

ASSESSMENT OF THE OAK CHIPS INFLUENCE ON THE COLOUR AND SENSORY PARAMETERS OF FETEASCA NEAGRA BY RAPID MATURATION SIMULATION

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Abstract

Feteasca Neagra is a Romanian black grape variety, which is vinified in various styles, but seems to evolve better when matured in oak barrels. To rapidly assess the evolution of this variety in the presence of wood, an experiment was designed to account for the contact with wood, as well as the presence of lower or medium oxygen levels. Oak chips of different origins (American and French) were used, each with a medium and high degree of toasting, respectively. Freshly obtained wines were kept in contact with 3 g/l of these oak chip samples for 2 months, either protected from air or in conditions simulating the oxygenation level obtained during barrel maturation. The colour of the samples was assessed spectrophotometrically after 2, 4, 6 and 8 weeks of wood contact. Sensory analysis of wines was also performed after 2 months of wood contact. After this short experimental period it was observed that oxygen is correlated with a reduced clarity and is inducing a more yellow hue in the wines, irrespective of the time of assessment. Furthermore, chips are changing the initial colour of wines bringing some yellow hues of their own, especially in the case of medium-toasted oak chips, but in time this effect fades away. The intensity of colour is, however, increased by the highly-toasted chips, the effect being also reduced by oxidative maturation. The sensory analysis showed that the overall quality of the control sample is the most affected by the presence of oxygen, while the aftertaste also scored lower than in the case of wines produced with chips contact. During this short time of experimentation the toasting degree of the chips did not significantly influence the colour parameters and sensory scores, but the origin of the chips is starting to show a sensory influence when wines mature, proven by a slight increase in the sensory scores for samples treated with American chips in the presence of moderate oxygenation level. Considering these findings it seems that Feteasca Neagra would indeed benefit from wood contact, especially with oak of American origin. The abstract is too long (maximum 180 words or 10 rows)

Key words: French oak; American oak; chips toasting level; CIELab parameters; sensory analysis.

INTRODUCTION

Wine maturation is a well-known winemaking practice for improving wine quality and organoleptic characteristics (Breton *et al.*, 2018). The maturation of red wines in oak barrels is a technique commonly used in many wineries (del Álamo-Sanza *et al.*, 2004; OIV, 2019a resolution 8/2001), but alternative methods are also available (Oberholster *et al.*, 2015; OIV, 2019a resolution 9/2001; OIV, 2019b 3/2005, 430/2010, 406/2011). The most common oak species used for barrel making are the French and Eastern European oak *Quercus petraea* Liebl. and *Q. robur* L., respectively or the American oak, *Q. alba* L. (Jordão *et al.*, 2005; Cadahia *et al.*, 2009; Alañón *et al.*, 2011). Besides the positive effects of oak wood on wine flavour, traditional wine maturation in

oak barrel allows wine compounds to interact with constant low doses of oxygen, which is an important factor for producing quality red wines (Bautista-Ortín *et al.*, 2008) with stable colour and sensory characteristics. Many studies revealed that barrel walls are permeable to oxygen and permit the transfer of the oxygen from the air to wine at moderate rates, between 15 and 45 mg/l/year, on the condition that the barrels are new, hermetically sealed and with wet staves (Ribereau-Gayon, 1933; Vivas and Glories, 1997; del Alamo-Sanza and Nevares, 2018). Due to the porosity of oak wood a dose of ≈ 0.135 mg O₂ are transferred by any gram of wood into the wine at the moment of first contact (García-Estévez *et al.*, 2017). A decreasing permeability for oxygen is observed in old barrels with a rate of about 10 mg/l/year for five-year-old barrels (Vivas and Glories,

1997; del Alamo-Sanza and Nevares, 2018). However, in order to reduce the time and costs, maturation of red wines can be accomplished directly in the inox tanks, by using oak-based alternative products (powder, chips, cubes or staves) in combination with micro-oxygenation (MOX) technique. Introducing oak wood products directly in wines proved to be a suitable practice, economically beneficial for early release of young red wines on the market (Oberholster *et al.*, 2015; Kyraleou *et al.*, 2016). Anyway, for applying these techniques with positive results, winemakers also need a micro-oxygenation unit fitted to the stainless tanks, as well as experience and knowledge regarding oxygen management. Time of contact, oak type and toasting, dosage levels in wine are also important factors. Many oak wood studies evaluated the volatile compounds released into the wines (Jordão *et al.*, 2005; Jordão *et al.*, 2006a; Chira and Teissedre, 2013), mostly phenols (Jordão *et al.*, 2007; Jordão *et al.*, 2012; Chira and Teissedre, 2013; 2015; Izquierdo-Cañas *et al.*, 2016), but also other compounds with impact on wine chemical matrix and sensory properties (de Coninck *et al.*, 2006; Gonçalves and Jordão, 2009; Oberholster *et al.*, 2015).

