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ORIGINAL ARTICLE

Effect of the cold pre-fermentative maceration and aging on lees times on the phenolic compound profile, antioxidant capacity and color of red sparkling wines

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Abstract This was the first study evaluating the impact of cold pre-fermentative maceration using refrigeration on the nutraceutical quality and color of red sparkling wines elaborated with the cultivar Syrah, and the evolution of these variables with different autolysis times. The sparkling wines were elaborated using the traditional method with different maceration times (NM, 24 and 72 h) and aging on lees (3 and 18 months of autolysis). In the sequence, it was conducted the characterization of the phenolic compound profile by HPLC–DAD ($n = 21$), the antioxidant capacity (ABTS, DPPH, and FRAP assays), and the color (CIELab and CIEL*C*h systems). The total phenolic content (TPC) and antioxidant capacity (AOX) were higher with longer maceration (M72) and autolysis (18 months) times, reaching 453.54 mg L^{-1} of TPC, and AOX above 2.11 mmol TEAC L^{-1} by the three in vitro assays conducted. *Cis*-resveratrol, kaempferol-3-*O*-glucoside, quercetin-3-β-D-glucoside, isorhamnetin-3-*O*-glucoside, and petunidin-3-*O*-glucoside showed a good correlation ($r > 0.8$; $P < 0.05$) with the antioxidant capacity and were found in higher concentrations in the sparkling wines elaborated with maceration. In addition, maceration promoted a more intense red (a*) and saturated (C*) color. Thus, the results indicated that cold pre-fermentative maceration and autolysis positively influenced the bioactive potential and the color of the red sparkling wines. This practice should be better explored through the elaboration of this product.

Keywords Syrah (Shiraz) · Traditional method (*Champenoise*) · Autolysis · Bioactive compounds · In vitro antioxidant activity

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Introduction

There has been a search for diversification in the production of sparkling wines in recent years, exploring new varieties and styles (Sartor et al. 2019a; González-Lázaro et al. 2019; Eder and Rosa 2021; Prokes et al. 2022). Although most are white or rosé, the elaboration of red sparkling wines could be a new alternative for production and marketing (González-Lázaro et al. 2019). The increasing interest in the production of red sparkling wines has encouraged research concerning their chemical composition and the contributions of these characteristics to increase their quality and complexity (Rizzolo et al. 2018; González-Lázaro et al. 2019; Pérez-Magariño et al. 2019). The red grape of the cultivar Syrah is already widely used to elaborate still red wines (Garrido-Bañuelos et al. 2021; Alves et al. 2022) and for sparkling wines in countries such as Australia and Brazil (Eder and Rosa 2021). However, there is still no research

aimed at understanding the behavior of this variety in the elaboration of red sparkling wines. The use of red grapes to elaborate this product should aim at adequate technological and phenolic maturity, such that the base wine reaches a moderate alcoholic content (10.0–11.5ºGL) and low pH value (bellow 3.5), with the acidity and phenolic compound content well-balanced to guarantee the sensory characteristics desired by the winemaker (González-Lázaro et al. 2020).

Thus, the use of different enological techniques has been put into practice to obtain high quality red sparkling wines (Rizzolo et al. 2018; González-Lázaro et al. 2019; Pérez-Magariño et al. 2019). In the elaboration of still red wines, different non-traditional pre-fermentative enological techniques have been studied to understand their effects on the color and phenolic compound composition of the beverage (Heredia et al. 2010; Cejudo-Bastante et al. 2014). One example is cold pre-fermentative maceration, which could be adopted to extract water soluble compounds in the absence of alcohol at low temperatures, mainly phenolic and volatile compounds (Ribéreau-Gayon et al. 2006). This technique has been used in the elaboration of sparkling wines to obtain white, rosé, or red products depending on the time and temperature employed. In addition, different protocols can be adopted, influencing the contents of the compounds extracted and providing chemical and sensory changes to the beverage (Ruiz-Moreno et al. 2017; Pérez-Magariño et al. 2019; González-Lázaro et al. 2019). Of these protocols, cold pre-fermentative maceration with the use of dry ice provided good results for the elaboration of red sparkling wines (González-Lázaro et al. 2019). Refrigeration is another option for cold pre-fermentative maceration, and it was compared with the use of dry ice to elaborate Syrah still red wines (Heredia et al. 2010). The authors observed a greater potential for refrigeration due to better extraction of phenolic compounds and greater color stability. However, the technique of cold pre-fermentative maceration with the use of refrigeration has still not been exploited for the elaboration of red sparkling wines. The phenolic compounds are already being studied as a quality marker for white and rosé sparkling wines (Sartor et al. 2019a, b). Additionally, a greater content of phenolic compounds can extend the shelf-life of food products (Galanakis 2018).

On the other hand, in the elaboration of sparkling wines by the traditional method, the time of aging on lees (autolysis) is also a determinant factor for the composition and complexity of the product. Thus, giving rise to more aromatic and balanced sparkling wines, with a greater potential for aging and color stability (Gallardo-Chacón et al. 2010) and the duration of this step and the reactions involved has been the object of various studies (Ruiz-Moreno et al. 2017; Sartor et al. 2019a, 2021). Furthermore, the interaction between the cell surface of the yeast lees during autolysis has been investigated, to understand its antioxidant action, since this could protect the sparkling wine against oxidation by consuming small amounts of oxygen during autolysis (Gallardo-Chacón et al. 2010). However, it was shown that the antioxidant capacity of the yeast lees decreased with an autolysis time greater than 36 months (Pons-Mercadé et al. 2021). Hence, the authors showed that the duration of this step was a determinant factor in the maintenance of the sparkling wine bioactive quality. In addition, recent studies evaluated the phenolic and volatile compositions and color of red sparkling wines after nine months of autolysis (Pérez-Magariño et al. 2019; González-Lázaro et al. 2019), nonetheless the autolysis time was not a focus of these studies. To the present moment, only red sparkling wines elaborated with four and nine months of autolysis have been registered (Eder and Rosa, 2021), so it is important to explore a longer time for this step to provide greater complexity and stability to the product. In addition, optimizing operational parameters in winemaking technologies is essential for the maintenance of bioactive compounds, such as phenolic compounds (Galanakis 2021).

