Volatile composition, foam characteristics and sensory properties of Tempranillo red sparkling wines elaborated using different techniques to obtain the base wines

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ABSTRACT

The aim of this work was to study oenological techniques to obtain adequate base wine for red sparkling wine elaboration. Four winemaking techniques were carried out: pre-fermentative cold maceration with dry ice and *delestage* with premature grapes; and sugar reduction in must and partial dealcoholisation of wine with mature grapes. Their effect on oenological parameters, volatile composition, foam and sensory characteristics was valuated. Reduction of sugar content and partial dealcoholisation allow obtaining base wines with more adequate alcohol content. No differences were found between the oenological parameters during the ageing time. Pre-fermentative cold maceration and partial dealcoholisation had a greater influence on the volatile composition of the base and red sparkling wines. Oenological technique did not affect the foam instrumental parameters. Red sparkling wines from premature grapes showed higher vegetal aromas, and pre-fermentative cold maceration the best foam sensory descriptors.

Keywords: red sparkling wines; oenological techniques; volatile compounds; foam; sensory analysis

INTRODUCTION

In the last years, a continuous increase of the world market of sparkling wines has been observed. According to the reports published by the International Organization of Vine and Wine [1-2], sparkling wine production increased around 46% since 2002 and 7% from 2015 to 2016.

In Spain, the market of natural sparkling wines is also growing quite fast due to the increase of both national consumption and export volume. “Cava” wines continue cornering the largest percentage of sales of quality Spanish sparkling wine. However, more and more Spanish wineries are elaborating natural sparkling wines following the traditional method in order to extend their range of products and open new markets. Most of the sparkling wines elaborated in our country are white and rosé ones, being the production of red sparkling wines practically non-existent.

Red sparkling or semi-sparkling (“frizzante”) wines are elaborated in other countries like Australia, South-Africa, Argentina, Italy or Portugal, with a great acceptance by consumers. Most of these wines are semi-sparkling wines and with slight red colour and lacked of complexity, with the exception of sparkling Shiraz wines from Australia.

Wine sector is a highly competitive market, and the winemakers are looking for new markets or products to diversify their production. In sparkling wine production, the use of innovative grape varieties [3-4] or the development of new products could be good alternatives. Recently, Olarte et al. [5] have been studied different types of factors that influence the acceptance of a new product such as red sparkling wine. Therefore, the study of quality red sparkling wine elaboration and chemical composition will be of interest.

One of the initial problems in red sparkling wine elaboration is to obtain suitable base wines that should have moderate alcohol content, good colour intensity and good mouthfeel. The alcohol degree of base wines for sparkling wine elaboration should be between 10-11.5 %vol. However, the red grapes harvested at that time of technological maturity have not achieved the adequate phenolic maturity, and they are considered in a prematurity stage.

Therefore, different winemaking techniques should be studied to obtain suitable base wines for the elaboration of natural red sparkling wines.

Pre-fermentative cold maceration is used in red winemaking to favour the extraction of soluble water compounds such as some phenols, anthocyanins and aroma precursors [6-8]. This technique will reduce the extraction of green tannins from premature grapes and therefore the astringency in wines [9]. However, these effects depend on many factors, such as grape variety, ripeness degree, maceration time, etc. [7, 10-11], even on the type of fermenter used [8].

The *delestage* technique with partial removal of seeds can be useful to improve the quality of wines elaborated with red premature grapes, producing softer, less astringent and more fruity wines due to a lower tannin and higher aroma extraction [12-14].

Other alternative could be the use of red grapes harvested at their optimum degree of maturity (considering as phenolic maturity) that will have a high alcohol degree for sparkling wine elaboration and use techniques that could reduce the alcohol degree. In this case, techniques as the reduction of sugar content in musts [15-16] or the partial dealcoholisation of wines [17-19] could be interesting and would allow obtaining base wines with more adequate alcohol content.

No studies have been found related to the elaboration, composition and/or changes occurring during the ageing of red sparkling wines, probably due to the difficulty of obtaining a base wine with the appropriate characteristics, as it was previously commented.

Therefore, the aim of this work was to study different oenological techniques to obtain the adequate base wine for red sparkling wine elaboration. The oenological parameters, volatile composition and foam characteristics of the different red sparkling wines elaborated were evaluated after their ageing on lees in bottle, and after their ageing in bottle without lees. A sensory analysis at the final ageing moment was also carried out.

MATERIAL AND METHODS

Chemicals

The volatile compound standards were purchased from Fluka (Buchs, Switzerland), Sigma-Aldrich (Steinheim, Germany) and Alfa Aesar (Lancashire, United Kingdom). HPLC-grade reagents were provided by Carlo Erba Reagents (Barcelona, Spain), and the remaining reagents were supplied by Panreac (Madrid, Spain). Water Milli-Q was obtained via a Millipore system (Bedford, MA). Helium BIP (99.9997%), air zero (99.998%) and Premier plus hydrogen (99.9992%) were provided by Carburos Metálicos S.A. (Valladolid, Spain).

Winemaking process

All base and sparkling wine elaborations were carried out in the experimental winery of the Oenological Station (ITACyL) sited in Rueda (Valladolid, Spain). All base wines were elaborated in stainless steel tanks of 150 L in duplicated.

Grapes from *Tempranillo* grape variety were harvested in two maturity moments: premature grapes (PM) with alcohol degree and acidity suitable to elaborate a sparkling wine (pH 3.2, 6.5 g/L of tartaric acid, 10.9 % probable alcohol and 3.5 g/L malic acid), but that they do not have the adequate phenolic maturity (green seeds that contribute to more astringent and bitter compounds); and grapes at their optimum degree of maturity (M) (pH 3.4, 6.0 g/L of tartaric acid, 12.4 % probable alcohol and 2.2 g/L malic acid).

Two winemaking techniques were carried out for each type of grapes: pre-fermentative cold maceration with dry ice (PM-DI) and *delestage* (rack and return) with partial removal of seeds (PM-D) with the premature grapes; and sugar reduction in must (M-SR) and partial dealcoholisation of wine (M-AR) with the mature grapes. In both moments, control wines (PM-C and M-C) were also elaborated, following the traditional red winemaking process. The red grapes were destemmed, crushed, and slightly sulphited (0.05 g/L). Commercial *Saccharomyces cerevisiae* yeasts (FERM ES 488, Enartis, Italy, 0.2 g/L) were inoculated to undergo alcoholic fermentation at a controlled temperature < 25 ºC ± 2 ºC. The maceration-fermentation time was 7 days. Once alcoholic fermentation was completed, the mass was pressed and the wine was racked into a new tank. Commercial *Oenococcus oeni* bacteria (Viniflora CH16, CHR Hansen, Denmark) were inoculated to carry out the malolactic fermentation.

The pre-fermentative cold maceration was carried out by addition of 3 mm dry ice pellets (Carburos Metálicos S.A., Valladolid, Spain) to the destemmed and crushed grapes. Dry ice was added in the quantity necessary to decrease the temperature to 5 ºC ± 2 ºC, and this temperature was maintained for 3 days before the beginning of alcoholic fermentation. Further dry ice was added as necessary to maintain the temperature to 5ºC ± 2 ºC. After that, the traditional red winemaking is followed.

The *delestage* was carried out after 2 days of the beginning of alcoholic fermentation, during three consecutive days. Additionally partial removal of seeds was done during *delestage*. The remaining processes were the same than the traditional elaboration.

The sugar reduction in must was carried out by a nanofiltration process using a SR3 membrane (Koch Membrane System), selected considering the results obtained in previous works [20]. The details of the membrane process are found in Salgado et al. [21]. To reduce the probable alcohol degree, the initial must was mixed with the obtained permeate in the membrane process, in adequate proportions, looking for a reduction of two probable degrees. After that, the traditional red winemaking is followed.

