

Organoleptic characterization of wines in contact with oak wood fragments immersed in plasma activated water (PAW)

Lucía González-Arenzana¹, Rosa López¹, Rocío Escribano-Viana¹, Beatriz Larreina¹, Patrocinio Garijo¹, Ana Rosa Gutiérrez¹, Pilar Santamaría¹, Carmen Olarte¹, Fernando Alba-Elías², Ana González-Marcos², Elisa Sainz-García², Ana Sainz-García², Rodolfo Múgica-Vidal², Ignacio Muro-Fraguas², and Isabel López-Alfaro¹

¹ICVV, Instituto de Ciencias de la Vid y el Vino (Universidad de La Rioja, Gobierno de La Rioja, CSIC). Finca La Grajera, 26071, Logroño, Spain

²Departamento de Ingeniería Mecánica, Universidad de La Rioja. Edificio Departamental. San José de Calasanz 31, 26004 Logroño, La Rioja Logroño, Spain

Abstract. Oak barrels are a valuable material for wine ageing, although their difficult cleaning and disinfection favours microbiological contamination causing wine quality depreciation. Atmospheric pressure cold plasma is a suitable technique to reduce microbiota, but there is little research on its impact on the nutritional and sensory characteristics of foods. The aim of this study was to analyse the organoleptic characteristics of red wines in contact with plasma-activated waters (PAW) treated wood, of different origins and toasting levels. These red wines were compared with others that had been in contact with two types of control wood; some immersed in distilled water and others subjected to sulphur dioxide combustion. The results showed that oak wood treated with PAW did not cause defects at the olfactory and gustatory level of the red wines, which presented good harmony, sufficient body and balanced fruity and spicy notes, sometimes even superior to those described for red wines in contact with control wood (sulphited or submerged in distilled water). Therefore, the treatment of the wood with PAW did not have a negative impact on the sensory quality of the wines, regardless of the origin and toasting of the wood.

1 Introduction

The growing demand for natural products is posing to the industry the challenge of providing safe, healthy, and minimally processed wines.

During the aging in oak wood barrels, the wine is enriched in aromas, taste sensations, balance and aromatic complexity so appreciated by the consumer.

The problems that arise during the aging of the wine are essentially associated with microbiological or contamination since the specific characteristics of oak wood make it difficult to disinfect and clean it. In fact, microorganisms take refuge in the natural pores of the wood and can cause negative organoleptic alterations in the wine, such as the synthesis of volatile phenols (stable aroma, leather, etc.), the increase of volatile acidity (aroma vinegar) or the biogenic amines formation [1]. Although used barrels do not have the same potential as that of new ones, they still have an excellent use value for numerous wines and alcoholic products at a lower cost, so its good maintenance is essential for this purpose.

The burning of a sulfur disc inside empty barrels has been practice widely used in wineries for barrel sanitization. The combustion of sulfur produces sulfur dioxide, which is the one that has biocidal effects on wood. However, the appearance of Directive 98/8/CE2 of the European Commission, which prohibits the use of sulfur dioxide for the sanitization of barrels, has led to the need for new solutions that allow this task to be viable from an economic and operational point of view, guaranteeing effective sanitization. This scenario has led to the development of new alternative technologies for the sanitization of barrels (thermal, ozone, ultrasound, etc.) but none of them have been able to respond adequately to the needs of the wine sector.

Therefore, studying new technologies for eliminating the wine-spoiler microorganisms without affecting wine quality [2] is interesting. One of these technologies that has shown its interest in this regard, is the use of atmospheric pressure cold plasma (APCP).

Plasma is referred as the fourth state of matter comprised of positive and negative ions, electrons, excited and neutral atoms, free radicals, molecules in the ground and excited states and the UV photons [3]. However, due to the porous structure of the wood treatments of surface are not adequate so that the use of Plasma Activated Water (PAW) [4], a water that contains mainly reactive species, could be an alternative method for oak barrel sanitization.

