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Peer-reviewed version available at *Beverages* **2018**, *4*, 89; doi:10.3390/beverages4040089

Evolution of Phenolic Compounds Profile and Antioxidant Activity of Syrah Red and Sparkling Moscatel Wines Stored in Bottles of Different Colors

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Abstract

The objective of this study was to evaluate the evolution of the Syrah red and sparkling Moscatel wines stored for 12 months in green, amber and clear bottles. The phenolic compounds profile and antioxidant activity *in vitro* were determined. Commercial wines were bottled in an automatic filling machine, stored and analyzed every three months for one year. Several phenolic compound families were quantified through reversed-phase high performance liquid chromatography (RP-HPLC) coupled to diode-array detection (DAD) and fluorescence detection (FD). The different bottle colors studied did not influence the evolution of the sparkling Moscatel and Syrah red wines, since the main variations obtained were related to storage time. The main changes were observed in the Syrah wine, where storage time was associated with an increase in hue (*h**), decrease in catechins, increase in procyanidins and, most notably, a decrease in the anthocyanin malvidin 3-glucoside. In general, the wines showed good stability in relation to the antioxidant activity *in vitro*.

Keywords: bottle color, shelf life, *Vitis vinifera* L., quality control.

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1. Introduction

The wine sector has increasingly sought to obtain wines with high sensory quality as well as typical characteristics, since these attributes are the main aspects expected by consumers [1]. Products which do not fulfill the sensory expectations of the consumers may be rejected.

The São Francisco Valley (SFV), located in the Northeast Region of Brazil, is an unusual and emerging area [2]. This region currently produces 2 million liters of sparkling Moscatel wines and 1.16 million liters of young red wines (mainly with Syrah grapes). The characteristics of these wines make them appropriate for rapid consumption [3], and storage is recommended for a maximum period of two years.

The shelf life of a wine can be left undetermined, since its chemical composition is very complex [4] and several favorable or unfavorable reactions can occur during the storage time. In addition, the temperature, bottle position, exposure to light, moisture and container color may affect the qualitative characteristics of the wine. Furthermore, these factors may promote changes in the phenolic composition through copigmentation, polymerization and oxidation reactions. These reactions have a negative or positive impact from the consumer's viewpoint [5-6].

Some authors have evaluated the factors related to wine aging, such as: the adverse influence of light and the influence of the bottle color on the evolution of the color and volatile composition of wine [7-12]. However, studies to measure the effect of bottle colors on the profile of several families of phenolic compounds related with wine quality, such as flavanols, flavonols, phenolic acids, anthocyanins, and stilbenes, have not been previously reported.

Another parameter directly influenced by storage is the antioxidant activity *in vitro*, and some studies have shown that there is a considerable decrease over time [4,13].

In this context, the objective of this study was to evaluate the evolution of Syrah red and sparkling Moscatel wines stored for 12 months in bottles of different colors, in relation to the phenolic compounds profile obtained through RP-HPLC/DAD/FD and the antioxidant activity *in vitro*.

2 – Materials and Methods

2.1 Chemicals

Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid), 2,2-diphenyl-1-picrylhydrazyl (DPPH) and 2,2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) were obtained from Sigma-Aldrich (St. Louis, MO, USA). Potassium persulfate, ethanol, acetone, sodium carbonate and Folin-Ciocalteu reagent were obtained from Merck (Darmstadt, Germany). Methanol and acetonitrile (both HPLC grade) and orthophosphoric acid were provided by Vetec Química Fina Ltda (Rio de Janeiro, Brazil), JT Baker (Phillipsburg, NJ, USA) and Fluka (Buchs, Switzerland), respectively. The water used in this study was purified with the aid of an Elga PURELAB® Option-Q (USA) purification system. The standards of ferulic, cinnamic and gallic acids were obtained from Chem Service (West Chester, PA, USA), and the *p*-coumaric and chlorogenic acids, viniferin and piceatannol were obtained from Sigma-Aldrich (St. Louis, MO, USA). The standards kaempferol 3-glucoside, rutin, pelargonidin 3-glucoside, (+)– catechin, cyanidin 3-glucoside, (-)- epicatechin, (-)- epigallocatechin, (-)– epicatechin gallate, isorhamnetin 3-glucoside, delphinidin 3-glucoside, malvidin 3-glucoside, petunidin 3-glucoside, peonidin

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3-glucoside, procyanidin A2, procyanidin B1, procyanidin B2, quercetin 3-glucoside, and *cis* and *trans* resveratrol were obtained from Extrasynthese (Genay, France).

2.2 Samples

Two types of bottles were acquired from the company Owens-Illinois (Recife, PE, Brazil), one bottle specifically for sparkling wine (Trilogy Glass & Packaging Inc., Sparkling wine bottle 750 mL Prosecco) and the other for table wine (BD Reference L+G, 750 mL Bordeaux), both with a holding capacity of 750 mL and in clear, green and amber-colored glass. Single varietal Syrah (red) and sparkling Moscatel (white) wines were obtained from the Miolo Wine Group (Casa Nova, BA, Brazil) located in the São Francisco Valley (SFV; latitude 9º15' S; longitude 40º50' W). The bottling was carried out by the cited company with the aid of an automatic filling machine, Suprema 30 (Bertolasso, Italy). After the bottling process, the wines were closed with a natural cork and stored vertically on shelves which received natural light indirectly (±8h/day), at temperatures which varied from 24 to 30ºC, over a period of 12 months. The treatments for the Syrah red wine were as follows: stored in clear bottles and evaluated after 3, 6, 9 and 12 months (SCB3, SCB6, SCB9 and SCB12, respectively); stored in green bottles and evaluated after 3, 6, 9 and 12 months (SGB3, SGB6, SGB9 and SBG12, respectively); and stored in amber bottles and evaluated after 3, 6, 9 and 12 months of storage (SAB3, SAB6, SAB9 and SAB12, respectively). The treatments for the sparkling Moscatel wine were as follows: stored in clear bottles and evaluated after 3, 6, 9 and 12 months (MCB3, MCB6, MCB9 and MCB12, respectively); stored in green bottles and evaluated after 3, 6, 9 and 12 months (MGB3, MGB6, MGB9 and MGB12, respectively); and stored in amber bottles and evaluated after 3, 6, 9 and 12 months (MAB3, MAB6, MAB9 and MAB12, respectively).

*2.3 Basic parameters, CIE L*a*b* color, total phenolic and monomeric anthocyanins*

The determination of the density, alcohol content, pH, titratable acidity and free and total sulfur dioxide was carried out following the methods recommended by the International Organization of Vine [14]. The color was measured with the aid of the coordinates L*, a*, b*, C* and h* (CIELab) in a colorimeter, model Delta Vista 450G, with the software program i7 (Delta Color, Brazil), using an acrylic cuvette with a 1 cm optical path length.

The total phenolic content was determined using the colorimetric method of Folin-Ciocalteu [15], using gallic acid as the standard, and the results were expressed as gallic acid equivalent (GAE mg L^{-1}).

For the determination of the monomeric anthocyanins, the differential pH method was used [16], and the values were expressed as malvidin 3-glucoside in mg L^{-1}.

