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# Optimization of Making Barrel-Fermented Dry Muscatel Wines

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The optimization of making barrel-fermented muscatel wines requires determining what type of must clarification is most suitable for the quality of the wine, as well as what type of barrel will yield the most acceptable wines. This is achieved by adding pectolytic enzymes to clarify part of the muscatel must statically; the rest is clarified by vacuum filtration. The musts obtained are fermented in French and American oak barrels and, once fermentation has ceased, they are kept with their lees for 2 months, with periodic stirring. Eleven conventional parameters and 31 volatile compounds were quantified, and a sensory analysis of the wines was produced, which led us to conclude that static clarification with pectolytic enzymes from the muscatel musts produces the best-structured wines and the larger content of higher alcohols, esters, and terpenic compounds. The wines fermented in American oak barrels received the highest overall marks, which may be due to the greater aromatic complexity given off by the compounds in the wood.

**KEYWORDS: Barrel fermentation; clarification; volatile compounds; white wine; muscatel**

## INTRODUCTION

The fermentation of white wines in small oak barrels and the subsequent aging are traditional practices (*1*, *2*) that are more and more commonly being used for making both aromatic wines and other, more neutral varieties.

The extraction of compounds from the wood depends on the type of wood used, its structure, origin, drying process, years of use, and level of toasting (*3*−*5*). The compounds extracted from the wood go through various transformations during the fermentation and aging, when enzyme reaction reduces the aldehydes to alcohols (*6*, *7*).

The wines obtained in this way retain varietal aromas together with the aromas that come from fermentation, wood extracts, and extracts given off during the lees aging. The autolysis of the yeasts, which occurs during lees aging, produces volatile compounds, especially long-chain esters due to slow esterification of fatty acids freed by the yeast. However, yeast cell wall degradation frees intracellular hydrolytic enzymes, which release terpene molecules from their glycosidic precursors (*8*, *9*). When wine is in contact with the lees, it becomes enriched in polysaccharides from the yeasts, especially in mannoproteins. This is of great interest in wine-making as the phenol compounds from the wood are fixed and cause the wine to clarify naturally, thus limiting the astringency of the tannins from the barrel. Stirring the lees intensifies autolysis and strengthens the intensity and persistence of the aroma (*10*, *11*).

Interaction between the fermentation metabolites, the compounds extracted from the wood, and those coming from yeast autolysis gives rise to complex balances of aromas (*12*).

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Muscatel grapes are the basis for the famous sweet wines and liqueurs from the Valencia region. These grapes are also used for making wines that are dry, fresh, light, fruity, and strongly aromatic. The authenticity of the muscatels is linked to the presence of terpenic compounds; the aromatic potential of these compounds depends on glycosylate compounds from terpenes in the grape.

The pulp from the muscatel grape is very rich in pectin, and so its musts contain abundant lees, which must be reduced by clarification. Large amounts of lees during fermentation produce more higher alcohols and volatile fatty acids, which are compounds detrimental to wine quality. Conversely, reducing the amount of lipids in the must stimulates the yeast to synthesize esters, which improve the quality of the wines (*13*). However, overclarification eliminates the fine lees, which contain a significant amount of terpenic compounds. This would have a negative impact on the fermentation process (*14*). The pectolytic enzymes are of great use in breaking down the pectin, extracting a larger amount of must, and freeing aromatic precursors.

A well-structured muscatel depends not only on terpenic compounds but also on aromatic compounds formed during fermentation (*15*). Barrel fermentation and subsequent lees aging may be an alternative in making dry muscatel wines because it produces new aromatic compounds giving a greater complexity to the varietal aromas. It also produces polysaccharides that contribute to the texture and full body of the wines.

There has been a proposal to make dry muscatel wines in barrels with the aim of expanding the product line of muscatels in the Valencia region. The optimization of this process requires determining the type of clarification that is best for preserving