This was the first time that PAW was put into contact with oak wood barrel surface. This study was aimed to analyze from a sensory point of view the impact of the immersion of the oak fragments on PAW in the organoleptic profile of red wines, compared to wines that aged with wood treated with the burning of a sulfur disc and with wood immersed in distilled water.

2 Materials and methods

2.1 Wine sampling

Wines that had been in contact with oak wood from two origins (American and French) with two toasts (Medium and Plus), were analyzed.

The treatments consisted of the immersion of the different oak wood in several liquids during 30 min. These liquids were the distilled water and the PAW generated by direct plasmation of distilled water. Moreover, oak wood fragments were also submitted to the burning of a sulfur disc. The essay was performed with triplicates so that three fragments of wood were introduced in a glass jar containing 3 liters of young red wine. They were in flotation, with an inert atmosphere, and covered with a lid. The wines were stored for 8 weeks in a chamber at 35 °C.

2.2 Sensory analysis

The sensory analysis was carried out using the approved wine black glasses to mask the color of the wine while studying the olfactory and taste phases.

To evaluate the existence of significant differences between the samples, a discriminative test was carried out: multiple difference test [5], in which the magnitude of the difference of the different samples with respect to the control at olfactory and gustatory level was asked, using an ordinal qualitative variable of 6 points: None (0), Very Slight (1), Slight (2), Moderate (3), Much (4) and Very Much (5). The discriminative analysis was carried out by a panel of 10 tasters. The interpretation of the results was carried out using the non-parametric analysis for more than two independent samples of Kruskal-Wallis test and the post hoc Dunn's multiple comparison test with the Rsoftware (v.4.2.2; R Core Team, 2022) [6].

In addition, a descriptive test was carried out using two tasting sheets, one for the intensity, quality and harmony for the olfactory phase and other sheet for the taste phase, both using a structured 7-point scale.

3 Results and Discussion

3.1 Discriminative test with multiple difference test

The discriminative results of the magnitude of the difference for the olfactory phase are shown in the Table 1. In the wines aged with the American Medium toasted and with French Plus toasted oak fragments, significant differences were not found between samples. In contrast, when the wood was American Plus toasted and French Medium toasted the wines aged with oak treated with the burning of a sulfur disc (SO₂) were significantly "much different" to the other samples.

Table 1. Magnitude of the difference (median) of the olfactory phase for the wines after ageing with American (A), and French (F), with Medium (M) and Plus (P) toasted oak fragments that were treated with distilled water (CW), with PAW and with the sulphur burning of a sulfur disc (SO₂) and Kruskal-Wallis test and post hoc Dunn's multiple comparison test.

Samples	AM	AP	FM	FP
CW	0	0.5a	1a	0
PAW	1.5	2a	1a	3
SO ₂	1.5	4b	4b	1

*Different letters mean significant differences between samples for each oak type ($p < 0.05$).

The discriminative results of the magnitude of the difference for the gustatory phase are shown in the Table 2. In the wines aged with the American Medium toasted oak fragments, significant differences were not found between samples. When the wines were aged with American Plus toasted oak fragments, everyone was significantly different, being the ones aged with the burning of a sulfur disc (SO₂) treated oak fragments identified as "moderate-much different" to the control. For the wines aged with French Medium toasted oak fragment, the one aged with oak treated with the burning of a sulfur disc (SO₂) was significantly different to the others and it was identified as "moderate different". When the oak was French but with Plus toasted oak fragments, the ones aged with the PAW treated oak fragments were also significantly different and identified as "moderate different".

Table 2. Magnitude of the difference (median) of the gustatory phase for the wines after ageing with American (A), and French (F), with Medium (M) and Plus (P) toasted oak fragments that were treated with distilled water (CW), with PAW and with the burning of a sulfur disc (SO₂) and Kruskal-Wallis test and post hoc Dunn's multiple comparison test.

Sam202ples	AM	AP	FM	FP
CW	1	1.5a	1a	0.5a
PAW	2	2.5ab	2a	3b
SO ₂	1	3.5bc	3b	1.5ab

*Different letters mean significant differences between samples for each oak type ($p < 0.05$).