2.4 Antioxidant Activity in vitro

The antioxidant activity (AOX) of the wines was measured through the methods of free radical capture using 2,2-diphenyl-1-picrylhydrazyl (DPPH) and 2,2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS). In both methods, the analytical standard Trolox was used to construct the calibration curves, following the methods described by Kim, Guo, Packer [17] and Re et al. [18]. The results were expressed as equivalent in millimoles of Trolox per liter of wine (mmol TE L^{-1}).

2.5 Determination of phenolic compounds profile by RP-HPLC/DAD/FD

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The individual phenolic compounds in the wines were determined by reversed-phase high performance liquid chromatography (RP-HPLC) on a Waters Systems (model Alliance e2695) coupled to diode array detection (DAD) and fluorescence detection (FD). The analysis was performed according to the methodology described by Silva et al. [19], using the software program Empower™ 2 (Milford, MA, USA) for data treatment. For the separation of compounds, a Gemini NX C-18 column (150 mm × 4.6 mm × 3 µm) and a Gemini NX C-18 guard column (4.0 mm × 3 mm × 3 µm) were used, both manufactured by Phenomenex (Torrance, CA, USA).

The oven temperature was maintained at 40 ºC and the solvent flow at 0.6 mL min^{-1}, with a total run time of 65 min. The gradient used was 0 min: 100% A; 18 min: 87.5% A, 2.5% B, 10.0% C; 30 min: 83.5% A, 3.2% B, 13.3% C; 36 min: 75.0% A, 5.0% B, 20.0% C; 48.5 minutes: 65.0% A, 8.3% B, 26.7% C; 50 min: 65.0% A, 8.3% B, 26.7% C; and 65 min: 100% A. Solvent A consisted of a solution of 25 mmol L^{-1} of potassium dihydrogenphosphate with the pH adjusted to 2.05 with phosphoric acid, solvent B was methanol and solvent C was acetonitrile. The detection and quantification of the compounds was carried out through the use of external standards.

2.6 Statistical analysis

All of the analyses were carried out in triplicate and the results were expressed as average ±standard deviation of the values obtained. The results obtained were submitted to analysis of variance (one-way ANOVA) and compared applying the Tukey test ($p<0.05$), with the aid of the software program SPSS Inc., version 17.0 (Chicago, IL, USA). In order to evaluate the behavior of the phenolic profile in relation to the storage time in the different colored bottles, the application of multivariate statistics was carried out using principal component analysis (PCA), as described by Coelho et al. [20].

3. Results and Discussion

3.1 Basic parameters of wines

The Syrah red and sparkling Moscatel wines were analyzed using classical techniques before their bottling to determine the basic characteristics of the samples and verify that they adhered to standards established by Brazilian legislation. The results obtained are shown in Table 1.

*3.2 Color evolution based on the CIE L*a*b* system*

The results for the wine color are given in Table 2. For the sparkling Moscatel, there was a similar increase in the b^* and C^* values for the three bottle colors, with ranges of 6.9-12.87 and 7.0-12.87 for b^* and C^*, respectively. The increase in the $+b^*$ values indicates an increase in the yellow color, which was also observed by Del Caro et al. [8] who studied the stability of Moscato and Malvasia white wines over a period of 18 months. This observation could be due to the oxidation of the flavanols in the wine. The increase in C^* values represents an increase in the shine (higher clarity) over time, which could be due to sedimentation of the colloids in the wine during storage.

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Table 1. Basic parameters of the sparkling Moscatel and Syrah wines.

Basic parameters	Sparkling Moscatel	Legal limit	Syrah	Legal limit
pH	3.40 ± 0.02	Na	3.90 ± 0.02	Na
Titratable acidity (g L^{-1})	7.6 ± 0.1	4.1–9.8	5.2 ± 0.2	3.0–9.8
Alcohol content (% *v/v*)	9.1 ± 0.1	7.0–10.0	13.1 ± 0.1	8.6–14.0
Volatile acidity (g L^{-1})	0.5 ± 0.1	max. 1.2	0.6 ± 0.1	max. 1.2
Free SO_2 (mg L^{-1})	47 ± 2	Na	52 ± 2	Na
Total SO_2 (mg L^{-1})	159 ± 1	max. 350	96 ± 1	max. 350

na = not applicable.

In relation to the color of the Syrah wine, a similar decrease in the *a** values during storage was observed for all bottle colors, with values in the range of 7.99-3.48. This decrease in the *a** values reflects a decrease in the red color of the wine and this change is more evident considering the increase in the hue angle (*h**) (in the range of 40.07º and 64.36º). According to Sant'Anna, Gurak, Marczak, Tessaro [21], an increase in the *h** values indicates an increase in the orange hue, which is evidence of the evolution of a wine. Marquez et al. [10] evaluated the stability of sweet red wines over 12 months and obtained results similar to those of this study. This color change can be attributed to a decrease in the anthocyanin content.

3.3 Evolution of the individual phenolic compounds

The profiles for the individual phenolic compounds in the sparkling Moscatel and Syrah red wines are provided in Tables 3 and 4, respectively.

3.3.1 Sparkling Moscatel

The profiles for the evolution of the phenolic compounds during the storage of the sparkling Moscatel wine in the bottles with different colors did not show considerable changes in the flavanols, phenolic acids and stilbenes during the storage time studied (Table 3). However, the flavanol catechin showed a significant increase during storage and this increase was similar in bottles of all colors. Previous studies conducted by Maury et al. [9], Clark et al. [7], Dias et al. [12] and Xing et al. [22] also evidenced an increase in the catechin contents during the aging process of white wines. This factor could explain the slight increase in the +*b** values (yellow color) for the sparkling Moscatel during storage (see Table 2), suggesting that aging occurred. Based on this finding, it is recommended that the sparkling Moscatel wine of the SFV is preferably consumed while still young.

3.3.2 Syrah Red Wine

In relation to the evolution of the phenolic compounds profile for the Syrah red wine (Table 4), the flavonols did not show notable changes during storage in the bottles of different colors. On the other hand, for the flavanol group, there was a decrease in the content of catechin and epicatechin. This behavior was also observed by Marquez et al. [10], who studied the stability of sweet Merlot, Syrah and Tempranillo red wines over 12 months. The decrease in the catechin and epicatechin levels could be due to tannin/tannin and/or tannin/anthocyanin polymerization occurring during the evolution of red wines [23].

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Table 2. Evolution of CIELab color of the sparkling Moscatel and Syrah wines stored in bottles of different colors for 12 months.