A part of the wine elaborated traditionally from mature grapes was dealcoholized. The dealcoholisation process was carried out by reverse osmosis, using the equipment Flavy MT (Bucher Vaslin), to reduce two alcohol degrees.

Quality sparkling wines elaborated by the traditional method are obtained after a second fermentation in closed bottles in contact with lees for at least 9 months (EC Regulation Nº 606/2009 for sparkling wines with a protected designation of origin). Therefore, after cold-stabilization and clarification of base wines (T0), the tirage liquor was added and the wines were bottled. The tirage liquor was formed by yeast *S. cerevisiae* var. *bayanus* (0.30 g/L, IOC 18–2007, Oenologique Institut de Champagne, Epernay, France), sucrose (23 g/L) and bentonite calcium activated (0.03 g/L) (Laffort, France). Previously, a starter was prepared with 10% of the base wine, 180 g/L of sucrose and 0.30 g/L of yeast. The bottles were kept in a cellar at a temperature (11-13 ºC) and relative humidity (75-85%) controlled for 9 months (T9). After that, the sparkling wines were riddled and disgorged (no expedition liqueur was added) and were maintained in the same cellar for 12 months (ageing in bottle without lees, T9+12). The pressure and residual sugars were measured periodically to control the second fermentation in two different bottles for each treatment. The pressure was measured by an aphrometer and the sugars by an enzymatic method.

Since the second fermentation takes place in individual bottles, three bottles of each sparkling wine experience at each sampling time were analysed.

Analysis of oenological parameters, colour parameters and phenolic composition

Standard oenological parameters in wines were determined according to official analysis methods [22]: pH, titratable acidity (as g/L tartaric acid), volatile acidity (as g/L acetic acid) and alcohol degree (% vol: mL ethanol/100 mL wine). The methods used to evaluate these parameters are accredited by ISO 17025 Norm and the uncertainty has also been calculated according to this Norm.

Colour parameters, colour intensity and tonality were evaluated using the Glories methodology [23]. Phenolic composition was evaluated by the quantification of total phenols by reaction to Folin-Ciocalteu and total anthocyanins by pH changes [24].

Analysis of volatile compounds

Major volatile compounds were quantified by direct injection of 1 µL of wine. An Agilent 7890A gas chromatograph with a flame ionization detector (FID) was used. Samples were injected in split mode (25:1), and volatiles were separated using an Agilent DW-WAX (30 m x 0.25 mm i.d. x 0.25 µm film thickness) capillary column. The chromatographic conditions were: helium as carrier gas at a flow rate of 0.7 mL/min, column temperature program, 40 ºC held for 4 min, heated at 1 ºC/min to 70 ºC, and then heated at 30 ºC/min to 200 ºC (held for 10 min), and the injection temperature was 250 ºC. Each compound was identified and quantified by a calibration graphs built with pure standard solutions, analysed under the same conditions.

Minor volatile compounds were extracted by liquid-liquid. extraction following the method and chromatographic conditions established by Rodríguez-Bencomo et al. [23]. Briefly, 250 mL of wine, 5 mL of dichloromethane, and 75 µL of a mixture of two internal standards (550 mg/L of methyl octanoate, and 450 mg/L of 3,4-dimethylphenol) were added to a flask. The extraction was carried out for 3 h with continuous stirring (150 rpm) in an orbital shaker. After this time, the organic phase was separated, concentrated until 400 µL and analysed by gas chromatography–mass detector (GC–MS). Chromatographic analyses were performed with a HP-6890N GC coupled to a HP-5973 inert MS detector equipped with a Quadrex 007CWBTR capillary column (60 m length, 0.25 mm i.d., and 0.25 mm film thickness), following the chromatographic conditions established by

Rodríguez-Bencomo et al. [25]. Quantification was carried out following the internal standard quantification method, using the quantification ions, and internal standards indicated in Pérez-Magariño et al. [3].

Measurement of foaming properties by instrumental method

Foam properties of sparkling wines were evaluated using Mosalux equipment (Station Oenotechnique de Champagne, Cormontreuil, France) according to Maujean et al. [~~24~~26]. Three parameters were measured: HM (mm), the maximum height reached by foam or foamability; HS (mm), the foam stability height and TS (seconds), the foam stability time [~~25~~27].

Sensory analyses

The sensory analysis was performed in a designed test room in accordance with ISO 8589 Standard (2010), and was carried out by twelve expert tasters (7 male and 5 females judges from 40 to 60 years old) from the Regulatory Councils of different Spanish D.O. and wineries, according to the methodology described in González-Sanjosé et al. [~~26~~28]. The wines were evaluated in duplicate in two different sessions and the serving temperature was 8-10ºC. Samples were presented in random order using a structured scale of ten points to evaluate the following ~~This work has focused on~~ olfactory attributes: olfactory intensity, vegetal, yeasty, fruity, oxidized, and reduced notes~~ were evaluated~~.

Sensory foam properties were valuated using the descriptors defined by Gallart et al. [~~27~~29], initial foam, foam area, foam collar, bubble size and effervescence speed with scores from 1 to 3.

The sparkling wines were tasted after 9 months of ageing on lees and 12 months of ageing without lees (T9+12).

Statistical analyses

ANOVA and the Least Significant Difference test were applied at significant level of $p < 0.05$. Factor analysis using varimax rotation criterion was performed and only factors with eigenvalues greater than 1 were selected. These statistical analyses were carried out using Statgraphics Centurion XVII.

Generalized Procrusters Analysis (GPA) was applied on the mean ratings for olfactory and foam attributes by using the Senstools 3.3.2. program (Utrecht, The Netherlands).

RESULTS AND DISCUSSION

Oenological parameters, phenols and colour parameters of ~~musts and~~ wines

Table 1 shows the oenological parameters, phenolic content and colour parameters of ~~the grapes and~~ the different base and red sparkling wines elaborated during the ageing on lees and later ageing in bottle without lees.

The red base wines obtained with premature grapes had an alcohol degree of 11 %vol, while those obtained with mature grapes had 13 %vol (M-C). Therefore, the processes of sugar reduction of must and dealcoholisation of wine were carried out looking for the reduction of two alcohol degrees to obtain quality sparkling wines with more adequate alcohol content. The M-AR wine was reduced their alcohol degree in 2 %vol and the M-SR in 1 %vol. The lower reduction of the alcohol degree in M-SR wine could be due to the usual deviations that occur during fermentation, and therefore a good correlation between the probable alcohol degree in musts and the real alcohol content in wines is not always found [~~28-29~~30-31]. The increase of the alcohol degree of sparkling wines due to the second fermentation in bottle was between 0.8-1.0 %vol from T0 to T9+12 wines (Table 1), as it was expected due to the addition of sucrose.

The titratable acidity of the base and red sparkling wines from the mature grapes were significantly lower than those from premature grapes, as it was expected. Taking into the account that the uncertainty value for these data is 0.2, no~~No~~ differences between treatments in titratable acidity of wines from the same maturity degree were found.

In general, no significant differences were found between the oenological parameters of the different wines during the ageing time, with the exception of volatile acidity that slightly increased during the ageing in bottle without lees.

The base and sparkling wines elaborated with mature grapes presented higher content of total phenols and anthocyanins than those elaborated with premature grapes, with the exception of M-AR. In addition, these wines also presented the highest colour intensity and the lowest tonality values. These differences were maintained during the ageing time.

Volatile compounds of wines

Table 2 shows the concentrations of the thirty two volatile compounds identified and quantified in the red base wines in order to study the influence of grape maturity and the oenological technique used as well as the odour threshold of each compound [32-34].

