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ORIGINAL RESEARCH ARTICLE

Exploring Corbières red wine typicity: evaluation of the impact of winemaking processes on the chemical composition and organoleptic properties of wines from different terroirs

Alice Argentero[1], Soline Caillé[1], Floriano Joaquim Engenheiro[1], Bénédicte Fontez[2], Peggy Rigou[1], Laetitia Mouls[1]*

[1]SPO, Univ Montpellier, INRAE, Institut Agro, 34000 Montpellier, France

[2]MISTEA, Univ Montpellier, INRAE, Institut Agro, 34000 Montpellier, France

ABSTRACT

Aromatic and polyphenolic compounds play an important role in the quality of red wines, contributing not only to flavour, but also to colour, astringency and bitterness. To explore the influence of climate and oenological practices on the chemical and sensory profile of wines, red blends from five terroirs of the Corbières appellation were analysed, taking into account two production years (2018 and 2019). The wines were a blend of at least two of four main grape varieties: Syrah, Grenache, Carignan and Mourvèdre. Volatile compounds were evaluated by HS-SPME-GC-MS, while wine pigments and derived pigments were evaluated by spectrophotometric measurements. In addition, the wines were compared using a quantitative descriptive analysis sensory profile method. The percentage of the blend significantly influenced the polyphenolic and aromatic characteristics of the wines, showing distinct 'typicity' characteristics between sub-regions. Maceration techniques, on the other hand, did not did not allow us to discriminate between sub-regions, but enabled a better understanding of chemical and sensory differences between samples.

KEYWORDS: aroma compounds, polyphenols, sensorial analysis, multifactorial analysis, terroir, wine, winemaking

*correspondence: laetitia.mouls@institut-agro.fr

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INTRODUCTION

1. Impact of terroir on wine quality: focus on pedo-climatic conditions

The quality of a wine from a specific viticultural region is the result of the "terroir effect", a complex set of interactions involving pedo-climatic conditions (topography, climate and soil), viticultural and oenological practices and the final quality of the grape (Vaudour, 2002). Indeed, climate and soil conditions influence the development of the vine and the ripening of the grapes, affecting the accumulation of primary (sugars, amino acids, polysaccharides, etc.) and secondary metabolites (aromatic, phenolic compounds, etc.). Polyphenols can have an important influence on red wine quality, contributing to colour, astringency and, to a lesser extent, bitterness (De Freitas and Mateus, 2011; Escribano-Bailon and Santos-Buelga, 2012; Noble, 1994). Aromatic compounds include primary (grape ripening), secondary (fermentation) and tertiary (ageing) aromas, which have a major impact on the odour and flavour of wines (Ferreira, 2010). Temperature, water and radiation are among the most important abiotic factors to interact with vine and fruit development. Several studies have found a lower biosynthesis of anthocyanins and stilbenes with temperatures above 35 °C during ripening, highlighting a genotype-dependent response (Fernandes De Oliveira *et al.,* 2015; Pastore *et al.,* 2017; Yan *et al.,* 2020). However, the effect on the aromatic profile seems difficult to establish, as it is difficult to separate temperature and radiation effects (Kwasniewski *et al.,* 2010). Interestingly, higher temperatures increase the perception of overripe fruity notes due to higher concentrations of ketones, furanones and lactone family compounds (Pons *et al.,* 2017). With regard to water deficit, while a moderate stress situation can have a positive effect on the quantity of aromas and polyphenols, a high stress situation will lead to a considerable reduction in these compounds. In fact, a strong water deficit will also lead to a lower sugar concentration due to a reduced carbon assimilation (Rienth *et al.,* 2021).

2. Impact of terroir on wine quality: a focus on winemaking practices

In light of the above, the role of the producer is important, since he must not only manage the operations in the vineyards to better adapt to the specific pedo-climatic conditions, but also choose the most suitable winemaking method to produce a quality wine, while respecting the limits imposed by the appellation (Caenegem, 2005). The winemaking process affects the diffusion of volatile and phenolic compounds from the grape skins into the must (Sacchi *et al.,* 2005). Blending is a very common technique, generally used to increase complexity (Hopfer *et al.,* 2012). In particular, studies have highlighted an increase in colour values, especially the co-pigmentation phenomenon, resulting in higher colour stability (Hopfer *et al.,* 2012; Monagas *et al.,* 2006). Interestingly, volatile compounds were affected differently in the same studies, with their final levels in blended wines depending more on the varietal-based blend than on the technique itself (Hopfer *et al.,* 2012).

3. Context presentation and aim of the study

The Corbières appellation is one of the most renowned territories in the South of France for red wine production. The red wines produced in this region are made from a blend of at least two of the four main grape varieties: Syrah, Grenache, Mourvèdre and Carignan. The climatic conditions and vine phenology within the Corbières AOC vary due to the heterogeneous geography of the region. At the time of writing, the appellation is undergoing a process of subdivision into five sub-regions, with the aim of defining the areas with homogeneous geographical conditions that are characterised by more precise specifications, in order to produce wines that can be perceived by consumers as having a strong typicity. In the present study, wines were selected and analysed over two vintages in order to determine the impact of climate and oenological practices on their chemical and sensory profile. By evaluating the oenological processes used through the studied sub-regions, the aim of this study was to integrate human knowledge into the characterisation of a terroir, and to evaluate both its role in the expression of certain chemical molecules or sensory descriptors in wines and its capacity to convey typicity features on a sub-regional scale. A better understanding of these relationships will provide the AOC with some insight into the role of its practices on wine quality.

MATERIALS AND METHODS

1. Climate data

SAFRAN climatic data for 2018 and 2019 were obtained through the SICLIMA platform developed by AgroClim-INRAE (Maury *et al.,* 2021). Data for each region were obtained from the weather station in the main villages. Rainfall data for April to September, and temperature (minimum, average and maximum) data for June to September are reported in Table S1.

2. Wine Samples

Wine samples were provided by 40 producers across the five sub-regions (Alaric, AL; Lagrasse, LA; Durban, DU; Lézignan, LE; Maritime, MA) within the AOC Corbières appellation. Two vintages were chosen for each *cuvée*. In total, 53 AOC Corbières red wines from the 2018 vintage and 39 from the 2019 vintage were studied. To ensure a good representation of all sub-regions under study, the selection process was based on the sample fulfilling several key criteria: a blend that included at least two of the four main varieties (Syrah, Grenache, Carignan, and Mourvèdre) and the absence of oak treatment (Table 1).

TABLE 1. Number of bottles analysed for each sub-region in 2018 and 2019 vintages. AL = Alaric, DU = Durban, LA = Lagrasse, LE = Lézignan, MA = Maritime.

Year	N° bottles					
	AL	DU	LA	LE	MA	Total
2018	10	11	8	10	14	53
2019	8	7	8	8	7	38

3. Winemaking parameters

Surveys were done to gather information on the different operations carried out during winemaking, from harvest to bottling. A selection of parameters was determined from the collected data, taking into account their statistical feasibility (ensuring sufficient repetitions for each variable across all samples) and impact on the ultimate quality of the wines. This study considered five different parameters which all play a crucial role in the alcoholic fermentation (AF) process: winemaking method employed (AF_type), percentage representation of each variety in the blend (grape variety), the specific type of yeast used for fermentation (yeast_type), cap management operations that influence extraction of colouring matter from grape skins or oxygen supply (AF_management), and duration from the end of malolactic fermentation to the end of the fermentation process (ageing). AF_type and grape_variety were treated as quantitative data, whereas yeast_type, AF_management, and ageing were treated as qualitative data (Table 2).

4. Oenological Parameters

Classical oenological parameters were measured by the "Natoli & Associés" laboratory, located in Montpellier, France, following International Organisation of Vine and Wine (OIV) reference methods.

5. 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, and adapting the wine dilution in order to obtain values of between 0.01 and 1. Absorbance measurements were performed on each sample with values of between 230 nm and 800 nm. Colour Intensity (CI) and Color hue were measured in pure wine, via a 0.1 cm path length cell and extraction of absorbance values at 420 nm (A420), 520 nm (A520) and 620 nm (A620) [CI = (A420 + A520 + A620) × 10, Colour hue = A420/A520]. Bisulfite Adducts (BA) were measured through a 0.1 cm path length cell, 30 min after addition of 50 µL acetaldehyde aqueous solution to 5 mL of samples [BA = (A420 + A520 + A620) × 10]. Acetaldehyde was added to trap sulfites and convert bisulfite adducts to flavylium ions. Samples pre-treated with acetaldehyde solution were diluted with a hydroalcoholic solution [14 % (v/v), 3 g potassium hydrogen tartrate, pH 3.4], and the part of the colour due to Co-pigmentation phenomenon (Copig) was measured 30 min later through a 1 cm path length cell [Copig = A520 (CI) × DF - A520 × 10]. The sulfite Non-Bleaching fraction of Pigments (NBP) was measured through a 0.1 cm path length cell, 30 min after the addition of 75 µL of sodium hydrogen sulfite solution (200 g/L) to 5 mL of wine (NBP = A520 × 10,). Finally, Total Polyphenol Index (TPI) at 280 nm, Total Red Pigments (TRP) at 520 nm were measured through 1 cm path length cell, 30 min after dilution of wines in 1 M HCl solution [TPI = A280 × DF (dilution factor); TRP = A520 × DF].

6. Volatile aroma compounds analysis by HS-SPME-GC-MS

6.1. Chemicals

Sodium chloride (CAS: 7647-14-5, ≥ 99.5 %), 1-octanol (CAS: 111-87-5, ≥ 99 %), and alkane solution C8:C20 were purchased from Sigma Aldrich, France. Ultra-Pure water was produced and dispensed in the lab by Milli-Q® IQ 7 003/05/10/15, Merck, France.

