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Influence of terroirs on the phenolic composition and astringency of wines made from representative plots of Corbières subregions

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ABSTRACT

Phenolic compounds play a significant role in the organoleptic properties (colour, astringency, and bitterness) and stability of wines through oxidative processes. These properties depend on both intrinsic factors, such as grape variety, and extrinsic ones, such as soil, climate, and winemaking techniques. The Corbières appellation is a renowned red grape variety region of the south of France. This work evaluated the effect of soil, climate, and vineyard practices in this region on the polyphenolic compositions of Syrah, Grenache, Carignan, and Mourvèdre mono-varietal wines produced at an experimental scale from the same vintage. A general and targeted study of the wines' polyphenol profiles was adopted, focusing on anthocyanins, derived pigments, and tannins. The studied regions can be discriminated in terms of some of the analysed parameters depending on grape variety. Moreover, through a Check-All-That-Apply (CATA) sensorial analysis, a link was traced between the tannin composition and the astringency perception of the wines.

KEYWORDS: polyphenols, tannins, wine, astringency, terroir



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INTRODUCTION

The chemical composition of a wine determines its quality and depends on grape variety (influenced to a greater or lesser extent by ripeness, soil conditions and climate), the winemaking process (greater or lesser extraction of compounds of interest) and type of reactions (evolution of compound structures) (Arnold & Noble, 1978; Canals *et al.*, 2005; Castellarin *et al.*, 2007; Llaudy *et al.*, 2008; Gawel, 1998; Jackson & Lombard, 1993; Kontoudakis *et al.*, 2011; Ribéreau-Gayon *et al.*, 2006; Robichaud & Noble, 1990; Seguin, 1986; van Leeuwen & Seguin, 2006; Vaudour, 2002; Vidal *et al.*, 2003; Zsófi *et al.*, 2011). The chemical composition of red wines is more complex than that of white wines, since maceration allows polyphenolic compounds to be extracted from the solid parts of the berries (skins and seeds) (Bindon *et al.*, 2014; He *et al.*, 2010). Among polyphenols, anthocyanins are responsible for the colour of red wines, while tannins play a major role in astringency (Arnold & Noble, 1978; Peleg *et al.*, 1999).

Polyphenolic compounds are secondary metabolites that are primarily synthesised in the grape berry. Their biosynthesis is linked to the grape variety (genetic factor), but is also influenced by soil, climate, and environmental parameters (De la Cerda-Carrasco *et al.*, 2015; Rodríguez Montealegre *et al.*, 2006). Indeed, terroir factors can have an impact on the flow of carbon towards specific pathways of polyphenol metabolism in the grape berry, ultimately modulating its polyphenolic composition (Li *et al.*, 2011). Climate conditions (mainly rainfall and temperatures) can influence water content in the soil, thus its availability for the vine (Pinasseau *et al.*, 2017). Water content can be modulated depending on the soil, the orientation of the plot, and the training system (Brillante *et al.*, 2016; Brillante *et al.*, 2018; Pereyra *et al.*, 2023). Indeed, deep soils enable deeper rooting and better access to water, and the sapling training system of vines seems to be better adapted to drought (Santesteban *et al.*, 2017).

Ultimately, skin and seed tannins are mainly biosynthesised at the start of berry formation, between flowering and *véraison*; nonetheless, the level of berry ripeness at harvest can have an influence on their extraction during fermentation.

From the start of the winemaking process until the wine is consumed, tannins, by virtue of their reactivity, undergo numerous chemical reactions. Oxidation reactions lead to intra- and inter-molecular reactions within the tannin structure, changing its conformation (Mouls & Fulcrand, 2012; Mouls & Fulcrand, 2015; Poncet-Legrand *et al.*, 2010). In parallel, tannins can also react with anthocyanins, through either direct reactions or polycondensation reactions, via acetaldehyde bridges. These reactions lead to the formation of derived pigments (Cheynier *et al.*, 2006; Picariello *et al.*, 2017; Salas *et al.*, 2003; Vidal *et al.*, 2002) that can lead to a colour change, and which can also have an influence on interactions and ultimately on astringency (Gao *et al.*, 1997; García-Puente Rivas *et al.*, 2006). Astringency is a complex phenomenon induced by the interaction between tannins and

salivary proteins, which can be modulated by the composition of the tannin fraction and by the complex matrix of the wine (Brossaud *et al.*, 2001; Rinaldi *et al.*, 2014). The astringency of a wine evolves to a greater or lesser extent over time and, in general, tends to soften as the wine ages, probably due to this evolution in the structure of the tannins, which modifies the interactions with salivary proteins (McRae *et al.*, 2012).

The quality of red wines therefore depends on many parameters, making it complicated to determine their role in the terroir impact (Canals *et al.*, 2005; Castellarin *et al.*, 2007; Jackson & Lombard, 1993; Kontoudakis *et al.*, 2011; van Leeuwen & Seguin, 2006; Zsófi *et al.*, 2011). In the first part of this work, we aimed to highlight differences in phenolic composition and gustatory descriptors between wines from different areas of the Corbières appellation, and to explain these differences through soil, climate and vineyard management practices. To do this, representative plots from five subregions of the Corbières appellation were selected. For each subregion, four grape varieties were studied (Syrah, Carignan, Grenache, and Mourvèdre) over two vintages. Each variety was harvested at the same level of ripeness and standardised winemaking processes were carried out to specifically study the effect of plot characteristics (soil, vine management, etc.) and climate by removing any influence arising from the winemaking practices. Chemical and sensory analyses were carried out on the single-varietal wines that were obtained: in particular, anthocyanins, derived pigments, and the tannin fraction were analysed and their impact on wine profile was evaluated, with a focus on the astringent perception.

In the second part of the study, since blending plays an important role in the Corbières appellation, a similar blend with the four monovarietal wines from each zone was carried out, and the blended wines were sensory analysed with the aim of determining whether the blend enhances the terroir typicity in terms of astringency.

MATERIALS AND METHODS

1. Vineyard sites

The sites studied are located in the south of France in five subregions of the Corbières appellation: Alaric (AL), Durban (DU), Lagrasse (LA), Lézignan (LE), and Maritime (MA). During the 2021 and 2022 growing seasons, four plots in each subregion were delimited, each plot containing one of the four main red grape varieties Syrah, Grenache, Carignan, and Mourvèdre. The vineyards were selected upon consultation with producers from the most representative villages of each subregion. The main vineyard characteristics are given in Table S1. Concerning the Grenache plot from the LA subregion, in the end, the chosen vineyard for the 2021 vintage was not considered in the study, because the climatic conditions required an earlier harvest to be carried out, meaning that the wines from this vintage were not representative. To address representativeness issues, another plot was selected for the 2022 experimental vintage.

The main characteristics of the vineyards are reported in Table S1.

2. Climate conditions during the 2021 and 2022 vintages

SAFRAN climatic data for 2021 and 2022 were obtained from the SICLIMA platform developed by AgroClim-INRAE (Maury *et al.*, 2021). Data for each vineyard were obtained from the proximal weather stations (located on average 4–5 km from each vineyard). Detailed climate information (average rainfalls, and average, maximum and minimum temperatures) for each subregion are shown in Figures S1 and S2.

3. Soil conditions

Soil information for each selected site was obtained from the Geoportail platform from maps produced by the Groupement d'Intérêt Scientifique sur les Sols (GIS Sol) and the Réseau Mixte Technologique Sols et Territoires in the context of the “Inventaire, Gestion et Conservation des Sols (IGCS)” programme – Référentiels Régionaux Pédologiques (RRP) section. Main soil information for the 20 plots is reported in Table S1.

4. Winemaking

Between 80 and 100 kg of grape berries were harvested from each experimental site. Due to the different varieties studied and the differing climatic conditions of the appellation sites, the grapes were harvested when the content of soluble solids was approximately 21–25 °Brix, titratable acidity was between 2.5 to 4.5 g/L and pH was between 3.30 and 3.70. All the fermentations were performed in the INRAE experimental winery of the Unité Expérimentale de Pech Rouge (UEPR), Gruissan, France. The grapes were independently processed at the research winery using a destemmer-crusher (Bucher Vaslin, Niederweningen, Switzerland). The must was then protected by adding 3–5 g/hL of SO₂, depending on grape sanitary conditions. The crushed grapes obtained from each repetition were transferred to 100-L tanks, and the following day they were inoculated with 20 g/hL of the commercial yeast *Saccharomyces cerevisiae* SafETM BC S103 (Fermentis, Lesaffre, France) along with 20 g/hL of GO-FERM PROTECTTM (Lallemand Oenology, France) for the alcoholic fermentation, which took place at a controlled temperature of 25 ± 2 °C. An analysis of the initial assimilable nitrogen concentration (mg/L) of the musts was carried out and, depending on the level of deficiency, Diammonium Phosphate (DAP) (Laffort France S.A.S., France) was added at the beginning or middle of fermentation, or at both stages, at a volumetric mass density value of around 1,060 g/L. During the 2021 vintage, three samples (CAR_LA, MOUR_LA, and MOUR_DU) of rectified concentrated must were added – as is legitimate in the appellation for that vintage (maximum 1 % vol.) – due to the sanitary conditions preventing the achievement of the minimum °Brix set. During fermentation, each vat was pumped down every two days and pumped over at a

volumetric mass density value of around 1,060 g/L. On average, alcoholic fermentation was considered finished when residual sugars were inferior to 0.50 g/L. After 10–12 days of maceration–fermentation, each tank was pressed using an experimental press (Speidel, Germany). Both the free-run and press wine were then transferred to a 50 L tank and inoculated with 1 g/hL of *Oenococcus oeni* strain, Lalvin VP41 (Lallemand Oenology, France). Malic acid measurements were performed weekly; the end of malolactic fermentation was considered as being when the wine contained less than 0.2 g/L of malic acid. Then, the wines were racked to separate lees and transferred to a 20-L tank. Five grams per hectolitre of SO₂ was then added and wines underwent one–two months of aging before being bottled. At bottling, both the CO₂ and SO₂ levels were adjusted. The bottles were kept at controlled temperatures (16–18 °C) until analysis.

