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Effect of wine maturing on the colour and chemical properties of Chardonnay wine

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Abstract: Untreated chardonnay wine was used as the raw material for this study. The wine samples were divided into three groups and monitored over 32 weeks. Three ways of wine maturation were used: glassware, wooden barrel and the addition of oak chips for 6 weeks, which can significantly increase total polyphenols ($P < 0.05$) and flavonoids ($P < 0.05$) concentration. The use of oak shavings had a comparable effect to the oak barrels. The use of oak shavings can replace wood barrels in the maturation process. The oak shavings also achieved lower oxygen concentration ($P < 0.05$) in wine. Ageing the Chardonnay increased ($P < 0.05$) the polyphenol concentration and had an impact on the wine colour under the different maturation conditions.

Keywords: quality; barrel; oak chips; colorimetry; polyphenols

Wine contains over a hundred ingredients, one third of which consist of antioxidants and phenolic compounds. Polyphenols are most commonly found in the skin and the pulp of grapes. These components are able to interact in the production process and play an important role in the antioxidant properties of wines (Ribéreau-Gayon et al. 2006). Chardonnay is a medium ripening grapevine variety. The variety is suitable for maturation in barrels, or for production of sparkling wines (Pospíšilová et al. 2005). There is a large difference in the content of the phenolic substances among the white and red grapevine varieties. Their quantity is influenced by the climatic conditions and technological adaptations after harvesting, the length of maceration, the temperature, the alcohol content, the pH, etc. (Downey et al. 2003; Villano et al. 2006). The flavonoid content is naturally higher in red wines due to the higher concentration of polyphenols in the skins and the way the red wine is produced. Frequently, their accumulation in the outer skin layers can be observed, because their biosynthesis is stimulated by solar radiation (Villano et al. 2006).

Maturation in oak barrels is an important step in wine-making. The species of oak that are traditionally used are *Quercus alba*, also called the American oak, *Q. sessilis* and *Q. robur* referred as French oaks belong to the oak species which are mostly used for wine maturation in Europe. The barrel production technology itself, treatment and frequency of the barrel usage also affect the chemical properties of the wood (Ancín et al. 2004; Spillman et al. 2004). Barrels can be toasted in several stages. Depending on the degree of toasting, the barrels can be divided into three groups (Chatonnet et al. 1994). The thermal degradation of the carbohydrates generates furans, the degradation of the lignin produces volatile phenols, and the dehydration of the acids present in the wood produces lactones (Cerdán et al. 2002). Using oak shavings (chips) can be an alternative solution to ageing in barrels. By using the oak shavings in white wines, the oxidation and colour changes that occur when

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ageing in barrels can be avoided (Pérez-Coello et al. 2000). Micro-oxidation, which is typical for maturing in barrels, is often replaced by micro-oxidation, when using oak shavings. Their porous surface also brings oxygen molecules that naturally react with the wine and influence its character.

MATERIAL AND METHODS

Untreated Chardonnay wine was used as the raw material. Wine has been provided by agricultural cooperative PD Vinohrady Choňkovce the Slovak republic. The wine was not treated. The wine was stabilised by adding $K_2S_2O_5$ in the amount of 40–200 mg L^{-1}. Seventy-five litres of the wine was divided into three groups and stored in three different containers. The first (control) group was stabilised with 4 mg L^{-1} $K_2S_2O_5$ and stored in a 25 L glassware for the entire duration of the experiment (32 weeks). The second (barrel) group was treated with $K_2S_2O_5$ at dose of 8 mg L^{-1} and stored in a new 25 L wooden barrel for 6 weeks. Then the wine was withdrawn into a glass demijohn and stored for another 8 months. The third (chips) group was filled into a demijohn (25 L), treated with $K_2S_2O_5$ at dose of 8 mg L^{-1} and oak chips at 2 g L^{-1} of wine were added and stored for 6 weeks. Subsequently, the wine was withdrawn and the oak chips were removed. The wine was stored in a clean glass demijohn for another 8 months. We used a new oak barrel, 25 L in volume, made from Slovak oak by a Slovak producer in Čečejovce (district Košice, surroundings). The barrel was treated one week before the experiment. The barrel was filled with a solution of 10 g L^{-1} of tartaric acid and water. The oak chips were commercially purchased from Protea (Gensac la Pallue, France). We chose medium toast chips. The temperature was limited to 16 °C during the experiment.