MATERIALS AND METHODS

This study used a young red wine produced from Feteasca Neagra in 2018 through classic red winemaking technology at Pietroasa Viticulture and Enology Research and

Development Station. The grapes were harvested on 25th September 2018, sulphited with 30 mg/kg SO₂, then crushed and destemmed. Crushed grapes were treated with an extraction enzyme Enovin Color 2 g/q and, after homogenization, with a mixture of condensed and hydrolysable tannins Tanicolor Super 15 g/hl and then inoculated with 20 g/hl active dry yeast Viniferm TTA (Agrovin). The maceration-fermentation was conducted at 25-28°C for 4 days in a roto-fermentor. After 4 days of skin contact during maceration, the fermented grapes were pressed and the resulted wine allowed to continue the alcoholic and malolactic fermentations in 100 hl tanks. Once the malolactic fermentation finished, the wine was racked off lees, acidified with 1 g/l tartaric acid and sulphited with 50 mg/l SO₂. The main wine parameters determined based on the standardized methods (OIV, 2018) were: 15.6% vol. alc.; 6.04 g/l total acidity expressed as tartaric acid; 0.78 g/l volatile acidity expressed as acetic acid; 25.9 g/l total dry extract; 24.1 g/l non-reducing extract; 1.8 g/l reducing sugars. For the maturation experiment, the base young wine was introduced in bottles of 1.0 litre capacity with a dose of 3 g/l oak chips and left for 2 months at 16°C. The chips used were of different types (two different levels of toasting, medium or high, and two botanical origin, French or American) and maturation was performed with two different levels of oxygen. Wine sample variants were prepared in triplicate and are detailed in Table 1.

Table 1. Types of oak chips and level of oxygen used for the variants of fast maturation of red wine

Codification of samples	Oak botanical origin	Toasting level	Oxygen level low*	Oxygen level moderate (barrel-like)**
FR_M	French oak	Medium	x	
FR_M_OX	<i>Quercus petraea</i>			x
FR_H		High	x	
FR_H_OX				x
AM_M	American oak	Medium	x	
AM_M_OX	<i>Quercus alba</i>			x
AM_H		High	x	
AM_H_OX				x

*Low oxygenation obtained by fully occupying the space in the bottles; **Moderate oxygenation obtained by leaving a headspace of 7 ml of air in 1-litre bottles, resulting in rough <4 mg O₂/l/month.

The level of oxygenation was implemented by taking into account the reference value of 50 mg/l/year of oxygen, considered valid for new oak barrels, which means that approximately

0.14 mg O₂/l/day are required to simulate an oak barrel oxygenation medium, with a very good oxygen transmission rate (OTR). To accomplish this OTR, the experiment was

designed to have the bottles opened every two weeks to permit the oxygen to enter into the headspace in a controlled manner. To be able to do this, the headspace was readjusted every time the bottle was opened in order to introduce about 1.92 mg O₂/1/2 weeks. To put this scenario into practice, based on the desired oxygen level per bottle, we have calculated the required headspace and the filling level, taking in to account the ambient temperature of 16°C. Our estimations used the ideal gas law, the transformation of mg into ml of oxygen, the calculated density of oxygen at our ambient temperature, the molar mass of oxygen which is 31.99 g/mol, temperature (16°C) and universal gas constant, 0.08206 l·atm·K⁻¹·mol⁻¹. The resulted density of oxygen ($\rho^{16^\circ\text{C}}\text{O}_2=1.348216\text{ kg/m}^3$) allowed us to estimate the volume of oxygen in ml ($\approx 1.42\text{ ml O}_2$) for our experimental conditions. However, knowing that the content of oxygen in the atmosphere is 20.95% by volume (Williams, 2019), the headspace would be about 6.78 ml of air. Because every 2 weeks when the bottles were opened wine was also taken for analyses, in order to simplify the methodology and keep the headspace in the bottles constant, the removed wine for analyses was replaced with the same amount of control wine, keeping in each bottle a headspace of 7 ml.