5. Oenological parameters

Classical oenological parameters were measured by the “Natoli & Associés” laboratory located in Montpellier, France, adopting the International Organization of Vine and Wine (OIV) reference methods. Oenological parameters of wines from both vintages are reported in Table S2.

6. Reagents, chemicals, and materials

Acetonitrile, methanol (absolute), ethanol (absolute) and hydrochloric acid were purchased from Merck/Sigma-Aldrich (Saint-Quentin-Fallavier, France). Anthocyanins were quantified using malvidin-3-*O*-glucoside, delphinidin-3-*O*-glucoside, cyanidin-3-*O*-glucoside, peonidin-3-*O*-glucoside, and petunidin-3-*O*-glucoside as standards, which were purchased from Extrasynthese (Genay, France).

7. Colour and total polyphenol index analysis by UV-visible spectrophotometry

UV-visible absorbance measurements were performed using a Shimadzu UV-1900 spectrophotometer following the protocol described by Atanasova *et al.* (2002). The absorbance measurements were done using a 0.1 cm and 1 cm path length cell, adapting dilution to wine to obtain values of between 0.01 and 1. The totality of the colour parameters were calculated following the protocol of Leborgne *et al.* (2023). Seven variables were determined in both vintages: Colour Intensity (CI), colour hue, Non-Bleachable Pigments (NBP), Bisulfite Adducts (BA), colour due to Copigmentation (Copig), Total Red Pigments (TRP), and Total Polyphenol Index (TPI). Results obtained for each variety in both vintages are reported in Tables 1–4.

8. Extraction and analyses of phenolic compounds from wines

8.1. Tannin extraction

The tannin fraction was extracted by SPE on a Fractogel column (6 mL Fractogel, 1.6 cm diameter column, 3 cm Fractogel height). The column conditioning was 3 × 5 mL H₂O = 0.05 % TFA. Five millilitres were placed on the column.

TABLE 1. Average relative amounts for colour parameters, total anthocyanins (glucosylated, acetylated, and coumaroylated), and tannins parameters for the Syrah mono-varietal wines in 2021 and 2022 vintages. Anthocyanins were calculated as mg/L eq. malvidin-3-O-glucoside. Concerning ANOVA results: * = p -value < 0.05; ** = p -value < 0.01; *** = p -value < 0.001; ns = not significant (for a p -value > 0.05). Different Latin letters in the same row indicate statistically significant differences between mean values (p < 0.05).

Variables	Syrah					Year	
	AL	DU	LA	LE	MA	Average	p -value
CI							
2021 ***	12.41 ^c	12.75 ^c	13.51 ^b	14.19 ^a	13.82 ^{ab}	13.34 ^b	***
2022 ***	14.25 ^b	8.53 ^c	20.69 ^a	9.33 ^c	20.14 ^a	14.59 ^a	
TPI							
2021 **	47.45 ^b	47.00 ^b	54.05 ^a	48.35 ^{ab}	53.00 ^{ab}	49.98 ^b	***
2022 ***	68.80 ^a	50.05 ^b	61.20 ^a	48.75 ^b	62.15 ^a	58.20 ^a	
NBP							
2021 **	2.51 ^b	2.72 ^b	2.84 ^{ab}	3.26 ^a	2.81 ^b	2.83 ^a	***
2022 ***	2.89 ^a	1.52 ^e	2.26 ^c	1.87 ^d	2.38 ^b	2.18 ^b	
Copig							
2021 ***	4.94 ^b	3.27 ^c	4.65 ^b	5.63 ^a	5.51 ^a	4.80 ^b	***
2022 ***	7.20 ^b	3.97 ^c	12.87 ^a	4.8 ^{bc}	12.49 ^a	8.27 ^a	
TRP							
2021 ***	19.70 ^{cd}	20.85 ^{bc}	24.70 ^a	17.80 ^d	23.25 ^{ab}	21.26 ^b	***
2022 ***	26.10 ^b	24.75 ^b	35.95 ^a	22.40 ^b	32.05 ^a	28.25 ^a	
Total glucosylated anthocyanins*							
2021 ***	262.77 ^{bc}	332.03 ^a	279.45 ^b	255.73 ^c	278.41 ^b	281.68 ^a	***
2022 ***	161.70 ^c	199.43 ^b	246.74 ^a	184.28 ^b	252.03 ^a	208.84 ^b	
Total acetylated anthocyanins*							
2021 ***	93.32 ^c	298.65 ^a	114.44 ^b	97.09 ^c	114.44 ^b	144.19 ^a	***
2022 ***	71.35 ^d	124.68 ^{bc}	144.40 ^a	119.57 ^c	134.120 ^{ab}	118.2 ^b	
Total coumaroylated anthocyanins*							
2021 **	38.59 ^b	66.24 ^a	53.81 ^{ab}	49.88 ^{ab}	41.70 ^b	50.05 ^a	ns
2022 ***	45.41 ^c	44.20 ^c	61.71 ^a	53.18 ^b	63.97 ^a	53.70 ^a	
Trihydroxylated/dihydroxylated anthocyanins							
2021 ***	8.98 ^d	16.49 ^a	11.18 ^c	9.16 ^d	13.62 ^b	11.88 ^a	ns
2022 ***	7.93 ^d	9.42 ^c	17.34 ^a	8.33 ^d	15.27 ^b	11.65 ^a	
Total tannins**							
2021 **	740 ^{ab}	627 ^b	887 ^a	813 ^a	880 ^a	789 ^a	ns
2022 ***	700 ^c	613 ^c	907 ^{ab}	820 ^b	993 ^a	807 ^a	
% Modifications of tannins							
2021 *	47.10 ^{ab}	55.04 ^{ab}	56.87 ^a	43.19 ^b	57.53 ^a	51.94 ^a	ns
2022 ***	47.67 ^b	47.98 ^b	58.08 ^a	41.32 ^c	55.21 ^a	50.05 ^a	
Apparent aDP							
2021 *	7.13 ^a	5.69 ^b	7.26 ^a	6.03 ^b	6.78 ^a	6.58 ^a	*
2022 ***	6.90 ^a	5.79 ^d	7.13 ^a	6.04 ^c	6.28 ^b	6.43 ^b	
*expressed as mg/L eq. malvidin-3-O-glucoside							
**expressed as mg/L							

TABLE 2. Average relative amounts for colour parameters, total anthocyanins (glucosylated, acetylated, and coumaroylated), and tannins parameters for the Grenache mono-varietal wines in 2021 and 2022 vintages. Anthocyanins were calculated as mg/L eq. malvidine-3-O-glucoside. Concerning ANOVA results: * = p -value < 0.05; ** = p -value < 0.01; *** = p -value < 0.001; ns = not significant (for a p -value > 0.05); n.a. = not available. Different Latin letters in the same row indicate statistically significant differences between mean values (p < 0.05).

Variables	Grenache					Year	
	AL	DU	LA	LE	MA	Average	p -value
CI							
2021 ***	5.51 ^b	7.71 ^a	n.a.	5.78 ^b	4.63 ^c	5.90 ^a	ns
2022 ***	4.87 ^c	11.03 ^a	8.14 ^b	2.24 ^c	3.69 ^c	6.59 ^a	
TPI							
2021 **	51.50 ^a	40.05 ^b	n.a.	40.25 ^b	40.65 ^b	43.11 ^a	***
2022 ***	54.30 ^a	52.35 ^a	52.95 ^a	46.40 ^b	28.95 ^c	46.99 ^a	
NBP							
2021 **	0.93 ^b	1.61 ^a	n.a.	1.08 ^b	0.86 ^b	1.12 ^a	***
2022 ***	1.02 ^c	2.12 ^a	1.26 ^b	1.06 ^c	0.50 ^d	1.19 ^a	
Copig							
2021 ***	3.68 ^b	3.45 ^{bc}	n.a.	4.80 ^a	3.18 ^c	3.78 ^a	***
2022 ***	1.86 ^c	6.67 ^a	4.47 ^b	2.36 ^c	2.24 ^c	3.51 ^a	
TRP							
2021 *	14.25 ^a	13.00 ^{ab}	n.a.	13.35 ^{ab}	12.15 ^b	13.19 ^a	***
2022 ***	11.65 ^b	12.70 ^b	14.70 ^a	12.65 ^b	7.20 ^c	11.78 ^b	
Total glucosylated anthocyanins*							
2021 ***	197.11 ^b	156.07 ^b	n.a.	268.90 ^a	153.24 ^c	188.80 ^a	***
2022 ***	109.39 ^c	99.26 ^d	146.28 ^a	137.28 ^b	105.44 ^c	119.53 ^b	
Total acetylated anthocyanins*							
2021 ***	5.27 ^b	4.07 ^b	n.a.	10.93 ^a	4.59 ^b	7.62 ^a	***
2022 ***	4.25 ^c	3.41 ^d	7.27 ^a	6.39 ^b	4.51 ^c	5.16 ^b	
Total coumaroylated anthocyanins*							
2021 ***	18.26 ^b	12.63 ^c	n.a.	42.59 ^a	18.14 ^b	23.62 ^a	***
2022 ***	10.01 ^d	8.44 ^e	16.44 ^a	11.98 ^c	13.03 ^b	11.98 ^b	
Trihydroxylated/dihydroxylated anthocyanins							
2021 ***	6.84 ^d	12.63 ^c	n.a.	13.71 ^b	16.20 ^a	12.35 ^a	ns
2022 ***	6.75 ^d	8.84 ^c	15.59 ^b	16.22 ^b	30.83 ^a	15.65 ^a	
Total tannins**							
2021 ns	587 ^a	567 ^a	n.a.	627 ^a	480 ^a	565 ^a	ns
2022 ***	527 ^b	533 ^b	427 ^b	500 ^b	660 ^a	529 ^a	
% Modifications of tannins							
2021 ns	30.44 ^a	26.38 ^a	n.a.	22.23 ^a	26.22 ^a	26.32 ^b	**
2022 ***	22.57 ^d	32.85 ^c	24.49 ^d	37.82 ^b	44.14 ^a	32.37 ^a	
Apparent aDP							
2021 **	6.54 ^a	5.22 ^b	n.a.	5.97 ^a	6.13 ^a	5.96 ^a	ns
2022 ***	5.88 ^a	4.47 ^b	4.69 ^b	4.63 ^b	5.80 ^a	5.09 ^a	
*expressed as mg/L eq. malvidin-3-O-glucoside							
**expressed as mg/L							