In general, the red base wines produced from mature grapes showed higher levels of ethyl esters of straight-chain fatty acids, mainly ethyl hexanoate and ethyl octanoate, and lower of alcohol acetates than those produced from premature grapes. No clear tendencies in these compounds were found in other studies, and in general, it will depend on the grape variety and grape composition, in particular, in free amino nitrogen and ammonium [~~30~~35], taking into account that the fermentation conditions were the same.

The red base wines elaborated from premature grapes showed the highest concentrations of C6 alcohols, mainly 1-hexanol and *cis*-3-hexenol. These compounds are responsible of herbaceous and vegetal notes, and can have a negative effect on wine quality ~~[31]~~. However, all the wines elaborated in this study had C6 alcohol concentrations below their odour threshold values [32].

Higher alcohols are the most abundant volatile compounds in wines, and in concentrations lower than 400-500 mg/L, they could have a synergic effect on fruity and floral notes of wines [~~32-33~~34, 36]. ~~No s~~Statistically significant differences were found in total higher alcohols between the red base wines produced from premature and mature grapes, showing the wines produced from premature grapes (PM-C) the highest content, with the exception of 1-propanol. ~~However, the wines produced from mature grapes showed slightly higher levels of 1-propanol and lower of isobutanol than those produced from premature grapes.~~

~~In general, the~~ The red base wines obtained from mature grapes showed the highest levels of vanillin derivatives (Table 2). These compounds can be formed from the glycosylated precursors present in grapes and may contribute to vanillin and sweet floral notes [~~34~~37]. The content of γ-lactones was also higher in the wines obtained from mature grapes than in those obtained from premature grapes. These compounds are formed during the alcoholic fermentation from their corresponding hydroxy-acids [~~35~~38], and contribute to sweet fruit notes.

The pre-fermentative cold maceration with dry ice (PM-DI) and the partial dealcoholisation of wine (M-AR) were the techniques that had the most influence on the volatile composition of the red base wines.

The base wines obtained with pre-fermentative cold maceration (PM-DI) stood out by the highest alcohol acetate content that is in agreement with other studies carried out using different grape varieties and cold maceration conditions [8-9], that could increase the fruity notes of these wines. On the other hand, these base wines presented lower total content of higher alcohols than PM-C wines. Higher alcohols are produced mainly during yeast fermentation of sugars and yeast metabolism of amino acids, therefore, it could be thinking that the pre-fermentative cold maceration do not favour the extraction of amino acids. However, the assimilable nitrogen composition of grapes also has an important influence on the production of these compounds, which is greater when there is a lack of available nitrogen [~~35~~38].

The partial dealcoholisation process modified the content of ethyl esters and alcohol acetates to a great extent (M-AR) [~~36~~39]. The ethyl esters of straight-chain fatty acids and alcohol acetates were reduced, while the ethyl esters of branched-chain fatty acids and ethyl lactate increased. The content of vanillin derivatives in M-AR wine was also lower than M-C wine due to the dealcoholisation process that in general produces a reduction in volatile compounds [~~36-37~~39-40]. In spite of this, an increase in the higher alcohol content in M-AR wines was observed.

Then, due to the high number of compounds and wine samples, multivariate analysis was carried out (thirty two volatile compounds x six wines x three ageing moments). Factorial analysis with all data was performed, in order to see if the information given by these compounds all together would allow differentiating the wines studied according to the grape maturity, the ageing time and/or the oenological technique used.

The factorial analysis selected six factors with an eigenvalue greater than 1, which explained the 89.3 % of the total variance. Table 3 shows the loadings for each variable on the selected factor, as well as the eigenvalue and the cumulative variance. The variables with higher loading values contribute most significantly to the explanatory meaning of the factors (marked in bold).

Figure 1a shows the distribution of the different sparkling wines studied in the plane defined by the first two factors, which explained the 59.3% of the total variance. As can be seen in this figure, the variables associated with factor 1 permit to differentiate the base and red sparkling wines by grape maturity, independently of the ageing time or the oenological technique used. The wines elaborated from mature grapes appear on the right side of the plane, showing higher and positive values of factor 1. Therefore, these wines presented higher values of vanillin derivatives (methyl vanillate, ethyl vanillate, acetovanillone), benzyl alcohol and γ–nonalactone, and lower of C6 alcohols and alcohol acetates (mainly hexyl acetate). These results agree with those found by Bindon et al. [3841] and Antalick et al. [3035], and could be due to a greater content of aroma precursors of these compounds in more mature grapes.

On the other hand, factors 2 and 3 allow differentiating the wines by the ageing time, as can be seen in Figure 1b. The variables associated with factor 2 permit to differentiate the base and sparkling wines, independently of the grape maturity or the oenological technique used. This factor was mainly correlated positively with ethyl esters of straight-chain fatty acids (ethyl octanoate and ethyl decanoate), alcohol acetates and fatty acids (octanoic and decanoic acids), and negatively with ethyl lactate, ethyl isovalerate, α-terpineol and γ-butyrolactone. The red base wines appear on the right side of the plane, showing positive values of factor 2, while the sparkling wines showed negative values of this factor. These results indicate that during the ageing of the wines a decrease in ethyl esters of straight-chain fatty acids, alcohol acetates and fatty acids and an increase in ethyl lactate, ethyl esters of branched-chain fatty acids, α-terpineol and γ-butyrolactone were produced. These results agree with those obtained by other authors in white and rosé sparkling wines produced from different grape varieties [39-4142-44]. Therefore, it seems that the changes observed in volatile compounds are due to the own process of ageing on lees of these type of wines independently to other factors such as grape variety, winemaking conditions, wine type (white, rosé or red wine), etc.

Factor 3 allows differentiating the red sparkling wines aged on lees (T9) and the red sparkling wines after their ageing in bottle without lees (T9+12). During the ageing in bottle without lees, the wines were taking place in the negative zone of factor 3, which were due to the decrease of some fatty acids (mainly isovaleric and hexanoic acids), C6 alcohols, 4-vinylphenol and 2-phenylethanol, compounds positively associated to factor 3 (Table 3). Some of these changes occurred during the ageing in bottle without lees were also observed in white and rosé sparkling wines [~~41~~44].

As can be seen in the factorial analysis results, the differences in volatile composition of the wines studied by grape maturity and ageing time were more important than the differences found by the oenological technique used. Therefore, in order to study the differences or not produced in the final sparkling wines by the effect of the different techniques used to elaborate the red base wines, the individual volatile compounds in the final sparkling wines were treated by ANOVA.

Table 4 shows the data of volatile compounds of red sparkling wines at the final moment (wines aged on lees for 9 months and aged in bottle without lees for 12 months, T9+12) and the ANOVA results. In general, the differences found between the red base wines produced from premature and mature grapes were maintained in the sparkling wines. Therefore, the final red sparkling wines from premature grapes presented greater content of alcohol acetates and C6 alcohols, and lower of ethyl esters of fatty acids and vanillin derivatives.

In spite of the changes observed in volatile compounds of the red sparkling wines during their ageing, the differences found in the red base wines by the effect of the different oenological techniques were also observed in the final red sparkling wines. The PM-DI wine presented higher concentrations of alcohol acetates and ethyl esters of straight-chain fatty acids and lower of C6 alcohols and higher alcohols than PM-C wines. On the other hand, the M-AR wine had mainly lower content of alcohol acetates and lactones and higher of ethyl esters of branched-chain fatty acids than M-C wine.

Foaming instrumental parameters of red sparkling wines

Table 5 shows the data of the maximum height of the foam (HM) and the stable height of the foam (HS) of the red sparkling wines elaborated in the two ageing moments studied. The data of time stability (TS) are not shown since did not show good repeatability. This fact has been reported in other works [~~25~~27, ~~42~~45].