6.2. Solid-phase microextraction (SPME)

The SPME analysis method used was taken from Yang *et al.* (2019) with some modifications. Each sample contained 1 mL of wine diluted in 9 mL of Ultra-Pure water, 1 g of NaCl and 10 µL of a 0.04 g/L 1-octanol, which was used as internal standard solution. Wine dilution with water aimed at reducing the ethanol effect on volatility and adsorption of aroma compounds on the fibre, and thus improving method sensitivity (Davis and Qian, 2019). Each vial was tightly capped with a PTFE-silicon septum. Sample preparation and analysis was performed in triplicate. SPME was performed using a Triplus autosampler (Thermo Fisher Scientific, USA). A 2 cm-long fibre made of divinylbenzene/Carboxen/polydimethylsiloxane (DVB/CAR/PDMS), 50/30 µm (Supelco., USA) was used for volatile compounds adsorption. The bipolar and triphasic DVB/CAR/PDMS fibre was selected because of its capacity to extract both polar and non-polar analytes. DVB retains the large and less volatile molecules, while the carbon layer captures small volatiles. This fiber was reported to be the best suited to analysis applications, as it extracts the most volatile compounds with best sensitivity (Zianni *et al.,* 2023). Sample temperature equilibration was performed at 40 °C for 15 min, with agitation at 250 rpm. Volatile extraction was performed under the same conditions but for 30 min. Compounds were desorbed by inserting the fibre directly into the GC injection port, in a splitless mode and at 250 °C for 3 min. The SPME fibre was reconditioned in a fibre conditioner at 270 °C for 30 min after each sample analysis.

6.3. Gas Chromatography-mass spectrometry (GC-MS) analyses

GC-MS analyses were performed using a GC Trace Ultra GC (Thermo Fisher, USA) coupled to an ISQ Series MS (Thermo Fisher, USA) equipped with a DBWAX capillary column (30 m, 0.25 mm i.d., 0.25 µm film thickness, Agilent Technologies, USA). Helium was used as carrier gas, at a constant flow rate of 1.2 mL/min. GC oven temperature was set at 40 °C for 3 min, then increased to 210 °C at 3 °C/min, and to 245 °C at 5 °C/min, and maintained at this temperature for 5 min. The temperatures of both transfer line and ion source were set at 250 °C. MS was performed in the Electron Impact mode with an ionisation voltage of 70 eV. Spectrum data were acquired in the full scan mode (47 - 400 *m/z*). A mixture of aliphatic hydrocarbons (C8:C20) was loaded onto the SPME fibre and injected following the same oven temperature program to calculate the Kovats' Retention Index (RI) of each identified compound. Volatile compounds were identified by matching the mass spectra

and retention indices to NIST 2.0 and Wiley libraries and further confirmed using the RI. Spectrums were interpreted with Xcalibur V. 3.0 MS software (Thermo Fisher Scientific, USA). The relative amount of each compound was obtained by dividing its GC peak area by that of the internal standard (1-octanol). Results were expressed as per µg/L equivalents of internal standard added to the wine. Through the NMF statistical analysis, volatile compounds were divided into six groups. For each wine sample, the total relative amount of each group described above was calculated by totalling the amounts in µg/L of equivalent 1-octanol of each aroma compound in the group. The total volatile compounds in each of the six groups is reported in Table 2.

7. Sensory Analysis

7.1. Panel training and selection

Two panels of expert judges (unrelated to the wine industry) were selected based on their sensory performances: twelve judges (three men and nine women, average age of 55 years) in the first year and twenty judges (seven men and thirteen women, average age of 51 years) in the second. The judges were further trained to perform wine descriptive analyses (Norm *ISO 8586 1994*).

7.2. Quantitative Descriptive Analysis (QDA)

The wines were subjected to QDA to assess their similarities and differences, following the guidelines of ISO 13299 (2003). The expert panel agreed on 16 attributes to characterise the wine samples, including eleven olfactory descriptors and five gustatory descriptors (Table 2). To ensure panel uniformity, judges underwent training to understand and consistently use these attributes and familiarise themselves with the product space. Olfactory and gustatory reference standards (Table S2) were introduced to aid judges in identifying and memorising sensory attributes. The wines were subjected to QDA to assess their similarities and differences, following the guidelines of ISO 13299 (2003). The evaluations were conducted in individual testing booths. Each sample was served at room temperature (21.7 ± 0.6 °C) in black glasses to eliminate any potential visual bias. The glasses were covered with plastic caps and labelled with random three-digit codes. The wines were presented using a monadic service approach, following William's Latin square design, to ensure a balanced presentation order and prevent carry-over effects, as recommended by Macfie *et al.* (1989). For data reliability, the wines were analysed twice, with assessors rating each attribute on unstructured linear scales that ranged from "low" on the left to "high" on the right. Data acquisition was facilitated using FIZZ software (v.2.518; Biosystème, France).

8. Statistical Analysis

Statistical data analyses were performed using RStudio (version 2023.06.2) and XLSTAT software (version 2022, Addinsoft Paris).

8.1. Winemaking parameters

A Khi 2 test was carried out on the qualitative winemaking parameters, with a significant level set at 5 %. An ANOVA (zone factor) analysis was carried out on quantitative winemaking parameters, with a significant level set at 10 %. Mean values for the five sub-regions were then compared using the Tukey multiple comparison test. Given that appellation specifications are not very strict, we decided to establish a p-value of 0.1 to be able to take into account the variability within a zone. A Multiple Factor Analysis (MFA) was carried out on the winemaking parameters, considering both qualitative data (quali-vinif: Yeast type, length of ageing and fermentation management) and quantitative data (winemaking method, AF_type, and percentage of blending, grape variety). The sub-regions and the vintages were added as supplementary variables (Figure 1).

8.2. Polyphenol parameters

For polyphenol variables, an ANOVA (zone factor) analysis was carried out with a significance level set at 5 %. Mean values of the sub-regions were compared using the Tukey multiple comparison test. A Hierarchical Cluster Analysis (HCA) heatmap was employed on the mean values for each of the five sub-regions. The selected parameters for the Heatmap plot were Ward for the clustering method and Euclidean distance for the distance measure. Different colours and gradients were associated with the scale. The lower the value the lighter the colour, and the higher the value the darker the colour.

8.3. Volatile compounds

Given the complexity of studying the influence of the winemaking parameters on each volatile compound separately, we formed groups of aromas showing similar variations according to the parameters considered and having the same metabolic origin in grapes or wine. The Non-negative Matrix Factorisation (NMF) method was used to statistically identify the proximity between aroma behaviours. It should be noted that this method was only applied to aroma compounds that were common to both vintages. Clusters of variables were then rearranged by applying metabolic pathway knowledge. Six groups of aroma compounds sharing the same origin (varietal or fermentative) and metabolic pathway were thus determined: Fatty Acid Degradation (F.A.Deg.), Amino Acid Degradation (A.A.Deg.), Primary Aromas 1 (P.Aromas 1), Primary Aromas 2 (P.Aromas 2). Two groups identified by the NMF approach whose variables were not present in the previous six groups were added, even if the biological association of these groups of variables is not obvious: Statistical Group 1 (S.G1) and Statistical Group 2 (S.G2). These two groups included a mix of compounds sharing the same metabolic pathway (e.g., Ethyl heptanoate and heptanol) or statistical proximity in both vintages. Significant differences among the five sub-regions in the six volatile compound groups were then tested through an ANOVA (zone factor) analysis, with a significance level set at 5 %. Mean values of the sub-regions were compared through the Tukey multiple comparison test. An HCA heatmap was then performed for polyphenol parameters.

TABLE 2. Totality of groups considered, as well as the parameters included in each group and the variables determined for each parameter. For each parameter and variable, their statistical "name" is reported, so to allow a better understanding of statistical analysis adopted.

Group	Parameters	Statistical parameters	Variables	Statistical variables
Winemaking	Winemaking method	AF_type	Traditional	TRAD
			Carbonic Maceration	CM
			Cold Pre-fermentative Maceration	CPM
			Hot Pre-fermentative Maceration	HPM
	% of each variety in blending	Grape variety	Syrah	Syrah
			Grenache	Grenache
			Carignan	Carignan
			Mourvèdre	Mourvèdre
			Cinsault	Cinsault
	Type of yeasts used for fermentation	quali-vinif	Active Dry Yeasts	Active_dry_yeasts
			Indigenous Yeasts	Indigenous yeasts
	Operations carried out on the cap during maceration-alcoholic fermentation		*Remontage* as fermentation cap management practice	AF_Manag_R
			Pigeage as fermentation cap management practice	AF_Manag_P
			Délestage as fermentation cap management practice	AF_Manag_D
			Remontage-Délestage as fermentation cap management practice	AF_Manag_RD
			Remontage-Pigeage as fermentation cap management practice	AF_Manag_RP
			Pigeage-Délestage as fermentation cap management practice	AF_Manag_PD
			Remontage-Pigeage-Delestage as fermentation cap management practice	AF_Manag_RPD
	Length of aging (from the end of MF to bottling)		Short period of aging (< 6 months)	Aging_Short
			Medium period of aging (6 - 12 months)	Aging_Medium
			Long period of aging (> 12 months)	Aging_Long
Volatile compounds	Groups of volatile compounds determined through NMF analysis	Aroma	**Fatty Acid Degradation**: Ethyl 2-hexenoate, Ethyl Hexanoate, Hexyl Acetate, Ethyl Octanoate, Ethyl Decanoate, Decanoic Acid, Hexanoic Acid, Octanoic Acid, Ethyl 9-Decenoate, Ethyl Butanoate, 4-Methyl-1-pentanol, 3-Methyl-1-pentanol, 2-ethylhexan-1-ol	F.A.Deg.
			Amino Acid Degradation: Ethyl acetate, Ethyl 2-methylbutanoate, 2-Phenylethanol, Ethyl 2-hydroxy-4-methylpentanoate, Phenethyl acetate, Isoamyl acetate, 1-Pentanol Isomers, 2-methylpropan-1-ol, Nerolidol	A.A.Deg.
			P.Aromas 1: 1-Octen-3-ol, Estragole, Terpinen-4-ol, Fenchone	P.Aromas 1
			P.Aromas 2: Citronellol, β-Damascenone, Vitispirane, TDN, Alpha-terpineol, Benzyl alcohol, Linalool, DMS	P.Aromas 2
			S.G1: 4-Ethylphenol, Ethyl Heptanoate, Heptanol	S.G1
			S.G2: 1-hexanol, Ethyl Isovalerate, Ethyl Lactate, Diethyl Succinate, Butyrolactone, Methionol, 3-Nonanone	S.G2
Polyphenols	Colour parameters and Total Polyphenols Index	Polyphenols	Total Polyphenols Index	TPI
			Colour Intesntiy	CI
			Copigmentation	Copig
			Color Hue	Color Hue
			Non-Bleachable Pigments	NBP
			Bisulfite Adducts	BA
			Total Red Pigments	TRP
Sensorial analysis	Sensorial descriptors	Senso olfactory	Amyl	Amyl
			Cooked vegetables	Cooked vegetables
			Jammy Red Fruit	Jammy Red Fruit
			Empyreumatic	Empyreumatic
			Pastry	Pastry
			Spicy	Spicy
			Woody	Woody
			Lactic	Lactic
			Humus	Humus
			Laether	Laether
			Alcohol	Alcohol
		Senso gustatory	Acidity	Acidity
			Bitterness	Bitterness
			Fat	Fat
			Astringent	Astringent