TABLE 3. Average relative amounts for colour parameters, total anthocyanins (glucosylated, acetylated, and coumaroylated), and tannins parameters for the Carignan mono-varietal wines in 2021 and 2022 vintages. Anthocyanins were calculated as mg/L eq. malvidine-3-O-glucoside. Concerning ANOVA results: * = p -value < 0.05; ** = p -value < 0.01; *** = p -value < 0.001; ns = not significant (for a p -value > 0.05). Different Latin letters in the same row indicate statistically significant differences between mean values (p < 0.05).

Variables	Carignan					Year	
	AL	DU	LA	LE	MA	Average	p -value
CI							
2021 ***	14.25 ^b	9.27 ^d	9.55 ^d	16.20 ^a	13.54 ^c	12.56 ^b	***
2022 ***	12.80 ^b	12.74 ^b	13.19 ^b	13.62 ^b	16.05 ^a	13.68 ^a	
TPI							
2021 ***	37.80 ^{bc}	39.05 ^b	32.80 ^c	44.80 ^b	42.90 ^b	39.47 ^b	***
2022 **	52.60 ^a	47.15 ^{ab}	48.65 ^{ab}	46.70 ^b	44.55 ^b	47.93 ^a	
NBP							
2021 ***	2.29 ^b	1.73 ^d	2.54 ^a	2.50 ^a	2.17 ^c	2.05 ^b	***
2022 ***	1.02 ^c	2.12 ^a	1.26 ^b	1.06 ^c	0.50 ^d	2.25 ^a	
Copig							
2021 ***	4.80 ^b	3.47 ^c	4.89 ^b	5.63 ^a	5.55 ^a	4.87 ^b	***
2022 ***	6.88 ^b	7.49 ^b	6.98 ^b	7.77 ^b	9.82 ^a	7.79 ^a	
TRP							
2021 *	18.20 ^b	21.00 ^a	15.25 ^c	22.10 ^a	22.60 ^a	19.83 ^b	***
2022 ***	23.45 ^a	23.55 ^a	24.20 ^a	19.00 ^b	23.55 ^a	22.75 ^a	
Total glucosylated anthocyanins*							
2021 ***	214.96 ^c	320.29 ^b	205.58 ^d	364.45 ^a	180.49 ^a	257.15 ^a	***
2022 ***	203.60 ^c	227.98 ^a	210.05 ^b	145.75 ^d	232.27 ^c	203.93 ^b	
Total acetylated anthocyanins*							
2021 ***	7.84 ^c	16.07 ^b	7.90 ^c	34.61 ^a	5.56 ^d	14.40 ^a	ns
2022 ***	12.18 ^c	16.34 ^a	13.61 ^b	12.81 ^c	16.80 ^a	14.35 ^a	
Total coumaroylated anthocyanins*							
2021 ***	40.59 ^c	49.82 ^b	30.33 ^d	59.01 ^a	23.72 ^e	40.69 ^a	***
2022 ***	38.35 ^b	38.47 ^b	35.42 ^c	32.90 ^d	42.78 ^a	37.58 ^b	
Trihydroxylated/dihydroxylated anthocyanins							
2021 ***	12.18 ^d	18.45 ^a	16.37 ^b	13.71 ^c	18.85 ^a	15.91 ^a	ns
2022 ***	109.39 ^c	99.26 ^d	146.28 ^a	137.28 ^b	105.44 ^c	18.15 ^a	
Total tannins**							
2021 *	613 ^{ab}	560 ^b	593 ^{ab}	707 ^a	560 ^b	607 ^b	***
2022 ***	693 ^{bc}	567 ^d	627 ^{cd}	740 ^{ab}	807 ^a	687 ^a	
% Modifications of tannins							
2021 ***	48.01 ^{ab}	52.99 ^a	52.31 ^a	38.96 ^{bc}	35.45 ^c	45.54 ^a	ns
2022 ***	47.48 ^b	52.48 ^a	46.75 ^b	29.24 ^d	44.12 ^c	44.01 ^a	
Apparent aDP							
2021 ns	5.95 ^a	5.88 ^a	6.30 ^a	6.57 ^a	5.85 ^a	6.12 ^a	***
2022 *	5.91 ^a	5.48 ^{ab}	5.30 ^b	5.57 ^{ab}	5.55 ^{ab}	5.57 ^b	
*expressed as mg/L eq. malvidin-3-O-glucoside							
**expressed as mg/L							

TABLE 4. Average relative amounts for colour parameters, total anthocyanins (glucosylated, acetylated, and coumaroylated), and tannins parameters for the Mourvèdre mono-varietal wines in 2021 and 2022 vintages. Anthocyanins were calculated as mg/L eq. malvidine-3-O-glucoside. Concerning ANOVA results: * = p -value < 0.05; ** = p -value < 0.01; *** = p -value < 0.001; ns = not significant (for a p -value > 0.05). Different Latin letters in the same row indicate statistically significant differences between mean values (p < 0.05).

Variables	Mourvèdre					Year	
	AL	DU	LA	LE	MA	Average	p -value
CI							
2021 ***	21.94 ^a	7.89 ^e	9.19 ^d	13.74 ^b	13.19 ^c	13.20 ^a	ns
2022 ***	19.77 ^a	8.16 ^d	11.01 ^c	10.38 ^c	16.69 ^b	13.20 ^a	
TPI							
2021 ***	55.35 ^a	34.25 ^c	42.75 ^b	54.60 ^a	52.35 ^a	47.86 ^b	***
2022 **	63.45 ^a	43.80 ^c	54.35 ^b	42.90 ^c	59.15 ^{ab}	52.73 ^a	
NBP							
2021 ***	4.90 ^a	1.22 ^c	1.34 ^c	2.50 ^b	2.46 ^b	2.48 ^a	***
2022 ***	2.67 ^b	1.34 ^d	1.90 ^c	1.27 ^e	2.97 ^a	2.03 ^b	
Copig							
2021 ***	2.91 ^c	3.36 ^b	4.15 ^a	4.44 ^a	2.79 ^c	3.53 ^b	***
2022 ***	12.05 ^a	3.94 ^e	5.22 ^d	6.39 ^c	8.96 ^b	7.31 ^a	
TRP							
2021 ***	25.45 ^a	17.40 ^c	21.95 ^b	24.35 ^{ab}	23.30 ^{ab}	22.49 ^b	***
2022 ***	27.05 ^a	21.90 ^b	27.45 ^a	19.45 ^c	26.85 ^a	24.54 ^a	
Total glucosylated anthocyanins*							
2021 ***	219.04 ^c	227.32 ^c	290.97 ^a	257.81 ^b	304.48 ^a	259.92 ^a	***
2022 ***	252.34 ^b	205.27 ^d	266.29 ^a	215.03 ^{cd}	224.29 ^c	232.64 ^b	
Total acetylated anthocyanins*							
2021 ***	40.91 ^b	14.79 ^d	19.99 ^c	16.47 ^d	49.74 ^a	28.38 ^a	***
2022 ***	25.16 ^b	23.60 ^b	33.83 ^a	17.03 ^c	15.05 ^c	22.93 ^a	
Total coumaroylated anthocyanins*							
2021 ***	32.22 ^c	44.90 ^b	47.60 ^b	44.65 ^b	55.00 ^a	44.87 ^a	***
2022 ***	42.76 ^c	40.32 ^d	44.41 ^b	49.98 ^a	33.94 ^e	42.28 ^b	
Trihydroxylated/dihydroxylated anthocyanins							
2021 ***	4.70 ^d	9.63 ^a	5.96 ^b	5.78 ^{bc}	5.67 ^c	6.35 ^b	**
2022 ***	5.93 ^d	13.69 ^a	8.64 ^c	10.46 ^b	8.89 ^c	9.52 ^a	
Total tannins**							
2021 ***	673 ^b	380 ^c	553 ^b	627 ^b	887 ^a	624 ^b	**
2022 ***	687 ^b	573 ^c	653 ^{bc}	640 ^{bc}	847 ^a	680 ^a	
% Modifications of tannins							
2021 *	30.32 ^b	42.34 ^a	38.45 ^{ab}	39.58 ^{ab}	41.53 ^{ab}	38.44 ^b	**
2022 ***	33.65 ^d	45.92 ^b	37.37 ^{cd}	38.65 ^{cd}	53.83 ^a	41.88 ^a	
Apparent aDP							
2021 ns	6.83 ^a	6.74 ^a	6.22 ^a	6.03 ^a	7.18 ^a	6.60 ^a	**
2022 ***	6.23 ^b	6.17 ^b	6.09 ^b	5.36 ^c	7.08 ^a	6.19 ^b	
*expressed as mg/L eq. malvidin-3-O-glucoside							
**expressed as mg/L							

The gradient was 2 × 5 × mL EtOH/H₂O (15/85, v/v) + 0.05 % TFA, 2 × 5 × mL EtOH/H₂O (30/70, v/v) + 0.05 % TFA, 3 × 5 mL EtOH/H₂O (35/65, v/v) + 0.05 % TFA, 3 × 5 mL EtOH/H₂O (40/60, v/v) + 0.05 % TFA, 2 × 5 mL EtOH/H₂O (50/50, v/v) + 0.05 % TFA, 3 × 5 mL EtOH/H₂O (60/40, v/v) + 0.05 % TFA, 3 × 5 mL EtOH/Acetone/H₂O (30/45/25, v/v). Elution of glycosylated anthocyanins took place at 30 % and 35 % ethanol, acetylated anthocyanins at 35 % ethanol, and coumaroylated anthocyanins and a small proportion of tannin dimers at 40 % ethanol. The tannin fraction was eluted from 50 % ethanol. The tannin fraction was then lyophilised and weighed to calculate the concentration of the tannins (mg/L).

