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Article

Dissolved Oxygen Removal in Wines by Gas Sparging, Its Optimization and Chemical Impact

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Abstract: Sparging is a technique to remove an excess of dissolved oxygen from the wine with inerting gases before bottling to avoid negative consequences for its chemical and sensory properties. However, its effectiveness on these properties has not been studied in depth. This work investigates the effectiveness of different inerting gases (N_2, CO_2, and argon) in removing dissolved oxygen in different volumes of a model wine. The efficacy of these gases was also studied in white and red wine, as was their effect on the physicochemical characteristics. Sparging with N_2 in the model wine gave the best results in terms of cost–benefits, and with CO_2 the worst. The scaling in tanks of different sizes allowed us to establish that the N_2 expenditure ranged between 0.09 L and 0.23 L of gas per liter of model wine, establishing an index (L_{gas}/L_{wine}) that can be very useful for wineries to remove the dissolved oxygen. Sparging treatments in white and red wine showed very similar results to the model wine. The effect on the chemical properties of the wines was, in some cases, different for white and red wine and for each gas used. The incorporation of oxygen and the subsequent sparging produced a significant loss of some volatile compounds of sensory interest and increased the content of others that have a negative sensory effect. In addition, it had a negative effect on the chromatic properties of red wines.

Keywords: inerting gas; argon; nitrogen; oxygen; carbon dioxide; white and red wines; chemical composition

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1. Introduction

It is well known in the winemaking industry that a high concentration of dissolved oxygen (DO) can lead to accelerated oxidation of wines, especially white wines [1]. The wine gains oxygen throughout the winemaking process wherever it comes into contact with the air being racking the wine between tanks, as well as the materials used for racking critical points that must be taken into account to avoid large additions of oxygen before bottling [2,3]. According to the literature, it is recommended that the dissolved oxygen content in bottled red wines should be less than 1.25 mg/L or 0.6 mg/L in white and rosé wines, respectively [4–8], but this will depend on the winemaker, the style of wine to be made, and the technology available in the winery to be able to carry out good practices to avoid large incorporations of oxygen. In a study conducted by Letaief in 2016 [9], an audit of oxygen incorporation into wine during bottling was carried out in 18 wineries, and significant differences were observed between them. While some managed to maintain DO values of 0.2 mg/L in the wine during bottling, others were at values higher than 1.5 mg/L and could negatively affect the final properties of the wine. When the concentration of dissolved oxygen is higher than recommended, it is necessary to minimize it as much as possible before the wine is bottled. The removal of excess dissolved oxygen from the wine is usually conducted by applying a continuous bubbling of inert gas, mostly N_2, which allows this oxygen to be displaced out of the wine. This technique is known as sparging and started to be used in the winemaking industry in 1960 [10]. Apart from N_2, other gases less frequently used in winemaking, such as argon (Ar), CO_2, or a mixture of N_2 and CO_2, can be used to displace the oxygen dissolved in the wine. From an economic point of view, N_2 is preferred to Ar, as it is cheaper.

The sparging technique is based on Henry's law, which states that the solubility of a gas in a liquid is proportional to its concentration in the atmosphere above the liquid [11]. Thus, if N_2 is abundantly introduced into a wine, the other gases found in the wine, such as oxygen or CO_2, tend to mix with the N_2 bubbles to reestablish equilibrium, being eliminated from the wine by entrainment with the excess N_2 above its solubility in the wine [11]. Sparging is usually performed by two techniques consisting, on the one hand, of bubbling the inert gas in a tank by forming a column of bubbles of this gas and/or bubbling the gas in line during the movement of the wine [10,12–14]. In the case of the bubbling column, it is usually recommended to perform it in tanks with a high height/diameter ratio to favor the removal of oxygen. In this way, the process is carried out more efficiently because it allows a longer contact time between the gas and the wine, favoring the elimination of oxygen. This technique consists of introducing, through the lower part of the tank, the gas to be bubbled through a porous diffuser. The ascent of the gas through the wine generates an appreciable column of bubbles on the surface once the process has begun. In the case of in-line bubbling, the gas is introduced directly into a stream of wine flowing through a pipe or hose. The number of points where the inerting gas is applied can be one or several, depending on the distance the wine will travel from the starting point to the end. This technique also allows the elimination of oxygen that may be incorporated into the filtration system through which the wine passes [10,12–14].

It has been estimated that, for a wine that is saturated with oxygen, a single passage through a bubbling column reduces the dissolved oxygen concentration to a greater extent than through in-line bubbling [10]. In contrast, for a wine with lower dissolved oxygen levels, the two methods are equally effective. However, these results are not necessarily indicative of one method being more effective than the other, as there are numerous unaccounted factors that will affect the efficiency of each process. On the other hand, Cant 1960 [10] also evaluated the efficacy of bubbling a certain volume of N_2 to remove the oxygen present in a wine, either once or twice, being more effective than doing it twice.

There are other methods to remove oxygen dissolved in water, but they are not suitable for wine, such as boiling at atmospheric pressure, boiling at reduced pressure, or sonication at reduced pressure [14,15]. The latter is a process that requires a much higher energy expenditure than sparging and, in addition, can be detrimental to wine quality due to the destruction of positive aromatic components and the loss of ethanol [13]. Recently, membrane contactors have started to be used in the wine industry [16–19]. They are considered less invasive since no aroma compounds are removed from the wine during the process of use. However, apart from the cost of this equipment, regular cleaning of the membrane is required, and the wine must be well filtered before contacting the membrane [20].

If sparging is not performed with sufficient care, the taste and aroma of the wine can be altered. For this reason, whenever it is necessary to perform this practice, it should be conducted with a gas/liquid ratio as low as possible to avoid possible alterations in the wine matrix [20,21]. The efficiency of sparging depends on several factors, such as the bubble size of the inerting gas, the ratio between the gas flow rate and the wine flow rate (if the operation is performed during a wine racking), the application time (duration of contact between the inerting gas and the wine), the temperature of the wine, the pressure at which the inerting gas is applied, the number of times the inerting gas is applied, the initial amount of oxygen in the wine, the design of the entire winemaking installation, and the selection of the equipment to carry it [9–14,21]. The dispersed bubbles of the inert gas cause a partial pressure difference between the bubble and the dissolved gas, where the concentration of dissolved gas in the bubble is initially zero. This causes the dissolved gas to enter the bubble due to the concentration gradient and then leave the dissolution. There is a point where the concentration gradient between the bubble and the concentration of the dissolved gas is zero, and that is the point where the bubble is no longer useful in solution and needs to leave the liquid [21]. According to Girardon 2019 [20], the efficiency of this process can be improved by applying spiral turbulence with an in-line vortex, which can increase the efficiency by around 90%. Other authors have used a mixer–stirrer to perform this technique on model wine with the aim of removing oxygen [8]. However, it should be noted that high agitation can favor a heterogeneous rather than homogeneous bubble regime, which occurs at lower gas flow rates and is characterized by smaller bubbles rising uniformly from the gas diffuser to the surface [12]. The effectiveness of sparging will also depend on the matrix of the wine to which it is applied. As mentioned above, the most suitable time for sparging is usually at the end of the winemaking process since it is at this point that the wine has a lower protein content, a parameter that seems to have relevant importance in the oxygen desorption process in wine when N_2 is applied. In addition, parameters such as ethanol, glycerol, sugar, and dry extract could affect the viscosity of the wine, which consequently could affect the oxygen desorption process. Moreover, in aqueous solutions, such as wine, the presence of phenols, acids, alcohols, surfactants, and ions also affects the process [13].