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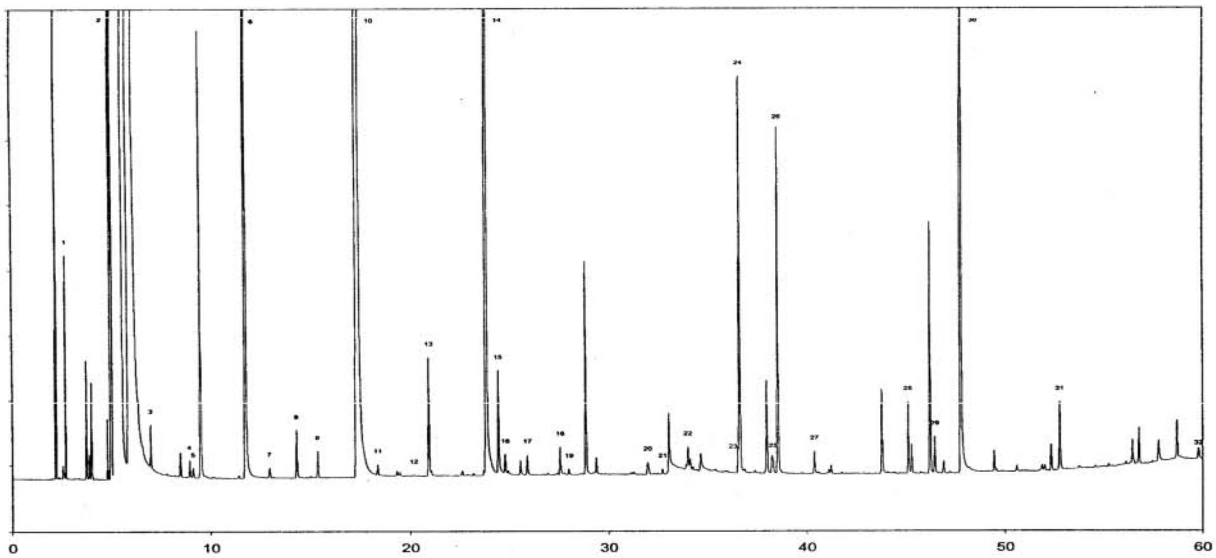


**Figure 1.** Chromatogram of minor components of muscatel wine fermented in an American oak barrel.

the varietal aromas and reduces the percentage of lees considerably also the most appropriate type of wood in which to make these wines.

## MATERIALS AND METHODS

The experiment was carried out using the muscatel grape proceeding from the Muscatel Sub-zone Appellation Controllée Valencia. Once the grapes were pressed and cooled to 7−8 °C, 5 g/hL of $SO_2$ was added to the must, which was divided into two parts. One part was subjected to vacuum clarification with soil containing medium-size diatom granules, and the other was subjected to static clarification by the addition of pectolytic enzymes.

Both types of musts were placed in new 225 L barrels made of French oak from Nevers (*Quercus petraea*) and American oak from Kentucky (*Quercus alba*) made by Magreñán (La Rioja, Spain) barrel makers. All of the barrels were medium toasted.

The experiment was carried out twice so that there were two musts with vacuum clarification and two others that had static clarification. Each must was fermented in two French oak barrels and two American oak barrels, making 16 wines in all.

The musts in each oak barrel were inoculated with 20 g/hL of *Saccharomyces cerevisiae*. The fermentation took place at 20−22 °C for 8 days. Malolactic fermentation did not take place because the wines were low in acidity with minimal amounts of malic acid. The yeast lees were left in with the wines for 2 months and stirred every 10 days. The wine was then bottled after 40 mg/L of $SO_2$ had been added.

The physical−chemical analyses were performed according to the official methods established by the *Bulletin de l'Office Internationale de la Vigne et du Vin* (*16*), classified by density, ethanol, pH, sugars, total and volatile acidity, total sulfurous content, color intensity, and total phenols.

Volatile components were quantified using the chromatography technique, in the gaseous phase with an HP-5890 chromatograph equipped with an ionization flame detector using nitrogen as a carrier gas.

Isobutyl, isopropyl, and isoamyl alcohols, acetaldehyde, ethyl and methyl acetates, methanol, 2-butanol, and 1-propanol were determined by the direct injection of 1 $\mu$L of wine containing 4-methyl-2-pentanol as an internal standard, in a Carbowax 1500 capillary column (length = 4 m, internal diameter = 0.32 cm) over Cromosorb to 15%, with 80−100 meshes (*17*).

Minor wine components were determined by making a prior extraction. 1-Butanol, *cis*-3-hexenol, 2-phenylethanol, isoamyl acetate, isobutyl acetate, hexyl acetate, ethyl propionate, ethyl butyrate, ethyl lactate, ethyl octanoate, ethyl decanoate, guaiacol, syringol, furfural, 5-methylfurfural, furfuryl alcohol, vanillin, *cis*-$\beta$-methyl-$\gamma$-octalactone, *trans*-$\beta$-methyl-$\gamma$-octalactone, linalool, $\alpha$-terpineol, citronellol, nerol, and geranioI were extracted in a continuous liquid−liquid extractor using organic solvents (diethyl ether and *n*-pentane 2:1 for 24 h). For an internal standard 1 mL of 2-octanol was added to 500 mL of wine. From this sample, 1 $\mu$L of the extract obtained after concentration by evaporation was injected in an HP-INNOWax (cross-linked polyethylene glycol) capillary column (60 m long and 0.25 mm internal diameter) (*18*, *19*).