The present study aimed to provide, for the first time, information concerning the impact of the time of cold pre-fermentative maceration with the use of refrigeration, on the extraction of phenolic compounds, antioxidant capacity and color of red sparkling wines elaborated with the cultivar Syrah, and also evaluate the evolution of these variables during different autolysis times.

Material and methods

Raw material

Four hundred and fifty kilos of Syrah grapes cultivated in the experimental area of Embrapa Semiárido (09° 09' S, 40° 22' W, 365.5 m, Petrolina—PE, Brazil), were harvested in September 2017, with a soluble solids content 20.1 ºBrix, pH 3.1 and total acidity of 8.6 g L^{-1} (expressed as tartaric acid) and used to elaborate red sparkling wines. The vines were cultivated on a vertical shoot positioning trellis system, grafted onto Paulsen 1103 rootstock, and drip irrigated.

Winemaking process

The base wines were elaborated in triplicate. The Syrah grapes were homogenized and divided into three batches, each with about 150 kg, one for each of the following treatments: Non-macerated (NM), with cold pre-fermentative maceration for 24 h (M24), and with cold pre-fermentative maceration for 72 h (M72). The NM batch grapes were destemmed, crushed, pressed, and clarified (8 °C for 48 h). The M24 and M72 grapes were destemmed and crushed and then submitted to cold pre-fermentative maceration with

refrigeration in a cold chamber (8 ± 2 ºC) in three 50 kg capacity stainless steel tanks and then pressed and clarified (8 ºC for 48 h). During destemming, 120 mg Kg^{-1} of potassium metabisulfite (Amazon Group Ltda., Brazil) and 0.02 mg L^{-1} of the pectolytic enzyme Everzym Blanc (Ever Brasil, Brazil) were added. After pressing, the musts of each treatment were transferred to three 20 L glass jars closed with glass airlocks. After clarification, alcoholic fermentation was carried out at 16 ± 2 ºC using the commercial yeast Maurivim PDM (Mauri Yeast Pty Ltd., Australia) *Saccharomyces cerevisiae* (0.20 g L^{-1}) and Gesferm (Amazon Group Ltd., Brazil) as the nutrient (0.20 g L^{-1}). After this step, protein stabilization was carried out using Maxibent plus bentonite (Amazon Group Ltd., Brazil) (0.8 g L^{-1}), and the tartaric acid stabilized at −4 ºC for 10 days.

For the second alcoholic fermentation, a tirage liqueur containing sucrose (24 g L^{-1}), Maurivim PDM yeast (0.25 g L^{-1}), Gesferm nutrient (0.25 g L^{-1}), and bentonite (0.10 g L^{-1}) was added to the base wine. The wines corresponding to each treatment were bottled in 750 mL bottles, each closed with a bidule and corona cap, giving 80 bottles per treatment, and maintained at 15 ± 2 ºC. This step was monitored by measuring the pressure with an aphrometer and the residual sugar content (OIV 2021). After concluding the second fermentation, the sparkling wines were maintained at 16 ± 2 ºC for 3 and 18 months for autolysis, and at the end of each of these times, they were riddled, disgorged, and corked.

Colorimetric parameters

The colorimetric parameters were determined using a portable colorimeter (Delta Color, Brazil) in the transmittance mode with illuminant D65, according to the CIELab and CIEL*C*h systems (CIE 2004), obtaining the values for luminosity (L*), red/green component (a*), blue/yellow component (b*), saturation (C*) and the hue angle (h).

Phenolic compounds

The total phenolic content (TPC) was determined according to Singleton and Rossi (1965) using a spectrophotometer. The compounds were quantified by constructing a calibration curve with gallic acid (Vetec, Brazil), reading the absorbance at 760 nm in a ThermoFisher Scientific spectrophotometer (Multiskan Go, USA), and the results expressed in mg L^{-1} gallic acid (GAE).

Using a high-performance liquid chromatography—HPLC (Waters Alliance e2695 model, Milford, CT, USA) and Diode Array detector—DAD (280, 320, 360, 520 nm), 21 phenolic compounds were quantified in the samples in a single 60-min run, as from adaptations of validated methods under the same analytical conditions by Natividade et al. (2013) and Da Costa et al. (2020). The validation parameters for the identification and quantification of the phenolic compounds can be found in the supplementary material (Table 1S). The column and pre-column used were Gemini-NX C18 (150 mm × 4.60 mm × 3 µm) and Gemini-NX C18 (4.0 mm × 3.0 mm) (Phenomenex, USA), respectively, using gradient elution and a mobile phase of 0.85% orthophosphoric acid (Fluka, Switzerland) in ultrapure water (Purelab Option Q Elga System, USA) (Phase A) and HPLC grade acetonitrile (J. T. Baker, USA) (Phase B). The oven temperature was maintained at 40 ºC and the flow rate at 0.5 mL min^{-1}. After filtering through a 13 mm diameter nylon membrane with a pore size of 0.45 µm (Phenomenex, USA), 5 and 10 µL of each wine sample were injected into the equipment.

The ferulic acid standard was obtained from Chem-Service (West Chester, USA). The standards of caffeic, *trans*-caftaric, p-cumaric, chlorogenic, and gallic acids, and piceatannol were acquired from Sigma-Aldrich (USA) and *cis*-resveratrol was acquired from Cayman Chemical (Michigan, USA). (−)-Epicatechin gallate, (−)-epigalatocatechin gallate, kaempferol-3-*O*-glucoside, quercetin-3-β-D-glucoside, isorhamnetin-3-*O*-glucoside, miricetin, rutin, malvidin-3-*O*-glucoside, peonidin-3-*O*-glucoside, delfinidin-3-*O*-glucoside, cianidin-3-*O*-glucoside, pelargonidin-3-*O*-glucoside and o *trans*-resveratrol were acquired from Extrasynthese (Genay, France).