Color	Reference (0 months)	Clear bottle				Green bottle				Amber bottle			
		3 months	6 months	9 months	12 months	3 months	6 months	9 months	12 months	3 months	6 months	9 months	12 months
Sparkling Moscatel													
L*	54.03±0.40	53.69±0.55^{a}	53.75±0.34^{a}	53.89±0.42^{a}	54.21±0.06^{a}	53.36±0.08^{ab}	53.58±0.31^{a}	53.63±0.48^{a}	54.08±0.26^{a}	54.08±0.12^{a}	52.47±0.09^{b}	53.53±0.15^{a}	53.53±0.45^{a}
a*	-1.12±0.21	-1.10±0.20^{a}	-1.45±0.23^{a}	-1.65±0.16^{ab}	-2.04±0.04^{bc}	-1.58±0.06^{ab}	-1.64±0.17^{ab}	-1.67±0.15^{ab}	-2.15±0.03^{c}	-1.62±0.10^{ab}	1.51±0.25^{a}	-1.72±0.18^{abc}	-2.05±0.03^{bc}
b*	6.90±0.47	8.05±0.50^{de}	8.94±1.11^{bcde}	9.44±0.73^{bcd}	11.87±0.23^{a}	8.35±0.10^{cde}	9.50±0.26^{bc}	10.20±0.16^{b}	12.70±0.31^{a}	7.74±0.24^{e}	9.71±0.58^{bc}	10.20±0.24^{b}	12.45±0.19^{a}
C*	7.00±0.44	8.20±0.54^{d}	9.39±0.68^{bc}	9.59±0.74^{bc}	12.04±0.23^{a}	8.49±0.11^{cd}	9.64±0.29^{b}	10.33±0.14^{b}	12.88±0.31^{a}	7.91±0.22^{d}	10.16±0.03^{b}	10.35±0.23^{b}	12.62±0.19^{a}
Syrah red wine													
L*	17.55±1.11	13.48±0.34^{cd}	14.24±0.37^{bc}	15.82±0.27^{a}	13.97±0.22^{bcd}	13.62±0.49^{bcd}	14.10±0.03^{bc}	13.57±0.1^{bcd}	13.66±0.09^{bcd}	14.36±0.31^{b}	13.24±0.09^{d}	15.91±0.26^{a}	13.75±0.38^{bcd}
a*	7.99±0.57	4.47±0.38^{bcd}	4.34±0.37^{cde}	3.52±0.05^{de}	3.48±0.44^{e}	4.33±0.23^{cde}	4.39±0.21^{cde}	5.49±0.46^{a}	4.49±0.35^{e}	5.39±0.33^{ab}	4.59±0.21^{abc}	4.56±0.39^{abc}	3.69±0.26^{cde}
b*	6.74±0.84	6.51±0.27^{a}	6.60±0.28^{a}	5.02±0.04^{c}	6.08±0.43^{ab}	6.27±0.24^{ab}	6.56±0.09^{a}	6.59±0.19^{a}	6.29±0.24^{a}	6.27±0.40^{ab}	6.67±0.13^{a}	5.49±0.38^{bc}	6.37±0.21^{a}
C*	10.46±0.90	7.39±0.27^{cd}	8.46±0.21^{ab}	6.39±0.20^{d}	6.39±0.35^{d}	8.45±0.37^{ab}	8.44±0.44^{ab}	7.78±0.21^{bc}	7.54±0.34^{bc}	7.69±0.41^{bc}	8.45±0.18^{ab}	8.84±0.56^{a}	7.36±0.31^{cd}
h*	40.07±2.54	57.74±0.26^{bc}	53.99±0.90^{cde}	54.72±0.46^{cde}	64.36±4.31^{a}	55.14±0.68^{bcde}	52.40±1.26^{de}	58.01±0.27^{bc}	56.52±0.82^{bcd}	51.26±0.15^{e}	54.47±2.67^{cde}	53.15±2.17^{cde}	59.92±1.16^{ab}

Averages followed by the same letters in a line do not differ between themselves according to the Tukey test (p<0.05).

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Table 3. Evolution of phenolic compounds (mg L^{-1}) in the sparkling Moscatel stored in bottles of different colors for 12 months.