8.2. Depolymerisation of tannin fractions

In this work we used the same thioglycolysis procedure described in the work of Deshaies *et al.* (2022).

8.3. Quantitative analyses of anthocyanins by UHPLC-MS

The samples were analysed using a Waters reversed-phase ultra-performance liquid chromatography system coupled to a Bruker amaZon X mass spectrometer. The liquid chromatography system comprised an ACQUITY UPLC (Waters, Milford, MA, USA) equipped with a photodiode array detector. The column was an ACQUITY UPLC HSS T3 (1.8 µm, 1 × 150 mm) preceded by a Waters column in-line filter (0.2 µm, 2.1 mm). All samples were analysed in triplicate. The mobile phase was composed of (A) water, containing 1 % (v/v) formic acid, and (B) 80 % (v/v) acetonitrile, 19 % (v/v) water, containing 1 % (v/v) formic acid. The elution gradient was as follows: 1 % B for 2 min, from 1 % to 8 % B for 13 min, from 8 % to 25 % B for 45 min, from 25 % to 95 % for 5 min, isocratic 95 % B for 4 min, from 95 % to 1 % for 1 min, isocratic 1 % for 5 min, with a flow rate at 0.13 mL/min. Injection volume was 2 µL, and the detection wavelength was from 200 to 600 nm. The column temperature was 38 °C. The MS conditions were as follows: Electrospray Ionisation (ESI), positive ion model; nebuliser (44 psi); dry gas flow (12 L/min); dry gas temperature (200 °C); scan 100–1000 *m/z*. Anthocyanins were quantified from the EICs after UHPLC-MS analysis. Quantification of malvidin-3-*O*-glucoside, delphinidin-3-*O*-glucoside, cyanidin-3-*O*-glucoside, peonidin-3-*O*-glucoside, and petunidin-3-*O*-glucoside was carried out using standards and the acetylated and coumaroylated derivatives were semi-quantified in malvidin-3-*O*-glucoside equivalent. Results were expressed as mg/L eq. malvidin-3-*O*-glucoside. Results obtained for each variety for both vintages are reported in Tables 1–4.

8.4. Analysis of tannins

After depolymerisation, the “apparent” Degree of Polymerisation (aDP), the percentage of modifications of the tannin fraction and the percentage of Epicatechin Gallate (% ECG) were determined from the UV chromatogram at 280 nm, according to the same protocols previously described by Deshaies *et al.* (2022) and Ben Aziz *et al.* (2017).

In particular, the percentage of modifications was calculated using the following formula:

$$\% \text{ modified tannins} = \frac{\Omega}{\Omega + \Delta} \times 100 \quad (1)$$

where Ω is the area of the hump under the peaks of the unmodified units; *i.e.*, the area of the tannins’ modified constitutive units; and Δ is the area of the peaks corresponding to the constitutive units of the tannins’ unmodified terminal and extension.

Finally, the percentage of epicatechin gallate (% ECG) was calculated as the ratio of the sum of the areas of terminal and constituent unmodified ECG units to the total areas of unmodified units. All samples were analysed in triplicate. Results obtained for each variety in both vintages are reported in Tables 1–4.

9. Sensorial analysis

9.1. Panel training and selection

Sensory analysis was performed by a sensory panel of expert judges unrelated to the wine industry, who were selected based on their sensory performance and further trained to perform wine descriptive analyses (Norm ISO 8586:2023). Twenty judges (7 men and 13 women, average age of 51 years) were selected in the first year, and twenty-two judges (7 men and 15 women, average age of 50 years) in the second year.

9.2. Analysis of mono varietal wines through check-all-that-apply (CATA) methodology

Monovarietal and blended wines were analysed using the CATA methodology (Meyners *et al.*, 2013). During the first two sessions, judges were trained to exercise, understand and consistently use attributes, and to familiarise themselves with the methodology. The list of attributes was compiled by i) taking the attributes found in a previous descriptive analysis on commercial wines from the AOC Corbières, and ii) adding sensorial descriptors that in other studies were associated with the varieties that we analysed (Argentero *et al.*, 2024). Standards were prepared by adding compounds to red wine and were used by the judges to help them identify and remember the sensory attributes. Wines were then analysed over five sessions (one session for each grape variety and one session for the blends). Analyses were conducted in individual testing booths. Samples (30 mL) were served at room temperature (18.3 ± 0.9 °C) in black glasses (to avoid any potential visual influence during evaluations) covered with a plastic cap and marked with random three-digit codes. Wines were presented in a monadic service, according to William’s Latin square design, to balance the presentation order and carry-over effect (Macfie *et al.*, 1989). Wines were analysed in duplicate in the same session. For each sample, the judges had to choose the most pertinent attributes, from a list of 33 olfactory terms, classified hierarchically into 15 olfactory families and 7 gustatory terms (astringency, fat, aqueous, acid, bitter, alcoholic, and sweet). Among the seven gustatory terms in this study, we particularly focused on astringency. Data acquisition was assisted by FIZZ software (FIZZ network, v. 2.518; Biosystème, Couternon, France).

9.3. Analysis of blended wines through check-all-that-apply (CATA) methodology

In both vintages, the same blend was made starting from the monovarietal wines, for each of the subregions of study. Each wine contained the four varieties in the following percentages: 30 % Syrah, 30 % Grenache, 30 % Carignan, and 10 % Mourvèdre. Percentages were established as a reflection of the cultivation trends through the appellation. Five wines were then made and analysed as monovarietal wines through a CATA sensorial analysis.

10. Statistical analysis

10.1. Sensorial analysis

An average Reproducibility index (R_i) was calculated to assess the individual performance of each judge: $R_i = (1/n * \Sigma[2 * \text{desrep}/(\text{desrep1} + \text{desrep2})]$, where desrep is the number of the same terms used by the judge for each replicate, desrep1 and desrep2 are the total numbers of terms used by the judge for the first and second replicates (respectively) and n is the number of samples duplicated. This parameter, ranging from 0 to 1, was used in previous work (Campo *et al.*, 2008). The sensory data obtained were binary and their analysis was performed using XLSTAT software (version 2022, Addinsoft, Paris, France). To analyse the significant differences between samples for each attribute, a Cochran's Q test was performed. When significant differences were revealed ($p < 0.05$), we performed multiple pairwise comparisons based on the critical difference of Sheskin.

10.2. Polyphenol parameters

For polyphenol variables, ANOVA analysis (zone and vintage factors) was used, applying a significant level of 5 %. Mean values of the five samples for each variety were compared through the Tukey multiple comparison test. These data analyses were performed using XLSTAT software (version 2022, Addinsoft Paris). The results obtained are reported in Tables 1–4.

10.3. Chemical and sensorial MFA analysis

For each grape variety, a Multiple Factor Analysis (MFA) was carried out on a selection of variables from the polyphenol (*anthocyanins, colour, and tannins*) and sensorial analyses (*Senso_gust*) by taking into consideration both vintages (Figures 1 to 4). The five subregions (*Vineyard*), three oenological parameters (*S/A, TAV and pH*) and the vintages (*Year*) were added as supplementary variables.

RESULTS AND DISCUSSION

1. Climate conditions in the 2021 and 2022 vintages

In terms of rainfall, 2022 stood out for its higher precipitation levels in March and April in comparison to 2021 (149.74 mm compared to 16.16 mm in March, and 55.98 mm compared to 29.08 mm in April, respectively).

This was succeeded by a dry period between July and September, during which a total of 36.28 mm of precipitation was recorded in the three-month period in 2022, in contrast to the 105.88 mm recorded in 2021 (Figure S1). Concerning rainfall throughout the appellation, differences between the five subregions were more marked in 2021, with MA and DU generally receiving a lower amount of water from January to October (253.6–284 mm) than in the other three subregions (309–312 mm). In 2022, vintage differences were less evident, with MA receiving lower amounts of rainfall (307.5 mm) and DU and LA generally receiving a higher amount of water (350 mm, like LA subregion, 347 mm).

In terms of temperatures, 2022 was characterised by average minimum and maximum temperatures that were three degrees higher than in the 2021 vintage between May and August (except for June, when the two vintages had similar average values) (Figure 2b). Besides these variations, temperatures varied slightly between the five subregions within each vintage. In both seasons, DU vineyards showed the lowest average temperature degrees during the vegetative period and LE (followed by AL) the highest.

2. Sensorial analysis: performance results

The ranking of the reproducibility index (R_i) associated with a judge allowed us to evaluate the accuracy of a judge's performance. The performances of the jury are quite homogeneous, even if they are slightly better in Year 2 (69 % of the common terms between the two replicates on average) than in Year 1 (51 % of the common terms between the two replicates on average).