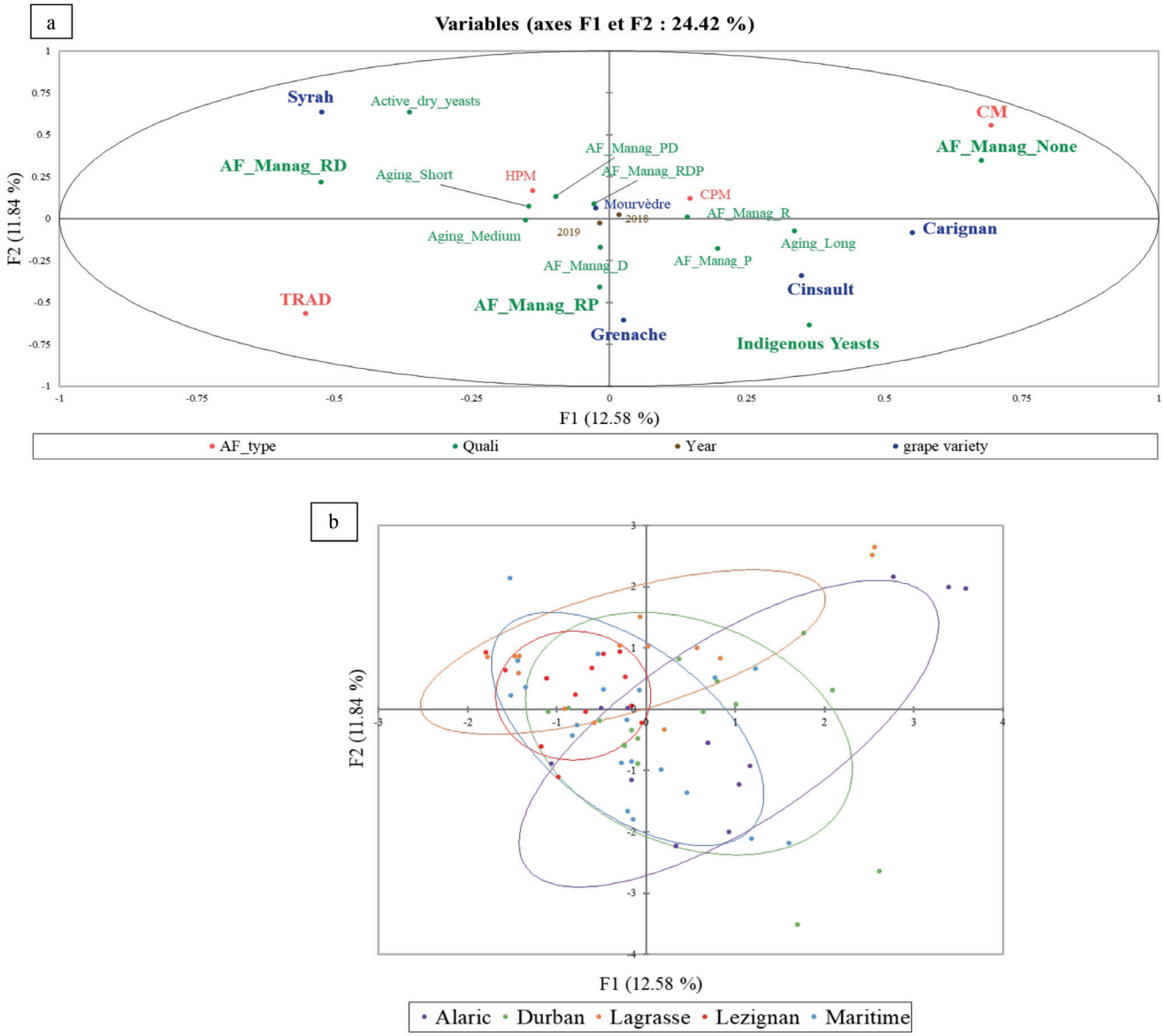


FIGURE 1. Multiple Factor Analysis (MFA) of winemaking qualitative and quantitative parameters.

a) Graph of variables. Parameters are reported with colour codes. Green: Qualitative parameters, "Quali", including the type of yeast and the AF_Management practice. Blue: the percentage of blending, "grape variety". Red: the winemaking method, "AF_type". Brown: the year, "Year". Variables in bold are those that contribute to the construction of the axes. b) Graph of individuals. Distribution of wine samples in F1 and F2 axes, with addition of confidence ellipses. The five sub-regions are colour coded.

8.4. Sensorial descriptors

Sensory descriptor data were converted by the FIZZ software into marks from 0 to 10.

Panel performance (discrimination, repeatability and consensus) was first checked with a three-way ANOVA (three factors: wine, judge, repeat) and an analysis of their interactions. Then, a two-way ANOVA (two factors: zone, judge) was run on the totality of the descriptors. Mean values for the five sub-regions were then compared through the Tukey multiple comparison test, and the chemical analysis comprised an HCA analysis.

8.5. General analysis

A general MFA was run on the winemaking parameters (*quali_vinif, AF_type and grape variety*), chemical variables, such as polyphenolic parameters (*Polyphenols*) and volatile compounds groups (*Aroma*), and sensorial descriptors (*Senso*), adding the five sub-regions, the two vintages (*year*) and the oenological parameters (*oeno*) as supplementary variables (Figure 2). Pearson's correlation coefficients were calculated for all the groups of variables in order to evaluate significant correlations. Finally, a global HCA heatmap was employed on the mean values of the sub-regions for all the variables that were found to be significant in either both or one of the two vintages.

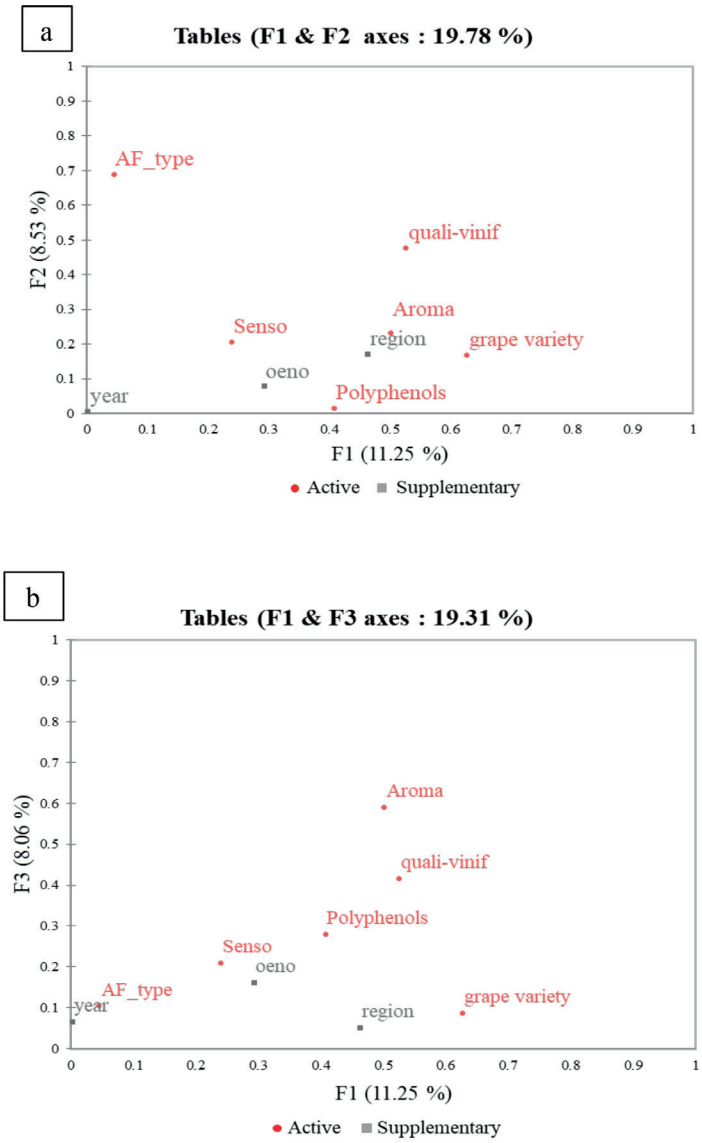


FIGURE 2. Multiple Factor Analysis (MFA) of winemaking parameters, polyphenols parameters, volatile compounds groups and sensorial descriptors. Oenological variables, regions and year are reported as supplementary parameters.

a) Representation of groups of variables on F1 and F2 axes and b) Representation of groups of variables on F1 and F3 axes.

