but also to the temperature of storage. The observed decrease in ester concentration usually corresponds to the lower perception of the fruity character, typical of young wines.

Total terpenes decreased significantly in both wines, as already reported (Rapp & Marais, 1993), but, as stated above, linalool and α-terpineol increased in the MV wine, probably due to conversion from glycosylated compounds in the case of linalool (Rapp, 1988) or from geraniol and linalool in the case of α-terpineol (Stevens *et al*., 1972). This evidence is interesting because of the possible positive impact of these floral aroma compounds on the sensory profile of the aged MV wine. Fatty acids decreased significantly during bottle storage, in contrast to what has been reported in the literature (Hernanz *et al*., 2009; Recamales *et al*., 2011; Pérez-Magarino *et al*., 2013). Minor compounds such as lactones, volatile phenols and aldehydes had also decreased by the end of the storage period.

Concerning bound volatiles, a total of 26 compounds, one only in traces, were detected in the MS and MV wines

TABLE 4
Evolution of bound volatiles[x] (µg/L) of Muscat of Sorso-Sennori wine after 18 months of bottle storage.

Volatile compounds	Storage time (months)	
	0	**18**
C-6 Alcohols		
1-Hexanol	121.8a	32.8b
(E)-2- Hexen-1-ol	16.7a	4.5b
(E)-3 Hexen-1-ol	3.8a	0.6b
Total	**142.3a**	**37.9b**
Alcohols		
2-Methyl-1-propanol	6.3a	Trb
1-Butanol	16.6a	3.1b
3-Methyl-1-butanol	176.0a	31.4b
1-Pentanol	27.7a	3.9b
1-Octen-3-ol	10.4a	1.0b
1-Heptanol	11.8a	7.5b
1-Octanol	5.0a	3.5b
Benzylalcohol	182.7a	25.5b
2-Phenylethanol	287.2a	40.0b
Total	**723.7a**	**115.9b**
Terpenes		
(E) Linalool oxide	19.2a	4.2b
(Z) Linalool oxide	58.5a	5.1b
Linalool	307.4a	11.8b
Ho-trienol	Tr	NP*
α-Terpineol	22.5a	NP
Epoxylinalol I	120.2a	6.4b
Epoxylinalol II	9.2a	Trb
β-Citronellol	26.6a	0.5b
Nerol	570.4a	47.4b
Geraniol	589.1a	73.4b
Totals	**1723.1a**	**148.8b**
Aldehydes		
Benzaldehyde	12.1a	NPb
Neral	12.5a	NPb
Totals	**24.6a**	**NPb**

[x] Means followed by different letters within each row are significantly different according to Tukey's test at $p \leq 0.01$.
[NP] Not present.

TABLE 5
Evolution of bound volatiles[x] (µg/L) of Malvasia di Bosa wine after 18 months of bottle storage.

Volatile compounds	Storage time (months)	
	0	**18**
1-Hexanol	NPb	31.6a
(E)-2- Hexen-1-ol	13.9a	2.0b
(E)-3 Hexen-1-ol	30.6a	3.0b
Total	**44.5**	**36.6**

TABLE 5 (CONTINUED)

Volatile compounds	Storage time (months)	
	0	**18**
Alcohols		
2-Methyl-1-propanol	9.5a	0.5b
1-Butanol	38.5a	2.0b
3-Methyl-1-butanol	134.8a	11.0b
1-Pentanol	44.2a	2.4b
1-Octen-3-ol	10.7a	0.9b
1-Heptanol	12.9a	4.1b
Benzylalcohol	109.2a	5.9b
2-Phenylethanol	169.6a	21.3b
Total	**529.4a**	**48.1b**
Terpenes		
(E) Linalool oxide	NP	Tr
(Z) Linalool oxide	NPb	2.2a
Linalool	33.7a	0.8b
Terpinen 4-ol	35.0a	NP
Ho-trienol	NP	NP
α-Terpineol	2.9a_	Tr
2,6-Dimethyl-3,7-octadien-2,6-diol	27.3a	NPb
Epoxylinalol I	NPb	1.5a
Epoxylinalol II	48.8a	NPb
β-Citronellol	tr	Tr
Nerol	5.6a	2.2b
Geraniol	33.6a	2.3b
2,6-Dimethyl-1,7-octadien-3,6-diol	41.8a	NP
Totals	**229.0a**	**9.0b**

[x] Means followed by different letters within each row are significantly different according to Tukey's test at $p \leq 0.01$.
[NP] Not present.