A UV-VIS spectrophotometer Analytik Jena AG Specord 250 equipped with WinAspect software version 2.2.7 was used to determine CIELab parameters. The CIELab parameters (OIV, 2018: method OIV-MA-AS2-11) were calculated automatically for each wine from the transmittance spectrum measured every 5 nm in the range of 380-780 nm, using glass cuvettes of 2 mm optical path length.

The sensorial analyses were performed after the maturation period, by a team of five evaluators, using a simplified U.C. Davis 20-point system (Table 2) developed for rating of large number of experimental samples (Amerine *et al.*, 1959; Bamforth and Cook, 2019; AWS, 2020). Accordingly, the evaluators analysed the following sensory traits awarding scores for each as follows: visual aspect (clarity and colour), maximum 3 points; olfactory characteristics (intensity and quality), maximum 6 points; olfacto-gustatory and mouthfeel traits (intensity and quality), maximum 6 points; aftertaste, maximum 3 points and overall impression (harmony), maximum 2 points. Wine rating according to their scores and the perceived quality can be: Extraordinary (18 to 20 points); Excellent (15-17 points); Good (12-14 points); Acceptable (9 to 11 points); Deficient (6 to 8 points); Poor and Objectionable (0 to 5 points).

Table 2. Implementing scores according to the perceived quality characteristics of the wine (AWS, 2020)

Assigned score	Visual (0-3 points)	Olfactory (0-6 points)	Olfacto-gustatory and mouthfeel (0-6 points)	Aftertaste (0-3 points)	Overall impression (0-2 points)
6	-	Extraordinary	Extraordinary	-	-
5	-	Excellent	Excellent	-	-
4	-	Good	Good	-	-
3	Excellent	Acceptable	Acceptable	Excellent	-
2	Good	Deficient	Deficient	Good	Excellent
1	Poor	Poor	Poor	Poor	Good
0	Objectionable	Objectionable	Objectionable	Objectionable	Poor

RESULTS AND DISCUSSIONS

The influence of the oak origin, regardless of oxygenation level

The evolution of clarity (parameter *L*, limpidity or lightness, Figure 1, left) followed a specific pattern in all samples during the experimental period, uncorrelated with the oxygenation level,

chips presence or degree of chips toasting. An increase in clarity during the first 48 days of

maturation was observed in all samples, followed by a decrease during the next 16 days. On the last 16 days of the maturation, control samples, without oak chips contact, recorded a higher drop in clarity than the samples treated with oak chips. However, the French oak seems to provide the best final clarity (*L*), while the American oak showed a behaviour closer to that of the control samples. An explanation can be the different level of tannin in the oak of different origins (able to precipitate some of the proteins and modify the colour intensity). Other

explanation for the reversal of the wine lightness after the first 48 days could be the beginning of precipitation of unstable colour matter in young red wines, a temporary phase in their path towards stabilisation, as well as an increase in colour intensity due to wine oxidative evolution.

On the other hand, the yellow component of the colour, the parameter $+b$ (Figure 1, right) followed a decreasing trend during the first 48 days, while after another 16 days of maturation the positive evolution was reversed, the parameter b showing a sharp increase. The other

CIELab parameter, $+a$, which correlates with the red colour, followed an increasing trend in the first 48 days, but remained constant afterwards (Figure 1, right). On the 64th day, the reddest were the repetitions of the control wine (Figure 1 right, parameter $+a$), followed by those treated with French oak and then by those obtained in the presence of the American oak. In the same time (Figure 1, right), the less yellow samples (parameter $+b$) samples were those treated with French oak, thus better preserving the hue of the red young wines, in which the yellow shades are not appreciated, being considered a sign of premature evolution.

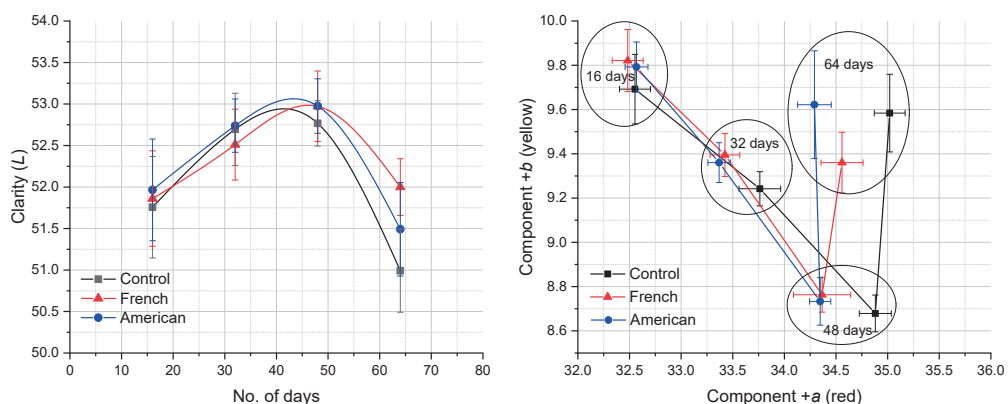


Figure 1. The influence of the oak origin, regardless of oxygenation level, on the CIELab parameters: left - clarity (L) evolution; right - red and yellow colour evolution in the ab colour space