Antioxidant capacity

The in vitro antioxidant capacity of the wines was determined using three spectrophotometric assays: DPPH (2,2-diphenyl-1-picrylhydrazyl) radical capture (Brand-Williams et al. 1995), FRAP—Ferric Reducing Antioxidant Power (Benzie & Strain 1996), and the ABTS [2,2'-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid)] radical capture method according to Re et al. (1999).

For the DPPH radical capture method (Sigma-Aldrich, USA), 0.1 mL of sample was mixed with 3.9 mL DPPH (0.06 mM) in a methanolic solution and incubated in the dark for 60 min at 23 ± 2 ºC, the absorbance being read at 515 nm. The FRAP reagent consists of acetate buffer (300 mM, pH 3.6), TPTZ (2,4,6-tri(2-pyridyl)-1,3,5-triazine) (Sigma–Aldrich, USA) (10 mM in 40 mM HCl) and $FeCl_3$ $6H_2O$ (20 mM), adding (2.85 mL) to 0.15 mL of the sample. The mixture was incubated for 30 min in the dark at 23 ± 2 ºC, and the absorbance was read at 593 nm. For the ABTS assay, 5 mL of a 7 mM stock solution of ABTS (Sigma–Aldrich, USA) were mixed with 176 µL of 70 mM potassium persulfate, and the mixture was incubated in the dark for 16 h. Readings were taken at 743 nm after 0 and 6 min for the control and after 6 min of incubation in the dark for the samples. The analytical

Table 1 Phenolic profile and antioxidant capacity of Syrah red sparkling wines elaborated with different times of cold pre-fermentative maceration and its evolution during aging on lees

Parameters	Maceration time[b]	Base wine	Aging time on lees[a]	
			3 months	18 months
Phenolic compounds[c]				
Gallic acid	NM	0.69 ± 0.03b	0.76 ± 0.01 Bb	3.78 ± 0.03 Ab
	M24	1.12 ± 0.05a	1.52 ± 0.02 Ba	3.99 ± 0.10 Ab
	M72	1.19 ± 0.11a	1.53 ± 0.02 Ba	4.26 ± 0.01 Aa
trans-caftaric acid	NM	90.48 ± 3.26a	83.93 ± 1.85 Aa	81.66 ± 2.03 Aa
	M24	47.33 ± 0.41b	46.78 ± 1.56 Ab	45.42 ± 0.50 Ab
	M72	34.58 ± 1.98c	34.79 ± 3.02 Ac	35.69 ± 3.84 Ac
Caffeic acid	NM	6.84 ± 0.19a	6.66 ± 0.14 Aa	6.31 ± 0.16 Aa
	M24	5.27 ± 0.03b	5.10 ± 0.23 Ab	5.42 ± 0.15 Ab
	M72	4.05 ± 0.30c	4.11 ± 0.28 Bc	5.12 ± 0.61 Ab
Chlorogenic acid	NM	0.58 ± 0.06b	0.81 ± 0.04 Bb	2.07 ± 0.08 Ab
	M24	0.85 ± 0.02a	1.18 ± 0.02 Ba	2.90 ± 0.12 Aa
	M72	0.91 ± 0.07a	1.11 ± 0.06 Ba	2.80 ± 0.44 Aa
p-Coumaric acid	NM	0.28 ± 0.02a	0.32 ± 0.01 Bb	0.53 ± 0.04 Ab
	M24	0.30 ± 0.00a	0.41 ± 0.03 Ba	0.70 ± 0.02 Aa
	M72	0.29 ± 0.01a	0.36 ± 0.00 Bab	0.57 ± 0.07 Ab
Ferulic acid	NM	0.31 ± 0.01a	0.40 ± 0.00 Ab	0.31 ± 0.01 Ab
	M24	0.32 ± 0.00a	0.70 ± 0.02 Aa	0.47 ± 0.01 Ba
	M72	0.32 ± 0.02a	0.65 ± 0.04 Aa	0.45 ± 0.04 Ba
(−)-Epicatechin gallate	NM	1.63 ± 0.03a	0.66 ± 0.02 Ab	0.73 ± 0.03 Ac
	M24	1.43 ± 0.06b	1.18 ± 0.08 Ba	1.32 ± 0.01 Aa
	M72	1.15 ± 0.06c	0.79 ± 0.04 Bb	1.17 ± 0.12 Ab
(−)-Epigalatocatechin gallate	NM	1.09 ± 0.04a	1.69 ± 0.02 Ac	1.58 ± 0.02 Ab
	M24	1.18 ± 0.02a	1.98 ± 0.13 Ab	1.61 ± 0.03 Bb
	M72	1.18 ± 0.02a	2.22 ± 0.09 Aa	1.74 ± 0.09 Ba
Kaempferol-3-*O*-glucoside	NM	0.34 ± 0.01c	0.33 ± 0.01 Ac	0.32 ± 0.01 Ac
	M24	0.77 ± 0.03b	0.77 ± 0.02 Ab	0.67 ± 0.05 Bb
	M72	0.96 ± 0.06a	0.90 ± 0.03 Aa	0.84 ± 0.04 Aa
Quercetin-3-β-D-glucoside	NM	2.10 ± 0.21b	1.86 ± 0.23 Ab	1.68 ± 0.11 Ac
	M24	9.46 ± 0.65a	9.72 ± 0.54 Aa	8.67 ± 0.91 Ab
	M72	10.68 ± 0.58a	10.68 ± 0.61 Aa	10.55 ± 0.87 Aa
Isorhamnetin-3-*O*-glucoside	NM	0.64 ± 0.04b	0.76 ± 0.05 Ab	0.73 ± 0.05 Ac
	M24	2.63 ± 0.12a	3.75 ± 0.16 Aa	3.55 ± 0.31 Ab
	M72	3.16 ± 0.24a	4.11 ± 0.19 Aa	4.30 ± 0.40 Aa
Myricetin	NM	0.42 ± 0.00b	ND	0.42 ± 0.00 b
	M24	0.53 ± 0.01a	0.49 ± 0.00 Aa	0.49 ± 0.01 Aa
	M72	0.54 ± 0.02a	0.47 ± 0.00 Aa	0.49 ± 0.02 Aa
Rutin	NM	0.23 ± 0.00b	0.35 ± 0.03 Ac	0.29 ± 0.01 Ab
	M24	0.29 ± 0.00a	1.08 ± 0.07 Ab	0.60 ± 0.02 Ba
	M72	0.31 ± 0.01a	1.20 ± 0.04 Aa	0.61 ± 0.05 Ba
cis-resveratrol	NM	0.13 ± 0.00b	0.16 ± 0.01 Ab	0.16 ± 0.02 Aa
	M24	0.17 ± 0.01a	0.18 ± 0.01 Aa	0.18 ± 0.02 Aa
	M72	0.17 ± 0.00a	0.19 ± 0.01 Aa	0.18 ± 0.02 Aa
Piceatannol	NM	0.30 ± 0.02b	0.29 ± 0.00 b	ND
	M24	0.39 ± 0.01a	0.50 ± 0.01 a	ND
	M72	0.39 ± 0.00a	0.52 ± 0.02 a	ND
trans-resveratrol	NM	0.27 ± 0.00b	0.27 ± 0.00 a	ND
	M24	0.30 ± 0.01a	0.27 ± 0.00 a	ND
	M72	0.29 ± 0.00a	0.26 ± 0.00 a	ND