3. Polyphenolic profile of wines depending on the plot

Each variety was treated and is presented here individually in order to overcome the variety impact on the discrimination of the five vineyards, and thus be able to gain a more specific understanding of the impact of the varying climate conditions – modulated by the different soil characteristics – on the polyphenolic and sensorial profiles of the wines originating from these territories.

3.1. Syrah expression

For the Syrah global MFA (Figure 1), F1 and F2 axes accounted for 52.95 % of the total variance (Figure 1a). The graph shows that the samples were generally divided depending on the vintage: the majority of the 2021 samples are situated in the upper section of the graph, while the 2022 samples are in the lower section, except for SYR_AL_22 (Figure 1b). This distribution could be related to anthocyanin content, with 2022 wines showing significantly lower total amounts than 2021 wines ($p < 0.0001$), of anthocyanidin-3-*O*-glucosides (208.84 mg/L in 2022, 281.68 mg/L in 2021) and acetylated anthocyanins (118.2 mg/L in 2022, 144.19 mg/L in 2021). Colour parameters were also affected by the vintage, showing an opposite trend to anthocyanins, with 2022 wines having significantly higher mean values of TPI (58.19), CI (14.59), Copig (8.27), and TRP (28.25), compared to those from the 2021 vintage (49.97, 13.34,

4.8, and 21.26, respectively) (Table 1). Regarding tannin compounds, the total concentration and the percentage of modification parameters were not affected by vintage (Table 1). As reported above, 2022 was a warmer vintage, with higher temperatures between August and September coupled to lower rainfall; as a result, the grapes were characterised by a higher mean S/A ratio (56.4 in 2021, 72.4 in 2022), due to both higher sugar concentrations and lower total acidities, compared to 2021. Tannin accumulation in grapes occurs from flowering to *véraison* and is influenced by several factors (*e.g.*, water stress, Sadras *et al.*, 2013). As a result, even though the S/A ratio was higher in the 2022 vintage, which possibly led to a higher extraction of tannins during alcoholic fermentation, the climatic conditions in the 2021 vintage may have promoted a higher accumulation of tannins, resulting in a similar final concentration in wines. However, vintage had a similar effect on the plots, which were divided into two smaller groups: DU, LE, and AL, which were relatively close to each other in

both vintages (on the left-hand side of the graph), and LA and MA, which formed one group in both vintages on the right-hand side of the graph (Figure 1b). In fact, in both vintages, Syrah wines from LA and MA vineyards showed significantly higher concentrations of total anthocyanins (434–447 mg/L in 2021, 452 mg/L in 2022) compared to LE and AL wines (402–406 mg/L in 2021, 278–357 mg/L in 2022), as well as significantly higher total tannins (900–990 mg/L) and percentage of modifications (55–58 %) compared to AL and DU (600–740 mg/L) and to LE, AL, and DU (41–55 %), respectively (Table 1). In our study plots, samples were collected at similar sugar concentrations in both vintages; nonetheless, at harvest, the S/A ratios of LA and MA were slightly higher (59–65 in 2021, 72–76 in 2022) than those of the LE, AL and DU samples (52–55 in 2021, 66–75 in 2022); this indicates that the phenolic maturity of the LA and MA samples may have been more advanced, thus resulting in a higher concentration and better extraction of anthocyanins.

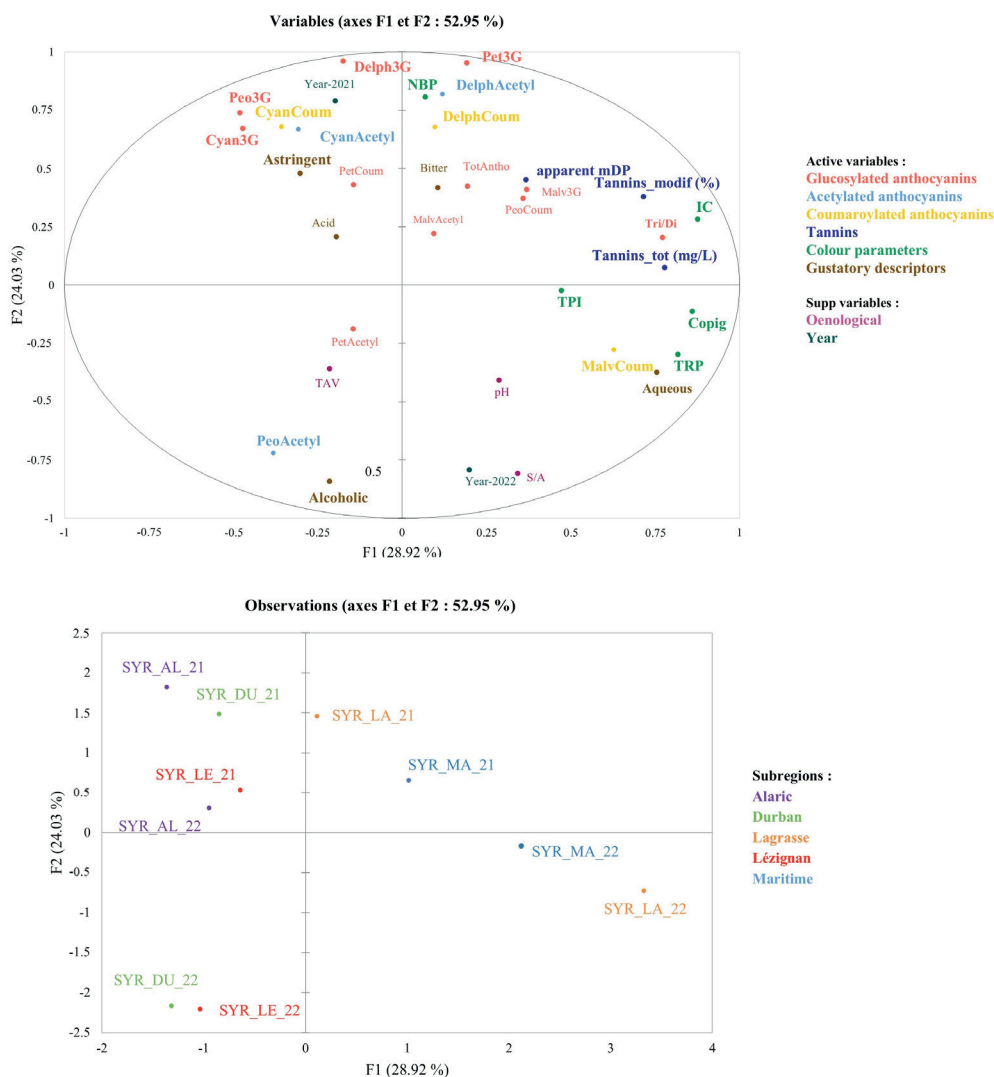


FIGURE 1. Multiple factor analysis (MFA) on polyphenols parameters and sensorial descriptors for Syrah variety wines. Oenological variables, climate indexes, and year are reported as supplementary parameters. a) Graph of variables on F1 and F2 axes. Groups of variables are reported with code colours (see legend). Variables reported in bold were those that contributed to the construction of the axis. b) Graph of individuals. Distribution of wine samples in F1 and F2 axes. The five subregions were reported with a colour code.

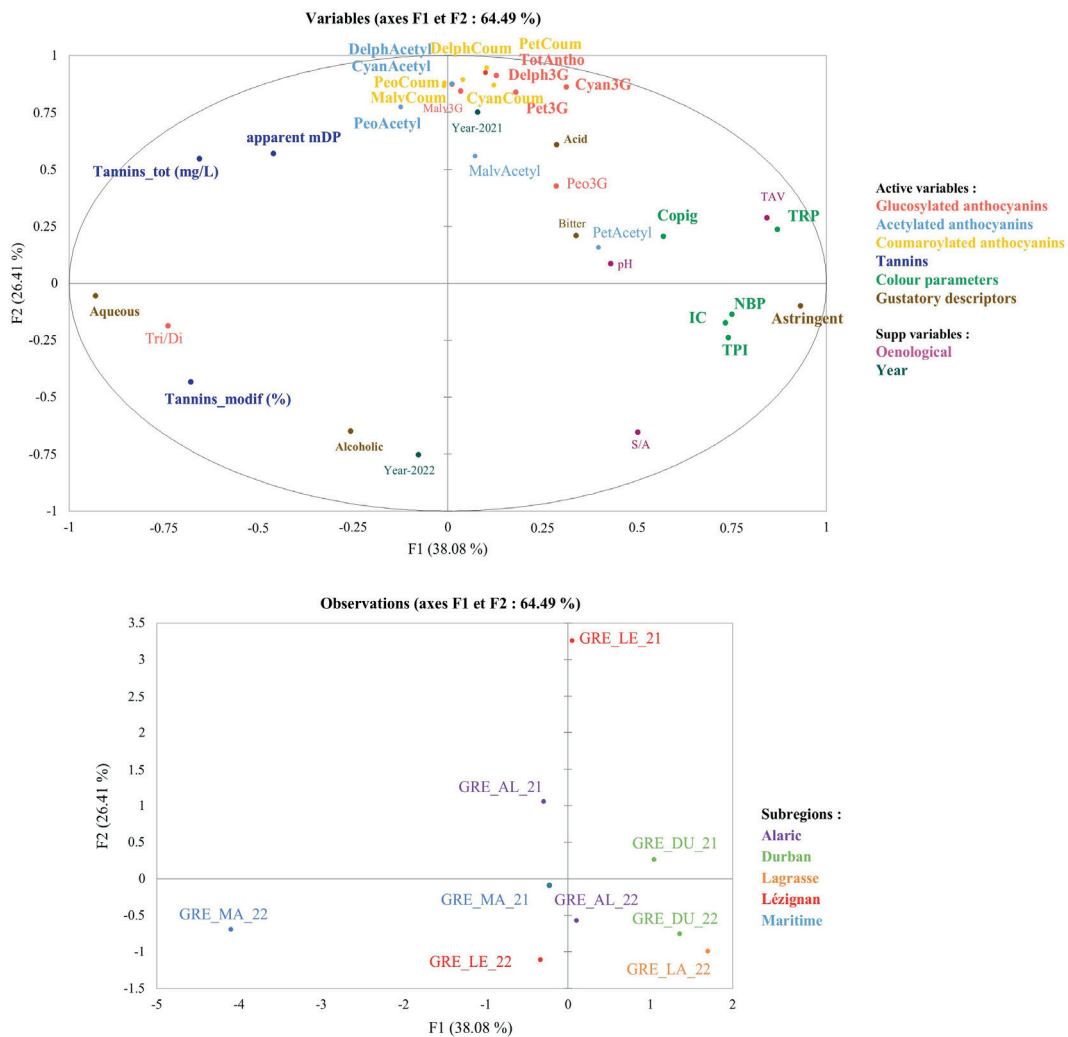


FIGURE 2. Multiple factor analysis (MFA) on polyphenols parameters and sensorial descriptors for Grenache variety wines. Oenological variables, climate indexes, and year are reported as supplementary parameters. a) Graph of variables on F1 and F2 axes. Groups of variables are reported with code colours (see legend). Variables reported in bold were those that contributed to the construction of the axis. b) Graph of individuals. Distribution of wine samples in F1 and F2 axes. The five subregions were reported with a colour code.