In general, the tasters did not identify the wines aged with oak fragments treated with PAW as “much different” wines compared with the control wines treated with distilled water. This result was positive because it means that the application of this new cleaning technology is feasible. Curiously, the wines aged with oak fragments that were treated with the burning of a sulfur disc were significantly discriminated as “much or moderate different” to the others, even more in the gustatory phase.

Despite the burning sulfur disc is the most traditional and antique method for the sanitization of oak barrels during the wine ageing, its application requires trained staff, safety facilities and security devices [7]. According to those authors, efficacy as microbicide and its impact on the wine quality has not been deeply analyzed. Some other authors [8] have described the formation of hydrogen sulphide that reacts with pyrazines of toasted wood to form thipirazines, but results about the impact on the gustatory phase have not been clearly described.

3.2 Descriptive test for wines after ageing with oak wood fragments

When the wood fragments were American Medium toasted oak, wines were in any case perceived as significantly different. This was an interesting result because this type of wood is very used in the wineries [9] and it perfectly supported the PAW treatment. In contrast, when the toasted was “Plus”, the impact of the treatments on the olfactory phase was significant.

In fact, the quality of the olfactory phase of wines aged with American Plus toasted oak fragments treated with the burning of a sulfur disc (SO₂) was described as lower than the other samples (Fig. 1). This low quality of aromas was explained because it was described as less fruity, spicy, woody and more chemical and drier than the other wines. Despite this, the description of the olfactory intensity, the gustatory phase, and the harmony were quite similar between treatments and control, although the wine after the PAW treatment was considered slightly more harmonious than the other samples.

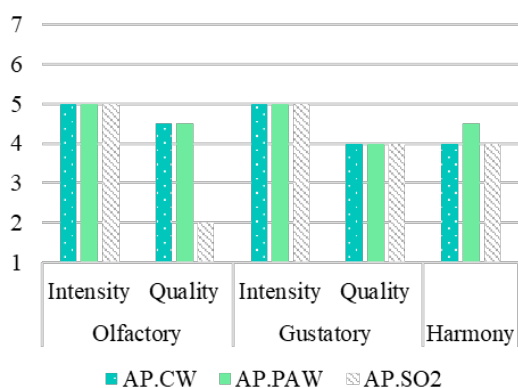


Figure 1. Median of the scores obtained for olfactory, gustatory and harmony description of wines after ageing with American Plus (AP) Toasted oak fragments treated with distilled water (CW), with PAW and with the burning of a sulfur disc (SO₂).

Regarding the olfactory phase of wines aged with French Medium toasted oak fragments it was shown that the control wine had the lowest intensity and the SO₂ wines the lowest quality. In terms of the gustatory phase description, the intensity of wines was similar, being the wine with the PAW treatment perceived as less intense but the one with the highest quality (Fig. 2) what was a very promising result. Again, the wine aged with oak treated with the burning of a sulfur disc (SO₂) was described as the one with the lowest quality in both phases and the lowest harmonious, being described as the less fruity in the olfactory phase and the driest in the taste phase.

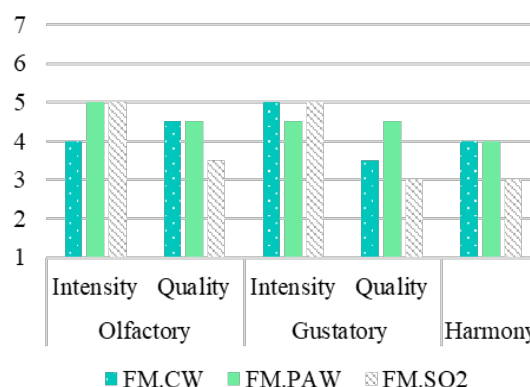


Figure 2. Median of the scores obtained for olfactory, gustatory and harmony description of wines after ageing with French Medium (FM) Toasted oak fragments treated with distilled water (CW), with PAW and with the burning of a sulfur disc (SO₂).