Determination of wine colour. The colour of the wine samples was measured by a Chroma meter CR-410 (Konica Minolta, Sensing, Inc., Japan) using CIELAB L^*, a^*, b^* values. For the measurement of the colorimetric data, (measurement area Ø 50 mm, illuminance D65, standard observer angle 2°) an installed cell holder (CR-A501) and a tube cell (CR-A502; Konica Minolta, Sensing, Inc., Japan) was applied along with the measurement parameters. The Colour Data Software CM-S100w SpectraMagic™ NX (Konica Minolta Sensing Inc., Japan) was applied too. The chroma meter was standardised using a white standard plate (CR-A43; Konica Minolta, Sensing, Inc., Japan) before the measurement. The results reported are the average values of six measurements.

Concentration of oxygen. The oxygen concentration was determined using an Oxi 3205 Set by WTW (Germany). A gauge was inserted directly into the container in which the wine was stored. Each wine sample was analysed six times.

Total polyphenols, flavonoids and antioxidant activity determination. The total phenolic content was determined using a Folin-Ciocalteu reagent (Singleton et al. 1999). The method is based on an oxidation-reduction reaction. Phenolic substances are oxidised in the alkaline medium while reducing the phosphotungstic-phosphomolybdenum complex to produce a blue colour. The samples were analysed spectrophotometrically at an absorbance of 750 nm. Gallic acid was used as the standard according to Singleton (1995). The determination of the total flavonoids was performed using the aluminium chloride and sodium nitrite method using gallic acid as the standard according to Zloch et al. (2004). To determine the antioxidant activity, the simple method of the DPPH (2,2-diphenyl-1-picrylhydrazyl) radical uptake was used. The DPPH solution was prepared before each determination by a concentration of 0.0025 μg L^{-1}. The colour changes were determined spectrophotometrically by measuring the absorbance at 517 nm. The magnitude of the antioxidant activity was expressed as the inhibition percentage of the DPPH radical according to Brand-Williams (1995). Each wine sample was measured six times. For the measurement, glass cuvettes were used.

The starting values of the wine before ageing are shown in Table 1.

Statistical analysis of the results. The results of the work were processed by GraphPad Prism 8.3.0 (2019). The results of the colour determination were processed by using the IBM SPSS Statistics 23 for Windows (SPSS, USA). A one-way analysis of variance (ANOVA) and Tukey's test for multiple comparisons of the means with a confidence interval set at 95% was conducted. The oxygen concentration, total polyphenols and flavonoids in the individual wine samples were compared

Table 1. The starting values of the wine before ageing under the three different conditions

Initial values	Mean ± SD
Oxygen concentration (mg L^{-1})	0.73 ± 0.03
Total polyphenols (g L^{-1})	0.28 ± 0.02
Total flavonoids (g L^{-1})	0.20 ± 0.02
Antioxidant activity (%)	48.26 ± 0.03

SD – standard deviation

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using a one-way ANOVA assay. Tukey's comparison test was used to compare the statistical differences between the means. The statistical significance level was set at 95 %. The obtained data were presented as mean ± standard deviation (SD).