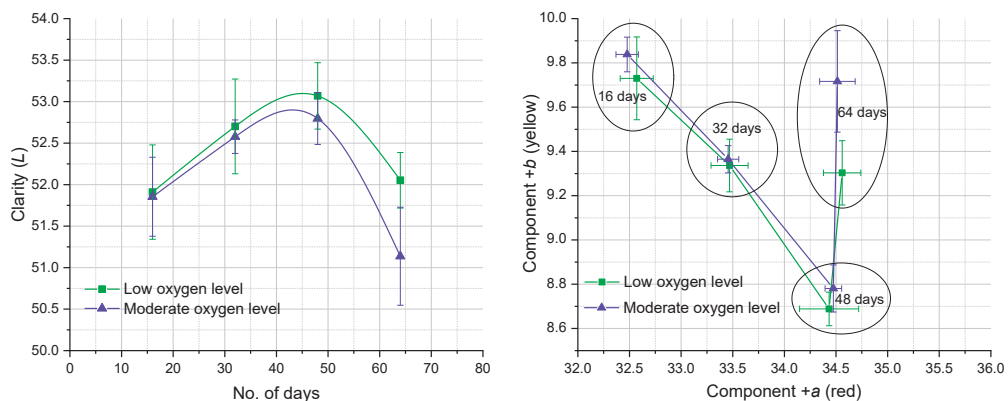


Figure 2. The influence of the oxygenation level, regardless of the oak chips type, on the CIELab parameters: left - clarity (L) evolution; right - red and yellow colour evolution in the ab colour space

The influence of the oxygenation level, regardless of the oak chips type

Both moderate and low oxygen levels (Figure 2, left) increase the clarity (L) similarly during

the first 48 days, while on the last 16 days, when the wines start to stabilize, the samples exposed to low oxygen levels, have the tendency to keep a better clarity, than those exposed to more oxygen (moderate levels).

Thus, introducing a moderate level of oxygen in the wine leads to a lower lightness, which can also correlate to a higher colour intensity. On the other hand, the results from Figure 2, right, show an increase of redness (parameter $+a$) and a decrease of yellowness (parameter $+b$) during the first 48 days in a similar way for both groups, low- and moderate-oxygen level. On the last 16 days, the redness (parameter $+a$)

stagnates, while yellowness (parameter $+b$) increases significantly in all cases, but more pronounced for samples exposed to moderate oxygen level, a sign of higher oxidation. These results suggest an accelerated maturation process in samples exposed to a moderate level of oxygen, as compared to the cases of only low oxygen levels.

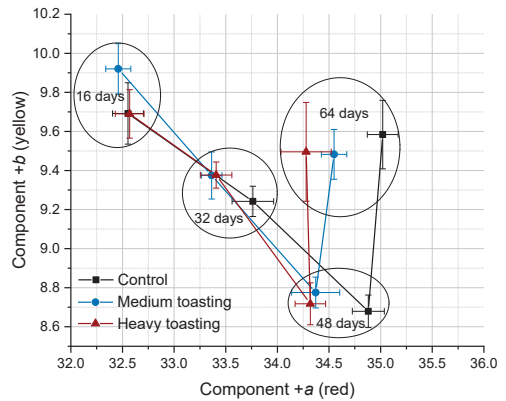
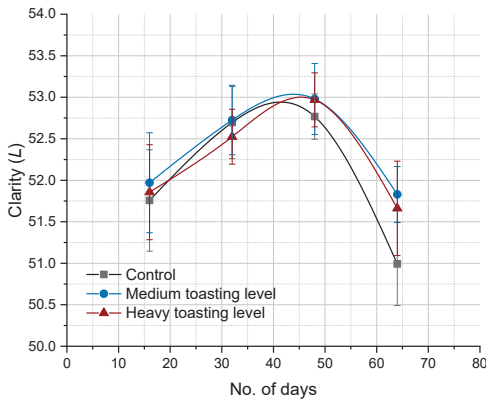