Only some slightly differences have been found between the different red sparkling wines elaborated. After nine months of ageing on lees (T9), the control sparkling wines showed the lowest values of HM, although the differences found between all the sparkling wines were not very high. No statistically significant differences were found in HS values.

After nine months of ageing on lees and twelve months of ageing in bottle without lees (T9+12), it was showed an increase of HM values in all the sparkling wines. The~~, and~~ PM-DI and PM-D were the wines with the highest values, although not statistically significant differences were found between treatments. The HS values were maintained constant or slightly increased.

No studies have been reported in the literature focus on the foam characteristics of red sparkling wines. However, the HM and HS values of the red sparkling wines were similar to those of white and rosé sparkling wines [~~41~~44, ~~43-44~~ 46-47]. The changes found of HM and HS in the red sparkling wines by effect of the ageing time showed also a similar trend than those observed in a previous work in white and rosé sparkling wines [~~41~~44].

Sensory analysis of red sparkling wines

Figure 2 provides a GPA consensus configuration of the relationship of the red sparkling wines as determined for their olfactory and foam perceptions. In the olfactory GPA space (Figure 2a), wines were properly located in the vectorial dimension defined by the two factors, which accounted for 44.73 % of the total variance. The consensus plot showed a clearly different distribution of red sparkling wines. In general, the red sparkling wines produced from premature grapes showed higher correlation with vegetal notes~~,~~ than wines from mature grapes, with the exception of M-SR that also are related with vegetal notes that agree with the volatile data previously commented. The M-AR sparkling wines was characterized by a higher olfactory intensity, dominated by oxidized, fruity and yeasty aromas, while M-C sparkling wines was described to be related with reduced notes.

Figure 2b shows the attribute average space obtained from the foam of the red sparkling wines, where PC1 explained 23.57% of the total variance and PC2 accounted for 19.54%. The consensus plot showed the red sparkling wines quite spread, thus indicating a marked difference among wines regardless of grape maturity. PM-D and M-C sparkling wines had high initial foam. PM-C and M-AR sparkling wines were more correlated with effervescence while PM-DI was described to be related with the best foam area, bubble size and foam collar. Although generally it is not easy to relate sensory results with chemical data, these foam sensory values are consistent with the higher HS values in PM-DI sparkling wines (Table 5). M-SR samples did not emphasize any particular foam sensory descriptor.

In summary, the differences in volatile composition of the wines studied by grape maturity and ageing time were more important than the differences found by the oenological technique used. From the point of view of volatile composition and foam characteristics, the base wines obtained from mature red grapes showed more positive characteristics than those obtained from premature grapes, with the exception of the excessive alcohol degree obtained for this type of wines. The reduction of sugar content in musts and the partial dealcoholisation of wines allow obtaining base wines with more adequate alcohol content, but the additional processes or equipment required imply costs that do not justify their use.

The red sparkling wines produced from premature grapes showed higher C6 alcohols content correlated with vegetal notes and lower volatile compounds associated with fruity aroma than those obtained from mature grape. However, the pre-fermentative cold maceration allows obtaining wines with similar volatile composition to red sparkling wines produced from mature grapes and with the best valued in the foam instrumental and sensory descriptors. Therefore, considering that this technique is used by many winemakers, the pre-fermentative cold maceration with dry ice could be the best option to obtain an adequate base wine for red sparkling wine elaboration.

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Table 1. Mean values of the oenological parameters (± uncertainty), phenols and colour parameters of red base wines and red sparkling wines

Ageing moment [a]	**Treatment** [b]	**pH**	**Titratable acidity** [c]	**Alcohol** [c]	**Volatile acidity** [c]	**Total phenols** [c,d]	**Total anthocyanins** [c,d]	**Colour intensity** [d]	**Tonality** [d]
T0	PM-C	3.5±0.1	5.1±0.2	11.1±0.2	0.32±0.04	1124 b	137 b	8.7 b	0.63 c
	PM-DI	3.4±0.1	4.9±0.2	11.1±0.2	0.32±0.04	1273 d	145 b	8.8 b	0.61 c
	PM-D	3.5±0.1	5.3±0.2	10.8±0.2	0.36±0.05	1069 a	147 b	7.1 a	0.63 c
	M-C	3.7±0.1	4.6±0.2	13.0±0.2	0.39±0.05	1253 d	214 c	11.5 c	0.53 a
	M-SR	3.7±0.1	4.6±0.2	12.0±0.2	0.20±0.03	1210 c	210 c	12.7 d	0.51 a
	M-AR	3.7±0.1	4.7±0.2	11.0±0.2	0.32±0.04	1161 b	115 a	12.4 d	0.58 b
T9	PM-C	3.3±0.1	5.5±0.2	11.7±0.2	0.32±0.04	1158 c	92 a	6.6 a	0.64 b
	PM-DI	3.3±0.1	5.3±0.2	11.9±0.2	0.32±0.04	1169 c	94 a	6.7 a	0.64 b
	PM-D	3.3±0.1	5.5±0.2	11.6±0.2	0.30±0.04	929 a	100 a	6.7 a	0.64 b
	M-C	3.5±0.1	4.7±0.2	13.7±0.2	0.32±0.04	1205 d	119 b	9.8 b	0.57 a
	M-SR	3.5±0.1	4.7±0.2	13.0±0.2	0.31±0.04	1196 d	148 c	10.0 b	0.55 a
	M-AR	3.5±0.1	4.7±0.2	12.0±0.2	0.32±0.04	975 b	86 a	10.3 c	0.59 a
T9+12	PM-C	3.4±0.1	5.5±0.2	11.9±0.2	0.57±0.06	1021 c	71 c	6.8 a	0.67 c
	PM-DI	3.4±0.1	5.2±0.2	11.9±0.2	0.57±0.06	1002 c	73 c	6.9 a	0.66 c
	PM-D	3.5±0.1	5.4±0.2	11.8±0.2	0.58±0.06	880 a	57 a	6.7 a	0.68 c
	M-C	3.7±0.1	4.5±0.2	13.9±0.2	0.62±0.07	1075 d	84 d	10.0 b	0.61 b
	M-SR	3.7±0.1	4.5±0.2	12.8±0.2	0.61±0.07	1064 d	102 e	10.2 b,c	0.58 a
	M-AR	3.6±0.1	4.7±0.2	12.0±0.2	0.65±0.07	952 b	65 b	10.4 c	0.61 b

[a] T0: base wines; T9: wines aged on lees for 9 months; T9+12: wines aged on lees for 9 months and aged in bottle without lees for 12 months

[b] PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized

[c] Titratable acidity (as g/L tartaric acid), alcohol (% vol: mL ethanol/100 mL wine), volatile acidity (as g/L acetic acid), total phenols (mg/L of gallic acid), total anthocyanins (mg/L of malvidin-3-glucoside)

[d] Values with different letters in each ageing moment indicate statistically significant differences ($p < 0.05$)

Table 1. Oenological parameters of grapes and red base wines and red sparkling wines

			Grapes		
Maturity stage	**ºBrix**	**pH**	**Titratable acidity**[c]	**Probable alcohol**	**Malic acid (g/L)**
Premature grapes	19.9	3.2	6.5	10.9	3.5
Mature grapes	22.2	3.4	6.0	12.4	2.2
			Wines		
Treatment[a]	**Ageing moment**[b]	**pH**	**Titratable acidity**[c]	**Alcohol**[c]	**Volatile acidity**[c]
PM-C	T0	3.5	5.1	11.1	0.32
	T9	3.3	5.5	11.7	0.32
	T9+12	3.4	5.5	11.9	0.57
PM-DI	T0	3.4	4.9	11.1	0.32
	T9	3.3	5.3	11.9	0.32
	T9+12	3.4	5.2	11.9	0.57
PM-D	T0	3.5	5.3	10.8	0.36
	T9	3.3	5.5	11.6	0.30
	T9+12	3.5	5.4	11.8	0.58
M-C	T0	3.7	4.6	13.0	0.39
	T9	3.5	4.7	13.7	0.32
	T9+12	3.7	4.5	13.9	0.62
M-SR	T0	3.7	4.6	12.0	0.20
	T9	3.5	4.7	13.0	0.31
	T9+12	3.7	4.5	12.8	0.61
M-AR	T0	3.7	4.7	11.0	0.32
	T9	3.5	4.7	12.0	0.32
	T9+12	3.6	4.7	12.0	0.65

[a] PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized.