RESULTS

1. Winemaking parameters

In the MFA plot (Figure 1), the first three axes (F1, F2 and F3) account for 33 % of the total variance. Interestingly, the variables for both the 2018 and 2019 vintages are situated in the centre of the graph, thus indicating that the year factor was not associated with the other factors. F1 (12.58 %) contrasted Carbonic Maceration (CM), no AF management practices (AF_Manag_none), and Carignan and Cinsault with Traditional (TRAD) and Remontage-Délestage fermentation management practice (AF_Manag_RD). On the positive side, F2 (11.84 %) was defined by active dry yeasts and Syrah, and on the negative side by Remontage-Pigeage AF management practice (AF_Mang_RP) and Indigenous yeasts (Figure 1a). F3 (9.45 %) further explained the correlations among variables by contrasting Mourvèdre, Cold Pre-fermentation Maceration (CPM) and Pigeage as AF management practice (AF_Manag_P) with Ageing_Short, Délestage as AF management practice (AF_Manag_D) and Remontage as AF management practice (AF_Manag_R) (Figure S1). Plotting the confidence ellipses of the sub-regions revealed no clear differences between them, but rather trends linked to some specific variables for each parameter. In the MFA plot, the samples are in a diagonal pattern, with one diagonal running from the lower right to the upper left quadrant and the other from the lower left to the upper right (Figure 1b). Wines from AL, LA and DU in the upper right part of the graph were either partially or fully produced through CM. Consequently, in accordance with the principles of CM, no operations were carried out on the cap during AF. However, the ANOVA carried out on these variables did not reveal any significant differences between the sub-regions. In particular, regarding the CM variable,

it can be seen that there were wines from LA and AL that were produced using 100 % CM. However, they represented a small percentage of the totality of the samples and were therefore an exception rather than a traditional feature of these sub-regions. Samples in the lower right part of the graph, mainly from DU, AL and MA, showed higher percentages of Carignan, Grenache and Cinsault in their blends, and in some cases underwent spontaneous AF. In particular, as shown by the Khi 2 test carried out on this parameter, in both vintages there was a significantly higher number of wines from AL made with indigenous yeasts ($p < 0.05$). However, one cuvée from DU also underwent spontaneous AF in both vintages, possibly explaining the barycentre position of this sub-region on the graph. Moreover, wines with a higher percentage of Grenache in this part of the graph were also correlated with the AF_Manag_RP practice. Conversely, on the other side of the graph, wines from LE, MA and LA had a higher percentage of Syrah and AF_Manag_RD. Concerning the AF_Manag_RD variable, the Khi 2 test showed a significant difference ($p < 0.05$) for LE in both vintages. Finally, the AF_Management parameter provides information on the different management practices used for the cap during fermentation, but only partially, because the frequency and duration of each practice are not specified. Concerning the percentage of each variety used in the blend, the ANOVA analysis pointed out significant differences ($p < 0.1$) among the sub-regions. In particular, for both vintages LA contained a higher percentage of Syrah (59 %) than DU and AL (28.4 and 25 %, respectively) (Figure 1a). By contrast, in 2018, AL wines contained a higher percentage of Carignan (37.5 %) in their blend than LA (10.7 %); meanwhile, in 2019, DU wines showed the highest average percentage of Cinsault (6 %) in their blend, this variety not being present in the blends of the wines from the other sub-regions.

The statistical analysis of the winemaking parameters allowed trends for each one of the sub-regions to be identified. All of these elements were then linked to the chemical and sensory composition of the produced wines in order to highlight any sub-regional specificities.

2. General MFA of winemaking parameters, chemical variables and sensorial descriptors

A MFA was done to understand whether there was a link between the winemaking parameters and the chemical and sensory parameters, and if so, whether these links could be attributed to specific areas (Figure 2). The first three axes provided 27.89 % of the total variance. The way in which the groups of variables are distributed shows that vintages (year) contributed the least to the three axes. Concerning winemaking, AF_type contributed to the F2 axis (2a, 38 %), and it was associated with quali-vinif. The grape variety contributed mainly to the F1 axis (26 %), being correlated to Aroma and Polyphenols. These latter contributed to both F1 (21 %) and F3 (2b, 35 %) axes, with Aroma being mainly associated with Senso and quali-vinif, and Polyphenols with grape variety. Finally, Senso did not contribute to the three axes, being mainly represented in the F5 axis. By adding the supplementary variables, the sub-regions can be seen to be distributed along the F1 axis, described by: Aroma, grape variety, Senso and Polyphenols; oeno being mainly related to Polyphenols (F1) and sensorial descriptors.

3. Polyphenols analyses of AOC Corbières wines

3.1. Classification of the sub-regions according to polyphenol parameters

An HCA heatmap was performed to understand how the five sub-regions were separated according to the polyphenol parameters (Figure 3). In 2018, two main clusters were observed: the first one including wines from LA and the second formed by the wines of the other sub-regions (Figure 3a). Moreover, the latter could be rearranged into two clusters: one containing MA and LE and the other AL and DU. By observing the variables dendrogram, two groups of variables can be identified: colour hue on the left, and the other parameters on the other right. It is conceivable that the separation of clusters in the sub-regions was driven by these two groups. In particular, LA was clearly separated from the other sub-regions by a lower colour hue value and higher NBP, TPI, TRP, BA, Copig and CI values. Furthermore, this trend could be highlighted also for the other sub-regions, with LE and MA's wines having generally higher values than wines from AL and DU. The ANOVA results confirmed this tendency for TPI, TRP and BA. In particular, LA showed significantly ($p < 0.05$) higher average values for TPI (58.65), TRP (16.12) and BA (12.38) than DU (44.68, 10.46 and 9.17, respectively) (Table S3).

In 2019, two main clusters were again highlighted. However, MA and LE were in the same cluster as LA, while AL and DU formed the second cluster (Figure 3b). The dendrogram of variables reveals a similar separation between the two vintages, TPI and the colour hue variable being together on the right side of the map. In this vintage year, the Copig and CI variables made the main contribution to cluster generation. By examining the map, it can be seen that LA, MA and LE had higher average values for these parameters (5.72, 4.99, 4.96 for Copig, 12.7, 11.44 and 11.30 for CI, respectively) than AL and DU (4.66 and 3.95 for Copig, 10.18 and 8.84 for CI, respectively). In 2019, the ANOVA analysis highlighted significant differences ($p < 0.05$) for different parameters compared to 2018: CI, NBA and Copig. In particular, LA showed significantly higher average values ($p < 0.05$) for CI (12.67) and Copig (5.7) than DU (8.84 and 3.95 respectively). Concerning NBP, significant differences ($p < 0.05$) were found between LA (3.5) and AL (2.5). Interestingly, AL showed different trends for the two vintages, especially in the TPI parameter: contrasting with the 2018 value (49.86), AL showed the highest average TPI value (53.19) in 2019 relative to wines from the other sub-regions (52.82 LA, 49.16 DU, 48.17 LE and 44.77 MA). However, this parameter was not significant among sub-regions in 2019, underlining the high standard deviations among the wines (Table S4).

In summary, AL and DU were clearly separated from LA in both vintages, while LE and MA were separated from LA in 2018 but in the same cluster as LA in 2019, probably due to their higher Copig and CI average values.

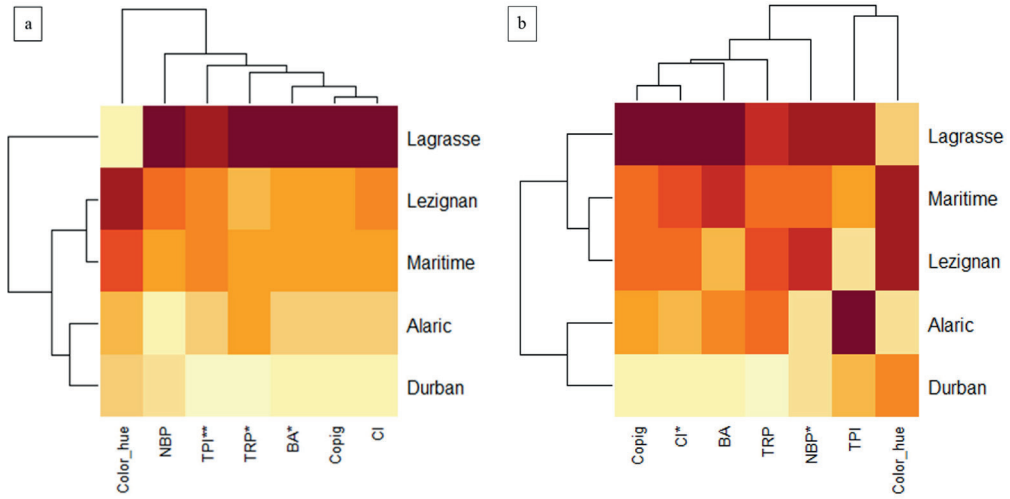


FIGURE 3. Hierarchical clustering heatmap performed on a) 2018 and b) 2019 polyphenol variables for the five AOC Corbières sub-regions (Ward algorithm and Euclidean distance analysis).

Variables that were significant among the five sub-regions from the ANOVA are reported with the asterisks: *** = $p < 0.001$, ** = $p < 0.01$, * = $p < 0.05$. The rows in the heatmap represent the sub-regions and the columns indicate the variables.