The discriminative test of the wines aged with French Plus toasted oak fragments showed only significant differences in the gustatory phase (Fig. 3). Probably, the toasted level of the wood with this origin was the key for blurring the differences between the wine samples at the olfactory phase. The control was the sample described with a slightly highest intensity but also the one with the lowest quality (Fig. 3) although samples were not differentiated by the harmony. In this case, the control sample was described as the one with the minor body and unctuousity.

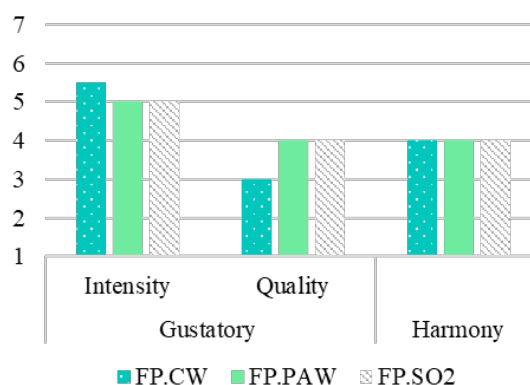


Figure 3. A: Median of the scores obtained for gustatory and harmony description of wines after ageing with French Plus (FP) Toasted oak fragments treated with distilled water (CW), with PAW and with the burning of a sulfur disc (SO₂).

4 Conclusions

Taking into consideration the results found in this study, the PAW treatment of the wood did not exert important differences in wines compared to a control with distilled water. In contrast, the wines aged with oak fragments that were treated with the burning of a sulfur disc were significantly discriminated as much or moderate different to the others, even more in the gustatory phase.

American Medium toasted oak wines were in any case perceived as significantly different and when the toasted was Plus the olfactory and gustatory phases of PAW wines were described as equal to the control. But the SO₂ treatment generated wines perceived as less fruity, spicy, woody and more chemical and drier than the other wines.

Wine aged with French Medium toasted oak fragments treated with PAW were described as the ones with the highest quality at the gustatory phase. Nevertheless, the wines from the SO₂ treatment were established as the one with the lowest quality in both phases and the lowest harmonious, being described as the less fruity in the olfactory phase and the driest in the taste phase. Wines aged with French oak fragments but with the Plus toasted provides wines after treatments that were described as the control wines but that reached higher gustatory quality than control wines.

Apparently, the origin and toasted of the oak fragments exert some impact on the sensory analysis results, and the SO₂ treatment was the one with the most

negative effects on the perception of the intensity and quality of the gustatory and olfactory phases.

In general, the treatment of the wood with PAW did not have a negative impact on the sensory quality of the wines, regardless of the origin and toasting of the wood.

Referencias

1. Malfeito-Ferreira, M. *Wine Spoilage Yeasts and Bacteria in Encyclopedia of Food Microbiology* **38**, 805-810 (2014)
2. M. Clodoveo, T. Dipalmo, C. Rizzello, F. Corbo, P. Crupi, *Inn. Food Sci. Emerg. Tech.* **38**, 41-56. (2016)
3. D. Dobrynin, G. Fridman, G. Fridman, A. Friedman, *New J. Phys.* **11** (2009)
4. U. Schnabel, R. Niquet, C. Schmidt, J. Stachowiak, O. Schlüter, M. Andrasch, J. Ehlbeck, *Int. J. Env. & Agric. Res.* **2** (2016)
5. M.C. Meilgaard, C. Thomas, C., C. Gail Vance, C. *Sensory Evaluation Techniques* (1999)
6. R Core Team. *R: A language and environment for statistical computing. R Foundation for Statistical Computing* (2022)
7. E. Stadler, U. Fischer, U. (2020). *J. Agric. Food Chem.* 5283-5295 (2020)
8. R.S. Jackson, *Chemical Constituents of Grapes and Wine in Wine Science Principles and Applications* (2014)
9. A. Martínez-Gil, M., del Alamo-Sanza, R. Sánchez-Gómez, I. Nevares, I. *Beverages* **4** (2018)