Compounds	Reference (0 months)	Clear bottle				Green bottle				Amber bottle			
		3 months	6 months	9 months	12 months	3 months	6 months	9 months	12 months	3 months	6 months	9 months	12 months
Flavonols													
Kaempferol	0.2±0.0	0.1±0.0^{b}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.1±0.0^{b}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0 ^{a}
Quercetin	1.4±0.2	0.4±0.0^{d}	1.0±0.2^{abc}	0.9±0.1^{c}	1.1±0.0^{ab}	0.3±0.0^{d}	1.0±0.7^{bc}	0.9±0.1^{c}	1.2±0.1^{a}	1.0±0.0^{bc}	0.9±0.1^{bc}	0.9±0.1^{c}	1.1±0.1 ^{ab}
Isorhamnetin	0.1±0.0	0.12±0.0^{b}	0.1±0.0^{a}	0.1±0.0^{ab}	0.1±0.0^{a}	0.1±0.0^{a}	0.1±0.0^{a}	0.1±0.0^{a}	0.1±0.0^{ab}	0.1±0.0^{a}	0.1±0.0^{a}	0.1±0.0^{ab}	0.1±0.0^{a}
Myricetin	0.2±0.0	0.2±0.0^{b}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{b}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0 ^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}
Rutin	0.2±0.0	0.1±0.0^{e}	0.2±0.0^{bcd}	0.2±0.0^{cd}	0.2±0.0^{cd}	0.1±0.0^{e}	0.2±0.0^{bc}	0.2±0.0^{ab}	0.2±0.0^{e}	0.2±0.0^{bc}	0.2±0.0^{cd}	0.2±0.0^{cd}	0.3±0.0^{a}
Σ Flavonols	**2.1**	**0.9**	**1.7**	**1.6**	**1.8**	**0.9**	**1.7**	**1.7**	**1.8**	**1.7**	**1.7**	**1.6**	**1.9**
Flavanols													
Catechin	1.5±0.5	0.5±0.0^{c}	1.8±0.1^{b}	1.7±0.1^{b}	6.0±0.0^{a}	0.5±0.0^{c}	1.8±0.0^{b}	1.7±0.1^{b}	5.8±0.0^{a}	1.7±0.1^{b}	1.7±0.1^{b}	1.6±0.2^{b}	5.8±0.0^{a}
Epicatechin	0.9±0.2	0.3±0.0^{e}	1.0±0.0^{abc}	0.9±0.1^{bc}	0.5±0.1^{d}	0.3±0.0^{e}	1.0±0.0^{a}	0.8±0.0^{bc}	0.9±0.0^{abc}	0.9±0.0^{abc}	0.9±0.1^{abc}	0.8±0.0^{c}	0.8±0.1^{c}
Procyanidin A2	0.4±0.0	0.3±0.0^{fg}	0.4±0.0^{de}	0.5±0.0^{a}	0.3±0.0^{f}	0.2±0.0^{g}	0.4±0.0^{bcd}	0.4±0.0^{bc}	0.3±0.0^{f}	0.4±0.0^{e}	0.4±0.0^{cde}	0.5±0.0^{ab}	0.3±0.0^{f}
Procyanidin B1	1.0±0.3	0.3±0.0^{de}	0.7±0.0^{bc}	0.5±0.1^{de}	3.7±0.1^{a}	0.3±0.0^{e}	0.6±0.0^{c}	0.5±0.0^{d}	3.6±0.1^{a}	0.8±0.0^{b}	0.6±0.0^{c}	0.4±0.1^{de}	3.7±0.1^{a}
Procyanidin B2	4.1±0.6	1.4±0.0^{c}	6.1±0.1^{a}	6.0±0.3^{a}	2 .4±0.0^{b}	1.4±0.0^{c}	6.1±0.2^{a}	5.8±0.3^{a}	2.3±0.1^{b}	5.6±0.2^{a}	5.8±0.3^{a}	6.0±0.1^{a}	2.4±0.1^{b}
Epigallocatechin	0.7±0.1	0.4±0.0^{fg}	0.7±0.0^{c}	1.1±0.0^{b}	1.2±0.0^{b}	0.3±0.0^{g}	0.4±0.0^{efg}	0.5±0.0^{cdef}	1.4±0.1^{a}	0.6±0.0^{cd}	0.7±0.2^{c}	0.5±0.0^{cde}	1.2±0.0^{b}
Epicatechin gallate	0.7±0.0	0.5±0.0^{fg}	0.8±0.0^{cd}	0.9±0.0^{a}	0.5±0.1^{fg}	0.5±0.0^{fg}	0.6±0.0^{e}	0.8±0.0^{b}	0.5±0.0^{g}	0.7±0.0^{d}	0.7±0.0^{d}	0.8±0.0 ^{bc}	0.6±0.0^{f}
Σ Flavanols	**9.4**	**3.7**	**11.3**	**11.6**	**14.6**	**3.5**	**11.0**	**10.5**	**14.8**	**10.6**	**10.9**	**10.7**	**14.8**
Phenolic acids													
Gallic acid	1.1±0.4	0.4±0.0^{g}	1.8±0.0^{abc}	1.8±0.0 ^{ab}	1.5±0.0^{cde}	0.4±0.0^{g}	1.7±0.0^{bcd}	1.9±0.0^{a}	1.4±0.0^{f}	1.6±0.0^{def}	1.8±0.1^{abc}	1.5±0.0^{ef}	1.4±0.1^{ef}
Caffeic acid	1.6±0.0	0.5±0.0d	1.8±0.1^{abc}	1.9±0.0^{ab}	2.0±0.0^{ab}	0.5±0.0^{d}	1.9±0.0^{ab}	2.0±0.1^{ab}	2.1±0.1^{a}	1.6±0.0^{c}	1.8±0.1^{bc}	1.9±0.2^{ab}	2.1±0.1^{a}
Caftaric acid	57.5±1.7	18.1±0.5^{d}	71.8±0.6^{ab}	66.8±1.6^{abc}	64.8±1.5^{c}	17.6±0.1^{d}	73.2±1.5^{a}	66.4±1.4^{abc}	66.3±1.3^{bc}	71.8±2.5^{ab}	71.7±3.7^{ab}	65.3±5.7^{bc}	67.4±0.7^{abc}
Chlorogenic acid	1.4±0.2	0.4±0.0^{e}	1.1±0.1^{cd}	0.8±0.0^{d}	3.9±0.1^{a}	0.4±0.0^{e}	1.1±0.0^{bc}	0.9±0.1^{d}	3.8±0.2^{a}	1.3±0.1^{b}	1.0±0.0^{cd}	0.8±0.1^{d}	4.0±0.0^{a}
p-Coumaric acid	0.3±0.0	0.2±0.0^{e}	0.3±0.0^{d}	0.3±0.0^{bcd}	0.4±0.0^{a}	0.1±0.0^{e}	0.4±0.0^{abcd}	0.4±0.0^{abcd}	0.4±0.0^{abc}	0.3±0.1^{d}	0.3±0.0^{cd}	0.4±0.0^{abcd}	0.4±0.0^{ab}
Ferulic acid	0.3±0.0	0.2±0.0^{b}	0.4±0.0^{a}	0.4±0.0^{a}	0.1±0.0^{c}	0.2±0.0^{c}	0.4±0.0^{a}	0.4±0.0^{a}	0.1±0.0^{c}	0.4±0.0^{a}	0.4±0.0^{a}	0.4±0.1^{a}	0.1±0.0^{c}
Σ Phenolic acids	**62.2**	**19.7**	**77.2**	**72.1**	**72.8**	**19.7**	**78.7**	**71.9**	**74.2**	**77.0**	**77.0**	**70.3**	**75.4**
Stilbenes													
trans Resveratrol	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd
cis Resveratrol	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd	Nd
Piceatannol	0.1±0.0	0.2±0.0^{c}	0.3±0.0^{b}	0.3±0.0^{b}	0.5±0.0^{a}	0.2±0.0^{c}	0.3±0.0^{b}	0.3±0.0^{b}	0.5±0.0^{a}	0.3±0.0^{b}	0.3±0.0^{b}	0.3±0.0^{b}	0.5±0.0^{a}
Viniferin	0.2±0.0	0.2±0.0 **b**	0.2±0.0^{a}	0.2±0.0^{a}	0.0±0.0 **c**	0.2±0.0^{b}	0.2±0.0^{a}	0.2±0.0^{a}	0.0±0.0^{c}	0.2±0.0^{a}	0.2±0.0^{a}	0.2±0.0^{a}	0.0±0.0^{c}
Σ Stilbenes	0.3	0.4	0.5	0.5	0.5	0.4	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Total Phenolics¥	280.0±8.1	245.0±18.1^{abc}	252.9±12.7^{ab}	231.4±5.4^{bcd}	214.7±8.3^{cd}	237.9±9.8 ^{abcd}	229.9±6.7^{bcd}	248.2±5.6^{ab}	150.0±1.1^{e}	212.5±20.5^{d}	212.3±6.7^{d}	267.0±1.9^{a}	225.2±11.2^{bcd}

Averages followed by the same letters in a line do not differ between themselves according to the Tukey test (p<0.05). Nd= Not detected. ¥Total phenolics quantified through the colorimetric method with Folin-Ciocalteu reagent.

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Table 4. Evolution of phenolic compounds (mg L^{-1}) in the Syrah red wine stored in bottles of different colors for 12 months.