Table 1 (continued)

Parameters	Maceration time[b]	Base wine	Aging time on lees[a]	
			3 months	18 months
Malvidin-3-*O*-glucoside	NM	9.76 ± 0.59b	4.19 ± 0.16 Ac	0.92 ± 0.03 Bb
	M24	48.45 ± 1.19a	26.10 ± 0.69 Aa	7.19 ± 0.36 Ba
	M72	49.97 ± 3.80a	25.00 ± 0.55 Ab	6.95 ± 0.86 Ba
Peonidin-3-*O*-glucoside	NM	0.96 ± 0.08b	0.34 ± 0.01 c	ND
	M24	2.46 ± 0.12a	0.98 ± 0.05 Aa	0.27 ± 0.01 Ba
	M72	2.37 ± 0.13a	0.87 ± 0.04 Ab	0.23 ± 0.02 Ba
Pelargonidin-3-*O*-glucoside	NM	1.36 ± 0.10b	0.68 ± 0.02 Ac	0.27 ± 0.01 Bb
	M24	5.46 ± 0.21a	2.78 ± 0.11 Aa	0.83 ± 0.04 Ba
	M72	5.65 ± 0.45a	2.62 ± 0.08 Ab	0.80 ± 0.09 Ba
Delphinidin-3-*O*-glucoside	NM	0.80 ± 0.05b	0.42 ± 0.01 Ac	0.22 ± 0.00 Bb
	M24	2.50 ± 0.11a	1.17 ± 0.07 Aa	0.45 ± 0.02 Ba
	M72	2.48 ± 0.16a	1.06 ± 0.05 Ab	0.43 ± 0.04 Ba
Petunidin-3-*O*-glucoside	NM	0.27 ± 0.01b	0.30 ± 0.00 Ab	0.26 ± 0.00 Ac
	M24	0.52 ± 0.02a	0.94 ± 0.03 Ba	1.08 ± 0.03 Ab
	M72	0.53 ± 0.03a	1.02 ± 0.01 Ba	1.20 ± 0.15 Aa
TPC[d]	NM	343.16 ± 4.32b	272.44 ± 6.39 Bc	301.69 ± 5.07 Ab
	M24	464.76 ± 13.91a	396.70 ± 11.15 Bb	436.79 ± 13.10 Aa
	M72	489.37 ± 28.82a	426.02 ± 13.02 Ba	453.54 ± 9.46 Aa
ABTS[e]	NM	1.70 ± 0.05c	1.27 ± 0.06 Bc	1.41 ± 0.02 Ac
	M24	2.64 ± 0.06b	2.28 ± 0.10 Bb	2.43 ± 0.07 Ab
	M72	2.84 ± 0.07a	2.47 ± 0.02 Ba	2.67 ± 0.09 Aa
DPPH[f]	NM	1.50 ± 0.05c	1.32 ± 0.03 Bc	1.51 ± 0.02 Ac
	M24	1.93 ± 0.08b	1.76 ± 0.03 Bb	1.95 ± 0.06 Ab
	M72	2.04 ± 0.03a	1.91 ± 0.03 Ba	2.11 ± 0.05 Aa
FRAP[g]	NM	2.02 ± 0.19c	1.36 ± 0.02 Bc	1.57 ± 0.02 Ab
	M24	2.96 ± 0.08b	2.18 ± 0.06 Bb	2.53 ± 0.05 Aa
	M72	3.26 ± 0.13a	2.31 ± 0.05 Ba	2.56 ± 0.05 Aa

[a]Results expressed as mean values ± standard deviation. Values followed by different letters indicate significant difference between the samples according to the Tukey's test ($p \leq 0.05$). Capital letters indicate differences and similarities between the sparkling wines with different aging time on less (3 and 18 months of autolysis). Small letters indicate differences and similarities between the base or sparkling wines elaborated with different cold pre-fermentative maceration treatments tested (NM, 24 and 72 h). *ND* Not detected

[b]*NM* Without maceration, *M24* pre-fermentative maceration for 24 h, *M72* pre-fermentative maceration for 72 h

[c]Phenolic compounds quantified by HPLC–DAD in mg L^{-1}

[d]Total phenolic content measured by Folin-Ciocalteu reducing capacity expressed in mg $L.^{-1}$ of gallic acid

[e,f,g]ABTS, DPPH and FRAP assays: Antioxidant capacity expressed as Trolox equivalents (mmol TEAC L^{-1})

standard used for the three methods to construct the calibration curves was Trolox (6-Hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) (Sigma-Aldrich, USA). The results were expressed in milli mole equivalents of Trolox per liter of wine (mmol TEAC L^{-1}). The absorbances were read in a Biospectro SP-220 spectrophotometer (Lab-lider, Brazil).

