Sensory characteristics changes of red Grenache wines submitted to different oxygen exposures pre and post bottling

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ABSTRACT

It is widely accepted that oxygen contributes to wine development by impacting its colour, aromatic bouquet, and mouth-feel properties. The wine industry can now also take advantage of engineered solutions to deliver known amounts of oxygen into bottles through the closures. This study was aimed at monitoring the influence of oxygen pick-up, before (micro-oxygenation, Mox) and after (nano-oxygenation) bottling, on wine sensory evolution. Red Grenache wines were prepared either by flash release (FR) or traditional soaking (Trad) and with or without Mox during elevage (FR + noMox, FR + Mox, Trad + noMox, Trad + Mox). The rate of nano oxygenation was controlled by combining consistent oxygen transfer rate (OTR) closures and different oxygen controlled storage conditions. Wine sensory characteristics were analyzed by sensory profile, at bottling (T0) and after 5 and 10 months of ageing, by a panel of trained judges. Effects of winemaking techniques and OTR were analyzed by multivariate analysis (principal component analysis and agglomerative hierarchical clustering) and analysis of variance. Results showed that, at bottling, Trad wines were perceived more animal and FR wines more bitter and astringent. Mox wines showed more orange shade. At 5 and 10 months, visual and olfactory differences were observed according to the OTR levels: modalities with higher oxygen ingress were darker and fruitier but also perceived significantly less animal than modalities with lower oxygen. Along the 10 months of ageing, the influence of OTR became more important as shown by increased significance levels of the observed differences. As the mouth-feel properties of the wines were mainly dictated by winemaking techniques, OTR had only little impact on “in mouth” attributes.

1. Introduction

It is widely accepted that oxygen contributes to wine development by impacting its colour, aroma, and mouth-feel properties. Oxygen management represents a major challenge in oenology as both excessive exposure and excessive protection lead to sensory defects.

Oxygen is introduced, in uncontrolled way, at various stages of winemaking, especially at pressing, during pumping over and other wine transfer operations, and at bottling [1]. Slow oxygen transfer also takes place during barrel ageing, contributing to colour stabilisation and astringency reduction processes associated with this practice. A mild oxygenation process, referred to as micro-oxygenation, has been proposed to simulate in a tank the continuous low oxygen uptake taking place in barrels [2,3]. This technique has become common practice to improve the quality of red wines, enhance colour intensity and stabilise wine pigments. In bottle, oxygen exposure is usually low but can be quite variable depending on the type of closure. Cork to cork variations in oxygen permeability are major sources of bottle to bottle variations [4,5]. Closure trials using cork, synthetic closures and screw caps have shown that sensory and chemical changes taking place during wine ageing are influenced by the type of stopper [6,7]. This has been attributed to differences in oxygen transfer rate through the closure [8,9]. The wine industry can now also take advantage of engineered solutions to deliver known and reproducible amounts of oxygen into bottles through the closures, that can be manufactured with specific and controlled oxygen transfer rate (OTR).

The aim of this study was to monitor the influence of oxygen exposure, before (micro-oxygenation, Mox) and after (nano-oxygenation) bottling, on wine sensory evolution. The rate of nano-oxygenation was controlled by combining consistent OTR...
closures and different controlled oxygen storage conditions. Since oxygen consumption is known to be initiated mostly by phenolic compounds [10,11], the same experiments were performed on wines differing by their phenolic content, obtained from the same grapes either by flash release (FR, higher phenolic content) [12] or by traditional soaking (Trad, lower phenolic content).

2. Experimental

2.1. Wine sample

2.1.1. Material

Ten thousand kilograms of grapes from *Vitis Vinifera* var. Grenache (2007), grown at INRA Pech Rouge Experimental Unit (Gruissan, Southern France) were harvested on two plots at commercial maturity in September 2007. Grapes from the first plot (22° Brix, pH = 3.6) were used for traditional winemaking and those from the second plot (25° Brix, pH = 3.7) were used for the flash release trial.