RESULTS AND DISCUSSION

Colour. The results of the L^*, a^*, b^* colorimeter parameter are presented in Table 2. Storing the wine with oak chips changes the L^* parameter (range from 0 – black to 100 – white) of the CIE L^*, a^*, b^* colour space. Lower values of the L^* parameter (barrel) were determined during the maturation. The wood porosity and associated micro oxidation significantly affected the lightness of the Chardonnay wine samples ($P < 0.05$). Although the a^* colorimetric parameter (range from negative – green to positive – red) and the b^* colorimetric parameter (range from negative – blue to positive – yellow) were affected. Statistically significant changes in the colorimetric parameters of the experimental wine samples were observed ($P < 0.05$). Maturation with oak chips and storage in a barrel resulted in increased a^* values of the wine samples. However, the values of the b^* colorimetric parameter showed a statistically significant decreasing trend ($P < 0.05$). The wood provides substances formed during the growth of a tree or in the production of a barrel. A typical example is the toasting of the barrels, when the wood is enriched with Maillard reaction products originating from the pyrolysis of wood (Vivas & Glories 1993). In our experiment, the colour changes in the wine samples were the most significant in the L^* colorimetric parameter. Higher L^* values were determined at the initial stage of the experiment in the control samples than at the end of the maturation. The lowest L^* values were obtained from the barrel with the chips samples. Due to the ageing in the barrel, the wine colour changed and the L^* values decreased. The sample had a lower lightness compared to the control. The a^* colorimetric parameter determined in the barrel sample group was the highest among the experimental wine sample groups. Due to the transition of the wood phenolic compounds, the a^* axis shifts the colour to a red shade. Skouroumounis et al. (2005) analysed the colour changes of wooded Chardonnay over five years. Oxygen reacts with the Chardonnay, which had the effect on the red spectrum of the CIE L^*, a^*, b^* system. We observed similar conclusions in our experiment in a significantly shorter time span than Skouroumounis et al. (2005). The b^* colorimetric values were the highest in the control sample group before the maturation.

Oxygen content. The results after maturing in the oak barrels and the oak "chips" for six weeks confirmed the effect of the maturing process on the oxygen concentration (Table 3). The sample with the oak shavings showed an initial increase in the oxygen concentration (2.77 mg L^{-1}) recorded during this experiment. At the beginning of the maturation process, the increase in the oxygen concentration was observed in the control group, which could be caused by the wood porosity. Subsequently, oxygen was dissolved into the wine samples. The oxygen saturation after six weeks of wine storage was higher than at initial stage of this experiment. The quality of the wine was significantly affected by the oxygen concentration. It mainly depends on the amount of the dissolved oxygen. The amount of oxygen is important for the proper dosing and consumption of SO_2. For reductive wines,

Table 2. The results of the L^*, a^*, b^* colorimeter parameters in the wine samples during storage (mean ± SD)

Parameter	Group	Ageing conditions			
		initial values	6 weeks	20 weeks	32 weeks
L^*	control	92.06 ± 0.09^{a1}	91.55 ± 0.53^{a12}	91.73 ± 0.64^{a1}	91.33 ± 0.58^{a1}
	chips	92.06 ± 0.09^{a1}	91.73 ± 0.25^{a12}	90.73 ± 0.25^{b2}	90.33 ± 0.58^{b2}
	barrel	92.06 ± 0.09^{a1}	91.00 ± 0.46^{b2}	90.47 ± 0.06^{b2}	90.33 ± 0.58^{b2}
a^*	control	1.61 ± 0.08^{a1}	1.57 ± 0.31^{a1}	1.60 ± 0.10^{a2}	1.57 ± 0.06^{a2}
	chips	1.61 ± 0.08^{b1}	1.58 ± 0.07^{b1}	1.87 ± 0.06^{a1}	1.87 ± 0.06^{a1}
	barrel	1.61 ± 0.08^{b1}	1.60 ± 0.01^{b1}	1.83 ± 0.06^{a1}	1.90 ± 0.10^{a1}
b^*	control	63.33 ± 1.17^{a1}	63.03 ± 0.47^{ab1}	63.13 ± 0.21^{a1}	61.67 ± 0.58^{b1}
	chips	63.33 ± 1.17^{a1}	60.43 ± 0.35^{b2}	59.87 ± 0.15^{bc2}	58.67 ± 0.58^{c2}
	barrel	63.33 ± 1.17^{a1}	60.44 ± 0.49^{b2}	60.33 ± 0.58^{b2}	59.33 ± 0.58^{b2}

The means sharing the same superscript in the columns ($^{1-2}$) and rows ($^{a-c}$) are not significantly different from each other (Tukey's test; $P < 0.05$)

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Table 3. The results of the chemical parameters in the wine samples during storage (means ± SD)