Compound quantification was based on the internal standard method. The efficacy of the method was verified from the analysis performed on standard solutions of the components and with the aid of an HP-5979 mass spectrophotometer associated with the chromatograph. **Figure 1** shows a chromatogram for minor wine components quantified by the internal standard method. **Figure 2** shows a mass spectrum of one of the compounds analyzed. The variance of the method was determined by the analysis of three replicates of one sample.

A panel of 20 expert wine tasters was assembled for a sensory evaluation of the wines. They used a standard tasting chamber and standard glasses. The properties graded were color, quality and intensity of aroma, quality and intensity of taste, and overall evaluation. A points system of positive numbers was used.

## RESULTS AND DISCUSSION

The compounds analyzed were grouped according to chemical affinity in conventional parameters, volatile compounds formed in the fermentation, terpenes, and volatile compounds extracted from the wood. The study of how to optimize the barrel-fermented dry muscatel wine-making process was carried out by comparing the two types of clarification. It was also performed using two types of barrels (made from either American or French oak). Finally, the corresponding ANOVA were used.

**Table 1** shows the effects of clarification type and type of oak barrel according to common parameters of the wines, as well as averages for the physical−chemical analyses made on the wines. The type of clarification does not significantly affect the conventional parameters with the exception of the sugars, which are slightly higher in the vacuum filtration wines. This may be due to the drastic elimination of lees that occurs with this type of clarification. This elimination has a negative effect on fermentation development and may hinder the total consumption of sugars (*14*, *20*).

Fermenting muscatel musts in American or French oak barrels generally does not influence the physical−chemical parameters

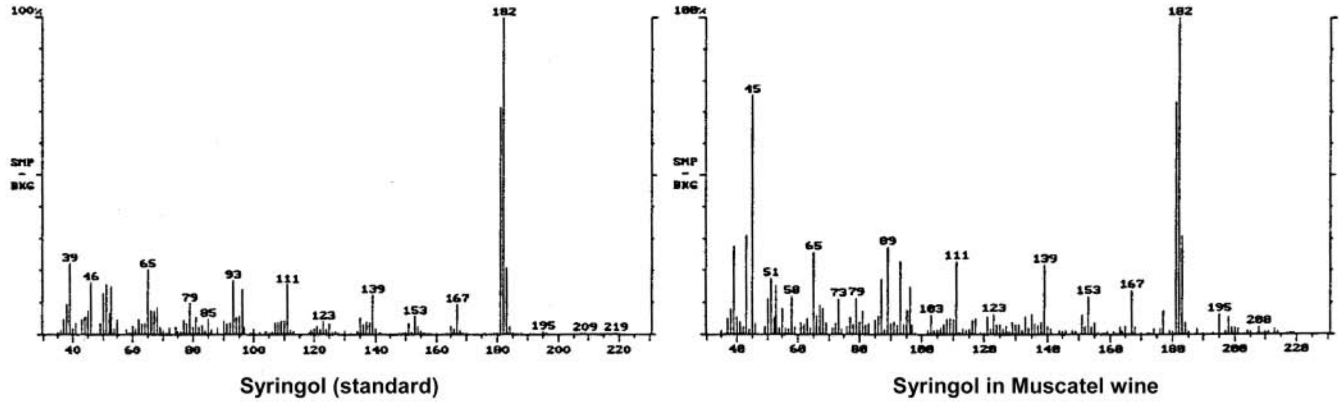


**Figure 2.** Mass spectrum of syringol standard and syringol in muscatel wine fermented in an American oak barrel.

**Table 1.** Influence of the Type of Clarification and Type of Oak Barrel on the Average Value of the Conventional Parameters of Muscatel Wines Fermented in Barrels