2.1.2. Winemaking trials

Two wines were prepared by traditional soaking (Trad) and flash release (FR), respectively. Each of these two wines was then divided in two batches submitted or not to micro-oxygenation (Mox/noMox), yielding four wines in total: FR + noMox, FR + Mox, Trad + noMox, Trad + Mox.

2.1.2.1. Preparation of traditional soaking trials. All the grapes harvested were destemmed and crushed using a destemmer-crusher.

2.1.2.2. Preparation of flash release trials. The treatment by FR consisted in destemming and crushing the grapes, heating them at 95°C during 6 min with biological vapour, and then submitting them to a strong vacuum (pressure closed to 60 hPa).

2.1.2.3. Fermentations. For both traditional soaking and flash release trial, the musts were distributed into two 50 hL fermentation stainless steel tanks equipped with temperature control, added with sulphite (5 g hL\(^{-1}\)) and with 500 g yeast (LB Rouge, Lallemand, 10 g hL\(^{-1}\)), and fermented to dryness. The cap was punched down daily to ensure mixture of the marc with the fermenting liquid phase. A supplementation of nitrogen nutrition was done (20 g hL\(^{-1}\) diammonium phosphate) after a decrease of 30 points of density. After 8 days of maceration, when alcoholic fermentation was finished (controlled by sugar analysis), the wines were racked (O\(_2\) pickup: 0.3 ppm), transferred to 20 hL tanks and added with lactic bacteria (Lalvin VP41, Lallemand) to induce malolactic fermentation. The malolactic fermentation was followed by paper chromatography. At the end of the malolactic fermentation, the wines were racked into other 20 hL stainless steel tanks and sulphites were added (3 g hL\(^{-1}\)).

2.1.2.4. Oxygen pick-up measurements. The total oxygen quantities introduced in the wines during each winemaking operation before bottling were measured by an orbisphere probe [13] (see Supplementary Electronic Material Table S1).

2.1.2.5. Micro-oxygenation (Mox) trials. Each of the wines was transferred from the 20 hL tank into four 2.7 hL tanks, adapted to perform Mox with a height of 3 m and a surface of wine of 0.09 m\(^2\). Mox was performed with a 10-channels Oenodev system, at 5 mg O\(_2\) L\(^{-1}\) month\(^{-1}\) during 3 weeks.

The noMox modalities were stored in the same cellar in the same kind of tanks.

2.1.2.6. Enological analyses. Free and total SO\(_2\) data, pH values, and alcohol percentage were obtained by Laboratoires Dubernet (Narbonne, France). Analyses were performed on three bottles of each wine modality and the mean of triplicate values are given in Table 1.

2.1.3. Bottling, closures and storage environment

The wines were bottled in 375 mL glass bottles (Saint-Gobain bordelaise 39), instead of 750 mL, in order to amplify the oxidation phenomena. Each of the four wines was divided in 4 batches in order to obtain 4 OTR conditions: one batch was closed with Nomacorc Light stoppers and stored in ambient air (21% oxygen). The three remaining batches were closed with Nomacorc Classic stoppers and stored respectively in ambient air (21% oxygen) and in stainless steel drums where oxygen levels were kept constant at either 4% oxygen or 0% oxygen. All the wines were stored in the same closed room at a constant temperature (23°C). The OTR were calculated using Fibox 3 trace fiber optic oxygen meter (Pre-Sens Precision Sensing GmbH, Regensburg, Germany) [14]. This luminescence-based technology is a very easy to use technology allowing non-invasive and non-destructive measurements of dissolved oxygen given by the oxygen sensor glued into the glass bottle prior to filling with wine and the head-space oxygen by a second sensor glued on the upper part of each bottle [15]. The OTR mean values of five replicates, along with the wines codes and physical characteristics of the stoppers, are given in Table 2.