Statistical analyses

The base wines were analyzed in triplicate by repetition of the vinification ($n = 3$) of each maceration treatment (NM, M24, and M72). For the analysis of the sparkling wines with the two autolysis times (3 and 18 months), three bottles of each replicate ($n = 3$) were taken at random for each

maceration treatment (NM, M24, and M72), which were also analyzed in triplicate. The results were first submitted to the Shapiro–Wilk normality test ($p \leq 0.05$), and those not presenting normality were transformed ($\sqrt{}$: gallic and chlorogenic acids and delphinidin-3-*O*-glucoside; Log($x+1$): piceatannol, *trans*-resveratrol, myricetin, and petunidin-3-*O*-glucoside). After the transformations, the data were submitted to the analysis of variance (ANOVA) and Tukey's test ($p \leq 0.05$) using the R Core Team (2021). The Principal Components Analysis (PCA) was used for exploratory purposes to analyze the data, and the Pearson correlation coefficients (r values) were calculated to correlate the phenolic compound composition with the antioxidant capacity at $p \leq 0.05$ for the sparkling wines using the XLStat software (Addinsoft Inc., Anglesey, UK, 2015).

Results and discussion

Phenolic compounds and antioxidant capacity

Table 1 shows the phenolic compounds identified and quantified by HPLC–DAD, the total phenolic content, and the antioxidant capacity of the Syrah red sparkling wines elaborated with different cold pre-fermentative maceration and autolysis times.

A total of six phenolic acids were quantified in the red sparkling wines. The use of cold pre-fermentative maceration increased the gallic, chlorogenic, and ferulic acid concentrations, and, to the contrary, decreased the *trans*-caftaric and caffeic acid contents. Furthermore, after 18 months of autolysis, the red sparkling wines showed increases in the gallic, chlorogenic, and *p*-coumaric acid contents and a decrease in the ferulic acid concentration compared with the initial period (3 months). The increase or decrease in some phenolic acids could be associated with the hydrolysis of tartaric esters and their corresponding hydroxycinnamic acids during the cold pre-fermentative maceration or even during the autolysis period (Sartor et al. 2019a).

The gallic acid concentration in the sparkling wines after 3 months of autolysis varied from 0.76 mg L^{-1} for NM to 1.53 mg L^{-1} in M72, whereas after 18 months of autolysis, it ranged from 3.78 mg L^{-1} in NM to 4.26 mg L^{-1} in the sparkling wine M72. These results are lower than those reported in South African sparkling wines (14.86 at 16.18 mg L^{-1}) (Minnaar et al. 2021). However, they are consistent with studies that showed an increase in concentration of the hydroxybenzoic acids, principally gallic acid in red Syrah wines elaborated with cold pre-fermentative maceration (Cejudo-Bastante et al. 2014) and during the autolysis of sparkling wines made from other cultivars (Sartor et al. 2019a), probably due to the hydrolysis of tannins (Garrido and Borges 2013). Thus, gallic acid could be a possible quality marker for sparkling wines aged on lees.

Caftaric acid is the hydroxycinnamic acid found in largest amounts in grapes and wines (Garrido and Borges 2013) and was the most abundant phenolic acid found in the present study. As a consequence of cold pre-fermentative maceration, its concentration varied from 83.93 mg L^{-1} in the sparkling wine NM to 34.79 mg L^{-1} in M72, after 3 months of autolysis. The fall in this concentration could be related to a greater oxidation rate in these wines since *trans*-caftaric acid is a substrate for polyphenoloxidase, the main oxidative enzyme present in the grapes (Sartor et al. 2019a). This acid is one of the phenolic compounds responsible for the astringency of wines (Garrido and Borges 2013), so the use of cold pre-fermentative maceration could represent an ally to decrease this gustatory sensation in red sparkling wines.

The flavanols (−)-epicatechin gallate and (−)-epigalatocatechin gallate were quantified in all the red sparkling wines, and in both cases, the highest concentrations were found in the sparkling wines M24 and M72. However, after 18 months of autolysis, the sparkling wines showed a different behavior when compared with the initial time (3 months), increasing the (−)-epicatechin gallate concentration and decreasing that of (−)-epigalatocatechin gallate in the sparkling wines elaborated with cold pre-fermentative maceration, independent of the time adopted in the technique. These variations could be related to the participation of these phenolic compounds in polymerization, hydrolysis, or even condensation reactions with the anthocyanins and could contribute to stabilizing the color of the wines (Monagas and Bartolomé 2009).

The flavonols can stabilize colors by taking part in copigmentation reactions with the anthocyanins (Garrido & Borges, 2013) and are part of the phenolic compound group recognized for their antioxidant, anti-carcinogenic, anti-inflammatory properties and immune functions in humans, against the Coronavirus infection (SARS-CoV-2) and other diseases (Galanakis et al. 2020). Thus, the present study showed a strong correlation ($r > 0.9$; $P < 0.05$) between kaempferol-3-*O*-glucoside, quercetin-3β-D-glucoside, and isorhamnetin-3-*O*-glucoside, with a strong antioxidant capacity for these red sparkling wines in the three assays carried out (ABTS, DPPH, and FRAP). Isorhamnetin-3-*O*-glucoside stood out with a greater correlation in the ABTS assay ($r = 0.951$; $P < 0.01$), FRAP ($r = 0.941$; $P < 0.01$) and DPPH ($r = 0.879$; $P < 0.03$). These flavonols showed greater concentrations in the red sparkling wines elaborated with cold pre-fermentative maceration (M24 and M72). Heredia et al. (2010) found similar results for this group of phenolic compounds, testing the same technique in the elaboration of still red wines from Syrah grapes. On the other hand, in general, the autolysis periods evaluated in this study showed no

significant variation in the concentration of flavonols. This behavior was also observed in other studies that evaluated different times of aging of white sparkling wines on yeast lees (Sartor et al. 2019a).