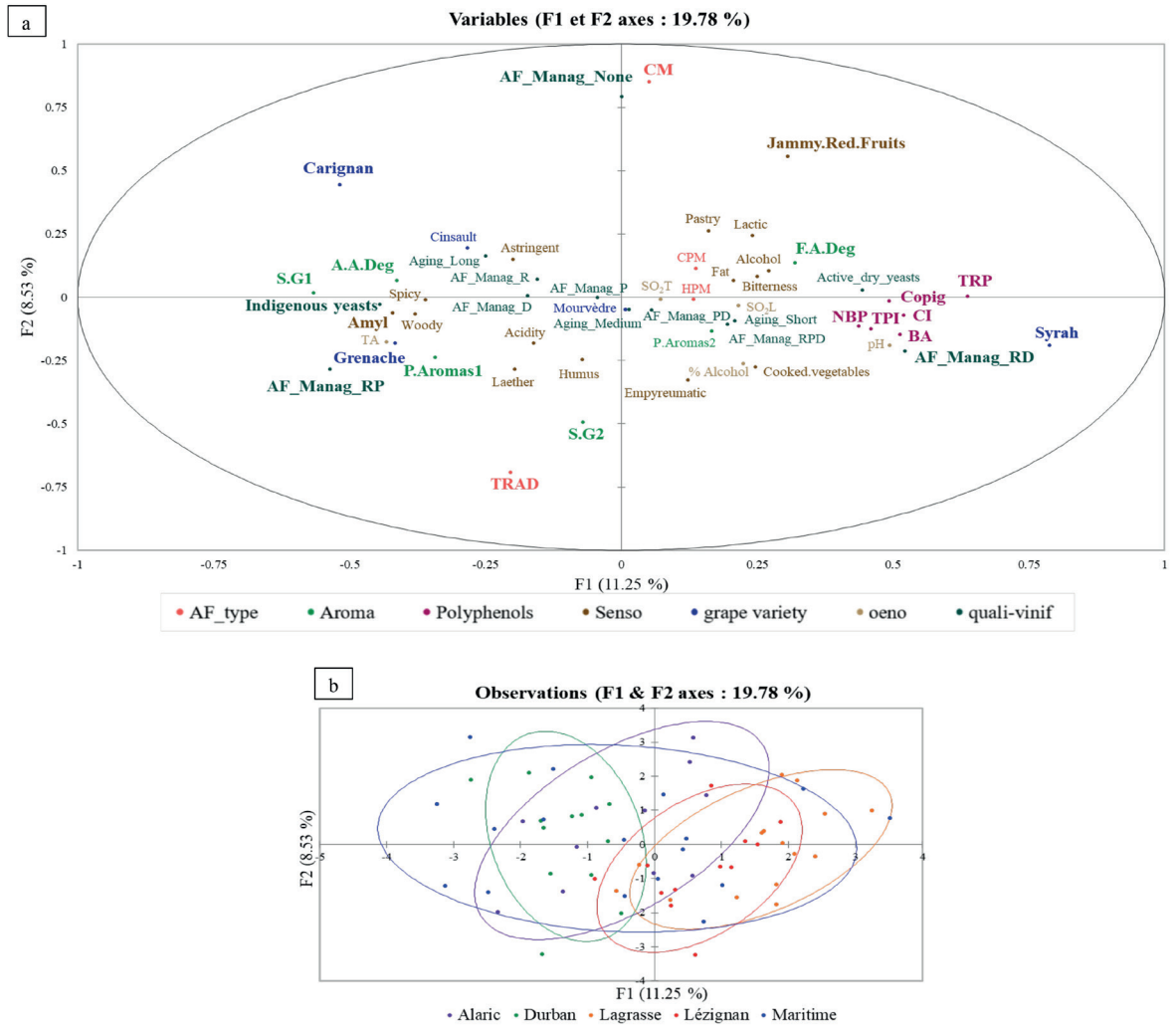


FIGURE 4. Multiple Factor Analysis (MFA) of winemaking parameters, polyphenols parameters, volatile compounds groups and sensorial descriptors. Oenological variables, regions and year are reported as supplementary parameters.

a) *Graph of variables* on F1 and F2 axes. Groups of variables are colour coded. Variables reported in bold are those that contribute to the construction of the axis. b) *Graph of individuals*. Distribution of wine samples on F1 and F2 axes, with addition of confidence ellipses. The five sub-regions are colour coded.

3.2. MFA of polyphenols and winemaking parameters

The general MFA revealed a positive association between polyphenols and both quali-vinif and grape variety groups of variables (Figure 2). Concerning the construction of the axes, polyphenols contributed mainly to the F1 and F3 axes (17 and 16 %, respectively). Figure 4a shows that F1 (11.25 %) contrasted all the polyphenol variables, Syrah and AF_Manag_RD with Carignan and Grenache, Indigenous yeasts and AF_Manag_RP. The positive link between certain polyphenol variables was also shown by the correlation matrix: in particular, in both vintages, CI had a highly positive correlation ($p < 0.05$) with NBP and Copig (Figure S2). Along the F3 axis (11.09 %), CI, TPI, NBP and Copig were negatively correlated with AF_Manag_D, AF_Manag_PD and HPM (Figure 5a). By plotting the confidence ellipses (Figure 4b and 5b) of the sub-regions, the positive side of the F1 axis was found to be mainly represented by LA and LE wines, along with two wines from MA, associated with polyphenol variables and Syrah; meanwhile, the negative side of the same axis was mainly represented by DU and AL, along with some samples from MA, associated with Cinsault and Carignan and indigenous yeasts and negatively correlated with polyphenol variables.

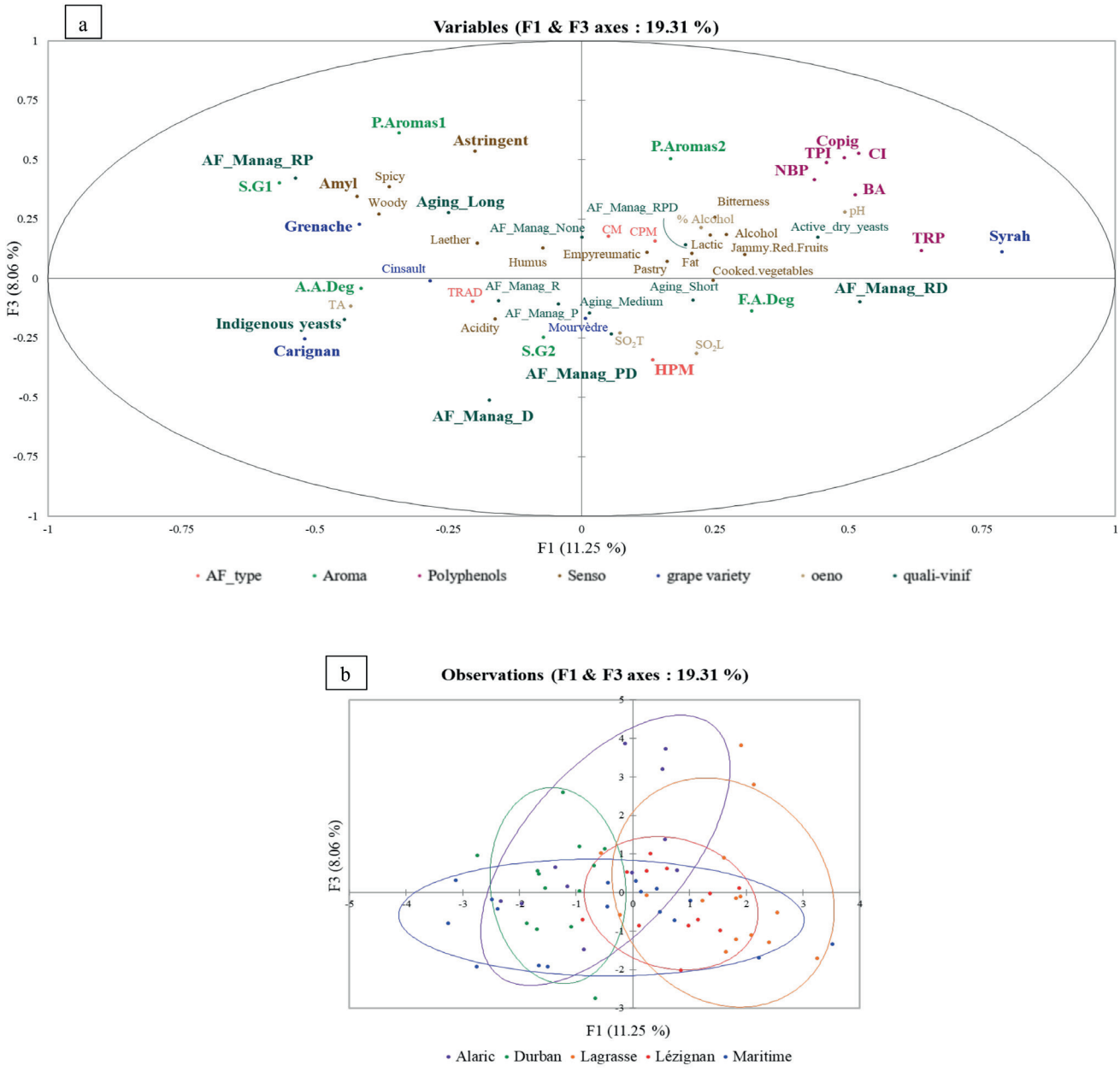


FIGURE 5. Multiple Factor Analysis (MFA) of winemaking parameters, polyphenols parameters, volatile compounds groups and sensorial descriptors. Oenological variables, regions and year are reported as supplementary parameters.

a) Graph of variables on F1 and F3 axes. Groups of variables are colour coded. Variables reported in bold are those that contribute to the construction of the axis. b) Graph of individuals. Distribution of wine samples on F1 and F3 axes, with addition of confidence ellipses. The five sub-regions colour coded.

4. Volatile compounds analyses of AOC Corbières wines

4.1. Classification of the sub-regions according to volatile compound groups

Forty-eight compounds were identified and their amounts relative to the internal standard were calculated in all 53 (2018) and 39 (2019) wines. The sub-regions were classified according to the mean values of the six previously defined aroma groups using HCA heatmap (Figure 6). In 2018, two clusters were identified: one comprising only LA, and the other containing the other sub-regions (Figure 6a). In the latter cluster, LE is statistically the furthest away from the others. Volatile compounds were also divided into two main clusters: F.A.Deg. on the right side of the heat map and the other groups on the left. The cluster on the left was further divided into two sub-groups: one comprising A.A.Deg. only and the other containing P.Aromas 1, SG1, SG2 and P.Aromas 2. It is clear that sub-region clustering was mainly driven by the variation in content of F.A.Deg. compounds, and, as confirmed by ANOVA, LA showed a significantly higher relative amount ($p < 0.05$) than DU, MA and LE. The LE sub-region was further separated from the other regions due to a lower relative content of the A.A.Deg compounds. By contrast, DU showed significantly higher relative amounts ($p < 0.05$) of A.A.Deg. and SG1 compounds than LE and LA.

In 2019 two main clusters were determined: one comprising LA and AL and the other MA, DU and LE (Figure 6b). In the latter cluster, MA is detached from DU and LE. The six groups of variables were separated like in 2018, with differences between the two clusters mainly driven by F.A.Deg. and A.A.Deg. compounds. In particular, as confirmed by ANOVA, LA showed higher relative amounts of F.A.Deg. than DU and LE, but lower amounts of A.A.Deg. than MA. Finally, like in 2018, DU showed higher relative amounts of S.G1 compounds than AL and LE.

In summary, LA and LE were at the opposite sides of the heat map in both vintages. AL was the closest to LA in 2018 and was in the same cluster in 2019. In 2018, DU was in the same cluster as MA and AL, but grouped with LE in 2019.

4.2. MFA of volatile compounds and winemaking parameters

The MFA highlighted a positive association between aroma and grape variety (F1) and quali-vinif (F1, F2 and F3) parameters (Figure 2). Aroma compounds contributed mainly to the F1 and F3 axes (21 and 35 %, respectively). The dispersion of the samples show that the wines were distributed along the F1 axis. In particular, with the addition of the confidence ellipses, the MFA showed a separation of the sub-regions into three significant groups. LA and LE were located on the positive side of the axis and correlated with the F.A.Deg. group, while DU was situated on the negative side and correlated with S.G1, A.A.Deg. and P.Aromas 1. The MA and AL samples were more centrally located and did not show a decisive trend (Figure 4b and 5b).