Compounds	Reference (0 months)	Clear				Green				Amber			
		3 months	6 months	9 months	12 months	3 months	6 months	9 months	12 months	3 months	6 months	9 months	12 months
Flavonols													
Kaempferol	1.2±0.1	1.1±0.0^{a}	1.0±0.0^{abc}	1.1±0.0^{a}	0.9±0.1^{cd}	1.0±0.1 ^{abc}	1.0±0.0^{abc}	1.1±0.0^{a}	0.9±0.0^{bcd}	1.1±0.0^{ab}	1.0±0.0^{abc}	1.0±0.1^{abcd}	0.9±0.1^{d}
Quercetin	32.5±1.4	23.2±0.1^{ab}	20.0±0.4^{c}	20.3±0.3^{c}	21.3±1.2^{bc}	24.4±1.0^{a}	20.8±0.3^{bc}	19.6±0.7^{c}	21.2±1.1^{bc}	24.3±1.0^{a}	20.6±0.8^{bc}	19.1±0.6^{c}	21.0±1.7^{bc}
Isorhamnetin	4.1±0.3	3.3±0.2^{a}	2.8±0.1^{b}	3.1±0.1^{b}	4.9±0.3^{b}	3.4±0.1^{a}	3.0±0.0^{b}	3.0±0.0^{b}	5.0±0.3^{b}	3.3±0.0^{a}	3.0±0.1^{b}	3.0±0.1^{b}	4.9±0.4^{b}
Myricetin	1.0±0.0	1.0±0.0^{a}	0.9±0.0^{b}	0.8±0.0^{c}	0.9±0.0^{bc}	1.0±0.0^{a}	0.9±0.0^{b}	0.8±0.0^{c}	0.9±0.0^{bc}	1.0±0.0^{a}	0.9±0.0^{b}	0.9±0.0^{bc}	0.9±0.0^{bc}
Rutin	1.7±0.2	1.3±0.3^{c}	1.7±0.1^{abc}	2.6±0.2^{ab}	2.9±0.7^{a}	1.6±0.2^{bc}	1.9±0.0^{abc}	2.2±0.5^{abc}	2.5±0.6^{ab}	1.8±0.1^{abc}	2.0±0.0^{abc}	1.9±0.1^{abc}	2.6±0.8^{ab}
Σ Flavonols	**40.4**	**29.7**	**26.5**	**27.9**	**30.9**	**31.4**	**27.6**	**26.7**	**30.5**	**31.3**	**27.5**	**25.8**	**30.2**
Flavanols													
Catechin	26.1±1.9	19.8±0.5^{ab}	17.5±0.9^{c}	17.3±0.7^{c}	3.5±0.1^{e}	21.0±0.7^{a}	18.7±0.0^{bc}	17.4±0.2^{c}	3.4±0.1^{e}	20.5±0.3^{a}	18.4±0.8^{bc}	15.6±0.7^{d}	3.3±0.1^{e}
Epicatechin	19.5±1.3	13.4±1.0^{ab}	11.3±0.7^{cd}	10.7±0.3^{de}	1.7±0.1^{f}	13.9±0.4^{a}	12.2±0.4^{bc}	10.6±0.0^{de}	1.6±0.1^{f}	13.6±0.2^{ab}	11.7±0.7^{cd}	9.8±0.6^{e}	1.5±0.1^{f}
Procyanidin A2	4.2±0.1	3.5±0.0^{abc}	3.1±0.0^{de}	2.9±0.2 ^{e}	1.0±0.0^{f}	3.6±0.1^{a}	3.3±0.0 ^{bcd}	3.1±0.3^{de}	1.0±0.0^{f}	3.6±0.1^{ab}	3.2±0.1^{cd}	2.9±0.1^{e}	1.0±0.0^{f}
Procyanidin B1	18.8±0.4	21.1±0.3^{a}	21.3±0.5^{a}	21.5±0.7^{a}	36.8±0.8^{b}	22.4±0.5^{a}	22.6±0.4^{a}	21.6±0.2^{a}	39.0±0.2^{a}	21.6±0.2^{a}	21.4±1.5^{a}	18.7±0.3^{d}	37.3±0.2^{ab}
Procyanidin B2	21.7±0.5	22.0±1.1^{de}	19.5±0.4^{f}	25.4±0.9^{c}	30.8±1.0^{b}	22.4±0.5^{de}	21.9±0.6^{def}	24.3±1.5^{cd}	33.4±0.5^{a}	21.9±0.5^{def}	21.4±0.5^{ef}	20.7±1.4^{ef}	31.9±0.1^{ab}
Epigallocatechin	4.6±0.5	2.2±0.0^{def}	3.9±0.1^{a}	3.7±0.3^{a}	1.4±0.0^{f}	3.1±0.0^{abcd}	3.5±0.2^{ab}	2.6±0.9^{bcde}	1.7±0.1^{ef}	3.4±0.1^{ab}	3.3±0.8^{abc}	2.3±0.1^{cdef}	1.7±0.2^{f}
Epicatechin gallate	3.2±0.8	2.8±0.2^{b}	2.6±0.1^{b}	2.7±0.1^{b}	3.4±0.3^{a}	2.9±0.1^{b}	2.7±0.1^{b}	2.6±0.1^{b}	2.9±0.2^{b}	2.7±0.1^{b}	2.6±0.2^{b}	2.5±0.1^{b}	2.9±0.1^{b}
Σ Flavanols	**98.0**	**84.8**	**79.3**	**84.1**	**78.5**	**89.4**	**84.8**	**82.1**	**83.0**	**87.3**	**82.0**	**72.37**	**79.6**
Phenolic acids													
Gallic acid	25.0±1.4	29.1±0.8^{e}	30.9±0.9^{cde}	33.9±0.7^{a}	33.5±0.6^{ab}	29.7±1.0^{de}	31.57.6±0.2^{bcd}	33.8±0.4^{a}	33.6±0.4^{a}	28.9±0.6^{e}	31.4±0.8^{cd}	32.1±0.1^{abc}	31.8±1.9^{abc}
Caffeic acid	9.6±0.3	9.1±0.0^{bc}	9.1±0.4^{bc}	8.5±0.1^{cd}	7.9±0.1^{d}	10.6±0.2^{a}	9.4±0.3^{bc}	9.5±0.2^{abc}	8.6±0.6^{cd}	9.9±0.5^{ab}	9.4±0.3^{bc}	9.4±0.2^{bc}	8.6±0.8^{cd}
Caftaric acid	91.2±0.7	84.6±1.2^{ab}	77.3±2.6^{cde}	76.4±0.3^{de}	75.4±1.1^{e}	85.6±1.0^{a}	80.8±1.6^{bc}	77.4±0.5^{cde}	76.6±1.1^{de}	86.8±2.3^{a}	79.6±1.6^{cd}	77.6±1.1^{cde}	76.1±0.6^{de}
Chlorogenic acid	4.4±0.3	4.4±0.1^{b}	5.7±0.2^{a}	5.6±0.3^{a}	5.5±0.2^{a}	5.8±0.1^{a}	5.6±0.1^{a}	6.1±0.2^{a}	5.7±0.6^{a}	5.3±0.5 ^{a}	5.8±0.3^{a}	5.6±0.2^{a}	5.9±0.5^{a}
p-Coumaric acid	1.4±0.0	2.4±0.0^{de}	2.3±0.0^{de}	3.2±0.0^{b}	3.8±0.0^{a}	2.3±0.0^{e}	2.4±0.0^{de}	3.1±0.1^{bc}	3.7±0.2^{a}	1.9±0.1^{f}	2.5±0.0^{d}	3.0±0.0^{c}	3.8±0.1^{a}
Ferulic acid	3.1±0.1	2.8±0.0^{a}	2.5±0.1^{b}	2.5±0.1^{b}	2.3±0.1^{c}	2.8±0.1^{a}	2.6±0.0^{b}	2.6±0.0^{b}	2.3±0.1^{c}	2.8±0.1^{a}	2.6±0.1^{b}	2.6±0.1^{b}	2.2±0.1^{c}
Σ Phenolic acids	**134.6**	**132.5**	**127.8**	**130.0**	**128.4**	**136.9**	**132.4**	**132.5**	**130.5**	**135.7**	**131.2**	**130.2**	**128.4**
Stilbenes													
trans Resveratrol	0.6±0.0	0.6±0.0^{ab}	0.6±0.0^{ab}	0.6±0.0^{bc}	0.5±0.0^{c}	0.6±0.0^{a}	0.6±0.0^{ab}	0.6±0.1^{ab}	0.5±0.0^{c}	0.6±0.0^{ab}	0.6±0.0^{ab}	0.6±0.0^{ab}	0.5±0.0^{c}
cis Resveratrol	0.8±0.1	0.4±0.0^{cd}	0.5±0.1^{ab}	0.4±0.0^{cd}	0.6±0.0^{a}	0.4±0.0^{cd}	0.4±0.0^{bcd}	0.5±0.0^{bc}	0.6±0.0^{a}	0.4±0.0^{cd}	0.4±0.0^{d}	0.4±0.0^{cd}	0.5±0.0^{a}
Piceatannol	2.0±0.1	1.6±0.0^{bc}	1.4±0.0^{de}	1.4±0.1^{d}	0.8±0.0^{f}	1.7±0.1^{a}	1.5±0.0^{cd}	1.4±0.1^{d}	0.8±0.0^{f}	1.7±0.0^{ab}	1.5±0.1^{cd}	1.3±0.0^{e}	0.8±0.0^{f}
Viniferin	0.8±0.0	0.8±0.0^{ab}	0.8±0.0^{c}	0.8±0.0^{c}	0.7±0.0^{d}	0.8±0.0^{a}	0.8±0.0^{cd}	0.8±0.0^{cd}	0.7±0.0^{d}	0.8±0.0^{cd}	0.8±0.0^{cd}	0.8±0.0^{c}	0.7±0.0^{d}
Σ Stilbenes	**4.2**	**3.4**	**3.3**	**3.2**	**2.6**	**3.5**	**3.3**	**3.3**	**2.6**	**3.5**	**3.3**	**3.1**	**2.6**
Anthocyanins													
Pelargonidin 3-glucoside	15.8±0.4	9.1±0.4^{a}	5.3±0.5^{b}	4.1±0.2^{c}	2.9±0.2^{d}	9.8±0.3^{a}	6.1±0.2^{b}	4.0±0.1^{cd}	3.4±0.1^{cd}	9.2±0.6^{a}	5.7±0.6^{b}	3.7±0.7 ^{cd}	3.2±0.1^{cd}
Cyanidin 3-glucoside	1.2±0.0	1.1±0.0^{a}	0.8±0.0^{de}	0.7±0.0^{efg}	0.6±0.0^{g}	1.0±0.1^{ab}	0.8±0.1^{cd}	0.7±0.0^{defg}	0.6±0.0^{g}	0.9±0.1^{bc}	0.8±0.0^{def}	0.7±0.0^{fg}	0.6±0.0^{g}
Delphinidin 3-glucoside	9.9±0.2	6.3±0.2^{a}	3.8±0.4^{cd}	3.26±0.07^{de}	2.1±0.1^{g}	6.7±0.2^{a}	4.5±0.2^{b}	3.1±0.0^{e}	2.4±0.1^{fg}	6.3±0.2^{a}	4.2±0.4^{bc}	2.9±0.4^{ef}	2.3±0.1 ^{fg}
Malvidin 3-glucoside	88.5±1.6	52.4±0.8^{a}	29.7±3.0^{c}	22.7±0.8^{d}	13.6±0.0^{f}	55.7±1.0^{a}	34.6±1.4^{b}	22.0±0.1^{d}	16.6±0.5^{ef}	55.0±1.8^{a}	32.3±2.5^{bc}	17.9±0.5^{e}	15.6±0.4^{ef}
Peonidin 3-glucoside	14.2±0.7	8.8±0.3^{a}	4.8±0.6^{b}	3.6±0.1^{c}	2.5±0.2^{d}	8.8±0.2^{a}	5.7±0.4^{b}	3.5±0.1^{cd}	2.8±0.1^{cd}	8.7±0.3^{a}	5.2±0.5^{b}	3.2±0.5^{cd}	2.7±0.1^{cd}
Petunidin 3-glucoside	4.3±0.8	2.3±0.1^{a}	1.4±0.1^{c}	1.32±0.01^{c}	1.0±0.0^{d}	2.5±0.1^{a}	1.6±0.0^{b}	1.3±0.0^{c}	1.1±0.0^{d}	2.4±0.1^{a}	1.5±0.0^{bc}	1.1±0.1^{d}	1.1±0.0^{d}
Σ Anthocyanins	**134.4**	**80.5**	**45.7**	**35.6**	**22.7**	**84.5**	**53.3**	**34.6**	**26.9**	**82.5**	**49.7**	**29.3**	**26.4**
Total Monomeric Anthocyanins$^{\Psi}$	243.6±1.0	179.0±3.0^{a}	129.0±6.2^{b}	94.2±1.0^{c}	38.2±1.0^{e}	182.6±5.0^{a}	135.1±5.4^{b}	105.8±5.4^{c}	65.8±2.0^{d}	186.3±4.9^{a}	126.6±3.0^{b}	97.7±6.6^{c}	73.8±1.0^{d}
Total Phenolics$^{¥}$	2439.8±22.0	1945.6±86.3^{d}	1955.3±62.3^{cd}	2125.2±27.9^{abc}	1933.2±37.6^{d}	2023.9±8.0^{bcd}	2050.9±9.3^{bcd}	2236.7±18.6^{a}	2029.2±93.1^{bcd}	1954.6±58.5^{cd}	2071.8±24.1^{abc} d	2193.4±51.2^{ab}	2094.3±115.4^{abcd}