Parameter	Time of the experiment		
	6 weeks	20 weeks	32 weeks
Oxygen concentration (mg L^{-1})			
Control group	2.99 ± 0.01^{a1}	2.58 ± 0.03^{a2}	2.31 ± 0.01^{a3}
Barrel group	5.08 ± 0.01^{b1}	4.36 ± 0.02^{b2}	3.85 ± 0.01^{b3}
Chips group	4.73 ± 0.02^{c1}	2.77 ± 0.01^{c2}	2.33 ± 0.02^{c3}
Total polyphenols (g L^{-1})			
Control group	0.28 ± 0.02^{a1}	0.27 ± 0.02^{b2}	0.27 ± 0.01^{b2}
Barrel group	0.36 ± 0.01^{b1}	0.38 ± 0.01^{b2}	0.37 ± 0.01^{a2}
Chips group	0.37 ± 0.03^{b1}	0.39 ± 0.04^{a2}	0.38 ± 0.02^{a2}
Total flavonoids (g L^{-1})			
Control group	0.20 ± 0.05	0.20 ± 0.03^{a}	0.19 ± 0.04^{b}
Barrel group	0.22 ± 0.01^{1}	0,22 ± 0.02	0.24 ± 0.01^{a2}
Chips group	0.21 ± 0.02^{1}	0.23 ± 0.04^{b2}	0.24 ± 0.07^{b2}
Antioxidant activity (%)			
Control group	46.86 ± 0.02^{a1}	43.75 ± 0.03^{a2}	41.29 ± 0.01^{a3}
Barrel group	55.41 ± 0.02^{b1}	52.36 ± 0.02^{b2}	50.36 ± 0.03^{b3}
Chips group	54.07 ± 0.01^{c1}	50.28 ± 0.03^{c2}	43.96 ± 0.01^{c3}

The means sharing the superscript in the columns ($^{a–c}$) and rows ($^{1–2}$) are statistically significant (Tukey's test; $P < 0.05$)

it is important that the oxygen content is as low as possible. It is important to remember that the effect of oxygen can also change the colour (Pérez-Coello et al. 2000). During ageing in barrels, it is necessary to monitor the oxidation of the wine and the ambient temperature, where the barrels are stored. The reason for the wine's oxidation could be the porosity of oak wood. Oxidation occurs either due to the oxygen transfer through the slots between the individual barrel joints, or due to the presence of catalysts in the wood. Oxidation is partly desirable because it intensifies and stabilises the colour while refining the taste. However, it has to be monitored, otherwise irreversible wine damage could occur (Oliveira et al. 2011). The barrel sample group had the highest measured oxygen concentration values. The increase in the oxygen in the sample stored in the oak barrel ($P < 0.05$) and the "chips" ($P < 0.05$) were recorded. Each sample had a decreased oxygen content after the storage. Subsequently, oxygen degradation was observed after 32 weeks of storage. The highest oxygen content was recorded in the samples maturing for 6 weeks in the oak barrel ($P < 0.05$). The samples with the oak chips had a comparable oxygen content along with the control samples group ($P < 0.05$). The wood provides phenols, which reacts with the oxygen in the wine and helps to prevent wine oxidation. This process is generally used in large-scale production, where the micro-oxidation is controlled. In particular, white wines have recently been produced in a strictly reductive form with minimal controlled micro-oxidation. On the contrary, for red wines, the partial or complete maturing of wine in wooden barrels is desirable. Micro-oxidation helps to soften the tannins, reduces the astringency and bitterness of red and some varieties of white wines (Moran et al. 2018).

Total polyphenols, flavonoids and antioxidant activity. The wine samples in the barrel and the wine with the addition of the oak chips matured for 6 weeks had a significantly increased amount of total polyphenols ($P < 0.05$) (Table 3). The oak chips had a stronger impact on the increased content of polyphenols in the wine samples. After 32 weeks of storage, the concentration of the total polyphenols was comparable to the samples of barrel group ($P < 0.05$). The results of the flavonoid concentration measurements are presented in Table 3. Flavonoids, as a part of polyphenols, are important for wine storage and conservation. The concentration of flavonoids increased in the barrel and the chips sample groups during the storage. We can conclude that the flavonoids helped with decreasing the oxygen content in experimental wine samples. The antioxidant activity was higher in both experimental groups than in the control one (Table 3), however, the changes in the antioxidant activity be-