2.1.3.1. Colour measurements. Spectrophotometric measurements were performed using a UV mc2 spectrophotometer (Safas) as previously described [3,16]. Colour intensity (CI) was calculated as the sum of absorbances at 420, 520 and 620 nm. These absorbance values were measured directly in a 1-mm light path cell and converted to absorbance values (A420, A520, A620) with a 10-mm light path. These absorbance values were used to determine the oxidation phenomenon. Each of the four wines was divided in 4 batches stored in the same cellar in the same kind of tanks.

Table 1

<table>
<thead>
<tr>
<th></th>
<th>Trad + Mox</th>
<th>Trad + noMox</th>
<th>FR + Mox</th>
<th>FR + noMox</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alcohol %</td>
<td>13.20 ± 0.01</td>
<td>12.98 ± 0.04</td>
<td>14.73 ± 0.0</td>
<td>14.64 ± 0.01</td>
</tr>
<tr>
<td>pH</td>
<td>3.66 ± 0.01</td>
<td>3.65 ± 0.01</td>
<td>3.78 ± 0.0</td>
<td>3.78 ± 0.01</td>
</tr>
<tr>
<td>Free SO(_2) (mg L(^{-1}))</td>
<td>41.33 ± 0.58</td>
<td>38.00 ± 0.00</td>
<td>41.00 ± 0.00</td>
<td>35.33 ± 0.58</td>
</tr>
<tr>
<td>Total SO(_2) (mg L(^{-1}))</td>
<td>93.67 ± 0.58</td>
<td>72.33 ± 0.53</td>
<td>74.33 ± 0.58</td>
<td>60.33 ± 0.58</td>
</tr>
<tr>
<td>TPI (A280nm)</td>
<td>34.41 ± 1.68</td>
<td>32.58 ± 3.00</td>
<td>43.85 ± 2.77</td>
<td>43.33 ± 2.15</td>
</tr>
<tr>
<td>CI</td>
<td>7.44 ± 0.04</td>
<td>7.57 ± 0.05</td>
<td>7.10 ± 0.02</td>
<td>7.38 ± 0.02</td>
</tr>
<tr>
<td>Clcor</td>
<td>11.75 ± 0.26</td>
<td>11.67 ± 0.17</td>
<td>10.87 ± 0.14</td>
<td>10.79 ± 0.09</td>
</tr>
<tr>
<td>L(^*)</td>
<td>77.90 ± 0.08</td>
<td>77.23 ± 0.23</td>
<td>79.38 ± 0.09</td>
<td>78.14 ± 0.11</td>
</tr>
<tr>
<td>a(^*)</td>
<td>22.51 ± 0.17</td>
<td>24.13 ± 0.45</td>
<td>19.21 ± 0.17</td>
<td>21.41 ± 0.31</td>
</tr>
<tr>
<td>b(^*)</td>
<td>1.32 ± 0.21</td>
<td>0.23 ± 0.29</td>
<td>3.29 ± 0.15</td>
<td>1.52 ± 0.20</td>
</tr>
<tr>
<td>H(^*)</td>
<td>3.35 ± 0.55</td>
<td>0.92 ± 0.48</td>
<td>4.08 ± 0.59</td>
<td>9.71 ± 0.52</td>
</tr>
</tbody>
</table>


Table 2
Wine codes, winemaking processes, synthetic stoppers used, % oxygen storage environment and calculated OTR.