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Averages followed by the same letters in a line do not differ between themselves according to the Tukey test ($p<0.05$). ¥ Total phenolics quantified through the colorimetric method with Folin-Ciocalteu reagent. Ψ Total monomeric anthocyanins quantified through the pH differential method.

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These polymerization reactions, in turn, lead to a change from an intense red color to an orange color [24], which explains the increase in the hue (*h**) observed for the Syrah wine after 12 months of storage (see Table 2).

In relation to the phenolic acids, caftaric acid was the major compound in the Syrah wine and it decreased during the storage time in the bottles of different colors (with values ranging from 91.2 to 75.4 mg L^{-1}). This behavior was also observed in red wines by Scrimgeour, Nordestgaard, Lloyd, Wilkes [25], who attributed this decrease to a high affinity of this compound toward the oxygen dissolved in wines. This oxidation reaction can adversely affect the sensory quality of the wine.

In relation to the stilbenes, the concentration of piceatannol decreased in the bottles of all colors. This compound is considered important since even at low concentrations its consumption has been associated with a decrease in the incidence of cardiovascular diseases [26]. In the Syrah red wine of the SFV, the concentration of piceatannol in the recently produced wine was 2 mg L^{-1}, and after 12 months of storage in bottles it was 0.76 mg L^{-1}. Guerrero et al. [27] observed a piceatannol concentration of 1.05 mg L^{-1} in red wine produced from the Jaen grape variety, after applying artificial UV light to grapes in order to stimulate its synthesis, which suggests that the values found for the Syrah red wine of the SFV can be considered good.