Sparging has been found to be effective in removing oxygen and CO_2 from wine and also in reducing excess SO_2, as well as certain sulfur aromas from reductive processes [8,11]. However, due to the scarce literature found, it is uncertain how this practice may affect wine composition, as well as the physicochemical and operational factors that may influence the efficacy of bubbling [8]. On the other hand, because red and white wines are chemically different, the oxygen removal process with N_2 or other inerting gases may vary according to the type of wine [22]. In the case of white wines, for example, N_2 can be used in order to remove dissolved oxygen; however, this process can also reduce CO_2 below the optimal level, which can affect the freshness and flavor of the wine. To avoid this scenario, it is usually recommended to use CO_2 alone or a mixture of CO_2 and N_2. With red wines, N_2 is usually the most optimal choice for sparging. However, some red wines also often require a small amount of dissolved CO_2, so a mixture of N_2 and CO_2 in a 2:1 ratio can sometimes produce more desirable results [22].

The potential effects of sparging on the concentration of aromatic compounds in wine remain relatively unknown, and there is speculation as to how this technique may affect such compounds that are of interest for wine quality. According to some authors [20], sparging, unless carefully applied, can remove certain compounds that positively influence wine flavor and aroma. However, work by Walls et al. (2022) [8] on white wine did not produce a significant modification of the volatile compounds studied after degassing the wine under certain conditions.

Based on the above, the objective of this work was to evaluate the effectiveness of different inerting gases, which are commonly used in the winemaking sector, with the capacity to remove dissolved oxygen in different volumes of a model wine stored in tanks with different sizes and dimensions. In addition, the most effective conditions for these gases were tested in a white wine and a red wine, and their effect on the physicochemical properties was evaluated.

2. Materials and Methods

2.1. Model Wines, White and Red, and Inerting Gases Used

The model wine used consisted of a hydroalcoholic solution at 12.5% v/v (food-grade alcohol), a total acidity of 5 g/L (using tartaric acid), and a pH of 3.5 (adjusted with sodium hydroxide), but without the rest of the compounds that are part of the matrix of a real wine that can consume oxygen.

A commercial white wine of the *Verdejo* variety and a red wine of the *Tempranillo* variety were used in the trials to evaluate the effect of dissolved oxygen removal with the different gases tested. The white wine had an alcoholic strength (AS) of 12.98% *v*/*v*, a pH of 3.29, a total acidity (TA) of 5.1 g/L, SO_2 L < 6 mg/L, and SO_2 T of 113 mg/L. The red wine had an alcoholic strength of 13.69% *v*/*v*, a pH of 3.77, a total acidity of 5.5 g/L, SO_2 L < 6 mg/L, and SO_2 T of 83 mg/L.

2.2. Oxygen Removal in Model Wine, White Wine and Red Wine

The methodology developed by Walls et al. (2022) [8] was followed with some modifications. Thus, a volume of 2.5 L of wine was placed in a Plexiglass tube with a maximum capacity of 4 L, forming a column of 1.5 m in height and 5 cm in diameter. The working temperature was 15.5 °C. The oxygen content was measured with two DP-PSt6 immersion probes connected to a measuring device (PreSens GmbH, Regensburg, Germany). All equipment was periodically calibrated according to the manufacturer's instructions. The methodology consisted, first, of incorporating atmospheric oxygen into the model wine through a porous diffuser. This diffuser had the same characteristics as the one used by Chiciuc et al. (2010) [23], i.e., stainless steel and an average pore size of 3.38 μm by bubbling air until reaching values of 3 mg/L (pO_2 = 63.33 hPa), values that can be reached with some ease during the different operations that the wine undergoes in its elaboration process. Once the wine was oxygenated, the inerting gases (N_2, CO_2, and/or Ar) were bubbled using the same porous diffuser at a flow rate of 0.03 L/minute (the flow rate was determined as the optimum to obtain a homogeneous bubble for the dimensions of the plexiglass tube) until reaching an oxygen content of 0.3 mg/L (pO_2 > 6.33 hPa), at which point bubbling ceased. For oxygen removal, N_2, CO_2, and Ar were used. All gases used were food-grade and were supplied in 50 L cylinders by Carburos Metálicos Air Products Group (Barcelona, Spain). The flow rate applied in each test was measured with a Siargo mass flow meter MF5700 series digital flow meter (Siargo Ltd., Santa Clara, CA, USA). This mass flow meter sensor directly measures mass flow with a very low pressure loss.

For the scaling tests in 40 L, 100 L, 500 L, 1000 L, and 1800 L tanks, other larger diffusers were used, and their characteristics are detailed in Table 1.

Table 1. Wine volumes used for sparging scaling, flow rate used, and pore surface area (centimeters) in contact with each liter of wine.

Tank Volume (L)	Heigh/Diameter of Tanks (cm)	Porous Diffuser Surface	Flow Rate (L/min)	Ratio S_{porous}/L_{wine}
2.5	200/5	11.9	0.03	4.75
40	50/36	24.5	0.48	0.61
100	68/45	25.9	1.2	0.12
500	105/80	58.1	6	0.12
1000	155/100	268.8	12	0.27
1800	160/123	268.8	21.6	0.15

The experiments carried out for oxygen removal by sparging in model wine were performed in triplicate and in white and red wine in duplicate at the experimental winery of the La Yutera campus (Palencia) of the University of Valladolid.

2.3. Physico-Chemical Analysis of White and Red Wine

The classic oenological parameters (AS, pH, total acidity, volatile acidity, free SO_2, and total SO_2), cooper and iron content, and total polyphenol index (TPI) were analyzed following the methods established by the OIV [24]. In addition, the absorbance spectra between 330 nm and 700 nm of all wines were performed. Spectrophotometric analyses were performed with a Perkin Elmer LAMBDA 25 UV/vis spectrophotometer. Color parameters (color intensity (CI) and hue (h)) were determined with the methodology described in Glories 1984 [25]. CieLAB *L**, *a**, and *b** color coordinates were measured following MSCV methodology [26]. Volatile higher alcohols were analyzed following the method described in Pérez-Magariño et al., 2019 [27], using a gas chromatograph with a flame ionization detector (GC–FID). In addition, minority volatile compounds, which play an important role in the sensory properties of wines, were analyzed according to the methodology established in del Barrio-Galán et al., 2021 [28].

2.4. Statistical Analysis

All the variables analyzed were treated using the analysis of variance (ANOVA) and the least significant difference (LSD) test at the significance level of $p < 0.05$. Statistical analyses were carried out using the STATGRAPHICS Centurion 18 program (Statgraphics Technologies Inc., The Plains, VA, USA).