<table>
<thead>
<tr>
<th>Wine code</th>
<th>Winemaking process</th>
<th>Nomacorc synthetic stopper</th>
<th>% Oxygen in storage environment</th>
<th>OTR (µg O₂ bottle⁻¹ day⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trad + Mox L</td>
<td>Trad + Mox</td>
<td>Light</td>
<td>21</td>
<td>11.9</td>
</tr>
<tr>
<td>Trad + Mox C</td>
<td>Trad + Mox</td>
<td>Classic</td>
<td>21</td>
<td>8.0</td>
</tr>
<tr>
<td>Trad + Mox C4</td>
<td>Trad + Mox</td>
<td>Classic</td>
<td>4</td>
<td>1.9</td>
</tr>
<tr>
<td>Trad + Mox C0</td>
<td>Trad + Mox</td>
<td>Classic</td>
<td>0</td>
<td>0.8</td>
</tr>
<tr>
<td>Trad + noMox L</td>
<td>Trad + noMox</td>
<td>Light</td>
<td>21</td>
<td>8.0</td>
</tr>
<tr>
<td>Trad + noMox C</td>
<td>Trad + noMox</td>
<td>Classic</td>
<td>21</td>
<td>11.9</td>
</tr>
<tr>
<td>Trad + noMox C4</td>
<td>Trad + noMox</td>
<td>Classic</td>
<td>4</td>
<td>1.9</td>
</tr>
<tr>
<td>Trad + noMox C0</td>
<td>Trad + noMox</td>
<td>Classic</td>
<td>0</td>
<td>0.8</td>
</tr>
<tr>
<td>FR + Mox L</td>
<td>FR + Mox</td>
<td>Light</td>
<td>21</td>
<td>11.9</td>
</tr>
<tr>
<td>FR + Mox C</td>
<td>FR + Mox</td>
<td>Classic</td>
<td>21</td>
<td>8.0</td>
</tr>
<tr>
<td>FR + Mox C4</td>
<td>FR + Mox</td>
<td>Classic</td>
<td>4</td>
<td>1.9</td>
</tr>
<tr>
<td>FR + Mox C0</td>
<td>FR + Mox</td>
<td>Classic</td>
<td>0</td>
<td>0.8</td>
</tr>
<tr>
<td>FR + noMox L</td>
<td>FR + noMox</td>
<td>Light</td>
<td>21</td>
<td>11.9</td>
</tr>
<tr>
<td>FR + noMox C</td>
<td>FR + noMox</td>
<td>Classic</td>
<td>21</td>
<td>8.0</td>
</tr>
<tr>
<td>FR + noMox C4</td>
<td>FR + noMox</td>
<td>Classic</td>
<td>4</td>
<td>1.9</td>
</tr>
<tr>
<td>FR + noMox C0</td>
<td>FR + noMox</td>
<td>Classic</td>
<td>0</td>
<td>0.8</td>
</tr>
</tbody>
</table>

Physical characteristics of Nomacorc low density polyethylene stoppers. Classic: length 37 mm, density 0.365 g cm⁻³; Light: length 38 mm, density 0.285 g cm⁻³.

Table 3
Attributes used in the sensory evaluation of wines. Definitions and reference used for training.

<table>
<thead>
<tr>
<th>Attributes</th>
<th>Definition</th>
<th>Reference (mixed with wine or pure)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colour intensity</td>
<td>Intensity of colour: from pale to dark</td>
<td>2.3 Hexen-1-ol</td>
</tr>
<tr>
<td>Shade</td>
<td>Shade of the colour: from orange to purple</td>
<td>Fruits syrups. Crushed fruits</td>
</tr>
<tr>
<td>Vegetable</td>
<td>Intensity of herbs and plants odours</td>
<td>Isoamyl acetate</td>
</tr>
<tr>
<td>Red fruits</td>
<td>Intensity of cherry, reccurantan, strawberry and blackcurrant odours</td>
<td>Caramel syrup</td>
</tr>
<tr>
<td>Amyl</td>
<td>Intensity of banana sweet and isoamyl acetate odours</td>
<td>Homofuranone</td>
</tr>
<tr>
<td>Caramel</td>
<td>Intensity of caramel syrup odour</td>
<td>Absolute ethanol</td>
</tr>
<tr>
<td>Burnt</td>
<td>Intensity of burned smoking and toasted bread odours</td>
<td>4-Ethylphenol decanoic acid</td>
</tr>
<tr>
<td>Alcohol</td>
<td>Intensity of alcohol odour</td>
<td>Tartaric acid</td>
</tr>
<tr>
<td>Animal</td>
<td>Intensity of leather: stable and animals odours</td>
<td>Quinine sulphate</td>
</tr>
<tr>
<td>Soursness</td>
<td>Intensity of sour taste</td>
<td>Grape stem tannin extract</td>
</tr>
<tr>
<td>Biterness</td>
<td>Intensity of bitter taste</td>
<td>Grape sugar</td>
</tr>
<tr>
<td>Astringency</td>
<td>Intensity of astringent taste</td>
<td></td>
</tr>
<tr>
<td>Sweetness</td>
<td>Intensity of sweet taste</td>
<td></td>
</tr>
</tbody>
</table>
and a time. The average was calculated on all wines (1)