| | type of clarification[a] | | type of oak[b] | |
|---|---|---|---|---|
| | filter | static | American | French |
| density (g/L) | 0.991 | 0.992 | 0.991 | 0.992 |
| ethanol (% vol) | 12.93 | 13.05 | 13.12 | 13.08 |
| total acidity (g/L tartaric acid) | 3.50 | 3.47 | 3.54 | 3.43 |
| volatile acidity (g/L acetic acid) | 0.63 | 0.55 | 0.67 | 0.62 |
| pH | 3.78 | 3.81 | 3.75 | 3.83 |
| $SO_2$ total (mg/L) | 83.2 | 92.1 | 90.4 | 84.8 |
| sugar (g/L) | 2.02* | 1.37* | 1.86 | 1.81 |
| colorant intensity | 0.21 | 0.23 | 0.21 | 0.24 |
| acetaldehyde (mg/L) | 14.35 | 18.65 | 17.4 | 15.6 |
| methanol (mg/L) | 51.7 | 56.3 | 53.5 | 55.9 |
| total phenols (mg/L gallic acid) | 331 | 359 | 238** | 399** |

[a] One asterisk (*) indicates significant differences between the two types of clarification ($p < 0.01$). [b] Two asterisks (**) indicate significant differences between the two types of wood ($p < 0.01$).

studied, with only the total amount of phenols being affected significantly. Because we are dealing with white wines, a very significant percentage of the phenols present in the wines came from the wood and were extracted during the fermentation process. Extraction level depends on the initial composition of the must, the pH and level of alcohol reached during fermentation, and the characteristics of the wood. In the experiment, the initial characteristics of the must, the physical−chemical parameters of the wines, and the toasting levels of the wood were similar. The only differences were in the structure and composition of the two types of oak (French and American) used in making the barrels. The wines fermented in French oak had a significantly higher total phenol content due to the higher concentration of phenols that this type of wood contains, as has been observed by other authors (*3*, *5*).

**Table 2** shows the effects and average concentrations of higher alcohols, acetates, and ethyl esters according to the type of clarification and the type of barrel. The influence of the type of clarification on the higher alcohols studied is significant in the concentration of isoamyl alcohol and in the total concentration of higher alcohols, which is greater in wines made by static clarification. The presence of larger amounts of higher alcohols in wines made from musts with a higher quantity of lees is consistent with results obtained by other authors (*13*, *21*−*23*).

With regard to the influence of the clarification procedure on esters concentration, the corresponding ANOVA showed

**Table 2.** Influence of the Type of Clarification and Type of Oak Barrel on the Average Value (in Milligrams per Liter) of the Higher Alcohols and Esters Analyzed in Muscatel Wines Fermented in Barrels

| | type of clarification[a] | | type of oak[b] | |
|---|---|---|---|---|
| | filter | static | American | French |
| 2-butanol | 1.13 | 0.46 | 1.04 | 0.56 |
| 1-propanol | 45.91 | 43.53 | 46.05 | 43.08 |
| isobutyl alcohol | 86.2 | 95.71 | 99.42** | 88.50** |
| 1-butanol | 4.45 | 3.56 | 4.20 | 3.81 |
| isoamyl alcohol | 145.5* | 187.8* | 175.9** | 164.7** |
| isopropyl alcohol | 1.39 | 1.38 | 1.48** | 1.33** |
| *cis*-3-hexenol | 0.06 | 0.06 | 0.06 | 0.06 |
| 2-phenylethanol | 24.76 | 26.04 | 27.74** | 23.05** |
| Σ higher alcohols | 309.4* | 358.5* | 355.9 | 328 |
| methyl acetate | 10.07 | 10.59 | 11.82 | 8.84 |
| ethyl acetate | 41.43 | 36.54 | 42.28 | 38.60 |
| isoamyl acetate | 2.21* | 2.46* | 2.39 | 2.28 |
| isobutyl acetate | 0.24* | 0.31* | 0.28 | 0.26 |
| hexyl acetate | 0.33 | 0.36 | 0.38 | 0.32 |
| ethyl propionate | 0.81* | 0.95* | 0.93** | 0.73** |
| ethyl butyrate | 0.35 | 0.27 | 0.34 | 0.33 |
| ethyl lactate | 8.49 | 8.30 | 8.60 | 8.39 |
| ethyl octanoate | 0.62 | 0.52 | 0.56 | 0.48 |
| ethyl decanoate | 0.19 | 0.16 | 0.19 | 0.15 |

[a] One asterisk (*) indicates significant differences between the two types of clarification ($p < 0.01$). [b] Two asterisks (**) indicate significant differences between the two types of wood ($p < 0.01$).

significant differences for the ethyl propionates and isobutyl and isoamyl acetates, which are found in large amounts when the must is subjected to static clarification. The formation of acetates depends on the content of their corresponding higher alcohols in the wines and their availability. However, fermentation conditions are also very important in the formation of esters. Also important is whether the clarification of the must allows for the presence of residual lees. In the wines subjected to static clarification, a greater concentration of higher alcohols and the presence of fine lees enable larger amounts of esters to form (*24*, *25*).