3.2. Grenache expression

Regarding the Grenache overall MFA (Figure 2), the F1 and F2 axes accounted for 64.49 % of the total variance. As with the Syrah wines, differences were observed for Grenache wines between vintages, but those from the 2021 vintage seemed more dispersed (Figure 2): the 2021 samples were characterised by varying S/A ratios (56,41–70,73), thus the evaluation of the impact of soil and climate parameters on the discrimination of these samples is more difficult due to a possible bias of differing maturity levels (Table S2). In general, the Grenache wines from 2022 showed significantly lower glucosylated, acetylated, and coumaroylated anthocyanin total concentrations ($p < 0.0001$), but a significantly higher percentage of modification of tannins (32 %) compared to the 2021 wines (26 %). Nonetheless, no significant differences in the total concentration (mg/L) of tannins and in the average values of the colour parameters were found between vintages, except for an increase in BA and TRP variables in 2022 (Table 2).

As a result, in both vintages, the DU wines showed significantly higher CI (7.7–11), BA (8.9–9.5), and NBP (1.6–2.1) values than the MA wines (3.6–4.6, 2.9–5.9, and 0.5–0.8, respectively) (Table 2). Concerning the anthocyanins, LA (only in 2022) and LE showed significantly higher concentrations of anthocyanidin-3-*O*-glucosides (268–137 mg/L), acetylated (10.92–6.39 mg/L), and coumaroylated (42.59–11.98 mg/L) anthocyanins than the AL, MA, and DU samples in both vintages (Table 2). However, concerning the trihydroxylated/dihydroxylated anthocyanins ratio (Tri/Di) in both vintages, the Grenache from MA showed a significantly higher value (16.2 in 2021 and 30.83 in 2022) than the Grenache from DU (12.6 in 2021, 8.8 in 2022) and AL (6.8, in both vintages). The Tri/Di ratio has been analysed in previous studies: in particular, Castellarin *et al.* (2007) have shown that in conditions of higher water stress, the metabolism shifts towards a higher accumulation of trihydroxylated anthocyanins. In the present study, no measurements were taken to assess the water stress status of the plants. However,

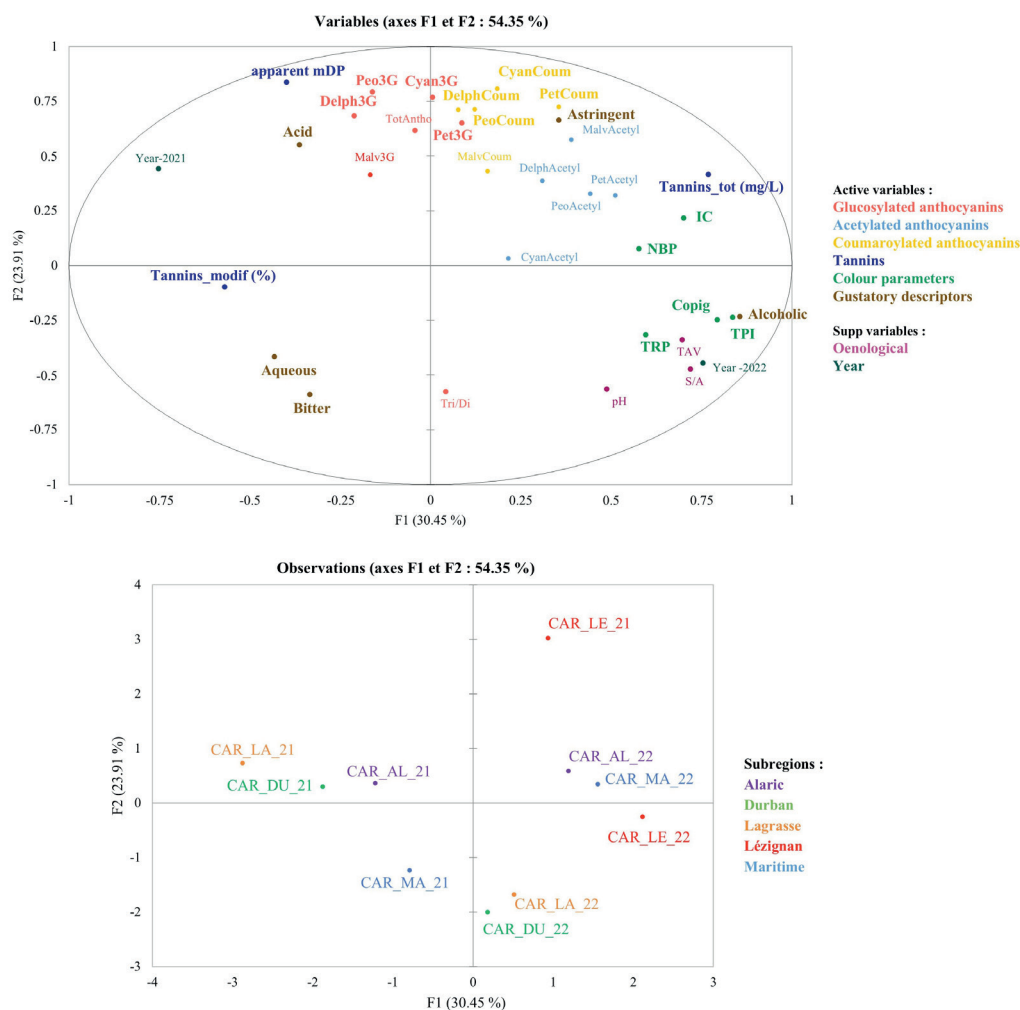


FIGURE 3. Multiple factor analysis (MFA) on polyphenols parameters and sensorial descriptors for Carignan variety wines. Oenological variables, climate indexes, and year are reported as supplementary parameters. a) Graph of variables on F1 and F2 axes. Groups of variables are reported with code colours (see legend). Variables reported in bold were those that contributed to the construction of the axis. b) Graph of individuals. Distribution of wine samples in F1 and F2 axes. The five subregions were reported with a colour code.

it is interesting to note that MA was generally characterised by lower cumulated rainfall from June to September than AL, which may have had an impact on the water availability in the soil. This is coupled with a lower average temperature range from *véraison* (July to September) in comparison to that of the DU subregion. In general, while no significant differences among the samples were recorded in the 2021 vintage, in 2022, the MA wine contained a significantly higher concentration of tannins (660 mg/L) and percentage of modifications (44 %) than all the other samples, in particular AL.

To summarise, in both vintages, the Grenache wines from the MA vineyard contained lower concentrations of total anthocyanins, resulting in lower colour pigments values (especially CI, TPI, and NBP) than the other samples. In addition, they were also characterised by a higher amount of tannins (but with a higher percentage of modifications), which was possibly linked to a lower astringent perception. Conversely, Grenache from DU maintained high values (especially TPI and NBP values) over the two vintages,

while showing low anthocyanin values. In both vintages, these two samples were harvested at similar maturity, but with a one-month gap in 2021 and a two-week gap in 2022. As proposed above, higher temperatures in 2022 could have induced a higher sugar accumulation, causing a shift between technological and polyphenolic maturity (Sadras *et al.*, 2013). This could explain the difference in Tri/Di ratio between the two samples, with Grenache from MA possibly being more highly subjected to water stress than Grenache from the DU vineyards.