[b] T0: base wines; T9: wines aged on lees for 9 months; T9+12: wines aged on lees for 9 months and aged in bottle without lees for 12 months.

[c] Titratable acidity (as g/L tartaric acid), alcohol (% vol: mL ethanol/100 mL wine) and volatile acidity (as g/L acetic acid).

Table 2. Volatile compounds of red base wines (T0) and odour threshold [a]

Compounds	**Odour threshold** [b]	**PM-C** [c]	**PM-DI**	**PM-D**	**M-C**	**M-SR**	**M-AR**
Ethyl butyrate	20	187 b	263 d	174 b	182 b	233 c	148 a
Ethyl 2-methylbutyrate	18	3.9 a	5.1 b	3.9 a	3.8 a	3.7 a	15.2 c
Ethyl isovalerate	3	6.8 a	7.8 b	7.0 a	8.3 b	9.6 c	19.0 d
Ethyl hexanoate	14	408 ab	442 b	389 a	547 d	506 c	371 a
Ethyl octanoate	5	540 bc	512 b	583 cd	663 e	603 d	343 a
Ethyl decanoate	200	216 b	200 b	213 b	196 b	211 b	117 a
Total ethyl esters		*1361 b*	*1430 c*	*1395 b,c*	*1599 d*	*1567 d*	*1014 a*
Ethyl lactate*	154	38 a	39 a	38 a	46 b	41 ab	79 c
Isoamyl acetate	30	1821 c	3790 d	1828 c	1316 b	1979 c	680 a
Hexyl acetate	-	38 d	52 e	38 d	11 b	16 c	4 a
2-Phenylethyl acetate	250	430 d	574 e	555 e	288 b	351 c	105 a
Total alcohol acetates		*2289 c*	*4416 e*	*2421 d*	*1615 b*	*2346 d*	*789 a*
Isovaleric acid	33	865 c	712 b	690 b	609 a	684 b	1482 d
Hexanoic acid	420	3083 a	2941 a	2895 a	3366 b	3489 b	3750 c
Octanoic acid	500	5478 b	5507 b	6315 c	6552 c	7224 d	3606 a
Decanoic acid	1000	1026 b	1206 c	1317 d	1071 b	1114 b	685 a
Total acids		*10452 b*	*10366 b*	*11216 c*	*11512 c*	*12315 d*	*9523 a*
1-Hexanol	8000	850 d	762 c	946 e	550 a	540 a	659 b
trans-3-hexen-1-ol	-	45 c	45 c	42 c	35 b	23 a	25 a
cis-3-hexen-1-ol	400	296 c	264 b	295 c	37 a	21 a	28 a
Total C6 alcohols		*1191 d*	*1071 c*	*1283 e*	*622 a*	*584 a*	*742 b*
Benzyl alcohol	200000	127 a	139 ab	152 b	305 d	248 c	338 e
Linalool	25	4.2 b	4.9 c	4.4 b	3.2 a	3.1 a	5.0 c
α-Terpineol	250	1.0 a	1.1 a	1.7 b	1.8 b	1.3 a	1.6 b
Citronellol	100	4.3 a	6.5 c	4.2 a	5.3 b	5.6 b	5.4 b
γ-Butyrolactone*	35	4.5 a	4.9 a	4.4 a	6.2 b	6.0 b	5.9 b
γ-Nonalactone	30	3.1 a	3.4 a	3.6 a	6.8 b	6.7 b	6.7 b
Methyl vanillate	3000	2.3 a	1.8 a	2.3 a	14.6 d	13.3 c	11.0 b
Ethyl vanillate	990	28 a	28 a	28 a	72 c	72 c	62 b
Acetovanillone	1000	35 a	33 a	33 a	44 b	49 c	47 c
Total vanillin derivative		*65 a*	*64 a*	*63 a*	*130 c*	*134 c*	*121 b*
4-Vinylguaiacol	10	16 b	12 a	15 b	30 d	38 e	21 c
4-Vinylphenol	180	174 b	163 ab	147 a	202 c	157 ab	290 d
Total volatile phenols		*205 b*	*175 a*	*162 a*	*231 c*	*195 b*	*312 d*
2-Phenylethanol*	14	50.1 b	49.3 ab	52.9 b	44.9 a	62.9 c	64.2 c
1-Propanol*	0.83	13.6 b	18.7 e	11.8 a	17.7 d	15.7 c	19.0 e
Isobutanol*	40	77.5 e	40.5 c	81.3 f	30.8 a	33.6 b	48.1 d

Isoamyl alcohols*	30	270 b	251 a	270 b	247 a	282 c	290 c
Total higher alcohols		*411 b*	*360 a*	*416 b*	*340 a*	*394 b*	*475 c*

[a] Mean values of two elaborations/tanks by treatment (n=2) in µg/L except those marked with an asterisk * that are expressed in mg/L. Values with different letters in each compound indicate statistically significant differences at $p < 0.05$

[b] Odour threshold values reported in literature [32-34]

[c] PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized

Table 2. Volatile compounds of red base wines (T0) [a]

Compounds	**PM-C** [b]	**PM-DI**	**PM-D**	**M-C**	**M-SR**	**M-AR**
Ethyl butyrate	187 b	263 d	174 b	182 b	233 c	148 a
Ethyl 2-methylbutyrate	3.9 a	5.1 b	3.9 a	3.8 a	3.7 a	15.2 c
Ethyl isovalerate	6.8 a	7.8 b	7.0 a	8.3 b	9.6 c	19.0 d
Ethyl hexanoate	408 ab	442 b	389 a	547 d	506 c	371 a
Ethyl lactate*	38 a	39 a	38 a	46 b	41 ab	79 c
Ethyl octanoate	540 bc	512 b	583 cd	663 e	603 d	343 a
Ethyl decanoate	216 b	200 b	213 b	196 b	211 b	117 a
Isoamyl acetate	1821 c	3790 d	1828 c	1316 b	1979 c	680 a
Hexyl acetate	38 d	52 e	38 d	11 b	16 c	4 a
2-Phenylethyl acetate	430 d	574 e	555 e	288 b	351 c	105 a
Isovaleric acid	865 c	712 b	690 b	609 a	684 b	1482 d
Hexanoic acid	3083 a	2941 a	2895 a	3366 b	3489 b	3750 c
Octanoic acid	5478 b	5507 b	6315 c	6552 c	7224 d	3606 a
Decanoic acid	1026 b	1206 c	1317 d	1071 b	1114 b	685 a
1-Hexanol	850 d	762 c	946 e	550 a	540 a	659 b
trans-3-hexen-1-ol	45 c	45 c	42 c	35 b	23 a	25 a
cis-3-hexen-1-ol	296 c	264 b	295 c	37 a	21 a	28 a
Benzyl alcohol	127 a	139 ab	152 b	305 d	248 c	338 e
Linalool	4.2 b	4.9 c	4.4 b	3.2 a	3.1 a	5.0 c
α-Terpineol	1.0 a	1.1 a	1.7 b	1.8 b	1.3 a	1.6 b
Citronellol	4.3 a	6.5 c	4.2 a	5.3 b	5.6 b	5.4 b
γ-Butyrolactone*	4.5 a	4.9 a	4.4 a	6.2 b	6.0 b	5.9 b
γ-Nonalactone	3.1 a	3.4 a	3.6 a	6.8 b	6.7 b	6.7 b
Methyl vanillate	2.3 a	1.8 a	2.3 a	14.6 d	13.3 c	11.0 b
Ethyl vanillate	28 a	28 a	28 a	72 c	72 c	62 b
Acetovanillone	35 a	33 a	33 a	44 b	49 c	47 c
4-Vinylguaiacol	16 b	12 a	15 b	30 d	38 e	21 c
4-Vinylphenol	174 b	163 ab	147 a	202 c	157 ab	290 d
2-Phenylethanol*	50.1 b	49.3 ab	52.9 b	44.9 a	62.9 c	64.2 c
1-Propanol*	13.6 b	18.7 c	11.8 a	17.7 d	15.7 c	19.0 e