3. Results and Discussion

3.1. Efficacy of Using N_2, CO_2, and Ar for the Removal of Oxygen in Model Wine

In order to establish the most suitable working flow rate, different flow rates were carried out with N_2, which made it possible to establish this flow rate at 0.03 L/min since it allowed the generation of small and homogeneous bubble sizes in the wine column and did not generate excess foam, as recommended in the literature consulted.

Next, other inerting gases, such as CO_2 and Ar, were tested in the same volume of model wine and under the same conditions of temperature and flow rate of gas incorporated as in the case of N_2, to evaluate their effectiveness as well. Figure 1 shows the kinetics over time for oxygen removal in the model wine with each of the inerting gases used and the volume that had to be applied per liter of wine in each trial (index L_{gas}/L_{wine}). As can be seen, the most effective gas for removing oxygen from the model wine up to values of 0.3 mg/L (pO_2 = 6.3 hPa) was N_2, which was necessary to apply 0.089 L of gas per liter of wine. The volume of Ar required to remove oxygen was similar to that of N_2 (0.099 L_{gas}/L_{wine}). On the other hand, CO_2 showed the worst results because it was necessary to incorporate this gas for a longer time, and therefore, it required significantly more volume to remove oxygen from the wine (0.243 L_{gas}/L_{wine}). N_2 is the least dense gas of the three gases used, which may explain its greater efficiency in displacing oxygen out of the wine. On the other hand, Ar and CO_2 are denser gases and form bigger bubbles than N_2, so there is less contact surface between the wine and the gas, and they do not succeed in displacing oxygen from the wine as quickly. As indicated in the literature, the smaller the bubble size, the better sparging results will be obtained precisely because there will be a larger interface between the wine and the gas [21,29,30]. In addition, CO_2 has the peculiarity of being highly soluble in wine [19], which is another factor that can affect its effectiveness in displacing oxygen. In contrast, the solubility of N_2 is very low or null in wine, but there is some ambiguity in these data depending on the literature consulted, indicating that it is slightly higher than the solubility of oxygen. Finally, it has been suggested that Ar is not soluble in wine [20,31,32]. This low or null solubility of both gases in wine may explain why the results obtained with both gases to displace dissolved oxygen have been very similar.

Today, there are wineries that have their own N_2 generator and those that do not usually buy bottles of this gas, which is more economical than others, such as Ar. For this reason, the most recommendable, from an economic cost–effectiveness point of view, is the use of this gas for the removal of oxygen dissolved in the wine, although periodic calibration of the generators is necessary to ensure the richness of the N_2 at the desired levels.

3.2. Scaling in the Use of N_2 for Oxygen Removal in Model Wine

Once it was proven that N_2 was the gas that provided the best results, scaling tests were carried out on larger volumes of model wine (40 L, 100 L, 500 L, 1000 L, and 1800 L) using N_2 under the same temperature conditions. The working flow rate (0.03 mL/min) was adjusted proportionally to the volume of wine being worked with. In addition, diffusers of different surface areas were used depending on the volume of wine (see Table 1). Figure 2 shows the kinetics of oxygen removal in the different volumes of model wine studied.

It also shows the amount of N_2 that had to be incorporated per liter of model wine, as well as the total volume of N_2 used. As expected, the greater the volume of wine, the greater the total amount of N_2 needed to be incorporated to remove oxygen, up to values of 0.3 mg/L. On the other hand, observing the index L_{gas}/L_{wine}, it was seen that, depending on the volume of wine from which oxygen removal was desired, the amount of N_2 to be incorporated ranged from 0.09 L to 0.23 L per liter of model wine. In general, it was seen that up to a volume of 500 L of wine, the amount of gas that needed to be incorporated to remove oxygen from the wine increased as the volume of wine increased. On the other hand, for larger wine volumes, the ratio L_{gas}/L_{wine} to be applied was similar. These results could be of great use to wineries that need to remove oxygen from their wines before bottling since, depending on the volume of wine contained in the tank, the amount of N_2 to be added during a given time to remove oxygen from the wine can be established. In this way, the wineries can apply the necessary gas to their wine following the index established in this work, knowing the initial dissolved oxygen in the wine and extrapolating the flow rate to be applied during a given time, depending on the volume of wine in the tank. However, these results will depend on the wine matrix, and therefore, these results are indicative for each winery.

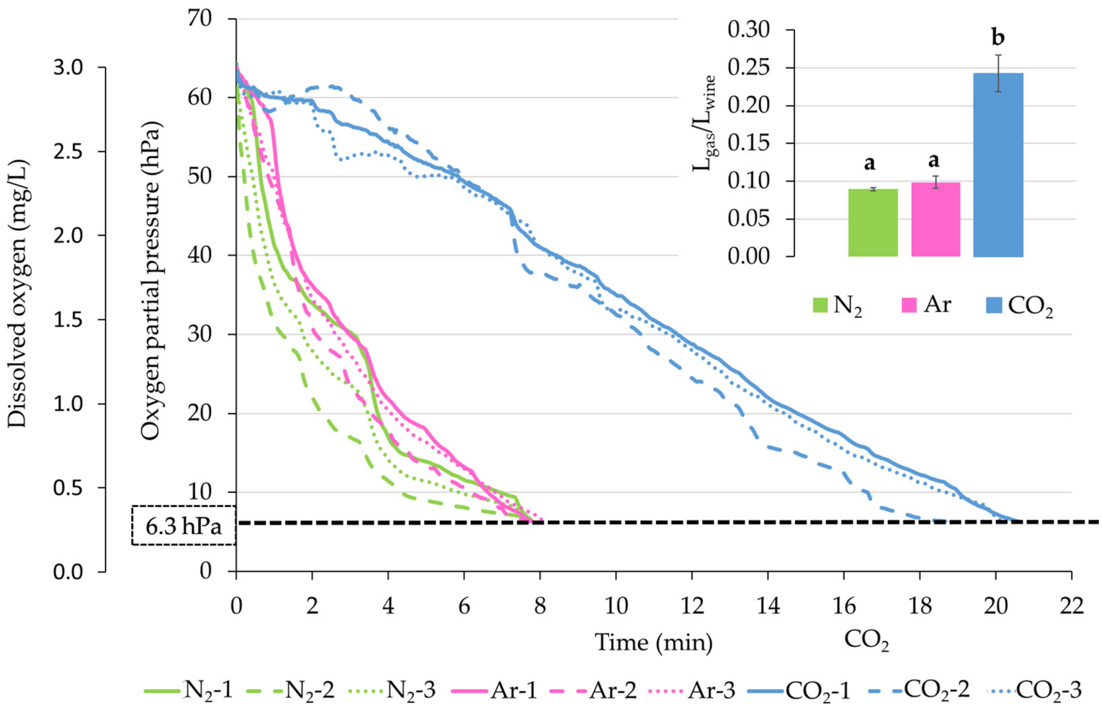

Figure 1. Kinetics of oxygen removal in model wine with the different inerting gases tested. Average of three repetitions were performed with each gas. Different letter in the bar graph indicated statistically significant differences.