3.3. Carignan expression

Regarding the Carignan global MFA (Figure 3), F1 and F2 axes accounted for 54.35 % of the total variance. The graph reveals a vintage effect, with all the 2021 samples located in the left-hand section of the graph (except for LE) and the 2022 samples in the right-hand section (Figure 3b). Indeed, the 2022 wines showed significantly higher average colour parameters values (especially TPI, TRP, and Copig) than the 2021 ones, as well as a significantly higher concentration of total tannins (700 mg/L in 2022 compared to 600 mg/L

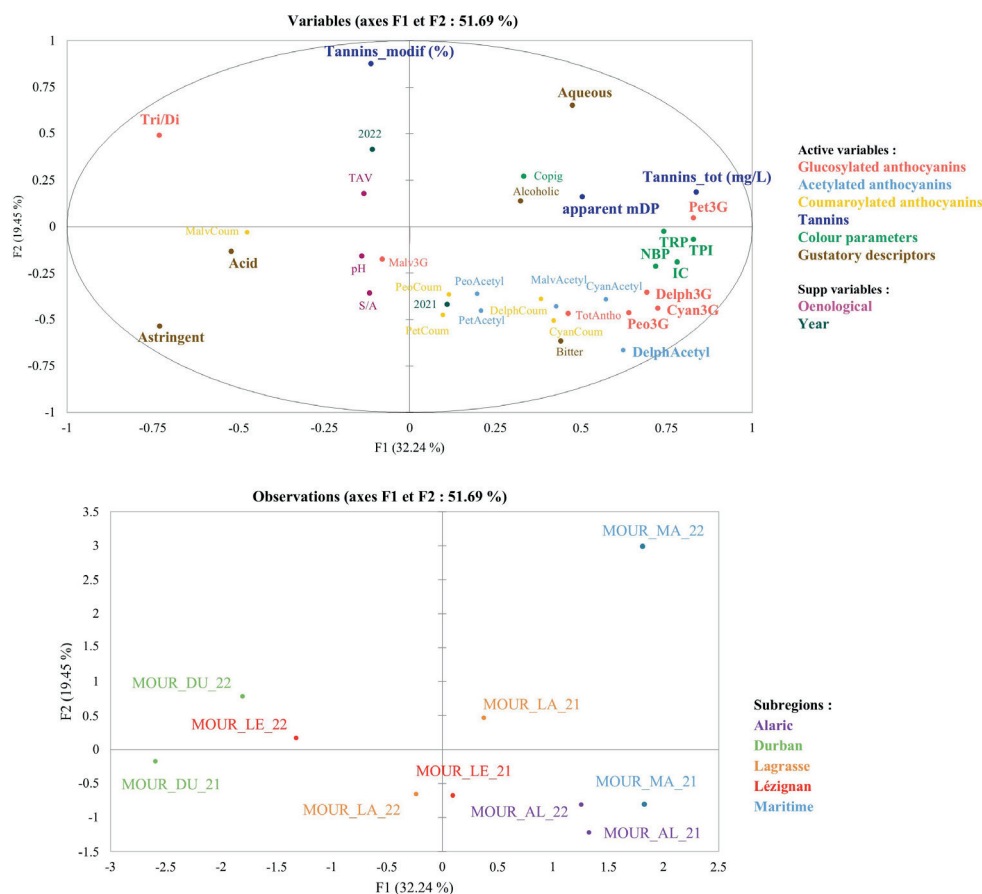


FIGURE 4. Multiple factor analysis (MFA) on polyphenols parameters and sensorial descriptors for Mourvèdre variety wines. Oenological variables, climate indexes, and year are reported as supplementary parameters. a) Graph of variables on F1 and F2 axes. Groups of variables are reported with code colours (see legend). Variables reported in bold were those that contributed to the construction of the axis. b) Graph of individuals. Distribution of wine samples in F1 and F2 axes. The five subregions were reported with a colour code.

in 2021); meanwhile, they contained significantly lower concentrations of total anthocyanidin-3-*O*-glucosides, especially delphinidin-3-*O*-glucoside and malvidin-3-*O*-glucoside (data not shown).

However, the grouping of the plots was similar in both vintages: the LA and DU samples formed a group that was clearly separated from the LE samples, while AL and MA were closer to either LA and DU (2021) or LE (2022) (Figure 3). These trends can be mainly explained by the fact that the LA and DU samples showed an increase in CI, TPI, NBP, Copig, and TRP, while those of MA, AL, and LE either remained stable (TPI, NBP, and Copig) or decreased (CI and TRP) between the two vintages (Table 3). As a general characterisation, in both vintages, the DU wines had, along with LE in 2021 and MA in 2022, significantly higher concentrations of anthocyanidin-3-*O*-glucosides and acetylated and coumaroylated anthocyanins than AL and LA wines. Concerning the Tri/Di ratio, in both vintages, the Carignan wines from the MA and DU vineyards showed significantly higher mean values (18 in 2021, 21–23 in 2022) than the wines from AL (12 in 2021, 11 in 2022). Finally, DU, LA, and AL showed a significantly higher percentage of modifications to tannins (48–52 %) than MA and LE (29–44 %) (Table 3).

Regarding this last group of samples, it is interesting to note that the Carignan varieties from MA and LE were characterised by similar average ages (7 and 15 years) compared to the ages of the other three vineyards (38, 53, and 71 years). Previous studies have evaluated the influence of vine age on the quality of final wines, highlighting either a higher polyphenol concentration (anthocyanins) in berries from younger vines than those from older ones or no differences due to age (Grigg, 2017; Heymann & Noble, 1987; Reynolds *et al.*, 2008; Sanmartin *et al.*, 2017). However, in a study by Riffle *et al.* (2022), wines from younger vines were found to have in general less tannins than those from older vines, possibly linked to seed number and weight per berry. Our results were in contradiction to this finding: in both vintages, LE wines contained a higher concentration of tannins, while wines from MA had either the lowest (in 2021) or the highest (2022) concentration. For both samples, these trends could be also due to differences in the S/A ratios between vintages (especially for MA sample), with the 2022 vintage showing a higher S/A ratio, thus a possible more advanced phenolic maturity, explaining the higher concentration of tannins. However, in both vintages, MA and LE showed lower percentages of modifications to tannins than the LA, DU, and AL samples,

which is linked either to the place of origin or to the nature of the constitutive flavan-3-ol monomers or/and the polydispersity of tannins (*i.e.*, the composition of the tannin fraction of the wine in terms of chemical structure and DP of tannins). Regarding Carignan from MA, it is worth noting that this vineyard particularly suffered from water stress in the 2021 vintage and that, as a result, the higher water deficiency may have affected the final tannin concentration, as well as the final anthocyanin concentration.

3.4. Mourvèdre expression

As regards the Mourvèdre global MFA (Figure 4), the F1 and F2 axes accounted for 51.69 % of the total variance. The MF graph shows that the vintage effect on the Mourvèdre variety was less pronounced than that on the other three varieties (Figure 4b). Different colour parameter trends were highlighted: while the average TPI, Copig, and TRP values showed a significant increase in 2022 compared to 2021, NBP showed a significant decrease and CI did not show any significant differences. Concerning anthocyanins, the differences in total anthocyanidin-3-*O*-glucosides between the vintages were significantly different but less marked than for the other varieties (232 mg/L in 2022 *versus* 259.92 mg/L in 2021) (Table 4). Finally, on average, wines from 2022 showed significantly higher amounts of tannins, as well as a higher percentage of modifications to these compounds, than the 2021 wines: nonetheless, the apparent aDP was significantly lower in the 2022 wines.

The graph shows that the grouping of the samples is based on plot rather than vintage, the wines from AL and MA being located in the lower right-hand side of the graph (except for MA_22, which is located on the upper right-hand side), those from LA and LE in the centre of the graph and those from DU on the left-hand side.

As a general characterisation, in both vintages, the wines from AL and MA plots showed a significantly higher concentration of tannins (673–887 mg/L) and apparent aDP (6.2–7.1) than the DU, LA, and LE wines (380–653 mg/L and 5.3–6.2, respectively, considering both vintages) (Table 4). This trend was accompanied by significantly higher values of CI (13–21 in 2021, 16–19 in 2022), TPI (54 in 2021, 59–63 in 2022), and NBP (2.4–4.9 in 2021, 2.6–2.9 in 2022), for AL and MA, while LA and DU wines showed general lower values (7–10, 34–43, and 1.2–1.3, respectively, considering both vintages). Regarding the percentage of modification of tannins, DU and MA had significantly higher values (41–53 %) than LE, LA, and AL wines (38 %, 37 %, and 33 %, respectively) in both vintages (Table 4). Nonetheless, in the 2022 vintage, despite differing in terms of percentages of modification, AL and MA were perceived as being more astringent than those from LE, LA, and DU. The total amount of anthocyanins in the latter three samples remained stable in both vintages; nonetheless, the lower total concentration of tannins may explain the lower TPI values between the two vintages.

4. Sensorial discrimination of wines based on their astringency

To go further in the interpretation of the impact of the variations in polyphenolic compounds on the sensorial profile of the final wines, a more detailed characterisation of the relationship between the polyphenolic compounds in the samples and final astringency perception was carried out and is reported here.

A Cochran statistical test was carried out on the results obtained for the astringency descriptor, a parameter evaluated during the sensorial analysis (Table 5). We decided to focus on the astringency descriptor because of the influence that polyphenols – especially tannins but also anthocyanins – can have on its degree of perception (Ma *et al.*, 2016; McRae & Kennedy, 2011).

While significant differences between Syrah, Grenache, and Mourvèdre wines were found, Carignan wines could not be differentiated based on this descriptor, despite significant differences in total concentration of tannins as well as percentage of modifications being found.

As far as Carignan wines are concerned, the perception of astringency cannot be explained solely by tannin composition in terms of concentration and percentage of modification, and further study of the types of tannin modification markers could provide more insights. Furthermore, as the matrix effect also has an impact on astringency, the study of other compounds involved in this phenomenon, such as polysaccharides and certain volatile compounds, could be envisaged.

Concerning the Syrah variety, AL wines were perceived as more astringent than LA wines in both vintages, especially in 2022 ($p = 0.001$): AL Syrah wine showed significantly lower total tannin concentrations than LA (700 mg/L *versus* 907 mg/L, respectively); nonetheless, AL wines showed a significantly lower percentage of modification (47.67 % *versus* 58.08 %, respectively), which may explain the final astringent perception. Indeed, even when present at lower concentrations, a higher percentage of modification of tannin fractions may affect the type of reactions they establish with salivary proteins, and therefore the astringency perceived.