\[ Y = \frac{1}{n} \sum_{i=1}^{n} \bar{x}_i \]  

(1)

where \( n \) is the number of wine modalities, and \( \bar{x}_i \) is the mean score of all judges, for a wine modality.

Then the difference between the average on all wines (\( Y \)) and the average for each wine was subtracted from the average of each wine to calculate the centred coordinate of each wine (\( Z_i \))

\[ Z_i = \bar{x}_i - (Y - \bar{x}) \]  

(2)

3. Results and discussion

3.1. Analysis of jury repeatability

For each analysis time point, interactions between subject and repetition and between wine and repetition did not show any significant effect on sensory parameters, indicating that the panel repeatability was good.

3.2. Sensory analysis at bottling

At bottling, results of two-factor ANOVAs (winemaking techniques and subject) showed a significant difference among the four wines for eight of the assessed attributes.

Multiple comparisons of mean values using HSD (Fig. 1), between winemaking techniques showed that Trad wines were perceived more animal and FR wines higher in astringency, slightly higher in red fruit aroma (in particular FR + Mox), and, but not significantly, in alcohol.

The difference in alcohol perception is related to differences in the alcohol concentration (Table 1) due to differences in Brix degrees at picking rather than to winemaking technologies (the grapes used to make FR were harvested at 25° Brix and those used to make Trad at 22° Brix).

The higher astringency of FR wines can be explained by an increased extraction of tannins which are primarily responsible for this sensation. Increased extraction of tannins in FR wines has been reported earlier [12] and is confirmed in the present experiment by the higher level of absorbance at 280 nm (Table 1) as well as by analysis of wine condensed tannin composition [21].

The higher animal perception in the Trad wines may result from contrast with the FR wines, which are perceived more fruity, as it was checked that they did not contain 4-ethyl phenol or 4-ethyl gaiacol, which are known contributors of animal flavour.

We observed a difference in bitterness between FR wines and Trad wines, with FR wines perceived more bitter than Trad wines, and FR + Mox wine significantly more bitter than Trad + Mox wine. The increased bitterness can be attributed to increased concentration of phenolic compounds, especially catechins and tannins in the FR wines [12], as these molecules are known to contribute bitterness in addition to astringency [22,23]. Moreover, the higher alcohol concentration in the FR wines may enhance the bitterness perception of phenolic compounds [24].

The jury perceived no significant difference in colour intensity or shade between the Trad and FR wines. However, FR wines showed lower colour intensity and appeared lighter (higher L*), less red (lower a*) and more yellow (higher b* and H*) than Trad ones (Table 1). The lack of discrimination of colour properties of red wines by human eye above certain colour intensity values has been reported earlier [25]

Micro-oxygenation, associated or not with FR, had an influence on shade, amyl and burnt: Mox wines were perceived lower in amyl and burnt odours. FR Mox was also perceived more orange than the other three wines. Spectrophotometric measurements revealed that Mox wines are lighter (colour intensity, L*), less red (a*) and more yellow (b*, H*) (Table 1) than noMox wines. Such differences were perceived by the jury only for the FR Mox wine which was the least coloured. Part of the colour differences may be explained by the higher amounts of free and total sulphites in Mox wines (Table 1), inducing bleaching of part of the anthocyanin pigments. The corrected colour intensity (Clor) values (obtained after addition of acetaldehyde to release anthocyanin pigments from colourless bisulphite adducts) were identical in the Mox and noMox wines, confirming this hypothesis.