As for the influence of the type of oak used for the barrel, the wines fermented in American oak barrels showed significantly higher values for ethyl propionate, 2-phenylethanol, and isobutyl, isoamyl, and isopropyl alcohol. On the other hand, the total amount of higher alcohol present in the wines fermented in American oak barrels does not significantly differ from the amount found in wines fermented in French oak barrels. Therefore, the effect of the barrel on the concentration of esters in the wine is very low.

**Table 3.** Influence of the Type of Clarification and Type of Oak Barrel on the Average Values (in Micrograms per Liter) of the Terpenic Compounds Analyzed in the Muscatel Wines Fermented in Barrels

| | type of clarification[a] | | type of oak[b] | |
|---|---|---|---|---|
| | filter | static | American | French |
| linalool | 250* | 326* | 286 | 256 |
| α-terpineol | 89* | 130* | 105 | 122 |
| citronellol | 73* | 87* | 91 | 79 |
| nerol | 116* | 124* | 118 | 105 |
| geraniol | 71 | 76 | 80 | 77 |

[a] One asterisk (*) indicates significant differences between the two types of clarification ($p < 0.01$). [b] Two asterisks (**) indicate significant differences between the two types of wood ($p < 0.01$).

**Table 4.** Influence of the Type of Clarification and Type of Oak Barrel on the Average Values (in Micrograms per Liter) of the Volatile Compounds Extracted from the Wood in the Muscatel Wines Fermented in Barrels

| | type of clarification[a] | | type of oak[b] | |
|---|---|---|---|---|
| | filter | static | American | French |
| guaiacol | 48.8 | 52.3 | 56.4 | 47.2 |
| syringol | 29.2 | 30.8 | 32.4 | 26.9 |
| furfural | 903 | 987 | 932 | 971 |
| 5-methylfurfural | 412 | 463 | 426 | 445 |
| furfuryl alcohol | 1.740 | 1.852 | 1.688 | 1.804 |
| vanillin | 355 | 341 | 395** | 301** |
| *cis*-β-methyl-γ-octalactone | 142 | 159 | 193** | 102** |
| *trans*-β-methyl-γ-octalactone | 76.5 | 61.4 | 58.3** | 84.1** |

[a] One asterisk (*) indicates significant differences between the two types of clarification ($p < 0.01$). [b] Two asterisks (**) indicate significant differences between the type types of wood ($p < 0.01$).

**Table 5.** Influence of the Type of Clarification and Type of Oak Barrel on the Average Values of Attributes Considered in the Sensory Analysis of Muscatel Wines Fermented in barrels

| factor: | | type of clarification[a] | | type of oak[b] | |
|---|---|---|---|---|---|
| level: | | filter | static | American | French |
| color | | 6.94 | 7.19 | 7.11 | 7.02 |
| aroma | quality | 4.82* | 7.39* | 6.21 | 5.87 |
| | intensity | 5.50* | 6.46* | 5.99 | 5.93 |
| taste | quality | 4.65* | 6.91* | 6.02 | 5.67 |
| | intensity | 5.48* | 6.09* | 5.92 | 5.64 |
| overall evaluation | | 4.91* | 6.63* | 6.64** | 5.22** |

[a] One asterisk (*) indicates significant differences between the two types of clarification ($p < 0.01$). [b] Two asterisks (**) indicate significant differences between the two types of wood ($p < 0.01$).

Wine characteristics are affected by the presence of higher alcohols or their esters. They contribute favorably to the quality of the wines, but when the total amount of higher alcohols in the wine passes a certain level (∼500 mg/L), structural defects may result (*26*). The greatest concentration of higher alcohols and ethyl propionate was found in wines made from musts subjected to static clarification and in the wines fermented in American oak barrels. This can increase the quality of the wines given that the concentrations are within beneficial levels for aroma quality.