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tween the sample groups were statistically significant. Flavonoids, cinnamic acid derivatives and also other phenol compounds are the main important wood components. By studying their reactions with free radicals, their positive influence on humans has been confirmed (Xia et al. 2010). The concentration of the total polyphenols increased to 0.38 ± 0.01 g L^{-1} in the barrel sample group and to 0.39 ± 0.045 g L^{-1} in chips sample group. This increase could be caused by the interaction between the wine and oak wood. The most important ingredients extracted from the barrels into wine include cis- and trans-β-methyl-octalactone (so-called whiskey lactone), volatile polyphenols, such as guaiacol, 4-methylguaicol and eugenol. Some of them are subject to wine reactions, so their concentration may vary depending on the length of contact with the wood (Chatonnet et al. 1994). According to Spillman (2004), oak wood mainly enriches wine with furfural, guaiacol and vanillin. The amount of each of these substances is directly dependent on the construction and origin of the wood. The use of oak shavings had similar results compared to the barrel group. The extraction of the substances is comparable to barrel group. The consumption of vanillin in wine is directly dependent on the time the wine is stored in the barrel. Its transfer from the barrel to the wine is the fastest at the start of the maturing process (Garde-Cerdán & Ancín-Azpilicueta 2006). Gambacorta et al. (2011) compared the impact of the technology applied on the total polyphenol content of selected wines. Based on their results, the number of total polyphenols is affected by the length of the maceration. Their obtained results also support the view on the correct selection of the grape variety. Giménez-Martínez (1996) achieved similar results by dipping oak slices in a distillate. Soaking the cuttings has the same effect, depending on the conditions created, as a few months maturation in the barrel. Chattonet (1994) compared the amount of vanillin in wines with and without shavings. As a result of his work, it was found that by the action of the yeast activity, vanillin is metabolised to vanillyl alcohol. Consequently, the concentration is lower than when the oak shavings were used. Flavonoids are the most widely studied group of polyphenols. Catechins, flavonols and anthocyanins are frequently occurring flavonoids in red wines (Guilford & Pezzuto 2011). The positive influence of the flavonoids on the circulatory system as well as their antioxidant activity is important. Yang (2009) focused on the comparison of the antioxidant activity and polyphenol content in fourteen wine samples. He compared both red and white varieties. The subject of their study was to determine the total polyphenols (0.20 g L^{-1}) and flavonoids (0.16 g L^{-1}) in the Chardonnay samples. The control wine had a flavonoid concentration of 0.20 ± 0.15 g L^{-1}. After six weeks of their experiment, the flavonoid content of the pure sample remained unaffected. For their "chips" sample, increased values were recorded from the original 0.20 ± 0.02 g L^{-1} to 0.22 ± 0.02 g L^{-1}. After 32 weeks of our experiment, the value of the control wine sample group had a reduced flavonoid content (0.19 ± 0.00 g L^{-1}). It also increased to 0.24 ± 0.01 g L^{-1} in the barrel sample group, respective to the wine samples with the addition of the chips. Our obtained flavonoid concentrations were higher than in the study of Yang (2009). The variety of the grapevine itself affects the total content of polyphenols, but the role of cultivation, as well as the choice of technological steps, are important too. The Chardonnay sample examined by Yang (2009) showed an antioxidant activity up to 61.00%. On the other hand, the highest antioxidant activity and ability to react with the DPPH radical was only 55.41% in our experiment. Although, a direct dependence between the number of polyphenols and the antioxidant activity was observed.

CONCLUSION

Ageing wine under three different maturation conditions changed the polyphenolic concentration, antioxidant activity and colour parameters of the Chardonnay wine samples. An increase in the total phenols and flavonoids in both experimental groups was observed when compared to the control wine sample group. High-Performance Liquid Chromatography analysis should be applied the further research of polyphenols. From an economic point of the view, we can conclude that the use of oak wood chips by wine producers should be more appropriate than the use of barrels.

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