3.3. Sensory analysis at 10 months

Wine sensory characteristics have been analysed after 5 and 10 months of ageing. Results are similar, but differences between the wines increased from 5 to 10 months. Therefore, we decided to dig extensively into the results at 10 months while the results at 5

Fig. 1. Main effects of winemaking technologies at bottling (T0), showing means for the eight significant attributes. (●) Trad + noMox; (□) Trad + Mox; (■) FR + noMox; (▲) FR + Mox. (+) Different labels (A, B, C) indicate means that significantly differ at \( p < 0.05 \), *\( p < 0.01 \); **\( p < 0.001 \); ***\( p < 0.0001 \).
Table 4
F-values and associated probabilities of the four-factor ANOVA at T10.

<table>
<thead>
<tr>
<th>Attributes</th>
<th>FR/Trad</th>
<th>Mox</th>
<th>OTR</th>
<th>Winemaking × OTR</th>
<th>Mox × OTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colour intensity</td>
<td>0.001***</td>
<td>0.001***</td>
<td>&lt;0.0001***</td>
<td>0.798</td>
<td>0.926</td>
</tr>
<tr>
<td>Shade</td>
<td>0.001***</td>
<td>&lt;0.0001***</td>
<td>&lt;0.0001***</td>
<td>0.477</td>
<td>0.848</td>
</tr>
<tr>
<td>Vegetable</td>
<td>0.572</td>
<td>0.987</td>
<td>0.019</td>
<td>0.192</td>
<td>0.688</td>
</tr>
<tr>
<td>Red fruits</td>
<td>0.406</td>
<td>0.271</td>
<td>&lt;0.0001***</td>
<td>0.759</td>
<td>0.479</td>
</tr>
<tr>
<td>Amyl</td>
<td>0.218</td>
<td>0.563</td>
<td>0.019</td>
<td>0.865</td>
<td>0.526</td>
</tr>
<tr>
<td>Caramel</td>
<td>0.214</td>
<td>0.247</td>
<td>&lt;0.0001***</td>
<td>0.759</td>
<td>0.479</td>
</tr>
<tr>
<td>Burnt</td>
<td>0.311</td>
<td>0.900</td>
<td>0.744</td>
<td>0.342</td>
<td>0.348</td>
</tr>
<tr>
<td>Alcohol</td>
<td>0.390</td>
<td>0.499</td>
<td>0.231</td>
<td>0.674</td>
<td>0.188</td>
</tr>
<tr>
<td>Animal</td>
<td>0.009**</td>
<td>0.195</td>
<td>&lt;0.0001***</td>
<td>0.080</td>
<td>0.201</td>
</tr>
<tr>
<td>Sourness</td>
<td>0.799</td>
<td>0.326</td>
<td>0.297</td>
<td>0.333</td>
<td>0.140</td>
</tr>
<tr>
<td>Bitterness</td>
<td>0.0004**</td>
<td>0.069</td>
<td>0.043</td>
<td>0.875</td>
<td>0.243</td>
</tr>
<tr>
<td>Astringency</td>
<td>&lt;0.0001***</td>
<td>0.238</td>
<td>0.125</td>
<td>0.529</td>
<td>0.996</td>
</tr>
<tr>
<td>Sweetness</td>
<td>0.287</td>
<td>0.951</td>
<td>0.144</td>
<td>0.228</td>
<td>0.606</td>
</tr>
</tbody>
</table>

*p < 0.05.
**p < 0.01.
***p < 0.0001.

months will be discussed later when examining the changes over time.