Concerning Grenache, significant differences in astringent perception were noted within wines only in 2022, with Grenache wines perceived as more astringent than wines from MA (Table 5). The lower astringency perceived in MA wines could be due to their higher percentage of modifications to tannins. While the DU and LA samples contained a lower amount of tannins (533 and 427 mg/L, respectively) than MA (660 mg/L), they also showed a lower percentage of modifications (24.49–32.85 %, against 44.14 %), which possibly promoted a stronger interaction with salivary proteins and thus inducing higher astringency (Ma *et al.*, 2016). This difference in tannic composition may be due to both the vineyards parameters which may have influenced tannin biosynthesis – and the harvest maturity

TABLE 5. Sensorial CATA analysis results for the astringency descriptor in 2021 and 2022 wines. Wines were evaluated and results reported as frequency of citation. Cochran’s Q test was performed. Concerning Cochran’s Q test results: * = p -value < 0.05; ** = p -value < 0.01; *** = p -value < 0.001. When significant differences were revealed (p < 0.05), we performed multiple pairwise comparisons based on the critical difference of Sheskin. n.a. = not available.

Vineyards	Astringency							
	Syrah		Grenache		Carignan		Mourvèdre	
	2021	2022	2021	2022	2021	2022	2021	2022
p -value	0.022 *	0.001 ***	0.442	0.002 **	0.287	0.353	0.001 ***	0.001 ***
Alaric	16 ^a	20 ^a	12	14 ^{ab}	11	13	15 ^{ab}	20 ^a
Durban	12 ^{ab}	14 ^{ab}	15	18 ^a	9	9	7 ^c	12 ^{bc}
Lagrasse	9 ^b	8 ^b	n.a.	17 ^a	10	11	10 ^{abc}	10 ^c
Lézignan	11 ^{ab}	11 ^b	13	15 ^{ab}	13	12	16 ^a	16 ^{abc}
Maritime	15 ^{ab}	15 ^{ab}	11	8 ^b	7	9	8 ^{bc}	19 ^{ab}

degree, thus modulating their extractability, as well as to ECG content and that of other compounds in the wine matrix (e.g., polysaccharides) (Bindon *et al.*, 2010). We calculated the percentage of ECG in these samples and observed that % ECG was slightly higher in GRE_DU than in the Grenache from MA (12 % versus 7 % ECG, respectively). Previous studies have pointed out a positive relationship between the intensity of astringency perception and the presence of galloylated subunits (% ECG) in the tannin structure due to the enhanced reaction with proteins (Rinaldi *et al.*, 2014; Vidal *et al.*, 2003). Concerning vineyard parameters, the DU and LA samples had been collected from vineyards with similar characteristics, which can mainly be attributed to their higher elevation (205 and 167 m, respectively) and to their having the same exposure (NE–SW). In addition, in the 2022 vintage, these two plots were harvested at similar dates and later than the MA plot (two weeks later). This difference in maturity can partly explain the difference in astringency.

Concerning the Mourvèdre variety, different trends in terms of astringency were highlighted depending on the vintage, with the LE wines in 2021 and AL in 2022 being those perceived as the most astringent. Interestingly, the DU wines were perceived as the least astringent in both vintages, along with MA in 2021 and LA in 2022 (Table 5). It may be possible to explain the higher astringency perceived in AL in 2022 by the significantly lower percentage of tannin modifications (33.65 %) (Table 4). Interestingly, in this vintage, the MA wine was perceived as being as astringent as the AL sample, in addition to both wines having similar % ECGs (18 % MA and 16 % AL, respectively); however, MA had the highest percentage of tannin modifications (54 % compared to 34–46 %). Nonetheless, the calculation of % ECG revealed that the MA and AL wine differed in terms of % extension units: MA had 9.40 % ECG extension units and AL 0.961 %. Previous studies have observed that astringency sensory evaluations can be positively affected by the proportion of ECG as extension units (Quijada-Morín *et al.*, 2012). In addition, these differences

may partly be due to a difference in phenolic composition of the wines between the two vintages. The wines from this plot underwent a strong increase in TPI value and in the colour fraction associated with non-bleachable pigments (NBP) between 2021 and 2022. The latter parameter comprises the coloured pigments which are no longer reactive, because they are linked to other compounds; therefore, this may be a better explanation for the highest percentage of modifications to tannins for MA in this vintage.

5. Impact of blending on the astringent perception

A sensory analysis study of the obtained blends was carried out on the single-varietal wines, with the aim of studying the impact of blending on astringency and of assessing whether the differentiations seen between the samples for this descriptor could be maintained once blended. The single-varietal wines showed differences in astringency depending on the plot, but what effect would blending have on the differences perceived?

The results of the CATA sensorial study showed a significant vintage effect: while the blended wines from the 2021 vintage showed no significant differences, the AL blended wine showed a significantly higher astringency than the DU blended wine in 2022 (Figure 5). For single varietal wines, the astringency of the AL wines was significantly higher for Syrah and Mourvèdre in both vintages, whereas it was significantly lower for DU for the Mourvèdre variety. Since Grenache from the DU subregion was perceived as being more astringent in both vintages, this result shows that Grenache seems to have less impact on astringency than Syrah. Therefore, depending on the vintage, astringency could vary with plot and blending may allow the final profile of wines to be modulated. Other experiments carried out by our research unit on many wines from the five subregions over two vintages, did not find any significant differences between the wines produced from these areas in terms of astringency (data not shown). This is very probably due to the differences in blending, which smooths out variable astringency depending on zone and grape variety.

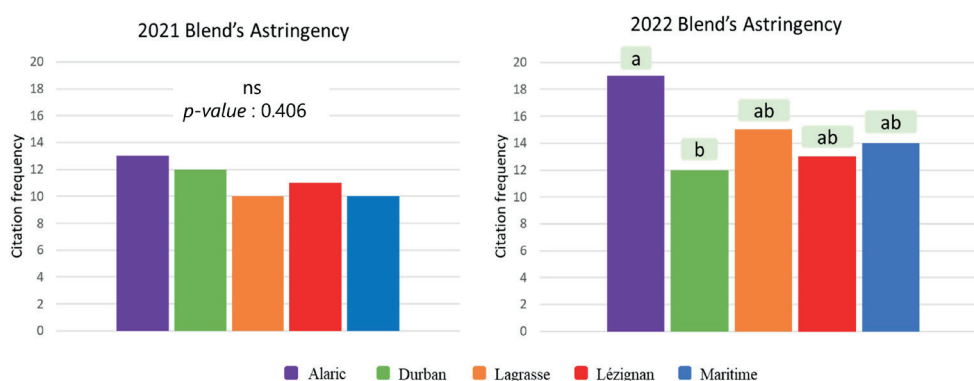


FIGURE 5. Barplots of sensorial CATA analysis results for the astringency descriptor in 2021 and 2022 common blended wines. Wines were evaluated and results reported as frequency of citation. Cochran's Q test was performed. When significant differences were revealed ($p < 0.05$), we performed multiple pairwise comparisons based on the critical difference of Sheskin.

CONCLUSION

The present study of polyphenolic and sensorial profiles of monovarietal wines from selected plots in representative areas of the five subregions of the Corbières appellation enabled us to further our understanding of the expression of these terroirs.

The impact of vintage on polyphenolic profiles was significant due to variations in rainfall and temperatures between the two vintages studied. Despite the vintage effects, major trends emerged for each variety across the regions. Indeed, both vintages of the wines produced from vineyards in the AL subregion showed lower anthocyanins and variable tannins, with higher astringency observed in the Syrah and Mourvèdre wines. The wines from the DU vineyards generally showed higher tannin modification, with Grenache and Mourvèdre being the most and least astringent. Meanwhile, the Syrah and Grenache wines from the LA vineyards contained higher anthocyanins and tannins, resulting in higher TPI and CI, with the Grenache wine being perceived as more astringent and Syrah as less astringent. Regarding the wines produced from the LE vineyards, Grenache and Carignan both contained a high content of anthocyanins and tannins, Syrah showed low astringency in 2022, and Mourvèdre was perceived as being highly astringent in 2021. Finally, the wines from the MA vineyards showed high tannin content with significant variations: the Grenache and Mourvèdre wines contained lower concentrations of anthocyanin, and astringency varied depending on variety and vintage, with Grenache and Mourvèdre being perceived as less astringent in 2022 and 2021, respectively.

These findings enable polyphenolic discrimination of each subregion, offering valuable insights into terroir factors. However, given the influence of the two distinct vintages, understanding the impact of soil and vineyard characteristics *versus* climate remains challenging. Despite its limitations, this study provides a preliminary understanding of subregional polyphenolic markers and astringent perceptions, suggesting terroir contributions. Future studies should analyse aroma compounds for a more comprehensive understanding of terroir impact on wine quality and finer discrimination between Corbières zones.

ABBREVIATIONS

- AL – Alaric
- DU – Durban
- LA – Lagrasse
- LE – Lézignan
- MA – Maritime
- TPI – Total polyphenols index
- CI – Colour intensity
- NBP – Non-bleachable pigments
- BA – Bisulfite adducts
- TRP – Total red pigments
- Copig – Colour due to copigmentation
- aDP – Average degree of polymerisation
- DAP – Diammonium phosphate
- MFA – Multiple factor analysis
- Tri/Di – Trihydroxylated/dihydroxylated anthocyanins ratio
- CATA – Check-all-that-apply

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Credit authorship contribution statement

Alice Argentero: investigation, conceptualisation, methodology, validation, formal analysis, writing – original draft. Ella Tyson: investigation, validation. Lucas Suc: investigation, validation. Frédéric Véran: methodology, validation. Mathilde Bauducel: investigation. Alain Samson: conceptualisation, validation, investigation. Soline Caillé: conceptualisation, formal analysis, validation, investigation, writing – review and editing. Peggy Rigou: conceptualisation, writing – review and editing. Laetitia Mouls: conceptualisation, methodology, validation, formal analysis, writing – original draft.

Declaration of competing interest

The authors declare that there are no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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