Figure 3. The influence of the oak chips toasting level, regardless of oak chips origin and oxygenation level, on the CIELab parameters: left - clarity (L) evolution; right - red and yellow colour evolution in the ab colour space

The influence of the oak chips toasting level, regardless of oak chips origin and oxygenation level

The level of toasting did not affect directly the clarity (L), the effect of an increased clarity coming from the contact with the oak wood of any type (Figure 3, left). The evolution of this parameter was similar to the one seen and explained already in Figure 1, left, the samples with oak showing a better clarity (L) than the

control, in the 64th day of evolution. However, as it results from Figure 3, right, the control samples were the reddest (parameter $+a$) and the yellowest (parameter $+b$) after 64 days of maturation, while the oak wood contact decreased both parameters a little, irrespective of their toasting level. Nevertheless, medium toasting seems to give slightly better results regarding the red parameter ($+a$) after 64 days of maturation.

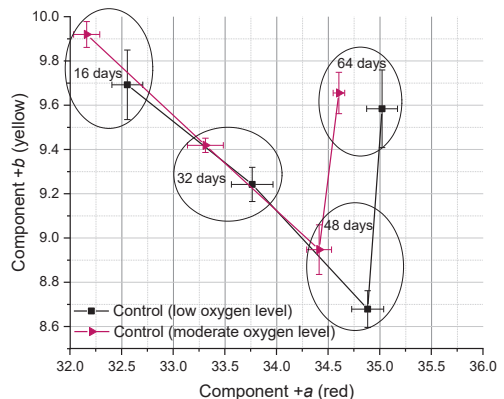
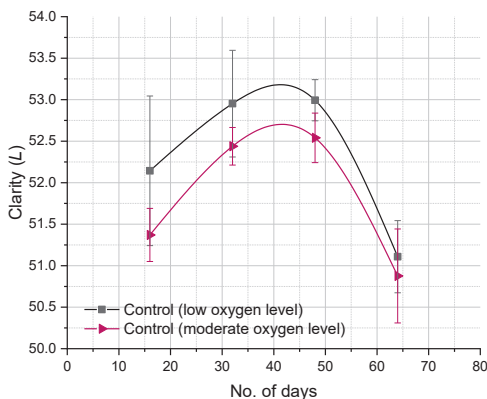


Figure 4. The influence of the oxygenation level on the control samples on the CIELab parameters: left - clarity (L) evolution; right - red and yellow colour evolution in the ab colour space

The influence of the oxygenation level on the control samples (without any wood contact)

The overall influence of oxygen can be observed in Figure 4, left, where moderate levels of oxygen induce lower colour lightness over the entire period of 64 days, which means that the colour is more intense in those samples. In the Figure 4, right, the parameter *+a* (red) increase is more pronounced in the low oxygen samples during all the 64 days. The parameter *+b* showed decreasing values during the first 48 days, increasing then sharply by the 64th day. The samples exposed to low oxygen level systematically maintained lower yellow hues as compared to the samples exposed to higher

doses of oxygen (moderate level), confirming that the yellow increases with the oxidation of wine.

In all cases presented, the constant increase in the red parameter values over the entire period of 64 days can be explained by the condensation reactions known to take place between tannins with anthocyanin. On average, oxygenated samples have a more intense colour, while, in low oxygenated samples, the clarity (*L*) and red (*+a*) values are higher.

The control samples without oxygen treatment showed the lowest values for parameter *+b*. The yellowness is influenced on one hand by slow oxidation and on the other hand by extraction of yellow-brown compounds from oak.

Table 3. Detailed sensory evaluation scores on the main categories and total for experimental wines