3.3. Experiments with Oxygen Removal in White and Red Wine

Once the experiments on model wine had been carried out, the oxygen removal tests were performed on real wine under optimum conditions. As in the case of the model wine, oxygen was added to both wines by bubbling atmospheric air up to 3 mg/L and then removed by applying different inerting gases (N_2, CO_2, and Ar) up to 0.3 mg/L at a flow rate of 0.03 L/min. All tests were performed in a volume of 2.5 L of wine (4 L capacity plexiglass tube), forming a column of 1.5 m and having a diameter of 5 cm, and were performed in duplicate. The white wines studied were as follows: control wine (WC), wine oxygenated with 3 mg/L of O_2 (WOX), wine sparged with N_2 (WN_2), with CO_2 (WCO_2), and with Ar (WAr). The red wines were: control wine (RC), wine oxygenated with 3 mg/L O_2 (ROX), wine sparged with N_2 (RN_2), with CO_2 (RCO_2), and with Ar (RAr).

Figure 3 shows the results obtained in terms of the time required to remove oxygen from white and red wine, as well as the expenditure of each inerting gas per liter of white and red wine. The amount of inerting gas that had to be applied to carry out the deoxygenation in both wines was equal (without statistically significant differences), and this result was also equal to that obtained in the model wine with the exception of the trials carried out with CO_2 (higher amounts needed in real wines). In other words, N_2 was the most effective gas, and CO_2 was the one that showed the worst results. Therefore, from a cost–benefit point of view, it is recommended to use N_2 for oxygen removal in wines.

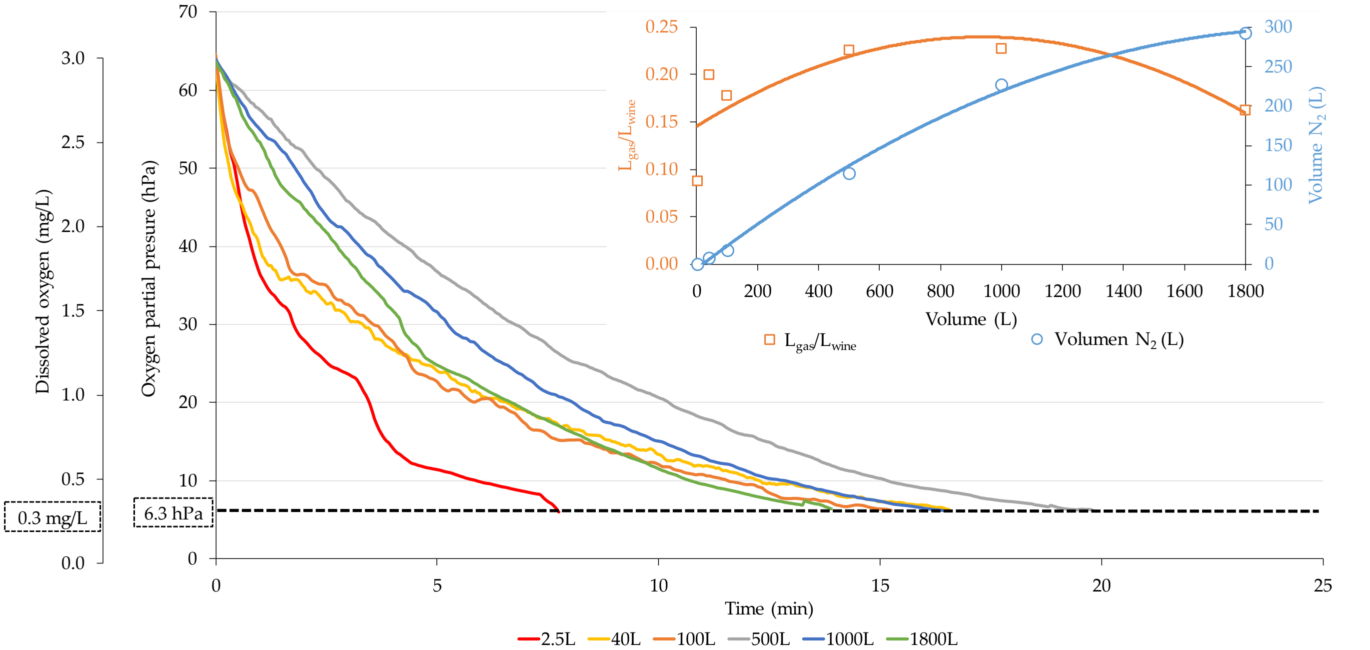

Figure 2. Kinetics of oxygen removal by sparging with N_2 in different volumes of model wine. Average of three repetitions were performed with each gas.

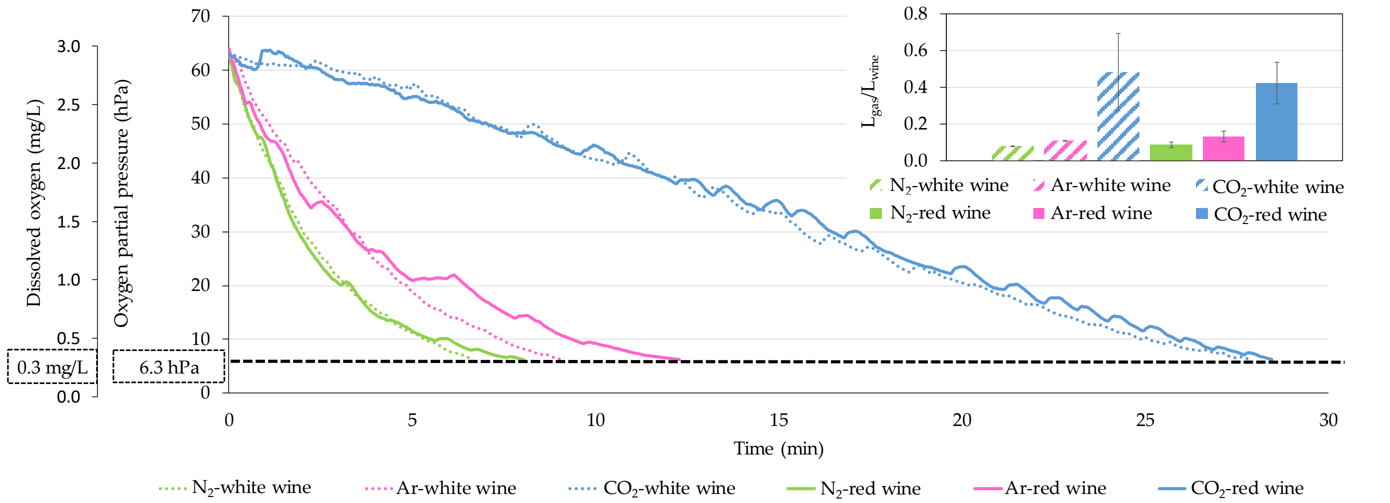

Figure 3. Kinetics of oxygen removal by sparging with N_2, CO_2, and Ar in a volume of 2.5 L of white and red wine. Average of three repetitions were performed with each gas.

3.4. Effect of Sparging on the Physicochemical Composition of Whites and Reds Wines

The few scientific studies found in real wine indicate that bubbling an inert gas to remove oxygen from a wine does not significantly affect the physicochemical composition of the wine [8]. Since the bubbling of any inerting gas usually results in a loss or evaporation of alcohol in the wine, it is possible that a modification of the volatile composition of the wine, as well as other compounds that influence the colorimetric and taste characteristics of the wine, may actually be occurring. For this reason, the classic oenological parameters—color, total polyphenol index (TPI), and volatile composition—of the white and red wines studied were analyzed.