In relation to the total monomeric anthocyanins in the wine, as well as for the individual anthocyanins, quantified through high-performance liquid chromatography (HPLC), there was a decrease during storage in bottles of all colors. Similar behavior has been observed in previous studies on red wine storage in bottles of a single color [10,28]. Specifically, during storage in clear bottles for 12 months the total monomeric anthocyanins decreased by 84.3% and this coincided with an 84.6% decrease in the content of malvidin 3-glucoside, which is the main anthocyanin responsible for the color of *Vitis vinifera* L. wines. These results suggest that the use of clear bottles may be detrimental to the preservation of the red wine color when the storage period exceeds a year.

3.4 Principal Component Analysis

Principal component analysis (PCA) was applied to the results obtained for the sparkling Moscatel and Syrah wines (Figure 1). For the sparkling Moscatel (Figure 1A), the first two principal components (PC1 + PC2) explained 92.81% of the data variance. For analysis purposes, only compounds with a loading > 0.70 will be considered. The PC1 with the highest weight of explained variance (57.80%) separated the treatments MGB3 and MCB3 in the negative part of the PC1, and this separation was associated with the negative loading for myricetin. The PC2 with the lowest weight of explained variance (35.01%) separated the wines with 6 and 9 months of bottle storage, in the positive part, associated with the loadings for ferulic acid, procyanidin A2 and B2, epicatechin gallate and viniferin. The wines with 12 months of storage were separated in the negative part (PC2) by the compounds procyanidin B1, catechin and chlorogenic acid. Based on the PCA, the separations obtained for the sparkling Moscatel were associated with the storage time.

In relation to the Syrah red wine (Figure 1B), the first two main components (PC1 + PC2) explained 93.8% of the data variance. The treatments with 3 months of storage in the bottles of the three colors (SCB3, SGB3 and SAB3) were separated in the positive part of the PC2, and the compounds responsible for this separation were petunidin, quercetin, myricetin, caftaric acid and ferulic acid.

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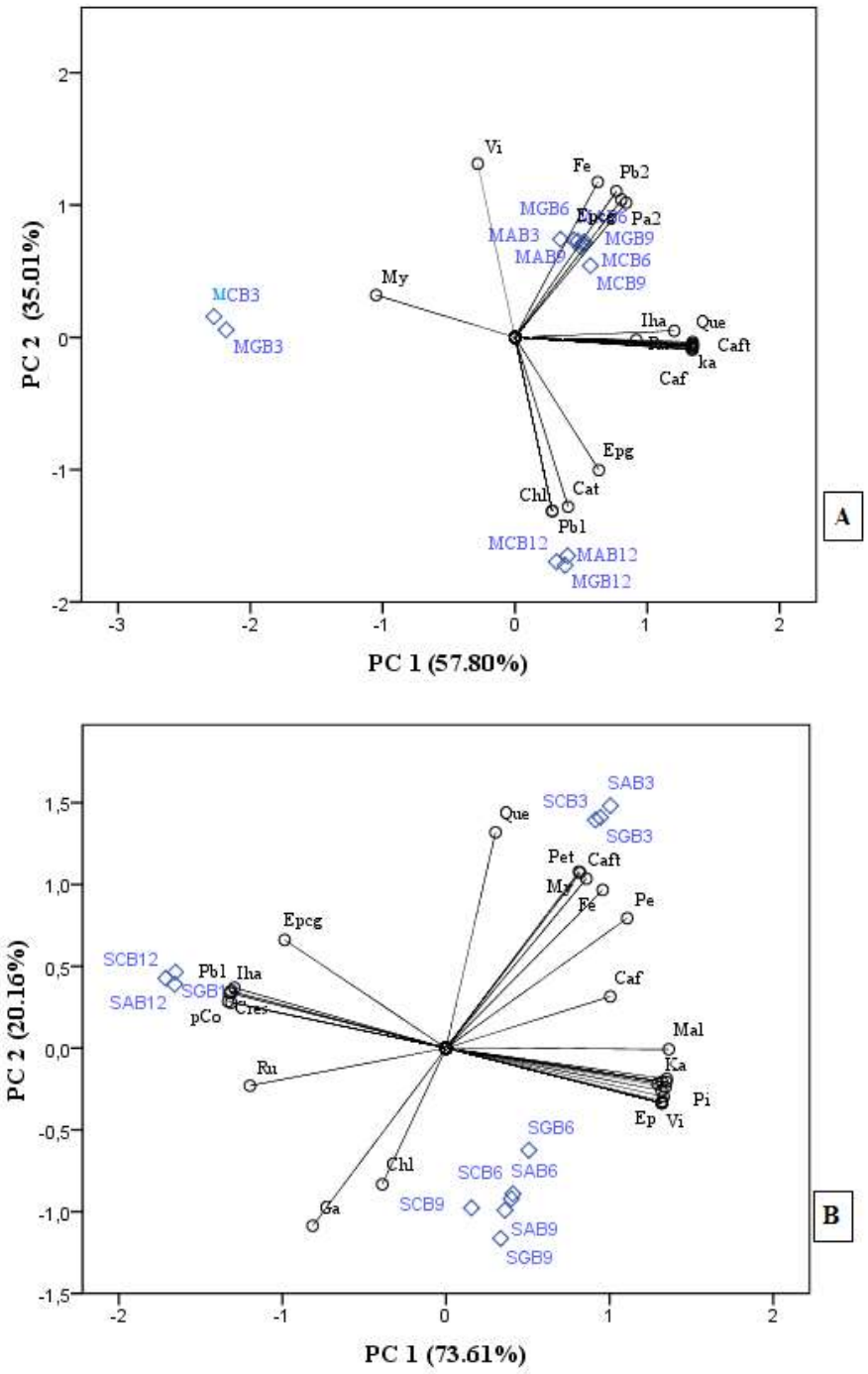

Figure 1. Principal component analysis (PCA) of phenolic compounds and the antioxidant activity results for sparkling Moscatel (A) and Syrah (B) wines stored in bottles of different colors for 12 months.

Legend: clear bottle 3 months (CB3), green bottle 3 months (GB3), amber bottle 3 months (AB3); clear bottle 6 months (CB6), green bottle 6 months (GB6), amber bottle 6 months (AB6); clear bottle 9 months (CB9), green bottle 9 months (GB9), amber bottle 9 months (AB9); clear bottle 12 months (CB12), green bottle 12 months (GB12), amber bottle 12 months (AB12). Kaempferol 3-glucoside (ka); quercetin 3-glucoside (Que); isorhamnetin (Iha); myricetin (My); rutin (Ru); caffeic acid (Caf); caftaric acid (caft); chlorogenic acid (Chl); *p*-coumaric (p-Co); ferulic acid (Fe); piceatannol (Pi); catechin (Cat); epicatechin (Ep); procyanidin A2 (Pa2);

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procyanidin B1 (Pb1); procyanidin B2 (Pb2); gallic acid (Ga); epigallocatechin (Epg); epicatechin gallate (Epcg); viniferin (Vi); pelargonidin (Pel); cyanidin (Cy); delphinidin (Del); malvidin (Mal); peonidin (Pe); petunidin (Pet); *trans*-resveratrol (Ter); *cis*-resveratrol (Cre). ◊ = treatments studied.