5. Sensorial analysis

5.1. Classification of the sub-regions according to sensorial descriptors

A QDA was carried out to determine sixteen sensorial descriptors in both vintages. Regarding the chemical parameters, an HCA heatmap was generated for the totality of sensorial descriptors (Figure 7). In 2018, three main clusters were observed: one containing LA and LE, one containing

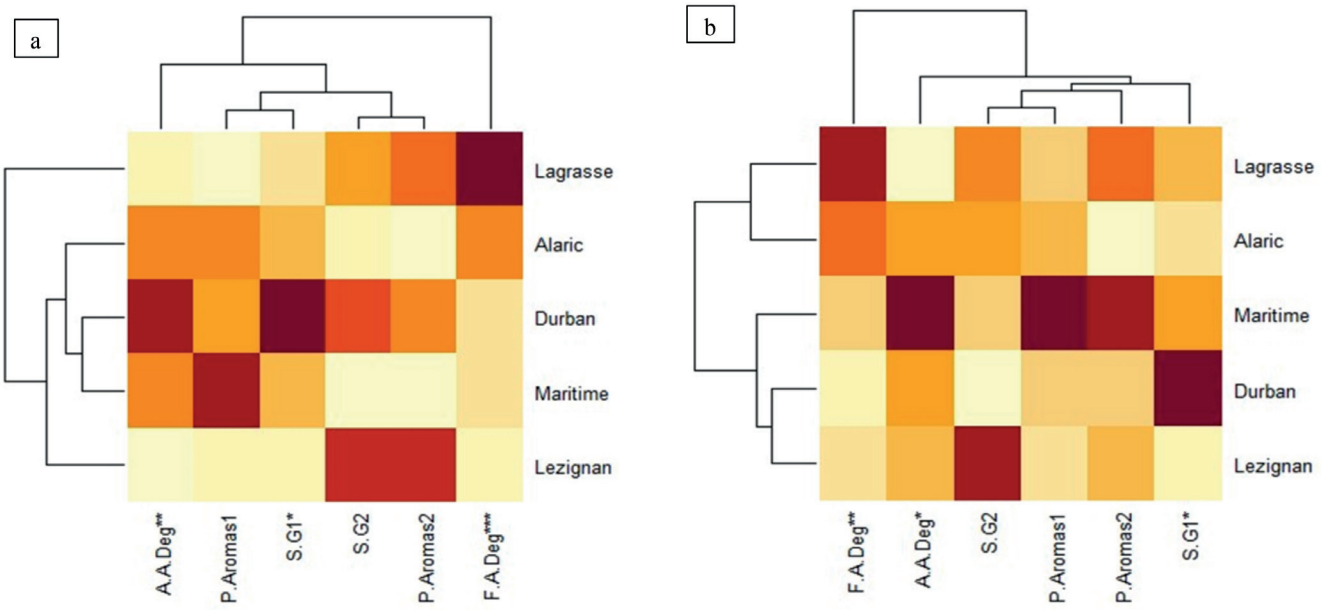


FIGURE 6. Hierarchical clustering heatmap performed using a) 2018 and b) 2019 volatile compounds groups for the five AOC Corbières sub-regions (Ward algorithm and Euclidean distance analysis).

Variables that were found significant among the five sub-regions from the ANOVA are reported with: *** = $p < 0.001$, ** = $p < 0.01$, * = $p < 0.05$. The rows in the heatmap represent the sub-regions and the columns indicate the variables.

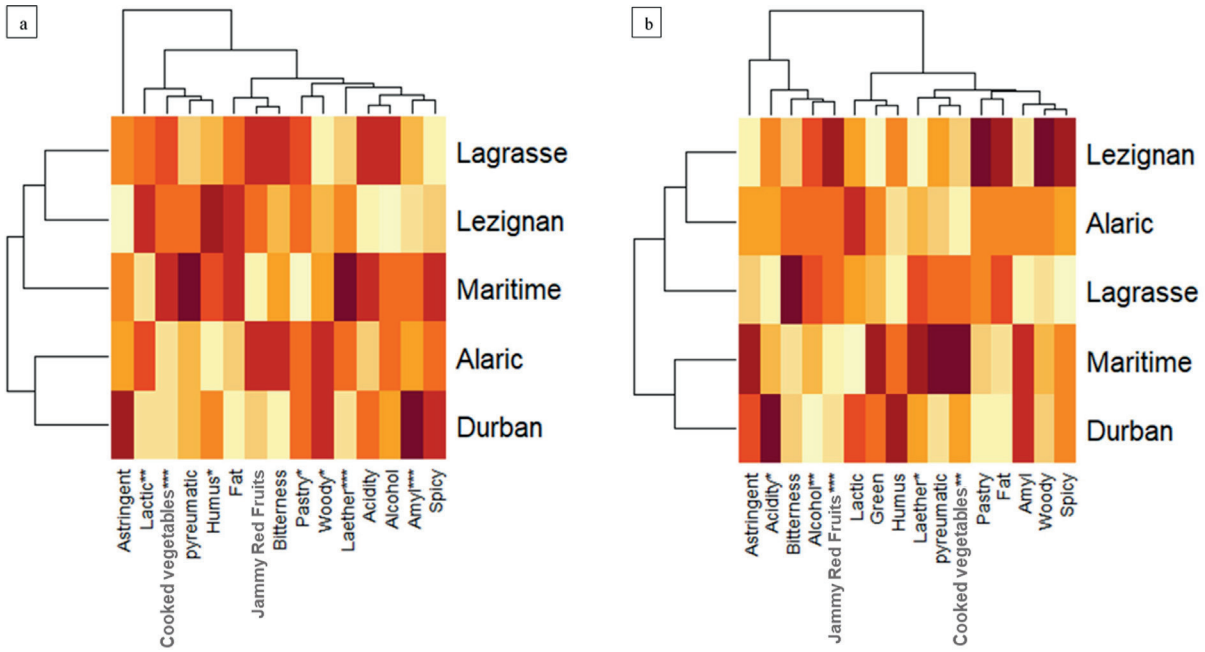


FIGURE 7. Hierarchical clustering heatmap performed using a) 2018 and b) 2019 sensorial descriptors for the five AOC Corbières sub-regions (Ward algorithm and Euclidean distance analysis).

Variables that were significant among the five sub-regions from the ANOVA are reported with asterisks: *** = $p < 0.001$, ** = $p < 0.01$, * = $p < 0.05$. The rows in the heatmap represent the sub-regions and the columns indicate the variables.

MA only, and another with AL and DU (Figure 7a). Two main groups of variables can be observed: astringency on the left, and the others on the right. These trends were partially confirmed by ANOVA that showed significant differences ($p < 0.05$) among the sub-regions for amyl, cooked vegetables, pastry, woody, lactic and leather notes. In particular, LE showed higher average intensities of humus and lactic notes than AL, MA and DU, respectively. DU showed higher amylic and pastry notes than LA and LE on one side and MA on the other. MA was, by contrast, perceived as having more pronounced cooked vegetables and leather characters than DU and AL on one side and DU and LA on the other. Finally, AL had more intense woody notes than LA.

In contrast to the 2018 vintage, two main clusters were highlighted in 2019: one containing LE, AL and LA, and the other MA and DU. A common trend could be observed for both vintages: LE and LA were in the same cluster, clearly separated from DU. The dendrogram of the variables also revealed two main groups: astringent, acidity, bitterness, alcohol and jammy red fruits on the left-hand side and the other descriptors on the right-hand side. Interestingly, the olfactory and gustatory descriptors were mixed in 2018, whereas in 2019 the gustatory descriptors were mainly found in the first group of variables (except jammy red fruits). These results were partially confirmed by ANOVA applied to this vintage: in particular, significant differences ($p < 0.05$) were found for cooked vegetables, jammy red fruits, leather, alcohol and acidity descriptors. In 2018, MA showed a higher intensity in leather than LE, and cooked vegetable notes than LE and AL. On the other hand, LE was characterised by higher intensities of jammy red fruits and alcohol descriptors than MA and DU. Finally, wines originating from LA were deemed less acidic than those from DU.

In summary, LE and LA were separated from DU in both vintages, with AL being close to DU in 2018 and grouped with LA and LE in 2019, and MA being separated from DU in 2018 and in the same cluster in 2019.

5.2. MFA of sensorial descriptors and winemaking parameters

The MFA revealed a positive correlation between the sensorial descriptors and quali-vinif (F1 and F3) and AF_type (F2) parameters (Figure 2). The Sensorial descriptors contributed the most to the F5 axis (47 %). Nonetheless, some descriptors could be related to winemaking parameters through the first three axes. In particular, the F1 axis (11.25 %) contrasted the Amyl descriptor, Indigenous yeasts, Grenache and AF_Manag_RP with Syrah and AF_Manag_RD (Figure 4). On the F2 axis (8.53 %), the jammy red fruits descriptor was positively associated with CM, AF_Manag_None and Carignan, while contrasting with TRAD. Finally, the Astringent character was shown to be positively correlated with Aging_long, and negatively correlated with AF_Manag_D, AF_Manag_PD and HPM on the F3 axis (8.06 %) (Figure 5). The addition of the confidence ellipses of the sub-regions revealed that the amyl descriptor was associated with wines from DU, MA and AL, possibly due to higher amounts of Grenache in their blend (Figure 4b). On the F2 axis, the jammy red fruits descriptor was associated with wines produced via CM (or using a winemaking method not 100 % traditional) or that showed a higher percentage of Carignan in their blends. Finally, the Astringent descriptor seemed to be related to DU, AL and MA (Figure 5b).

6. General HCA Analysis - Heat Map

A final HCA heatmap was conducted to determine how the sub-regions were separated based on the average values of each variable being significant. (Figure 8). In regard to winemaking parameters, only the blend percentage was kept, as it was the only significant quantitative factor. As a result, two main clusters were highlighted: LA in one group and the other sub-regions in the other which was further divided into two sub-clusters: one composed of AL and DU, and the other of MA and LE. By examining the totality of variables and their relationship with each other, it was possible to identify the main trends specific to each of the sub-regions studied.