**Table 3** shows the effects of clarification type and type of oak barrel on terpenic compounds of the wines as well as the average values of the terpenes analyzed. The wines subjected to static clarification showed significantly greater amounts of all terpenic compounds with the exception of geraniol, which, although present in higher amounts, was not statistically significant. The presence of terpenic compounds and their precursors in fine lees that remained in the must after static clarification, in addition to the presence of pectolytic enzymes which free terpenes after their precursors, produced a heavier concentration of these compounds in the wines (*15***,** *27*). Numerous studies have shown that muscatel grapes should not be overclarified, as the fine part of the lees contains a significant part of the varietal aroma, which it is better to retain during fermentation. The type of oak from which the barrels were made did not influence the concentration of terpenic compounds.

**Table 4** shows the effects of clarification type and type of oak barrel on the concentration of volatile compounds extracted from the wood. The concentration of volatile phenols and furanic and lactonic compounds in wines that have been in contact with the wood depends on the botanical and geographical origin of the wood, how the barrel was made, and the toasting level of the wood. The type of clarification had no significant effect on the concentration of these volatile compounds. Conversely, the type of barrel will affect all of the volatile compounds to a greater or lesser degree. Guaiacol and syringol will be present in greater quantities in wines fermented in American barrels, but the difference is insignificant when compared to French oak. Likewise, wines fermented in French oak barrels showed higher concentrations of furfural, methylfurfural, and furfuryl alcohol. However, differences were not significant. Vanillin is significantly greater in wines fermented in American oak, and its presence can be detected organoleptically, because it is concentrated at levels close to the threshold of perception (*26*).

Furfuraldehydes are formed during the toasting process and are reduced to furanic alcohols by enzymes during fermentation and aging in the barrel. This reduction produces a less structured wine and forms favorable aromatic compounds, which increase the quality of the wines. Vanillin also undergoes reduction by an enzymatic process becoming vanillic alcohol, which has much lower aromatic qualities (*6*, *7*, *26*).

β-Methyl-γ-octalactones are compounds specific to wood. Cis isomers are found in significantly greater amounts in wines fermented in American oak barrels, and their concentration is well over the threshold of perception. Wines fermented in French oak barrels showed higher concentrations of *trans*-β-methyl-γ-octalactone than of the corresponding cis isomer. Nevertheless, levels for the trans isomer were below its sensory threshold, which is much higher than the one for the cis isomer. Some authors use the *cis*/*trans*-β-methyl-γ-octalactone ratio to characterize wines made from French or American barrels. The American barrels give a higher ratio, which can be double that of French oak barrels (*5*, *28*).

**Table 5** shows the effects of clarification type and type of oak barrel on sensory perceptions to the wines as well as the average values of the attributes considered. Except for the color, the wines that had had their lees separated statically obtained significantly higher marks than those which had had their lees separated by filtration. The greatest concentration of higher alcohols, esters, and terpenic compounds found in the wines made from musts subjected to static clarification justifies higher marks for aroma, taste, and overall evaluation. According to Torres (*15*), the acceptability of muscatels depends on the concentration of terpenes and certain esters such as isoamylic and hexyl acetates, which are found in greater concentrations in wines undergoing static clarification.

The differences in color, aroma, and taste of the wines fermented in American and French oak barrels are minimal, but the overall evaluation for wines fermented in American oak barrels is significantly higher. The slightly higher concentration of higher alcohols and the vanilla and coconut aromas given off by the vanillin and the *cis*-$\beta$-methyl-$\gamma$-octalactone may contribute to a greater overall complexity in these wines and thus a higher sensorial evaluation.

From the chemical composition of the elaborated wines as well as their sensorial analysis described above, it can be concluded that static clarification through the addition of pectolytic enzymes produces the most highly rated barrel-fermented muscatel wines that contain the greatest amounts of higher alcohols, esters, and terpenic compounds.

Wines fermented in American oak barrels contained higher concentrations of volatile compounds than wines from French oak barrels. Volatile phenols follow the same trend as total volatile compounds. However, total content of phenolic compounds is higher in wines from French oak barrels. This apparent contradiction could be explained if French oak barrels would release higher amounts of nonvolatile phenols (*29*) and/or volatile phenols not considered in this work (eugenol, phenol aldehydes, etc.) (*30*) that might influence negatively the sensorial evaluation of these wines. Muscatel wines fermented in American oak barrels score higher in sensorial evaluations because of their superior gustatory and olfactory balance compared with the wines fermented in French oak barrels. In addition, the lower price of American oak barrels will reduce process cost.

The combination of static clarification with pectolytic enzymes, fermentation in American oak barrels, and subsequent lees aging with periodic stirring produces muscatel wines that are harmonious, balanced, and of a high aromatic complexity and presents a real alternative in the fermentation of muscatel grapes.

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