(Table 4 and 5). We found three *C-6* alcohols, nine higher alcohols, 13 terpenes and two aldehydes. Bound compounds are flavourless precursor compounds, thus are a reservoir of flavour. After 18 months, all the compounds had undergone a drastic reduction, as shown in Tables 4 and 5. About 90% of the total bound volatiles were detected both in the MS and MV wines.

The general loss of esters and terpenes is very important in these wines, as these compounds are responsible for the fresh, floral and fruity notes of wines. Terpenes, on the other hand, are responsible for the characteristic varietal aroma of Muscat and other aromatic wines such as Malvasia (Rapp *et al*., 1986; Pisarnitskii, 2001; Selli *et al*., 2006). As can be noticed in Table 6, different aroma compounds of both wines evolved under their OTV after the storage period, thus both wines lost some of their most distinctive sensory properties. Before ageing, Muscat wine was characterised, according to the OAV (Table 6), by 3-methylbutyl acetate, ethyl octanoate, ethyl hexanoate and linalool, while Malvasia wine was particularly rich in ethyl octanoate, which may confer fruity, banana, pineapple, peach and sweet notes, and

TABLE 6
Evolution of odour activity values (OAVs) of volatile compounds with more influence on the aroma of Muscat of Sorso-Sennori and Malvasia of Bosa wines after 18 months of bottle storage.

Compounds	Sensory descriptor[a]	Odour threshold (μg/L)	Muscat		Malvasia	
			0[x]	18	0	18
Linalool	Flowery	15[b]	21.73a[y]	8.93b	2.8b	11.3a
Geraniol	Citric	30[b]	7.63a	UT*b	UT*a	UTa
2-Phenylethanol	Roses, sweet	10000[b]	1.42a	UTb	2.27a	UTb
Terpinen 4-ol	Flowery	15[e]	UTa	UTa	39.93a	UTb
β-Citronellol	Lemon, lime	100[b]	1.85a	UTb	UTa	UTa
3-Methylbutyl acetate	Banana	2[d]	89a	29.5b	261a	70b
Ethyl butanoate	Kiwi	20[b]	7.4a	1.65b	UTb	2.2a
Ethyl hexanoate	Fruity, green, apple, banana	5[b]	41.2a	13.2b	84a	26.6b
Ethyl octanoate	Fruity, banana, pineapple, peach, sweet	2[b]	80.5a	24.5b	363.5a	110b
Ethyl decanoate	Sweet, grass	200[c]	UTa	UTa	1.95a	UTb
Hexanoic acid	Cheese	300[b]	2.05a	1.22b	4.50a	UTb
Octanoic acid	Grass acid	500[c]	UTa	UTa	4.55a	1.51b

[a] Genovese *et al*., 2007
[b] Guth, 1997
[c] Ferreira *et al*., 2000.
[d] Takeoka *et al*., 1989.
[e] Vilanova & Sieiro, 2006
* Under threshold of 1 μg/L
[x] In bottle storage time
[y] Means followed by different letters within each row are significantly different according to Tukey's test at $p \leq 0.01$

in 3-methylbutyl acetate, which may impart banana flavour (Genovese *et al*., 2007). After ageing, the total disappearance of geraniol can confer a floral/citric note. On the other hand, the increase of ethyl lactate in MS can enhance the overripe fruit flavour of the wine (Pérez-Coello *et al*., 2003).

CONCLUSIONS

This work provided new knowledge on changes during the bottle ageing of two important white wines, namely Muscat and Malvasia. Despite the reductions in compounds typical of bottle storage, we detected many changes in colour, phenolic and volatile compounds. Wine colour showed an increase of absorption in the 400 to 500 nm region and an increase of A420 due to oxidative browning. Bottle storage also influenced the phenolic content, which showed a decrease, in particular in the MV wine, of around 25%. This was probably due to the scarce antioxidant protection of the flavan-3-ols, which were present in this wine at a very low concentration.

Free and bound volatile compounds generally decreased after 18 months of storage (about 60% of free volatiles and about 90% of bound volatiles, both in the MS and MV wines), leading to a loss of the distinctive aromatic properties of these two wines, even if linalool and α-terpineol concentrations increased in the MV wine. The MV wine had the largest amount of free aromas, while the MS had a larger amount of bound volatiles. The two wines should be consumed before 18 months of storage if we want to preserve their optimal colour and, particularly, their sensory properties.

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