Concerning the stilbenes, the use of cold pre-fermentative maceration increased the piceatannol content, although its concentration was the same for both times tested (M24 and M72). After 18 months of autolysis, only *cis*-resveratrol was detected in the red sparkling wines, reaching 0.18 mg L^{-1} in the sparkling wines M24 and M72. The maintenance of the *cis*-resveratrol concentration, even after 18 months of autolysis, shows the bioactive potential of the red sparkling wines, since the in vivo antioxidant action associated with the consumption of this stilbene has already been proven (Gris et al. 2011). In addition, the present study showed a significant correlation ($r > 0.8$; $P < 0.05$) between the *cis*-resveratrol and in vitro antioxidant capacity of the red sparkling wines for the three assays carried out (ABTS, DPPH, and FRAP), especially ABTS ($r > 0.9$; $P < 0.02$).

The use of cold pre-fermentative maceration favored the extraction of anthocyanins, and of those quantified, malvidin-3-*O*-glucoside was the most abundant, as already reported in red wines (Cheng et al. 2022) and in another study on red sparkling wines (González-Lázaro et al. 2019). However, with the exception of petunidin-3-*O*-glucoside, autolysis decreased the concentration of the anthocyanins in the red sparkling wines, independent of the maceration time used. This decrease could be related to adsorption of the pigments by the yeast cell wall or even by possible hydrolysis and condensation reactions with other phenolic compounds, which could give rise to anthocyanin-derived pigments during the aging period (Monagas and Bartolomé 2009). The highest malvidin-3-*O*-glucoside concentration was found in sample M24 after 3 months of autolysis (26.10 mg L^{-1}), and even after 18 months of autolysis, the application of cold pre-fermentative maceration (M24 and M72) caused a six fold increase in the concentration of this anthocyanin when compared to the control (NM). On the other hand, petunidin-3-*O*-glucoside showed its highest value after 18 months of autolysis in the red sparkling wines produced using cold pre-fermentative maceration. Heredia et al. (2010) observed the formation of co-pigments derived from petunidin-3-*O*-glucoside in still red Syrah wines elaborated with cold pre-fermentative maceration, which could explain the conservation of this anthocyanin after aging on the lees. In addition, petunidin-3-*O*-glucoside showed good correlation with the antioxidant capacity of the red sparkling wines ($r > 0.92$; $P < 0.01$). Maintaining the anthocyanin content is important to conserve the antioxidant capacity of the sparkling wines, considering that this group of phenolic compounds shows great bioactive activity (Monagas and Bartolomé 2009). Thus, when autolysis is adopted in the elaboration of sparkling wines, it can be associated with enological practices such as cold fermentative maceration to decrease the impact of the fall in the anthocyanin content during this step.

According to Table 1, the red sparkling wines elaborated with the longest maceration (M72) and autolysis (18 months) times stood out for their TPC. In the sparkling wines with 3 months of autolysis, the concentrations varied from 272.44 mg L^{-1} for NM to 426.02 mg L^{-1} for M72, reaching 453.54 mg L^{-1} in M72 after 18 months of autolysis. In a study with sparkling white wines produced using Syrah grapes elaborated without maceration, the TPC was 134.79 mg L^{-1} (Nascimento et al. 2018). On the other hand, in a study evaluating Spanish red sparkling wines elaborated with Tempranillo grapes with cold pre-fermentative maceration using dry ice and nine months of autolysis, the TPC arrived at 1169 mg L^{-1} (Pérez-Magariño et al. 2019). Ruiz-Moreno et al. (2017) observed a greater TPC in sparkling wines elaborated with cold pre-fermentative maceration; in contrast with the present results, there was no influence of autolysis time on this variable in sparkling wines with up to 9 months of autolysis. Sartor et al. (2019a) reported smaller concentrations in sparkling wines with 15 months of autolysis. On the other hand, Gnoinski et al. (2021) found increasing TPC values in Australian sparkling wines with increasing autolysis times. The different reports in these studies could be explained by the cultivars studied, the cultivation regions, and, above all, by the diverse enological techniques applied to obtain the base wine.

The use of cold pre-fermentative maceration and a longer autolysis time increased the antioxidant capacity of the Syrah red sparkling wines according to the three assays evaluated: ABTS, DPPH, and FRAP (Table 1).

The antioxidant capacity values measured by FRAP varied from 1.27 mmol TEAC L^{-1} for NM to 2.47 mmol TEAC L^{-1} for M72 in the sparkling wines with 3 months of autolysis, reaching 1.41 mmol TEAC L^{-1} and 2.67 mmol TEAC L^{-1} (NM and M72, respectively) when compared with the final time (18 months). This behavior was probably due to the capacity of the yeast lees to absorb and liberate some phenolic compounds during the autolysis period (Gallardo-Chacón et al. 2010) and because, in some cases, the yeast lees only show a fall in their oxygen-consumption capacity after three years of autolysis (Pons-Mercadé et al. 2021). In addition, the antioxidant capacity measured by ABTS and DPPH in the M72 Syrah red sparkling wines with 18 months of autolysis (2.67 and 2.11 mmol TEAC L^{-1}, respectively) represented values three times greater than the Syrah white sparkling wines elaborated without the use of cold pre-fermentative maceration and without aging on the lees (Nascimento et al. 2018). Knowing that the characteristics of the base wine influence the antioxidant capacity of the sparkling wines after autolysis (Gallardo-Chacón et al. 2010), and from the results obtained in the present study, one can infer that the use of cold pre-fermentative maceration

increased the antioxidant capacity of the Syrah red sparkling wines aged on lees, possibly because this practice increased the concentrations of kaempferol-3-*O*-glucoside, quercetin-3-β-D-glucoside, isorhamnetin-3-*O*-glucoside and petunidin-3-*O*-glucoside.