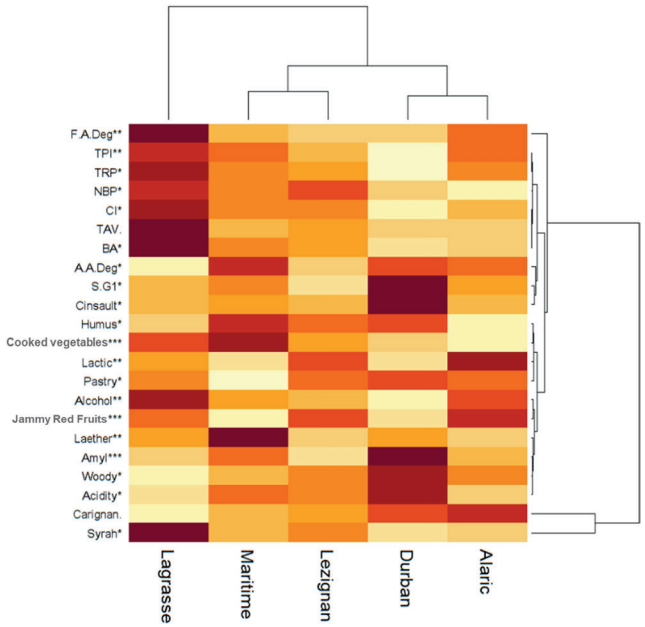


FIGURE 8. Hierarchical clustering heatmap performed using a) 2018 and b) 2019 significant variables for the five AOC Corbières sub-regions (Ward algorithm and Euclidean distance analysis).

Variables that were found significant among the five sub-regions from the ANOVA are reported with asterisks: *** = $p < 0.001$, ** = $p < 0.01$, * = $p < 0.05$. The rows in the heatmap represent the sub-regions and the columns indicate the variables.

LA wines featured a greater proportion of Syrah (59 %) in their blends, and exhibited higher average values of: NBP, CI, BA, TRP, TPI, alcohol percentage, alcohol gustatory perception and F.A.Deg. compounds. By contrast, they exhibited lower values for percentage Carignan (10-15 %), woody notes and gustatory acid perception and A.A.Deg. compounds.

Wines from MA exhibited higher relative amounts of A.A. Deg., and higher levels of humus, cooked vegetables and leather notes. By contrast, they were also found to have lower values for pastry, milky and fruity red notes.

LE exhibited higher average values for humus, lactic acid, and jammy red fruit notes and the NBP colour variable. Additionally, it displayed lower relative quantities of F.A.Deg., A.A.Deg., and S.G1 compounds, as well as a lower intensity of the amyl character.

DU featured higher average percentages of Cinsault and Carignan (together with AL), alongside elevated amounts of A.A.Deg. and S.G1 compounds, amyl and woody olfactory notes, and acidic mouthfeel perception. By contrast, it exhibited a lower percentage of Syrah, lower values for CI, TRP, TPI, pH, and lower lactic and jammy red fruit olfactory and alcohol gustatory descriptors.

Finally, the wines from AL exhibited a higher proportion of Carignan (27-37 %) in their blends, as well as more distinct notes of lactic, alcohol and jammy red fruits. However, they were characterised by a low proportion of Syrah (25-29 %), lower NBP mean value and lower humus and cooked vegetables notes.

DISCUSSION

1. Influence of winemaking on polyphenolic and gustatory profile

TPI and CI values of wines from both vintages corresponded with those of other red wines, whether mono-varietal or blended, originating from similar or southern latitudes with a warm climate (Costa *et al.,* 2014; Sáenz-Navajas *et al.,* 2012; Stavridou *et al.,* 2016). Our research has highlighted a certain variability in these parameters depending on the wine and sub-region: the TPI values of the wines ranged from 34.8 to 70, while the CI values ranged from 4.5 to 16.5. These variations can be explained by differences in the polyphenolic composition of the varieties studied, as well as differences in the winemaking processes, in particular the percentages of the blends and the winemaking method. In our research, Syrah was the most prevalent variety, accounting for an average of 40 %, followed by Grenache (30 %), Carignan (30 %) and Mourvèdre (15 %). Wines with a Syrah content exceeding 50 % exhibited the highest colour parameters, CI (13-16) and TRP (20-28) in particular. Compared to the other varieties, Syrah is well-known for its high anthocyanin concentrations (Sáenz-Navajas *et al.,* 2012). Jensen *et al.* (2008) compared the total polyphenolic content of various red wine varieties, finding that Syrah had the highest content while Grenache was low in total polyphenolic compounds (Costa *et al.,* 2014; Edo-Roca *et al.,* 2014). However, Abi-Habib *et al.* (2021) noted that despite Carignan having a greater anthocyanin concentration in their skins than Grenache, the extraction of polyphenols varied during maceration. Specifically, the extraction rates of anthocyanins and tannins were higher and faster in Grenache wines than in Carignan wines (Abi-Habib *et al.,* 2021). Maceration methods and fermentation length may have had different effects on the final polyphenolic profile of the wines (Table S2/S3). While Jensen *et al.* (2008) found Mourvèdre to contain the second-highest total polyphenol content, our study showed this variety to have a lower percentage in the blends (10-20 %) than the other varieties, which could explain its lower contribution to the overall polyphenolic profile.

This low content is linked to the late ripening period of Mourvèdre, occurring in September and October. During this phase, there is a drop in both temperature and rainfall (Figure S1). The HCA analysis of polyphenolic parameters did not reveal the distinct separation of the sub-regions, but they were grouped in similar clusters across both vintages. The groups displayed minor differences between the two vintages, likely due to variations in weather conditions or winemaking parameters. These factors could affect the metabolism of polyphenolic compounds found in grapes and their extraction during the winemaking process (Cosme *et al.,* 2020; Gutiérrez-Escobar *et al.,* 2021).

1.1. Impact of percentage of Syrah and CPM

The Syrah variety displayed a significant positive correlation ($p < 0.05$) with the TPI and colour parameters in both vintages. Since LA contained a greater proportion of Syrah in its blends, the higher TPI, BA and TRP values in 2018 may be due to this factor, as shown in Figure 5a. This trend can also be observed in 2019: in fact, LA and LE showed a higher percentage of Syrah in their blends, and thus higher final Copig, CI and NBP values (Figure 5b). Additionally, our research showed that TPI and astringency were correlated ($p < 0.05$), consistent with previous findings, although this descriptor was not significant across all sub-regions (Sáenz-Navajas *et al.,* 2015). Despite a higher percentage of Syrah in blends, there was no significant correlation ($p > 0.05$) with the astringency descriptor. This implies that wines with higher Syrah percentages were not always the most astringent. The sensation of astringency in the mouth is primarily caused by the binding between tannins and salivary proteins. The way in which tannins interact with proteins is dependent on their distinctive structural traits, such as their molecular weight, average degree of polymerisation, composition (including the percentage of galloyl group), and state of modification (Mouls and Fulcrand, 2015; Aziza *et al.,* 2016; Peña-Neira, 2019). Astringency can be influenced by grape composition and winemaking technique (Soares *et al.,* 2016). Among the samples with higher percentages of Syrah in them (> 50 %), CPM had been employed for both vintages. This process involves keeping the must at low temperatures (usually 10-15 °C) for several days prior to fermentation (Sacchi *et al.,* 2005). Numerous studies have examined the impact of this technique on polyphenol composition (Álvarez *et al.,* 2006; Gambacorta *et al.,* 2011). In the 2019 vintage, CPM was more widely used, possibly leading to average CI and Copig values in AL, LA, and MA being higher in 2019 than in the 2018 vintage (Table S2/S3). Moreover, Busse-Valverde *et al.* (2011) observed increased tannin extraction with this practice, but conflicting results on final astringency have been reported (Casassa *et al.,* 2015; González-Neves *et al.,* 2016). CPM was applied for different lengths of time in our study (24 hours-10 days). Wines undergoing 24 hours of CPM generally exhibited lower CI (8) compared to those subjected to 10 days of cold temperature (10-12 CI). This outcome is consistent with previous research. However, TPI and astringency did not exhibit the same pattern; therefore, since CPM was generally applied to all varieties in the blend and not just to one variety, it is possible to hypothesise a cultivar-specific influence. In summary, the CPM method, when adopted, had general positive effects on the colour components of wines, while its impact on the final astringency was not clear.

1.2. Impact of percentage of Carignan, CM and HPM

In the 2018 vintage, Carignan showed a negative correlation ($p < 0.05$) with TPI and colour parameters (Figure 4). As a sub-regional trait, AL were characterised as having a higher percentage of Carignan, followed by wines from DU. Of the wines containing the highest percentages of Carignan (> 50 %), the DU wines were among those with the lowest CI values (6-8), and had been produced applying TRAD, HPM or CM processes. A study from Geffroy *et al.* (2015) reported a strong impact of the cultivar, vintage and type of treatment on the impact of pre-fermentation heat-treatment on the final characteristics of wine. Specifically, Carignan subjected to pre-fermentation heat treatment at 70 °C, followed by pressing and fermentation in the liquid phase, resulted in lower anthocyanin levels and reduced TPI and CI compared to traditional maceration. However, the impact varied based on the treatment method. In the current study, wineries using heat treatment applied it uniformly to all varieties in the blend, either in the liquid or solid phase. Limited information was available on the operation itself, preventing a comprehensive understanding of its real impact. Regarding other winemaking methods used, Carignan showed a more widespread trend in the use of the CM process across the sub-regions. The polyphenolic profiles of wines subjected to CM in this study yielded contradictory results. Two DU samples exhibited the lowest CI (6-8) and TPI (35-47) values, while two AL and LA samples had higher CI (10.7) and TPI (57) values. This inconsistency aligns with findings from various studies on CM. In fact, several studies have indicated lower concentrations of polyphenols in CM wines, depending on factors such as temperature and maceration time (Pellegrini *et al.,* 2000; Tesniere and Flanzy, 2011). However, a recent study by González-Arenzana *et al.* (2020) reported higher polyphenolic content in CM wines than in traditional wines that had undergone destemming and crushing. These findings are supported by various previous studies (Etaio *et al.,* 2008; Gómez-Míguez and Heredia, 2004; Portu *et al.,* 2023). However, González-Arenzana *et al.* (2020) reported no significant variation in the overall amount of anthocyanins. Such outcomes were attributed to the fact that they had only analysed and compared Tempranillo wines: since the extractability of anthocyanins depends on the toughness of the skin, as well as on the ethanol formed inside the berry, the variety (and its maturity) may play a role in the final amount of anthocyanins in the final wines. If we consider the samples of our study, CM had been applied to either one (Carignan mostly), two (Carignan and Syrah) or the totality of the varieties in the blend. Therefore, the variations observed between the wines that underwent this process may be attributed to differences in the blend. Interestingly, the two cuvées with the highest TPI and CI were also the result of a CM process applied to