Isobutanol*	77.5 e	40.5 c	81.3 f	30.8 a	33.6 b	48.1 d
Isoamyl alcohols*	270 b	251 a	270 b	247 a	282 c	290 c

[a] Mean values of two elaborations/tanks by treatment (n=2) in µg/L except those marked with an asterisk * that are expressed in mg/L. Values with different letters in each compound indicate statistically significant differences at $p < 0.05$

[b] PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized.

Table 3. Factor loadings after varimax rotation of all red base and sparkling wines elaborated. Loadings lower than absolute values of 0.250 are not shown.

	Factor 1	**Factor 2**	**Factor 3**	**Factor 4**	**Factor 5**	**Factor 6**
Eigenvalue	9.8	9.2	4.5	2.3	1.7	1.2
Cumulative variance (%)	30.7	59.3	73.3	80.4	85.6	89.3
Ethyl butyrate		0.292	-0.321	**0.734**		
Ethyl 2-methylbutyrate	0.261	-0.379			**0.845**	
Ethyl isovalerate	0.286	**-0.621**	-0.384		0.515	
Ethyl hexanoate	0.415	0.436		0.358	-0.256	
Ethyl lactate		**-0.737**	0.530			
Ethyl octanoate		**0.920**				
Ethyl decanoate		**0.969**				
Isoamyl acetate	-0.410	**0.701**		0.463		
Hexyl acetate	**-0.612**	**0.723**				
2-Phenylethyl acetate	-0.468	**0.809**				
Isovaleric acid		-0.431	**0.801**		0.302	
Hexanoic acid			**0.938**			
Octanoic acid		**0.634**	-0.549			
Decanoic acid		**0.964**				
1-Hexanol	**-0.659**		**0.648**		-0.254	
trans-3-Hexen-1-ol	**-0.559**		**0.647**			
cis-3-Hexen-1-ol	**-0.923**					
Benzyl alcohol	**0.894**		0.344			
Linalool	-0.524					**0.764**
α-Terpineol	0.358	**-0.636**			0.579	
Citronellol		0.453				**0.767**
γ-Butyrolactone		**-0.766**	0.559			
γ-Nonalactone	**0.953**					
Methyl vanillate	**0.942**					
Ethyl vanillate	**0.861**		-0.312		0.298	
Acetovanillone	**0.862**	0.388				
4-Vinylguaiacol	**0.697**		-0.523			
4-Vinylphenol			**0.935**			
2-Phenylethanol		0.281	**0.754**		0.331	
1-Propanol		-0.491	0.309	**0.622**	-0.373	0.251
Isobutanol	-0.545			**-0.747**		
Isoamyl alcohols		-0.279	0.414	**-0.646**		

The bold numbers indicate the higher weight of each compound in each factor.

Table 4. Volatile compounds of red sparkling wines aged on lees for nine months and aged in bottle without lees for twelve months (T9+12) [a]

Compounds	**PM-C** [b]	**PM-DI**	**PM-D**	**M-C**	**M-SR**	**M-AR**
Ethyl butyrate	178 a	217 bc	167 a	203 b	229 c	212 bc
Ethyl 2-methylbutyrate	17 a	19 a	18 a	24 b	27 c	124 d
Ethyl isovalerate	27 a	27 a	25 a	36 b	39 b	65 c
Ethyl hexanoate	352 a	420 bc	443 c	405 b	475 d	475 d
Ethyl octanoate	232 a	266 b	268 b	258 a	321 c	263 b
Ethyl decanoate	37	36	40	36	37	38
Total ethyl esters	*844 a*	*985 b*	*1061 c*	*961 b*	*1128 d*	*1150 d*
Ethyl lactate*	69.0 a	91.8 d	79.9 bc	86.8 cd	75.0 ab	74.3 ab
Isoamyl acetate	828 c	1461 d	806 c	407 a	618 b	400 a
Hexyl acetate	11 b	13 c	8 a	nd [c]	nd	nd
2-Phenylethyl acetate	213 d	280 e	344 f	108 b	143 c	62 a
Total alcohol acetates	*1053 d*	*1754 e*	*1158 d*	*515 b*	*761 c*	*462 a*
Isovaleric acid	860 d	723 c	608 a	706 bc	659 ab	952 e
Hexanoic acid	2660 d	2216 b	1773 a	2648 d	2385 c	1858 a
Octanoic acid	3632 a	5217 c	6873 d	4928 c	5279 c	4402 b
Decanoic acid	212 a	296 c	292 c	209 a	254 b	298 c
Total acids	*7377 a*	*8437 c*	*9545 d*	*8491 c*	*8577 c*	*7511 b*
1-Hexanol	687 d	568 c	558 c	400 b	405 b	283 a
trans-3-hexen-1-ol	45 c	34 b	35 b	36 b	22 a	24 a
cis-3-hexen-1-ol	276 d	254 c	247 c	36 b	22 a	21 a
Total C6 alcohols	*1008 d*	*856 c*	*840 c*	*472 b*	*450 b*	*327 a*
Benzyl alcohol	107 a	120 ab	127 b	271 e	208 c	243 d
Linalool	4.3 b	5.5 d	4.9 c	3.2 a	2.7 a	4.2 b
α-Terpineol	1.1 a	2.1 b	1.9 b	3.5 d	3.0 c	4.1 e
Citronellol	3.2 a	5.5 b	3.4 a	3.0 a	3.3 a	3.1 a
γ-Butyrolactone*	8.2 b	9.7 c	6.6 a	11.3 d	9.5 c	8.5 b
γ-Nonalactone	3.6 ab	3.3 a	3.8 b	7.1 e	5.7 c	6.4 d
Methyl vanillate	2.0 a	1.7 a	2.0 a	12.4 b	12.2 b	13.1 b
Ethyl vanillate	25 a	31 ab	36 b	79 d	70 c	100 e
Acetovanillone	24 a	26 b	26 b	36 c	41 d	42 d
Total vanillin derivative	*50 a*	*59 b*	*65 b*	*128 c*	*123 c*	*156 d*
4-Vinylguaiacol	11 a	15 a	23 b	52 e	39 d	33 c
4-Vinylphenol	40 b	46 c	27 a	49 c	29 a	84 d
Total volatile phenols	*52 a*	*61 b*	*50 a*	*100 d*	*69 c*	*117 e*
2-Phenylethanol*	33.4 a	37.7 b	41.8 c	32.2 a	49.7 d	52.0 d
1-Propanol*	15.8 b	20.5 d	14.4 a	19.7 d	18.3 c	14.5 a