3.3.1. Winemaking and Mox effects

Significant differences (p < 0.05) between FR and Trad wines were found for five of the assessed attributes (Table 4): colour intensity, shade, animal odour, bitterness and astringency. Multiple comparisons of mean values using HSD (Fig. 2) show that Trad wines are perceived more animal and FR wines more bitter and astringent, as observed at T0. Olfactory differences due to winemaking decreased from T0: only differences in animal odour perception remained significant, and discrimination was lower than at T0.

Colour intensities were higher in the noMox wines than in the Mox wines and in the trad wines than in the FR wines but only Trad noMox and FR Mox were significantly different. Mox also exerted a significant effect on shade with the Mox wines perceived more orange than the noMox wines. Similar trends had been observed at T0, but differences in colour intensity perceived by the sensory panel were not significant. Spectrophotometric measurements at T10 (Fig. 2) showed little differences in colour intensity and that Mox wines were more orange (H*) than noMox wines and FR wines more orange than Trad wines.

This suggests that composition differences induced by the Mox treatment have induced differences in pigment evolution during wine ageing. Colour changes from red to purple then to orange have been observed earlier in wines submitted to micro-oxygenation over a rather long period [3].

3.3.2. OTR effect

OTR affected significantly eight of the assessed attributes: colour intensity, shade, vegetable, red fruits, amyl, caramel, animal and bitterness, as shown in Table 4 and Fig. 3. Multiple comparisons of mean values using HSD showed that OTR are separated into two groups:

- wines stored with classic 0% and classic 4% are lighter, show more purple shade, animal odour and slightly stronger vegetable character;
- wines stored with classic 21% and light 21% are darker, more orange, with higher red fruit and caramel odours and a little more amyl character.

Wines stored under higher OTR showed significantly higher colour intensity and appeared more orange (Fig. 3), in agreement...
with the visual perception. This confirms that moderate oxidation stabilises red wine colour but also directs reactions of phenolic compounds towards different pathways [3].

Oxygen exposure also modified wine odour: wines stored with higher OTR had higher red fruits and caramel odours and were lower in vegetable and animal odours (reductive characters).

OTR showed little impact on mouth-feel properties, even if the modality light 21% was more bitter than classic 21%.

3.3.3. Winemaking techniques × OTR interaction and Mox × OTR interaction

The effect of winemaking technique × OTR interaction was not significant for any attribute (Table 4). This means that, regardless of the winemaking technique, the extent of oxygen exposure, as controlled through closure OTR, had the same impact on wine sensory characteristics, after 10 months of ageing.

The same is true for Mox × OTR interaction. Whether wines are Mox or noMox, OTR had the same influence on the sensory characteristics.

3.3.4. Multivariate analysis

Multivariate analysis (AHC and PCA) permits a general evaluation of the results.

Differentiation of the wines is illustrated in the dendrogram of AHC. After 10 months of ageing, grouping appeared first based on OTR, then on winemaking techniques, with four main clusters separating OTR classic 0% and classic 4% from classic 21% and light 21% and Trad wines from FR wines. (see Supplementary Electronic Material Fig. S1)

In principal component analysis, two components had an eigenvalue higher than 1, and the first two principal components retained 81.4% of the variance. Fig. 4a shows the contribution of the attributes on the first two axes.

The first axis, representing 63.2% of the total variance was loaded positively with the attribute animal and negatively with the attributes red fruits, caramel and colour intensity.

Astringency, colour intensity and shade were correlated and contributed to the second axis (18.2% of the total variance).

Fig. 4. Contribution of the variables (a) and distribution of wines (b) after 10 months of ageing in the two dimensional coordinate system defined by the first two principal components.
Fig. 4b shows the projection of the wines in the space defined by the sensory attributes. The four AHC groups can be observed. Wines are clearly separated in function of their OTR along the first axis, which can thus be interpreted as an oxygenation axis, with oxygenation increasing from right to left. The second axis shows the separation of the winemaking techniques: Trad from Flash release wines and, within each group, Mox from noMox.