all the varieties in the blend. However, the final wines were perceived differently in terms of astringency. This outcome could be attributed to differing NBP values between the two wines, with AL2 exhibiting a lower NBP value (1.54) than LA7 (3.21), resulting in AL2 being perceived as more astringent (8/10) than LA7 (5.4/10). The NBP fraction included the anthocyanin-derived pigments that resist discolouration by hydrogen sulfide, and thus for which the 4' position in the A ring was not available due to a reaction with other compounds. Among these derived pigments, anthocyanins may also have reacted with low molecular mass pigments formed from pigmented tannins as the result of cleavage reactions (Bindon *et al.,* 2014). A modification in tannin structure may have led to various reactions with anthocyanins and to the formation of derived pigments, possibly influencing the astringency perception. Thus, even if CM did not prove significant in the sub-regions, it may have played a role in the extraction of polyphenols and thus ultimately in the astringency of red wines. AL and DU differed from the other sub-regions in their higher percentages of Carignan and lower average colour components. In fact, AL and DU shared similar features; however, within the sub-regions the samples differed in terms of polyphenolic composition, possibly due to the different winemaking techniques used (CM, HPM and TRAD), which may have varying impacts on the final polyphenolic composition.

1.3. Impact of percentage of Grenache and TRAD

Grenache was more commonly subjected to traditional winemaking techniques compared to other varieties, as can be observed in Figure 1. Although Grenache percentage in wines was not significantly different across the sub-regions, a positive correlation ($p < 0.1$) was found between wines with higher percentages of Grenache (40-50 % on average) and "astringency" mouthfeel sensation. Notably, the DU8 sample, being one of the most intense in terms of astringent sensation (8/10), had a higher percentage of Grenache (70 %) along with 30 % Cinsault, a variety also known for its low polyphenolic content, contributing less to the colour of wines (Etiévant *et al.,* 1988). As a result, the DU8 sample had a low CI (4.5) and also a low TPI (40), the latter being within the range of low values for red wines (35-75 approximately). The other samples with a lower TPI than DU8 (i.e., < 40) had higher CI values than DU8 (6.1-11.8) due to the fact that they also included Syrah and Carignan in their blend, but the resulting astringency sensation in the mouth was lower (5.7-7/10). Therefore, we can assume that the winemaking process to which DU8 was subjected caused a higher extraction rate, resulting in a greater quantity of tannins and a more pronounced astringent mouthfeel. If, on the other hand, we examine wines with the highest TPI values, we find that MA4-2 displayed the highest level of astringency intensity, despite containing only 40 % Syrah, but with 40 % Grenache. In the study by Jensen *et al.* (2008), the ratio of tannins in grapes to tannins in wines for Grenache was found to be 0.19, while it was between 0.20 and 0.28 for the other three varieties. These findings suggest a potentially higher tannin extraction rate for Grenache compared to the other varieties. As regards winemaking methods, our study found that Grenache was mainly vinified using the TRAD technique. Nevertheless, further research would be useful in order to evaluate the influence of each winemaking technique on this variety. This would provide confirmation regarding the impact of CM, CPM, and HPM on the decrease of the astringent sensation in comparison to the TRAD method.

2. Influence of winemaking on aromatic and olfactory profile

Of all the volatile compounds characterising the AOC Corbières wines, those released by yeasts during AF via the metabolism of amino acids and fatty acids were the most abundant and significantly different between sub-regions. The A.A.Deg group included acetate esters, higher alcohols and branched ethyl esters (Table 2). Higher alcohols were the most abundant of the A.A.Deg group, as has been previously reported in studies on different red wine varieties (Fariña *et al.,* 2015). Since the biosynthesis path of these compounds involves amino acids, it is conceivable that their final amount in wines is influenced by grape variety (Bouloumpasi *et al.,* 2015). The group of F.A.Deg compounds was characterised by ethyl esters, fatty acids and alcohols (4-methyl-1-pentanol and 3-methyl-1-pentanol). These compounds are derived mainly from unsaturated fatty acids and fermentable sugars and their final concentration in wines is related to yeast strain, fermentation temperature, oxygen management and sugar content (Godillot *et al.,* 2022; Styger *et al.,* 2011). Finally, volatile compounds derived from plant metabolism and grouped in P.Aromas 1 and P.Aromas 2 revealed no significant differences among the sub-regions. The MFA analysis revealed that the main separation between the sub-regions may be driven by the first axis; in particular, with samples showing higher relative amounts of F.A.Deg compounds on the positive side, and those showing higher relative amounts of A.A.Deg, SG1 and P.Aromas 1 on the negative side.

2.1. Impact of percentage of the blend and long ageing

Of the winemaking parameters, the percentage of a variety in a blend seems to have had a major impact on the aromatic profile. Indeed, wines with higher percentages of Syrah in their blend were positively correlated with F.A.Deg in 2019, and negatively correlated with A.A.Deg. and SG1 in 2018. This is evidenced by the fact that LA wines made from a high average percentage of Syrah (59 %) in the blend showed significantly lower amounts of AA.Deg and SG1 compounds, while DU wines made from a low average percentage of Syrah (28 %) contained significantly higher amounts of AA.Deg and SG1 compounds. In particular, a significant and negative correlation was found between Syrah and 2-phenylethanol. The final amount of this higher alcohol produced and released by yeast through the Erlich metabolic pathway is related to L-phenylalanine amino acid metabolism, whose content has been reported to be grape variety-related (Etiévant *et al.,* 1988). More precisely, Bouloumpasi *et al.* (2015) demonstrated that out of several red grape varieties from Greece, Syrah was among those with the lowest concentration of L-phenylalanine.

Therefore, based on these findings, the low 2-phenylethanol relative amounts found in wines made with a high percentage of Syrah may be explained by low amounts of L-phenylalanine in grapes. Conversely, samples with higher relative amounts of A.A.Deg and SG1 were made of higher percentages of Carignan and Grenache in their blend, and were perceived as having a stronger amylic olfactive character. In addition, the amylic descriptor was positively correlated with ethyl acetate, 1-heptanol and 2-methyl-1-propanol (or isobutanol) compounds. Similarly, De-la-Fuente-Blanco *et al.* (2016) have demonstrated the specific contribution of isoamyl alcohol and isobutanol to the spirit/solvent/alcohol notes in red wines and mentioned higher alcohols as being suppressors of lactic/red fruit odour notes. Our results are consistent with these findings, since the MFA (Figure 4a) showed that the amyl descriptor was negatively correlated with jammy red fruits and lactic notes.

Finally, wines made with higher percentages of Grenache in their blends were positively correlated with the P.Aromas 1 group of compounds in 2018. This trend was not necessarily linked to a specific sub-region, but rather spread throughout the appellation. In addition, samples with higher P.Aromas 1 were positively correlated with a long ageing period (Figure 5). This correlation may be influenced by the presence of terpenoids, such as terpinen-4-ol and fenchone, since previous studies have assessed their increase during wine ageing (Slaghenaufi and Ugliano, 2018). Samples showing higher amounts of P.Aromas 1 were perceived as having a higher humus olfactive notes in 2019. This tendency may be attributed to the presence of 1-octen-3-ol, a compound of fungus-contaminated grape origin that was previously reported as an off-flavour associated with humus/damp cave sensorial descriptors (Lisanti *et al.,* 2014). Nonetheless, the contribution of fenchone and estragole on this humus/damp note in the red wine matrix has not been reported thus far and would need further investigation.

2.2. Impact of the winemaking method

Other than the percentage of blending, the winemaking method can affect aromatic composition. In 2019, wines with higher percentages of Syrah were positively correlated ($p < 0.1$) with the F.A.Deg group of compounds, and this correlation was particularly influenced by ethyl hexanoate and hexanoic acid. In addition, samples showing the highest relative amounts of F.A.Deg compounds were mostly produced through a CPM or CM winemaking process. Several studies have highlighted a positive impact of these two winemaking methods on the overall aromatic profile of final wines (Moreno-Pérez *et al.,* 2013; Zhang *et al.,* 2019). More precisely, González-Arenzana *et al.* (2020) reported that Tempranillo wines produced using a CM process presented significantly higher concentrations of total esters, in particular ethyl butyrate and isobutyrate, which is consistent with the results reported by Antalick *et al.* (2014). Fermentation temperature is another factor affecting the production of ethyl esters. Indeed, Massera *et al.* (2021) evidenced that *Saccharomyces cerevisiae* strains released higher amounts of ethyl esters under low temperatures (15 °C), in particular ethyl hexanoate and ethyl butanoate when compared to high temperatures (25 °C). Thus, the use of CPM and CM methods in winemaking for the fermentation of predominantly Syrah blends appears have a positive impact on F.A.Deg. aroma compounds.

CONCLUSION

In summary, our study allowed us to differentiate wines from five sub-regions of the AOC Corbières appellation by means of a thorough analysis of their polyphenolic, aromatic, and sensorial profiles. It is worth noting that the percentage of the varieties in the blend was found to be a crucial factor influencing the polyphenolic and aromatic features of the wines. The significant variation observed in this parameter depending on the sub-region underlined its role as a distinctive 'typicity' trait for each sub-region. Though the winemaking method (traditional, carbonic maceration, etc.) was not found to be a typical trait among the five sub-regions, as it did not contribute to their overall differentiation, it could be clearly associated with specific chemical and sensory differences between the samples. The study emphasises the important role of winemaking parameters in the general differentiation of the sub-regions. However, it is essential to take into account that the observed disparities between the two vintages may also be indicative of differing weather conditions. As the analysed wines were produced from blends of different varieties cultivated in different vineyards, a more specific focus on the direct influence of terroir, including soil and climate characteristics, on wine quality would be essential. To better understand the overall expression of the terroir, it would be useful to carry out a single-varietal vinification of grapes from a single plot specific to each grape variety. This would enable a more nuanced investigation into their individual contributions. This methodology could enhance our understanding of the complex relationship between environmental factors and the quality of Corbières AOC wines.

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