3.4.1. Effect on Classical Oenological Parameters

Table 2 shows the classical oenological parameters and the color parameters analyzed in white and red wines. The effect observed in white and red wines after sparging was different, probably due to their different compositions. Thus, it was seen that in white wines, the use of CO_2 to remove oxygen produced an increase of 2% in the total acidity of the wine with respect to the rest of the wines that had the same value. This could be due to the fact that CO_2 is a highly soluble gas in wine and can produce an increase in total acidity [20], which can have an impact on the sensory perception of these wines. Therefore, it is something that must be taken into account depending on the type of white wine to which sparging with CO_2 is to be applied. On the other hand, in red wine, only the RN_2 wine showed lower total acidity (1.8%) than the control wine. The rest of the wines showed a value equal to the control wine.

All treated white wines, both oxygenated and deoxygenated with inerting gas, had between 16.7% (0.25 g/L) and 13.3% (0.26 g/L) less volatile acidity than the control wine (0.30 g/L), probably due to an evaporation effect. In contrast, no statistically significant differences were observed in the red wines. This result could be due to the fact that the volatile acidity in white wines is much lower than in red wines, producing a greater effect in the former than in the latter. On the other hand, as detailed in the literature [7,8], the use of inert gases can be effective in eliminating excess SO_2 in wines. In this case, it seems that such an effect was only observed with the use of Ar since both white wine WAr and red wine RAr showed a lower SO_2 T content than control wines WC and RC. This content was reduced by 2.7% in WAr wine and by 2.4% in RAr wine. Therefore, if the objective of the use of inerting gases in wines is to remove an excess of SO_2, it is most appropriate to use Ar. Statistically significant differences were not found in the content of metals such as copper and iron in the wines analyzed.

3.4.2. Effect on the Absorbance Spectrum, Color, and TPI of Wines

Both oxygenation and the subsequent removal of oxygen with the different inerting gases had a greater impact on the absorbance spectrum, color, and total polyphenols in red wines than in white wines. Thus, in the case of red wines, it was observed that both ROX and RN_2, RAr, and RCO_2 wines showed a lower absorbance along their entire spectrum with respect to the control wine (Figure 4a), which could have an influence on the color of the wines. Thus, the oxygen removal treatments significantly affected the CI of the wines, with RAr being the wine most affected by the oxygen removal process, suffering a greater decrease in CI, followed by RCO_2 and RN_2. On the other hand, ROX wine maintained lower CI values compared to RC wine but higher than wines treated with inerting gases. As is well known, oxygen is one of the components that can favor oxidation and participate in different reactions that can modify the color of red wines [33–35]. This difference between ROX and the rest of the wines treated with inerting gases could be due to the fact that these treatments favored the mixing of oxygen with the wine until it was completely removed, favoring, in turn, the degradation/oxidation of anthocyanins, which are mainly responsible for the color of red wines. However, the relationship between anthocyanin concentration and color is not linear, so polymeric pigments and anthocyanin reaction products are much more important, especially in finished wines. Oxygenation and the subsequent use of inert gases to remove oxygen also had an impact on the CIELab color parameters. Thus, it was seen that all treatments produced an increase in hue (h^*) and lightness (L^*), probably due to the loss of color-blue due to the action of oxygen and its subsequent removal with the sparging treatments.

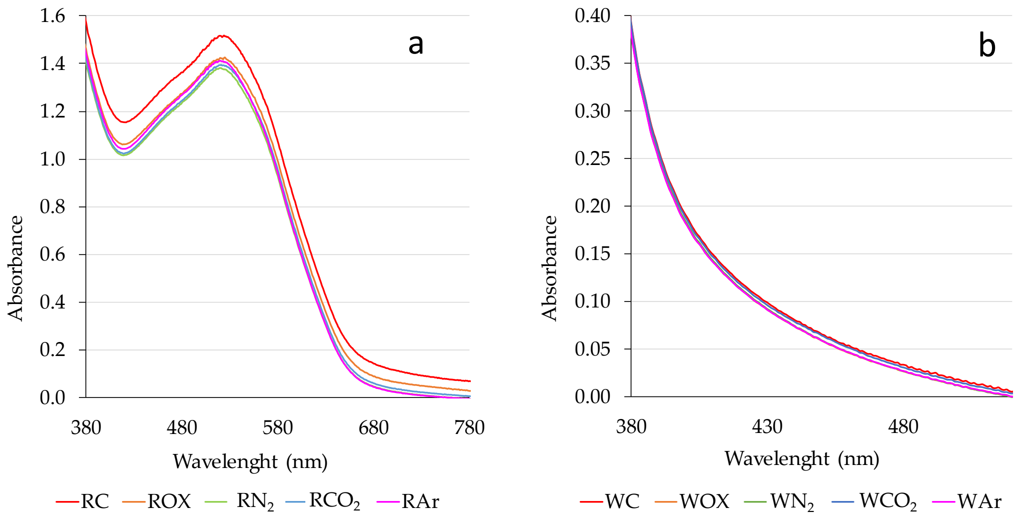

Figure 4. Spectral scan of the different red (**a**) and white (**b**) wines studied. White wine control (WC); white wine oxygenated (WOX); white wine sparged with N_2 (WN_2); white wine sparged with argon (WAr); white wine sparged with CO_2 (WCO_2). Red wine control (RC); red wine oxygenated (ROX); red wine sparged with N_2 (RN_2); red wine sparged with argon (RAr); red wine sparged with CO_2 (RCO_2).

The absorbance spectrum of the white wines was very similar in all wines, but certain significant differences were found (Figure 4b). Thus, it was seen that the control wine always showed the highest absorbance values in the whole spectrum, and the WOX and WAr wines had the lowest values. These differences in the spectrum made the color CI (absorbance at 420 nm) in both WOX and sparging-treated wines (WN_2, WAr, and WCO_2) lower than in the control wine. However, the parameters *a** and *b**, which measure the color of the wines in a more integrative way, did not show statistically significant differences, so it can be said that in this case, there was no color modification. Oxygenation and subsequent sparging with the different gases did not affect the TPI.

3.4.3. Effect on Volatile Composition

Regarding the analysis of higher alcohols in white wines (Table 3), no significant differences were observed in the content of 2-methyl-1-butanol and 3-methyl-1-butanol (isoamyl alcohols). These compounds can contribute positively or negatively to the aroma of wine, depending on their concentration [36,37]. Values above 300 mg/L in the content of higher alcohols (1-propanol, isobutanol, 2-methyl-1-butanol, and 3-methyl-1-butanol) usually contribute negative aromas. On the other hand, below this content, they can contribute positively to the aromatic complexity of the wines. However, an effect on another of the higher alcohols, 2-phenylethanol, was observed in the WOX, WCO_2, and WAr wines; its concentration is significantly lower than in the control wine and in the wine treated with N_2. This compound is characterized by providing the wines with floral aromas of roses and was found in all the wines at a concentration above the sensory perception threshold (14 mg/L) [28,38–40]. Therefore, wine oxygenation and subsequent sparging with CO_2 and Ar could negatively influence the sensory profile of these wines due to the significant loss of this compound. On the other hand, sparging with N_2 allowed for maintaining similar concentrations as the control wine and preserving the floral notes of the white wine.