Thus, after 10 month ageing, OTR effect was more important in wine differentiation than winemaking techniques.

Visual attributes separate wine samples according to OTR and winemaking techniques. Independently of winemaking technique, wines stored with classic 21% and light 21% were darker and more orange-red than those aged under classic 4% and 0%, which were more purple.

Fig. 6. Changes in red fruit and animal odour intensity over time according to winemaking technique and OTR. (♦) Classic 0%; (▲) Classic 4%; (□) Classic 21%; (×) Light 21%.
OTR had an important impact on olfactory attributes. Indeed, according to the rate of oxygen permeating through the closure, wines had different olfactory notes: in particular fruity characters were higher for wines with a more important oxygen exposure, and, in contrast, reduced characters were enhanced when little oxygen exposure is allowed (lower OTR).

Taste attributes appeared mostly related to the winemaking techniques and were little affected by OTR. FR wines tasted more astringent and bitter than Trad wines, due to higher extraction of grape tannins into the FR wines.

3.4. Evolution

When comparing the evolution for each wine modality, no difference was observed between Mox and noMox wines for the same OTR level. Therefore, only Trad + noMox and FR + noMox wines are presented below.

For both FR and Trad wines, oxygen exposure influenced changes in wine colour intensity (Fig. 5): with higher OTR, colour became more intense while it faded with lower OTR. This difference clearly increases between T5 and T10.

Wine shade shifts from red to more purple and then to more orange were observed with time (Fig. 5). These changes took place more rapidly in FR wines and with higher OTR. After 10 months, wines stored with lower OTR appeared more purple while those stored with higher OTR appeared more orange in concordance with spectrophotometric measurements of these wines (Fig. 3).

These results may be related to changes in polyphenolic composition as shown earlier with micro-oxygenation experiments [3]. Indeed, oxidation promotes formation of ethyl-bridged anthocyanin-flavanol derivatives which are purple pigments [26,27,28] and intensely coloured at wine pH [29] and of various pyrananthocyanins, which are orange pigments [30,31]. The former pigments are rather unstable in wine [28] so that pyrananthocyanins gradually become predominant.

Overall, the intensity of red fruit odour decreased between T0 and T5, then increased (Fig. 6). For both Trad and FR wines, the decrease was smaller and subsequent increase larger with higher OTR and larger differences were observed within the Trad wines. Animal odour decreased and increased, respectively, with higher and lower OTR (Fig. 6). Again differences were larger among the Trad wines and little changes were observed between T5 and T10 in the FR wines.

4. Conclusion

This study demonstrates the high incidence of moderate oxygen transfer through the closure on wine quality. Indeed, after one year of ageing in 375 mL bottles (corresponding roughly to two years in traditional 750 mL bottles), the wines which have received more oxygen were perceived higher in fruity and caramel odour and lower in animal character. They also showed more intense and more orange colour.

At bottling, wines made with flash release were perceived less animal and more fruity, bitter and astringent than the control wines. The last two characters can be attributed to increased extraction of phenolic compounds, and especially catechins and tannins, in the FR process. Taste differences associated with FR were maintained throughout ageing while aroma and visual differences observed after 5 and 10 months of ageing were mostly related to the OTR level. Along storage, the influence of OTR became increasingly important as shown by increased significance levels of the observed differences, exceeding that of the wine-making technique after 10 months of ageing.

This study opens perspectives for making different types of closures allowing better control of wine oxygen exposure after bottling, taking into account the type of wine and the expected storage duration. However, further studies of the chemical mechanisms involved in colour and aroma changes associated to moderate oxygen exposure are needed to better predict and control these processes.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.aca.2009.11.049.

References