Isobutanol*	79.7 d	41.6 c	83.4 e	32.2 a	36.3 b	35.6 b
Isoamyl alcohols*	299 c	255 a	298 c	251 a	294 c	277 b
Total higher alcohols	*428 d*	*354 a*	*437 d*	*335 a*	*399 c*	*379 b*

[a] Mean values of three bottles and two elaborations/tanks by treatment (n=6) in µg/L except those marked with an asterisk * that are expressed in mg/L. Values with different letters in each compound indicate statistically significant differences at $p < 0.05$ and values without letters indicate no statistically significant differences

[b] PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized

[c] nd: not detected

Table 4. Volatile compounds of red sparkling wines aged on lees for nine months and aged in bottle without lees for twelve months (T9+12) [a]

Compounds	**PM-C** [b]	**PM-DI**	**PM-D**	**M-C**	**M-SR**	**M-AR**
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Ethyl isovalerate	27 a	27 a	25 a	36 b	39 b	65 c
Ethyl hexanoate	352 a	420 bc	443 c	405 b	475 d	475 d
Ethyl lactate*	69.0 a	91.8 d	79.9 bc	86.8 cd	75.0 ab	74.3 ab
Ethyl octanoate	232 a	266 b	268 b	258 a	321 c	263 b
Ethyl decanoate	37	36	40	36	37	38
Isoamyl acetate	828 c	1461 d	806 c	407 a	618 b	400 a
Hexyl acetate	11 b	13 c	8 a	nd [c]	nd	nd
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Hexanoic acid	2660 d	2216 b	1773 a	2648 d	2385 c	1858 a
Octanoic acid	3632 a	5217 c	6873 d	4928 c	5279 c	4402 b
Decanoic acid	212 a	296 c	292 c	209 a	254 b	298 c
1-Hexanol	687 d	568 c	558 c	400 b	405 b	283 a
trans-3-hexen-1-ol	45 c	34 b	35 b	36 b	22 a	24 a
cis-3-hexen-1-ol	276 d	254 c	247 c	36 b	22 a	21 a
Benzyl alcohol	107 a	120 ab	127 b	271 e	208 c	243 d
Linalool	4.3 b	5.5 d	4.9 c	3.2 a	2.7 a	4.2 b
α-Terpineol	1.1 a	2.1 b	1.9 b	3.5 d	3.0 c	4.1 e
Citronellol	3.2 a	5.5 b	3.4 a	3.0 a	3.3 a	3.1 a
γ-Butyrolactone*	8.2 b	9.7 c	6.6 a	11.3 d	9.5 c	8.5 b
γ-Nonalactone	3.6 ab	3.3 a	3.8 b	7.1 e	5.7 c	6.4 d
Methyl vanillate	2.0 a	1.7 a	2.0 a	12.4 b	12.2 b	13.1 b
Ethyl vanillate	25 a	31 ab	36 b	79 d	70 c	100 e
Acetovanillone	24 a	26 b	26 b	36 c	41 d	42 d
4-Vinylguaiacol	11 a	15 a	23 b	52 e	39 d	33 c
4-Vinylphenol	40 b	46 c	27 a	49 c	29 a	84 d
2-Phenylethanol*	33.4 a	37.7 b	41.8 c	32.2 a	49.7 d	52.0 d

1-Propanol*	15.8 b	20.5 d	14.4 a	19.7 d	18.3 c	14.5 a
Isobutanol*	79.7 d	41.6 c	83.4 e	32.2 a	36.3 b	35.6 b
Isoamyl alcohols*	299 c	255 a	298 c	251 a	294 c	277 b

[a] Mean values of three bottles and two elaborations/tanks by treatment (n=6) in µg/L except those marked with an asterisk * that are expressed in mg/L. Values with different letters in each compound indicate statistically significant differences at $p < 0.05$ and values without letters indicate no statistically significant differences

[b] PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized.

[c] nd: not detected

Table 5. Mosalux foam parameters of red sparkling wines aged on lees for nine months (T9), and aged on lees for nine months and aged in bottle without lees for twelve months (T9+12)[a]

			T9			
	PM-C[b]	PM-DI	PM-D	M-C	M-SR	M-AR
HM[c]	75 a, A	80 ab, A	90 b, A	72 a, A	80 ab, A	109 c, A
HS[c]	36 A	37 A	37 A	38 A	35 A	37 A
			T9+12			
	PM-C[b]	PM-DI	PM-D	M-C	M-SR	M-AR
HM[c]	105 B	116, B	113, B	108, B	108, B	109, A
HS[c]	40 c, B	43 d, B	39 bc, A	38 bc, A	35 a, A	37 ab, A

[a] Values with different lowercase letters in each attribute and ageing moment indicate statistically significant differences at $p < 0.05$ and values without letters indicate no statistically significant differences. Values with different capital letters in each attribute and treatment indicate statistically significant differences at $p < 0.05$ by effect of the ageing moment

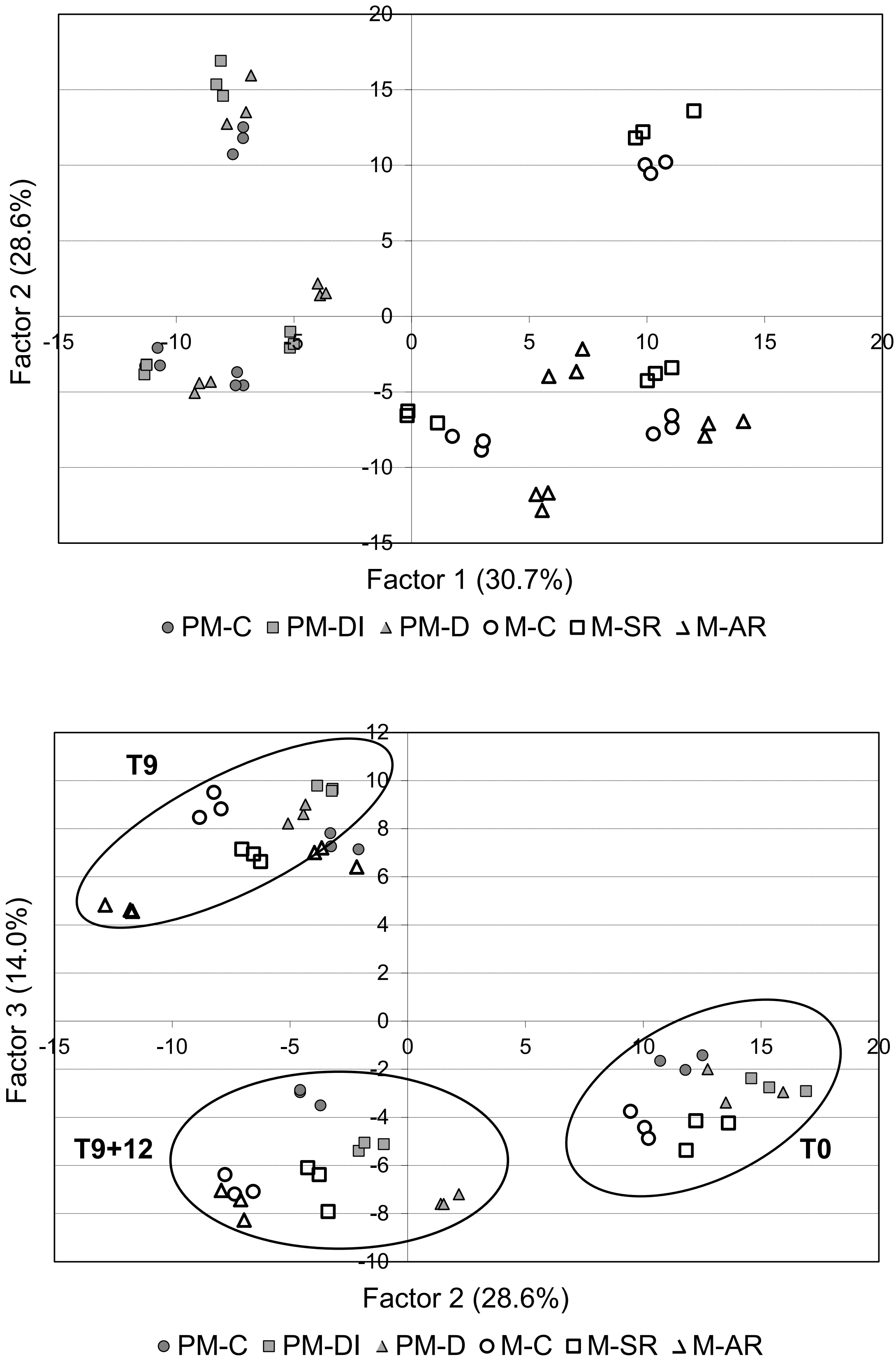
[b] PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized.