Principal components analysis

The principal components analysis was carried out to explain the influence of the times of cold pre-fermentative maceration and of autolysis on the phenolic compound composition and antioxidant capacity of the red sparkling wines (Fig. 1). The first principal component (PC1), which explained 57.56% of the variability of the sample, was responsible for separating the non-macerated sparkling wines (NM) for both autolysis times applied from the other samples. The NM sparkling wines were found in the negative part of PC1 and could be associated with greater concentrations of *trans*-caftaric and caffeic acid. The sparkling wines elaborated with cold pre-fermentative maceration (M24 and M72) were found in the positive part of the first component, showing that this practice had a greater influence on the phenolic compounds composition and antioxidant capacity of the red sparkling wines than the autolysis time. TPC, kaempferol-3-*O*-glucoside, quercetin-3β-D-glucoside and isorhamnetin-3-*O*-glucoside were the main components responsible for this separation, converging with the results of Table 1, which shows that these flavonols are present in greater concentrations in the sparkling wines elaborated with the use of cold pre-fermentative maceration. Figure 1 also shows that the vectors of these flavonols were the closest to those representing the antioxidant capacity according to the DPPH, ABTS, and FRAP assays, corroborating with the correlation between them, as cited before. In addition, the second principal component (PC2) separated the sparkling wines with different autolysis times. In the positive part of this component, the sparkling wines with 18 months of autolysis were associated with higher concentrations of gallic, chlorogenic, and p-coumaric acids, whose vectors were closest to these samples. Hence, these phenolic acids could be possible quality markers of autolysis time in red sparkling wines.

Colorimetry

The color of the red sparkling wines obtained by applying different cold pre-fermentative maceration times and

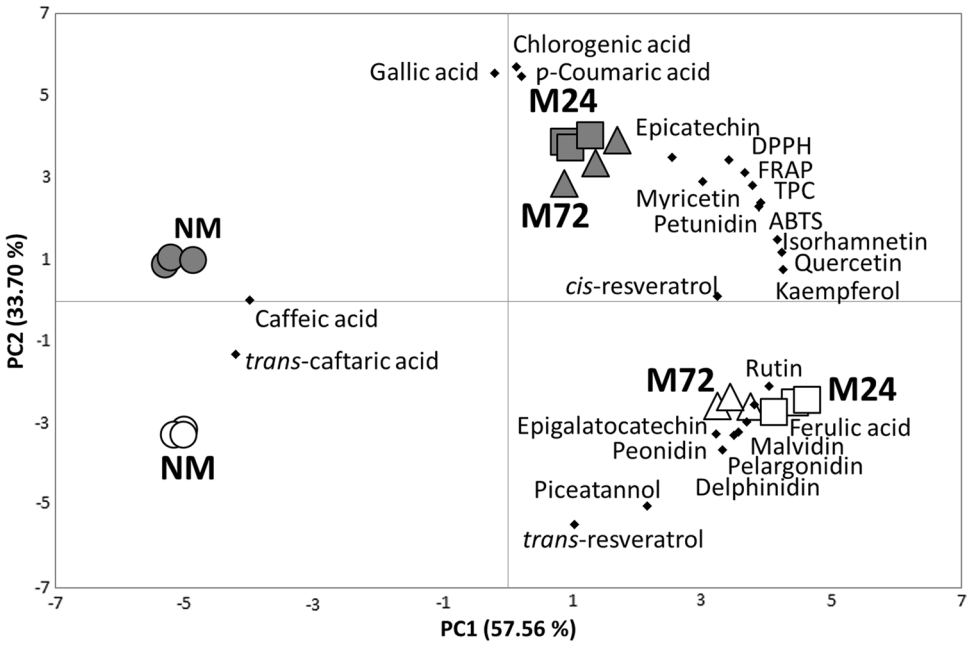

Fig. 1 Principal Component Analysis (PCA) showing the configuration of Syrah red sparkling wines elaborated with different pre-fermentative cold maceration times (NM, 24 and 72 h) and its evolution during aging on lees (3 and 18 months of autolysis) in function of antioxidant capacity, total phenolic content (measured by the spectrophotometric method) and phenolic compounds quantified by HPLC–DAD ($n=21$ compounds). TPC: total phenolic content, Epicatechin: (−)-epicatechin gallate, Epigalatocatechin: (−)-epigalatocatechin gallate, Kaempferol: kaempferol-3-*O*-glucoside, Quercetin: quercetin-3-β-D-glucoside, Isorhamnetin: isorhamnetin-3-*O*-glucoside, Malvidin: malvidin-3-*O*-glucoside, Peonidin: Peonidin-3-*O*-glucoside, Delphinidin: Delphinidin-3-*O*-glucoside, Petunidin: Petunidin-3-*O*-glucoside. NM: Without maceration, M24: pre-fermentative maceration for 24 h, M72: pre-fermentative maceration for 72 h. White circles, squares, and triangles = 3 months of autolysis; Dark gray circles, squares, and triangles = 18 months of autolysis