Table 2. Classical oenological parameters of the white and red wines studied.

	White Wine					Red Wine				
	WC	**WOX**	**WN_2**	**WCO_2**	**WAr**	**RC**	**ROX**	**RRN_2**	**RCO_2**	**RAr**
AS (% *v*/*v*)	12.98 ± 0.00 a	12.93 ± 0.00 a	12.99 ± 0.01 a	12.95 ± 0.04 a	12.91 ± 0.01 a	13.69 ± 0.00 a	13.56 ± 0.00 a	13.40 ± 0.04 a	13.63 ± 0.01 a	13.57 ± 0.16 a
TA (g/L)	5.1 ± 0.0 a	5.1 ± 0.0 a	5.1 ± 0.0 a	5.2 ± 0.1 b	5.1 ± 0.0 a	5.5 ± 0.0 b	5.5 ± 0.0 b	5.4 ± 0.0 a	5.6 ± 0.1 b	5.5 ± 0.0 b
pH	3.29 ± 0.00 a	3.29 ± 0.00 a	3.28 ± 0.00 a	3.29 ± 0.01 a	3.29 ± 0.01 a	3.77 ± 0.0 a	3.76 ± 0.0 a	3.77 ± 0.01 a	3.76 ± 0.01 a	3.780.01 a
SO_2 L (mg/L)	<6	<6	<6	<6	<6	<6	<6	<6	<6	<6
SO_2 T (mg/L)	113 ± 0 b	117 ± 0 c	111 ± 1 ab	113 ± 2 ab	110 ± 0 a	83 ± 0 b	92 ± 0 e	86 ± 1 c	88 ± 1 d	81 ± 0 a
Cupper (mg/L)	<7	<7	<7	<7	<7	<7	<7	<7	<7	<7
Iron (mg/L)	1.54 ± 0.03 a	1.53 ± 0.02 a	1.56 ± 0.08 a	1.57 ± 0.08 a	1.53 ± 0.06 a	1.57 ± 0.08 a	1.60 ± 0.07 a	1.70 ± 0.10 a	1.76 ± 0.10 a	1.64 ± 0.06 a
CI	0.121 ± 0.000 d	0.113 ± 0.000 a	0.113 ± 0.002 ac	0.115 ± 0.005 ab	0.118 ± 0.000 bc	16.0 ± 0.04 e	14.7 ± 0.02 d	14.3 ± 0.28 c	14.0 ± 0.14 a	14.2 ± 0.04 b
*L**	100 ± 0.4 a	100 ± 0.4 a	100 ± 0.1 a	100 ± 0.4 a	100 ± 0.20 a	36.0 ± 0.00 a	39.5 ± 0.00 b	42.0 ± 0.00 c	41.1 ± 0.00 d	41.6 ± 0.00 e
*a**	−1.26 ± 0.05 a	−1.35 ± 0.06 a	−1.35 ± 0.0.1 a	−1.34 ± 0.09 a	−1.33 ± 0.09 a	42.0 ± 0.05 a	43.8 ± 0.11 b	45.9 ± 0.11 c	45.2 ± 0.01 d	46.5 ± 0.04 e
*b**	10.01 ± 0.11 a	10.06 ± 0.13 a	10.08 ± 0.01 a	10.13 ± 0.18 a	9.92 ± 0.15 a	13.4 ± 0.19 a	14.2 ± 0.04 b	16.2 ± 0.06 c	15.1 ± 0.01 d	17.4 ± 0.03 e
*h**						17.7 ± 0.25 a	18.0 ± 0.08 a	19.5 ± 0.02 b	18.4 ± 0.00 c	20.5 ± 0.01 d
TPI	4 ± 0.03 a	4 ± 0.16 a	4 ± 0.03 a	4 ± 0.04 a	4 ± 0.04 a	62 ± 0.34 c	61 ± 0.62 c	58 ± 0.93 b	61 ± 1.02 c	54 ± 1.02 a

White wine control (WC); white wine oxygenated (WOX); white wine sparged with N_2 (WN_2); white wine sparged with argon (WAr); white wine sparged with CO_2 (WCO_2). Red wine control (RC); red wine oxygenated (ROX); red wine sparged with N_2 (RN_2); red wine sparged with argon (RAr); red wine sparged with CO_2 (RCO_2). Different letters indicate statistically significant differences. Different letters in the same row indicate statistically significant differences.

Table 3. Major volatile compounds in the white and red wines studied.

	White Wine					Red Wine				
	WC	**WOX**	**WN_2**	**WCO_2**	**WAr**	**RC**	**ROX**	**RN_2**	**RCO_2**	**RAr**
1-Propanol (mg/L)	46.1 ± 0 a	46.1 ± 0 a	46.3 ± 1 a	46.7 ± 1 a	46.2 ± 0 a	30 ± 0 a	30 ± 0 a	31 ± 1 a	30 ± 1 a	30 ± 1 a
Isobutanol (mg/L)	15.0 ± 0 a	16.0 ± 0 a	16.0 ± 1 a	16.0 ± 1 a	15.0 ± 0 a	60 ± 0 a	63 ± 0 a	64 ± 2 a	60 ± 0 a	60 ± 3 a
2-Methyl-1-Butanol (mg/L)	20.0 ± 0 a	20.0 ± 0 a	21.0 ± 1 a	20.0 ± 1 a	20.5 ± 1 a	49 ± 0 a	48 ± 0 a	50 ± 2 a	48 ± 0 a	49 ± 2 a
3-Methyl-1-Butanol (mg/L)	125 ± 0 a	128 ± 0 a	126 ± 1 a	126 ± 4 a	127 ± 1 a	208 ± 0 a	199 ± 0 a	206 ± 6 a	202 ± 2 a	207 ± 10 a
2-phenylethanol (mg/L)	17.9 ± 0.1 c	16.7 ± 0.4 ab	17.5 ± 0.7 bc	16.3 ± 0.3 a	16.4 ± 0.3 a	39.4 ± 1.9 ab	37.9 ± 1.5 a	38.9 ± 2.5 a	50.6 ± 0.9 c	42.5 ± 2.4 b

White wine control (WC); white wine oxygenated (WOX); white wine sparged with N_2 (WN_2); white wine sparged with argon (WAr); white wine sparged with CO_2 (WCO_2). Red wine control (RC); red wine oxygenated (ROX); red wine sparged with N_2 (RN_2); red wine sparged with argon (RAr); red wine sparged with CO_2 (RCO_2). Different letters indicate statistically significant differences.Different letters in the same row indicate statistically significant differences.