[c] HM: foam maximum height (mm); HS: foam stability height (mm)

FIGURE LEGENDS

Figure 1. Distribution of the base and red sparkling wines elaborated in the plane defined by (a) factors 1 and 2 and (b) factors 2 and 3. PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized.

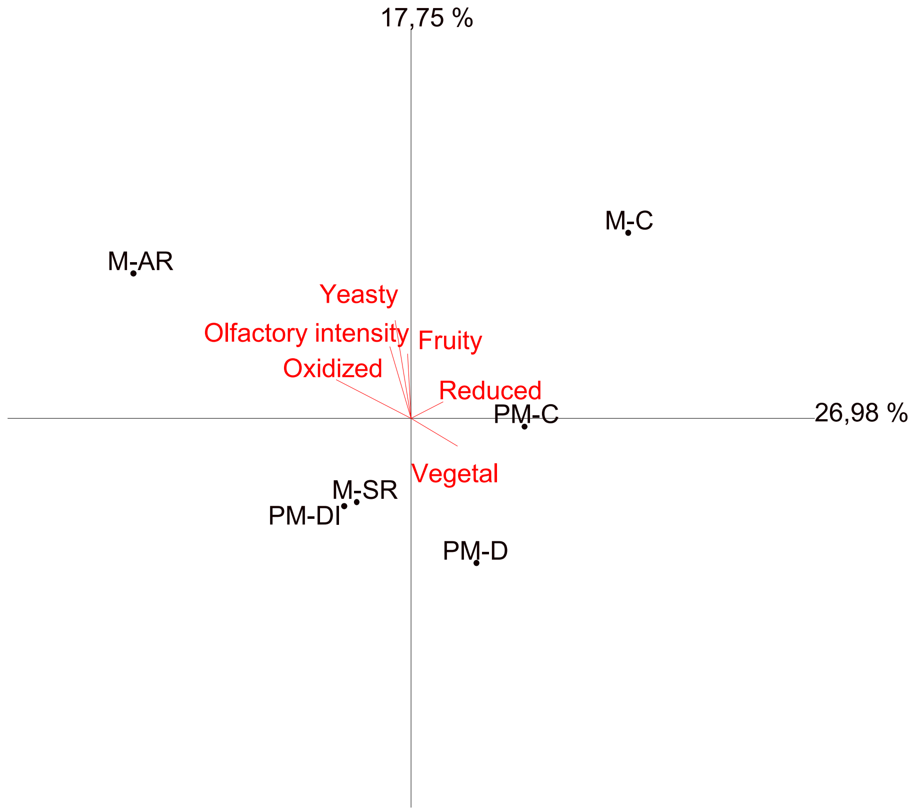
Figure 2. Generalized Procrustes analysis of the mean ratings for (a) olfactory phase and (b) for foam attributes in the different red sparkling wines aged on lees for nine months and aged in bottle without lees for twelve months (T9+12). PM: wines from premature grapes; M: wines from mature grapes; C: control wines; PM-DI: wines elaborated with pre-fermentative cold maceration with dry ice; PM-D: wines elaborated with delestage and partial removal of seeds; M-SR: wines elaborated with sugar reduction in must; M-AR: wines partially dealcoholized.


Factor 2 (28.6%)
Factor 1 (30.7%)
● PM-C ■ PM-DI ▲ PM-D ○ M-C □ M-SR △ M-AR
Factor 3 (14.0%)
Factor 2 (28.6%)
T9
T9+12
T0
● PM-C ■ PM-DI ▲ PM-D ○ M-C □ M-SR △ M-AR

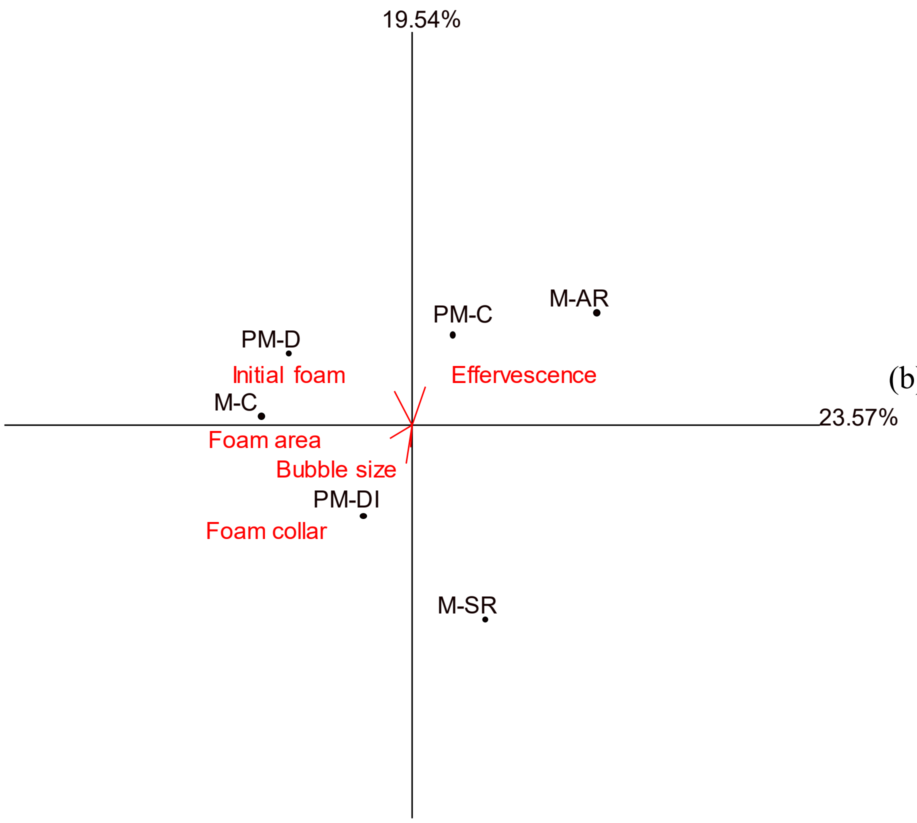
(a)

(b)

Figure 1


17,75 %
M-C
M-AR
Yeasty
Olfactory intensity
Fruity
Oxidized
Reduced
PM-C
26,98 %
Vegetal
M-SR
PM-DI
PM-D

(a)


19.54%
M-AR
PM-C
PM-D
Initial foam
Effervescence
M-C
23.57%
Foam area
Bubble size
PM-DI
Foam collar
M-SR

(b)

Figure 2