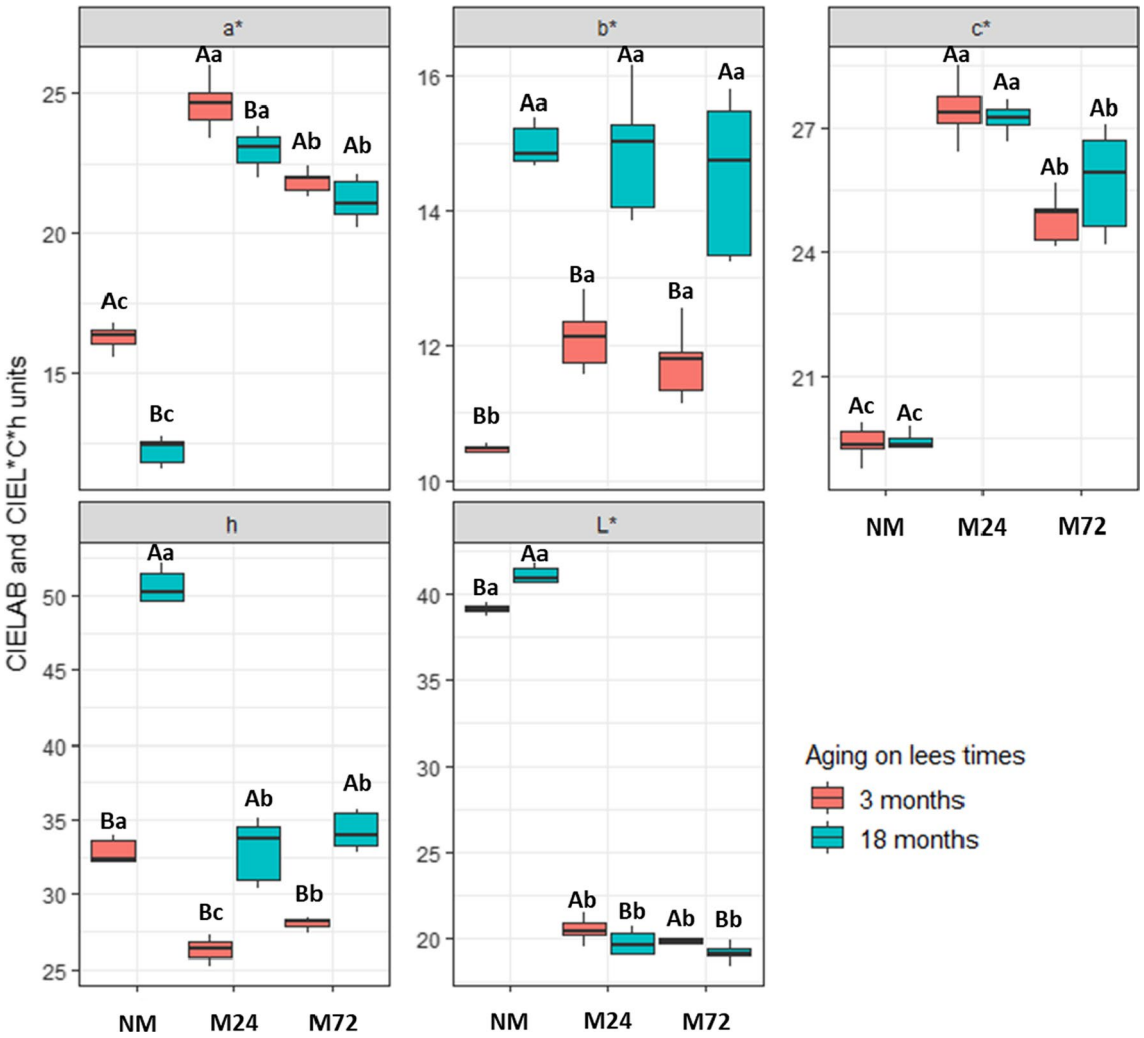

Fig. 2 Colorimetric analysis of Syrah red sparkling wines elaborated with different times of cold pre-fermentative maceration and its evolution during aging on lees. Different letters indicate a significant difference between the samples according to Tukey's test ($p \leq 0.05$). Capital letters indicate differences and similarities between the sparkling wines with different aging times on less (3 and 18 months of autolysis). Small letters indicate differences and similarities between the sparkling wines elaborated with different cold pre-fermentative maceration treatments tested (NM, 24 and 72 h). NM: Without maceration, M24: pre-fermentative maceration for 24 h, M72: pre-fermentative maceration for 72 h

their evolution during the two autolysis times applied, were evaluated by colorimetry (Fig. 2).

In general, the red sparkling wines elaborated with cold pre-fermentative maceration (M24 and M72) presented higher values on the coordinate representing the red color (a*) and color saturation (C*) and less luminosity (L*). Probably the nature of the phenolic compounds extracted with the different maceration times can explain this behavior, considering the hydroxycinnamic acids, flavonols, and flavanols. These compounds have an important role in stabilizing the color of the red wines, since they could take part in co-pigmentation reactions with the anthocyanins (Barrio-Galán et al. 2012; Garrido and Borges 2013).

Eighteen months of autolysis promoted an increase in the yellow coordinate (b*) and, consequently, the hue angle (h) became more distant from the red color (0º) and closer to 90º, showing that NM was the treatment that most suffered the impact of the autolysis time. During the 18 months of autolysis, the oxygen present in the bottles, in addition to being consumed by the yeast lees and by free sulfur dioxide, probably promoted reactions between the phenolic compounds, such as the condensation of anthocyanins with flavonols which form orange complexes and could thus cause changes in the color of the red sparkling wines (Garrido and Borges 2013; Pons-Mercadé et al. 2021).

Conclusion

Important changes in the bioactive compounds profile and color of the red sparkling wines were observed with the application of cold pre-fermentative maceration and as a function of the different autolysis times tested. An autolysis time of 18 months and the use of 72 h of cold pre-fermentative maceration increased the antioxidant capacity of the red sparkling wines elaborated with the Syrah cultivar. In addition, the use of maceration promoted a more intense red color in the red sparkling wines after 18 months of autolysis.

Thus the results of the present study demonstrated that the use of cold pre-fermentative maceration by refrigeration could be an optimum alternative for the elaboration of red sparkling wines by the traditional method. In addition, increasing the autolysis time promotes a better nutraceutical quality to the product.

However, further studies are required to better understand the impact of the cold pre-fermentative maceration and autolysis times on the volatile compound profile, sensory quality, and acceptability by the consumers of red sparkling wines elaborated by the traditional method.

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Author's contributions APAB: Conception of the research, methodology, investigation, winemaking, and analysis of the sparkling wines, data analysis, and manuscript writing. ISS: Winemaking, and analysis of the sparkling wines. LCC: Methodology and analysis of the phenolic compounds. ACTB: Resources, supervision, data analysis, and review.

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Availability of data and material The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Code availability Not applicable.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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