Figure 5 shows the content of the different groups of volatile minority compounds in white wines. Table S1 (Supplementary Material) shows the content of each of the compounds studied. In general, it was seen that the WOX wine and those treated with the inerting gases (WN_2, WAr, and WCO_2) to remove dissolved oxygen produced a significant loss of linear ethyl esters (EEL) and branched ethyl esters (EEB) with respect to the control wine, mainly due to the loss of hexanoate and ethyl octanoate, which are the majority compounds within the ethyl esters. These compounds are characterized by contributing fruity aromas to wines and have a very low perception threshold (5 and 2 μg/L, respectively) [28,38,41]. Therefore, the oxygenation and sparging treatments had a negative effect on the volatile compounds that contribute fruity notes, either due to their oxidation or their volatilization during sparging. On the other hand, the WAr wine presented a higher terpene content, mainly due to its higher α-Terpineol content. In general, this compound and the other terpene compounds are characterized by providing wines with floral aromas [28,38–40], but their content did not exceed the perception threshold. WN_2 and WCO_2 showed a higher content of C6 alcohols compared to the control wine, but these differences did not affect the potential sensory profile (values below the perception threshold). The wine WCO_2 had a lower content of vanillin derivatives than the control wine and the other wines. These compounds are of sensory interest for imparting vanilla aromas to wines [28,38–40]. However, their content in the white wines was well below the threshold of perception, so sparging with CO_2 had no effect from a sensory point of view. Sparging with N_2 and CO_2 also affected the Strecker aldehyde content, with these wines showing higher contents than the control wine and the Ar-treated wine. These differences were mainly due to the higher content of 3-methylbutanal and isobutyraldehyde, which were the major compounds within this group. These compounds are formed due to oxidation processes and contribute odors that are considered negative for the sensory profile of wines, such as dried fruit, wet wood, wet paper, etc., when they are above the perception threshold (4.6 and 6 μg/L, respectively) [42]. All wines had content above the threshold. In other words, although these aromas could already be perceived in the control wine, the treatments with N_2 and CO_2 were able to enhance them. However, perception and enhanced perception were not tested in this study, and there are many suppressing and enhancing effects that can make a compound aroma active, even below the threshold.

In the case of red wines, neither oxygenation nor subsequent sparging treatments affected the content of most of the higher alcohols. However, as with the white wines, significant differences were found in the 2-phenylethanol content, with the RCO_2 wine showing a higher value than the control wine and the rest of the wines. Therefore, the result found with this inert gas was different from what occurred in white wines.

As for the minority volatiles (Figure 6), sparging with N_2 and Ar in red wines had a negative effect on certain aromatic compounds of interest, such as alcohol acetates and terpenes, since their content was significantly lower than in the control wine and in the RCO wine. These treatments significantly affected isoamyl acetate, which, in addition to being the most abundant, has a low perception threshold (30 μg/L) and is characterized by fruity banana aromas [39,40,43]. As already mentioned for white wines, terpenes are varietal compounds that are characterized by imparting floral notes to wines [28,38–40], and in this case, linalool was the compound most affected by sparging with N_2 and Ar. However, these treatments would not have a negative effect since the content was below the perception threshold (25 μg/L).

Surprisingly, both the ROX wine and the wines that were treated with sparging showed a higher content of vanillin derivatives than the control wine.

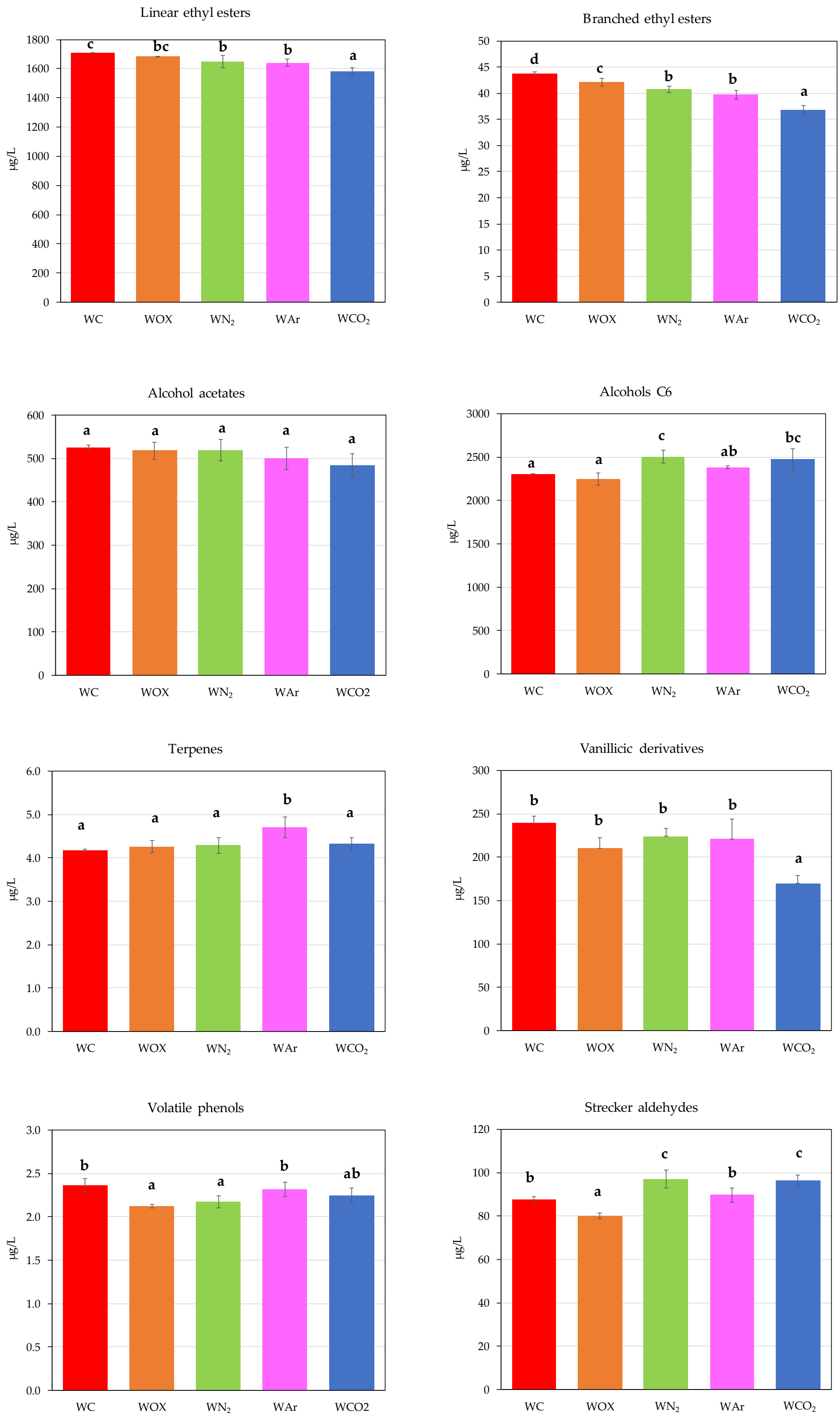

Figure 5. Content of the different groups of volatile minority compounds in the white wines studied. White wine control (WC); white wine oxygenated (WOX); white wine sparged with N_2 (WN_2); white wine sparged with argon (WAr); white wine sparged with CO_2 (WCO_2). Different letters indicate statistically significant differences.