The treatments with 6 and 9 months in bottles of all colors (SCB6, SGB6, SAB6; SCB9, SGB9, and SAB9) were separated in the negative part of the PC2, associated with chlorogenic acid and gallic acid. The treatments for 12 months in bottles of all colors (SCB12, SGB12, and SAB12) were separated in the negative part of the PC1 and were associated with higher values for isorhamnetin, rutin, *p*-coumaric acid, procyanidin B1 and B2, epicatechin gallate and *cis*-resveratrol and lower values for malvidin 3-glucoside, catechin and epicatechin.

Overall, the PCA of the results for the two wines studied showed that the evolution of the profile for the phenolic compounds studied was due to the storage time of up to 12 months and not the color of the bottles used to store the wines.

3.5 Antioxidant activity in vitro

The results for the antioxidant activity (AOX) of the wines studied are given in Figure 2. The AOX for the sparkling Moscatel measured with the DPPH method (Figure 2A) showed an increase during sixth months of storage for the clear bottles and ninth months of storage for the green and amber bottles. In relation to the AOX measured through the ABTS method, the AOX increased in the wines with six months of storage in the green and amber bottles and nine months in the clear bottles. Kallithraka, Salacha, Tzourou [29] studied the aging of white wine and also observed an increase in the antioxidant activity after six months of bottle storage.

In the case of the Syrah red wine, the AOX measured through both methods showed an increase as a function of the storage time for the amber bottles, while the wine stored in the clear and green bottles remained stable in this regard. The AOX values obtained in this study are in agreement with those reported by Padilha et al. [30] for commercial Syrah red wines of the SFV, and they are considered to be high. In general, the wines stored in bottles of different colors showed good stability with regard to the antioxidant activity *in vitro*, which is a good result since the AOX of wines is normally associated with consumer health protection in relation to several diseases [31].

4. Conclusions

The different bottle colors studied did not influence the evolution of the sparkling Moscatel and Syrah red wines since the main variations observed were associated with the storage time. The main changes observed in the Syrah red wine were an increase in the hue (*h**), decrease in catechins, increase in procyanidins and, most notably, a decrease in the anthocyanin malvidin 3-glucoside, which is the main compound responsible for the color of red wines produced from *V. vinifera* L. grapes. In general, the wines studied presented stability in terms of the antioxidant activity *in vitro*.

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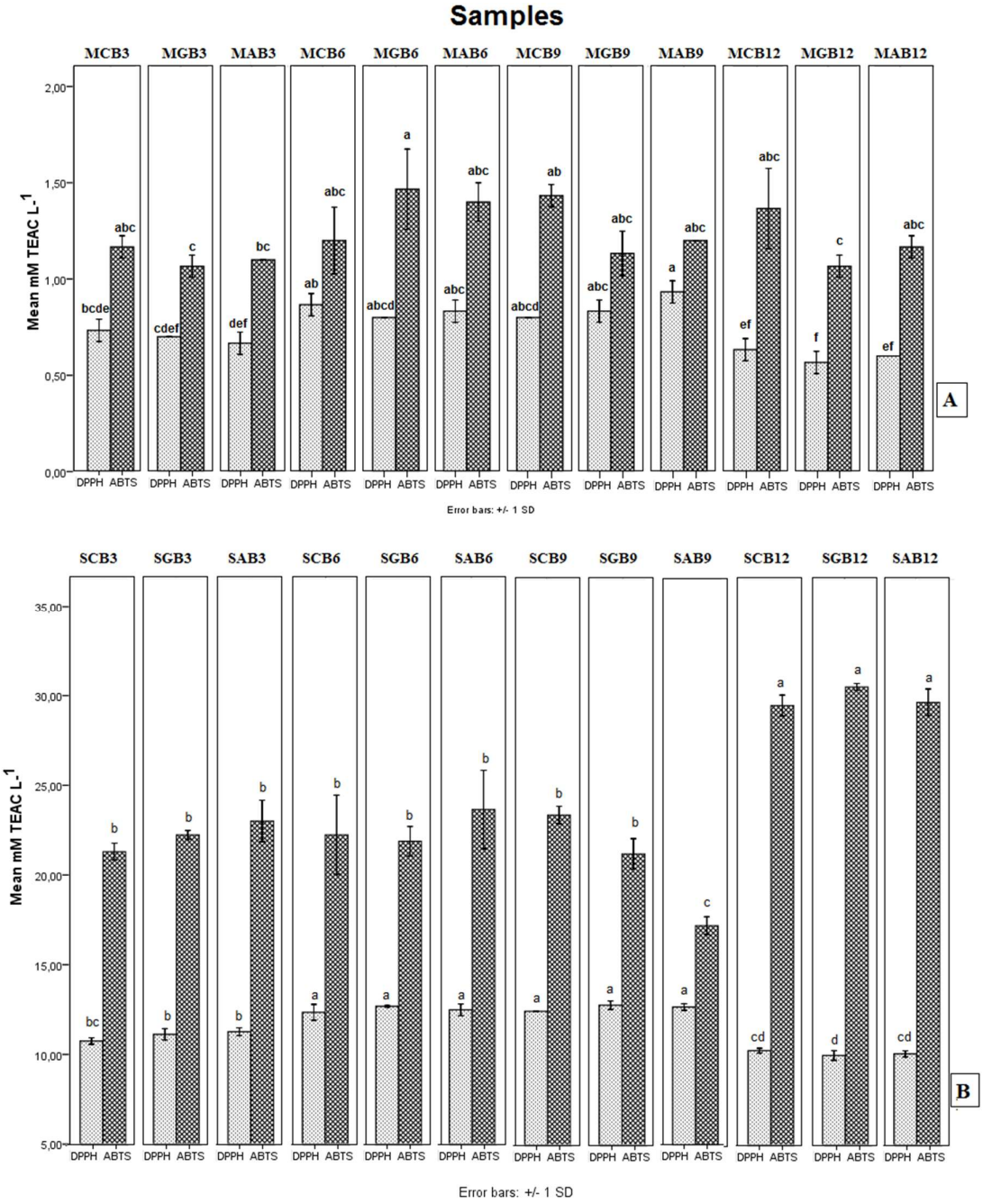

Figure 2. Antioxidant activity *in vitro* of the sparkling Moscatel (A) and Syrah (B) wines stored in bottles of different colors for 12 months.

Author contributions

Conceptualization, J.F.S. and A.M.S.N; Formal analysis, M.S.S.L. and M.d.C.P.D; Writing and Methodology, M.d.S.L. and G.E.P.

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Funding

CAPES (Coordination for the Improvement of Higher Education Personnel)

Acknowledgements

The authors would like to thank CAPES (Coordination for the Improvement of Higher Education Personnel) for the concession of scholarships and financial support. The authors would also like to thank the Miolo Wine Group, Piagentini and Botticelli for supplying the raw-material.

Conflict interest

The author declares that there is no conflict of interests regarding the publication of this case study article.

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