Samples	Visual (≤3 pct.)	Olfactory (≤6 pct.)	Olfacto-gustatory and mouthfeel (≤6 pct.)	Aftertaste (≤3 pct.)	Overall impression (≤2 pct.)	Total score (≤20 pct.)
CT	1.60±0.22 ^a	2.60±0.22 ^{cd}	2.80±0.27 ^{bc}	1.60±0.22 ^c	1.10±0.22 ^a	9.70±0.67 ^{cd}
FR_M	1.80±0.27 ^a	3.80±0.57^{ab}	3.40±0.42^{ab}	1.90±0.22^{abc}	1.10±0.22 ^a	12.00±1.41 ^{ab}
FR_H	1.80±0.27 ^a	4.40±0.65^a	4.24±0.34^a	2.24±0.25^{ab}	1.10±0.22 ^a	13.78±1.02 ^a
AM_M	1.90±0.42 ^a	4.30±0.57^{ab}	3.80±0.57^a	2.40±0.22^a	1.10±0.22 ^a	13.50±0.94 ^a
AM_H	1.80±0.27 ^a	4.20±0.57^{ab}	4.24±0.49^a	2.30±0.27^{ab}	1.10±0.22 ^a	13.54±0.75 ^a
CT_OX	1.60±0.22 ^a	2.30±0.45 ^d	2.10±0.22 ^c	1.50±0.35 ^c	1.00±0.00 ^a	8.50±0.61 ^d
FR_M_OX	1.80±0.27 ^a	3.30±0.57 ^{bcd}	3.40±0.89 ^{ab}	1.70±0.27 ^{bc}	1.10±0.22 ^a	11.30±1.44 ^{bc}
FR_H_OX	1.80±0.27 ^a	3.50±0.61 ^{abc}	3.30±0.45 ^{ab}	1.70±0.45 ^{bc}	1.00±0.00 ^a	11.30±1.20 ^{bc}
AM_M_OX	1.80±0.27 ^a	3.60±0.42^{abc}	4.20±0.27^a	1.90±0.22 ^{abc}	1.10±0.22 ^a	12.70±0.67 ^{ab}
AM_H_OX	1.90±0.22 ^a	3.70±0.27^{ab}	3.60±0.42^{ab}	1.90±0.42 ^{abc}	1.10±0.22 ^a	12.20±0.45 ^{ab}

^{abcd}ANOVA - Tukey Test (p < 0.05)

The sensory evaluation results are summarized in Table 3. Sensory scores of experimental wines showed that the oak chips considerably induce sensorial changes on the following levels: Olfactory, Olfacto-gustatory and mouthfeel and on Aftertaste.

In accordance to the sensory evaluation scores, the most appreciated wines were, as follow: 1) the group less exposed to oxygen and treated with either French oak or American oak (medium and heavy toast alike) - scores between 12.00-13.78, not significantly different; 2) the group exposed to moderate oxygen levels and treated with American oak (medium and heavy toast alike) - scores between 12.20-12.70, not significantly different, 3) the group exposed to moderate oxygen levels and treated with French oak (medium and heavy toast alike) - scores of 11.30, not significantly different and 4) the control wines, with scores of 9.7 (exposed to

low oxygen levels) and 8.5 (exposed to moderate oxygen levels), not significantly different.

The statistical differences among total scores showed that oak chips, regardless of botanical origin, under the condition of low oxygenation are the most suitable for maturation of wines produced from Feteasca Neagra.

CONCLUSIONS

The effect of oxygen during maturation. The wine samples exposed to more oxygen levels tend to have a decreased clarity (*L*) and a more intense colour, as well as an increased yellowness (parameter *+b*). The redness (parameter *+a*) is slightly decreased over 64 days of maturation in samples exposed to moderate oxygen level as compared to low level. The wines produced from Feteasca Neagra treated with a low dose of oxygen

showed a higher redness (parameter $+a$) and a lower yellowness (parameter $+b$), suggesting a better wine development under these conditions.

The effect of oak chips during maturation.

Oak chips slightly increase the clarity (L) during 2 months of maturation of wine samples as compared to control wines, while an increased stability regarding colour matter precipitations is expected after the treatment. On the other hand, oak chips increased yellowness (parameter $+b$) by extraction of yellow-brown compounds into wines, further influenced by oxidation. The yellowest samples were those treated with American oak, followed by control samples and then the samples treated with French oak.

The effect of chips toasting level. The different level of toasting did not significantly influence the clarity (L), redness (parameter $+a$) or yellowness (parameter $+b$), during 64 days of maturation. The changes compared with control samples are induced by the treatment with oak rather than the toasting level of oak.

The effect of treatments on sensory properties.

The best rated wines were those treated with oak wood (irrespective of the oak origin or toasting level) matured under low oxygen levels. Under the conditions of moderate oxygen exposure, the American oak chips proved more suitable than the French oak for Feteasca Neagra, but the scores at the sensory evaluation are still lower than for any wine treated with oak and exposed to low oxygen level. All the oak treated wines obtained higher scores than the samples not in contact with oak. However, because when exposed to more oxygen the American oaked wines behaved better than the French oaked wines, it is expected that the wines in contact with American oak will evolve better in time. Thus, we can safely recommend for the wines of Feteasca Neagra to be matured in contact with American oak, under low levels of oxygen.

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