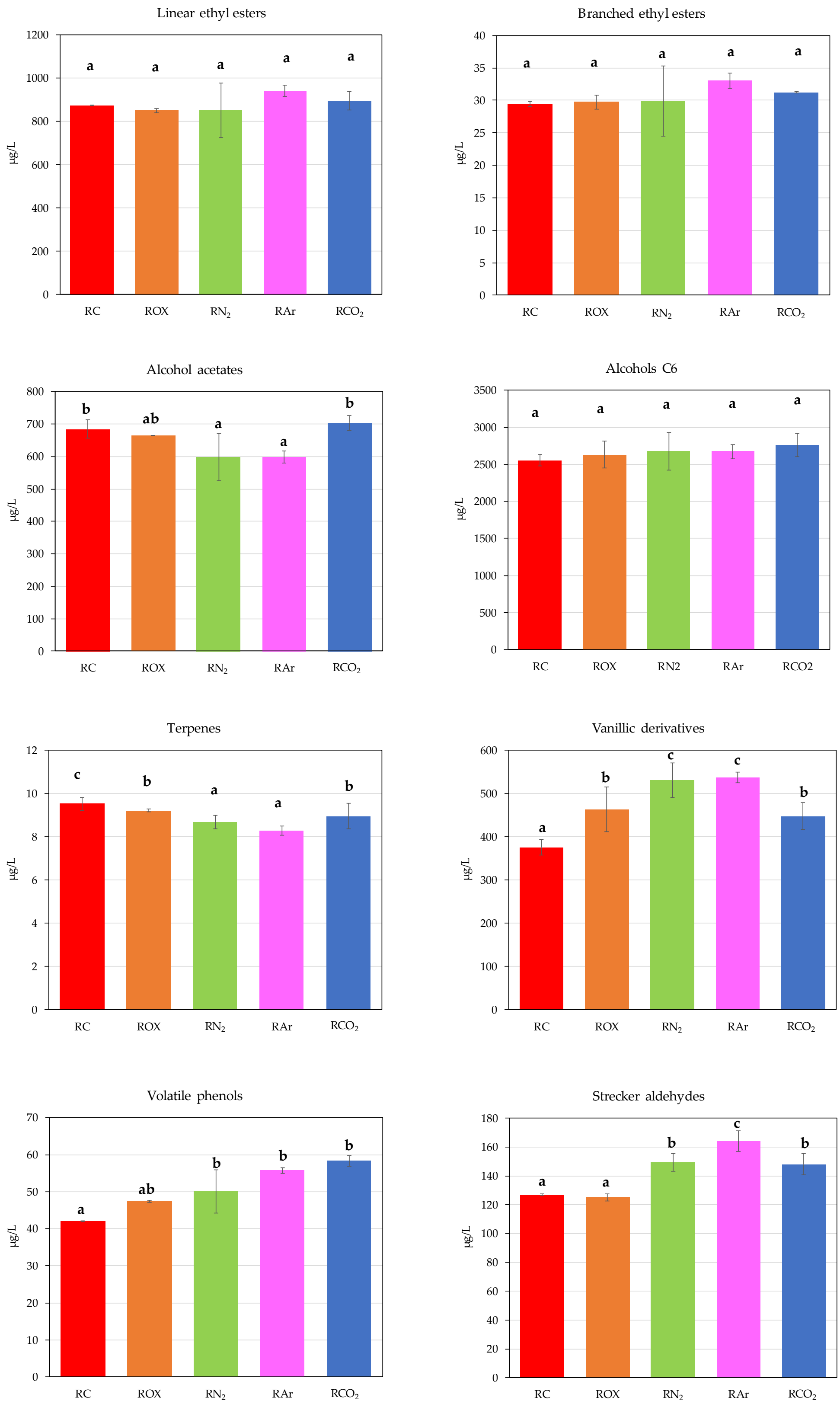

Figure 6. Content of the different groups of volatile minority compounds in the red wines studied. Red wine control (RC); red wine oxygenated (ROX); red wine sparged with N_2 (RN_2); red wine sparged with argon (RAr); red wine sparged with CO_2 (RCO_2). Different letters indicate statistically significant differences.

The effect of sparging on Strecker aldehydes was similar to that found in white wines, affecting above all the content of 2-methylbutanal, 3-methylbutanal, and isobutyraldehyde. In other words, the wines subjected to sparging with the different gases showed a higher content of these compounds than the control wine. As previously mentioned, these compounds can be generated during the oxidative processes that a wine can undergo [42]. Therefore, the increase in these compounds in the sparged wines could be due to the oxygenation of the wines with the inerting gases. However, until the oxygen is removed from the wine with the inerting gas, the oxygen is in contact with the wine for a longer time than in the case of the wine that was only oxygenated. Similar results were found for volatile phenols, where all the wines treated with sparging had a higher content than the control wine. This result was mainly due to the effect on compounds such as guaiacol, eugenol, and syringol, although only the eugenol content was above the perception threshold of 6 (µg/L), and its content was significantly higher in the RN_2 and RAr wines than in the rest of the wines.

4. Conclusions

In the search for a flow rate of N_2 to deoxygenate the model wine, it was possible to reduce the volume of N_2 gas required per liter of wine, although the time invested was greater, and thus the sparging process was improved.

N_2 proved to be the most effective inerting gas for deoxygenating both model wine and red and white wines, compared to other inerting gases, such as CO_2 and Ar, with CO_2 showing the worst results.

The scaling of sparging in model wine up to a volume of 1800 L allowed for the establishment of an index (L_{gas}/L_{wine}) ranging from 0.09 to 0.23 L of gas per liter of wine, depending on the volume of wine to be deoxygenated. This index can be used by wineries that need to deoxygenate their wines before bottling. However, the porous surface in contact with the wine (cm^2/L of wine) must be taken into account. The application time of the inert gas to deoxygenate the wine will depend on the initial oxygen content and the volume of the wine. Use in wineries would require more testing, especially with more cultivars and wine styles.

Oxygenation and the subsequent use of inerting gases to eliminate dissolved oxygen had a negative effect on the absorbance spectrum of red wines, mainly influencing their chromatic properties, decreasing the CI, increasing the hue, and thus the luminosity. An influence of these processes on the volatile composition of the wines was also observed, being, in some cases, different depending on the type of wine and the gas used. Within the majority of volatiles, differences were found mainly in the 2-phenylethanol content, but this depended on the gas used and the type of wine to which it was applied. In the minority volatiles, in general, oxygenation and subsequent sparging with inerting gases in white wines reduced the content of compounds of sensory interest, such as ethyl esters and, in the case of CO_2, vanillin derivatives. In addition, wines deoxygenated with inerting gases had a high content of Strecker aldehydes, although their production is usually related to oxidative processes, which contribute negative oxidative aromas. In red wines, both oxygenation and sparging with the different gases reduced the content of terpenes, and, in addition, treatments with N_2 and Ar reduced the content of alcohol acetates, both groups being of great sensory interest for their contribution of floral and fruity aromas. As in white wines, the use of inerting gases increased the content of Strecker aldehydes, which contribute notes of oxidized aromas.

Future studies should address the effect of these treatments in the long term during bottle aging of wines subjected to oxygenation and subsequent sparging with gases and evaluate their effect from a sensory point of view.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/beverages10010003/s1: Table S1: Content (µg/L) of minor volatile compounds in white and red wines studied.

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Data Availability Statement: Data are contained within the article.

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