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Use of acacia barrique barrels – Influence on the quality of Malvazija from Istria wines

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ABSTRACT

In order to improve Malvazija from Istria wine quality, we used two types of barrels: oak (of French and Croatian origin) and acacia (of Croatian origin). After 12 months ageing period all of the wines were analysed chemically and sensorially. Results showed marked differences between oak and acacia aged wines, especially in simple volatile phenol and oak lactones concentrations. During the ageing period a significant increase in furfural, 5-methylfurfural, guaiacol, eugenol and trans-eugenol was noticed. Results pointed out the importance of choosing the right barrels (oak or acacia) and time of leaving the wine in the barrels to achieve the desired goal. The highest rated wines were made in acacia barrels.

1. Introduction

Istria is one of the most remarkable wine growing regions in Croatia. This peninsula is located in the north region of the Adriatic Sea. The climate is predominantly Mediterranean, with temperate winters and hot summers. Malvazija from Istria (Malvazija istarska) is a domestic and widespread grape cultivar in this region that gives a white wine with distinguished organoleptical properties. Wines made from this grape are not generally very aromatic and in a normal process of grape transformation give wine with a limited potential for bottle ageing, i.e., a limited shelf-life. In order to improve wine quality, several modern microbiological (different yeast species and yeast combination; malolactic fermentation) (Swiegers, Bartowsky, Henschke, & Pretorius, 2005) and post fermentation treatment (ageing in different barrels) (Herjavec, Jeromel, Orlić, & Kozina, 2007) procedures can be carried out by the wine producers.

Wooden barrels have been produced at least since the time of Imperial Rome and oak has been used to construct wine barrels for over 2000 years (Jackson, 2000). Many types of wood have been used during this period, but those other than oak have generally seen use only in the construction of large storage barrels. In Europe, together with oak, chestnut and recently, acacia were also applied for this purpose (De Rosso, Panighel, dalla Vedova, Stella, & Flamini, 2009). Barrel ageing is a common practice in red wine production, but Burgundy white wines have traditionally also been fermented in barrel and, worldwide, it is becoming more and more common to use barrels in the elaboration of quality white wines. Several studies have described the influence of wood compounds on the aroma of white wine (Chatonnet, Dubourdieu, & Boidron, 1991; Pérez-Coello et al., 2000; Herjavec et al., 2007). Ageing in oak barrels is a traditional winemaking practice for improving wine organoleptic characteristics and quality. The role of oak wood in wine ageing is crucial in two aspects: (a) the transfer of oak aroma-responsible volatile compounds and astringency-related phenolic compounds to wine; and (b) gentle oxidation of certain compounds by atmospheric oxygen (which, passes through wood pores) resulting in a reduction of astringency and changes in colour (Bozalongo, Carrillo, Fernandez Torroba, & Tena, 2007). Oak compounds contributing to wine aroma are mainly furfural (dried fruit, burned almonds), guaiacol (burn overtones), oak or whisky lactone (woody and coconut notes), eugenol (spices, cloves and smoke character), vanillin (vanilla character) and syringaldehyde (also related to vanilla character). During wine ageing in barrel, volatile compounds extracted from oak wood contribute with aromatic notes of vanilla, smoke and spices (Hale, McCafferty, Larmie,

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Newton, & Swan, 1999). At present, studies on ageing of wines have focused fundamentally on the oak wood used to make the barrels. It is known that the quantity of compounds potentially extractable from oak barrels by wine depends mainly on the geographical origin and on the species of oak (Miller, Howell, Michaelis, & Dickmann, 1992; Mosedale & Ford, 1996), on the seasoning of the staves (Sefton, Francis, Pocock, & Williams, 1993), on the toasting of the barrel (Chatonnet, Boidron, & Pons, 1989; Hale et al., 1999) and on the age of the barrel (Pérez-Prieto, López-Roca, Martínez-Cutillas, Pardo-Mínguez, & Gómez-Plaza, 2003). The type of oak wood is also important, since the relative amounts of volatiles released by different types of oak usually differ hence so do the resulting wines. Tasters tend to prefer a balance between the "oak" aromas ordinarily produced by the oak lactones and the grape variety's own fruity aromas (Pérez-Coello et al., 2000). The oak species most commonly used in barrel making are *Quercus alba*, also known as American oak, *Quercus petraea* (or *Quercus sessiliflora*) and *Quercus robur* which grow in Europe, the most popular being French oak (Humphries, Jane, & Sefton, 1992).

Recently, De Rosso et al. (2009) have investigated the changes in chemical composition of a red wine aged in untoasted acacia, cherry, chestnut, mulberry, and oak wood barrels. Toasting represents a very important process that gives wines specific characteristics (Hale et al., 1999) when matured in contact with the toasted oak wood. In typical good winemaking practice raw or untoasted wood is seldom used, if ever. In this investigation we analysed the contribution of different toasted wood barrels (French and Croatian oak and acacia) on Malvazija from Istria wines. For the first time we analysed the influence of acacia barrels on the wine quality of Malvazija from Istria wines using chemical and sensory analyses.

2. Materials and methods

2.1. Barrels and fermentation

The new barrels used in this experiment (four of each kind) were made from French oak (*Q. petraea* from Allier), Croatian oak (*Q. petraea* from Slavonija) and acacia (*Robinia pseudoacacia* from two different locations in Croatia) and were obtained with the same specification (medium toasted level). The wine used in this experiment was a 2003 Malvazija from Istria white wine produced by Kozlovic winery, Momjan, Croatia. All the barrels were filled with the same wine immediately after the end of alcoholic fermentation. Wine samples were chemically analysed after 3 and 12 months of barrel ageing. Sensory analyses were performed after 12 months of barrel ageing.

2.2. Chemical analyses

Routine analyses of basic components (alcohol, reduced sugar, total acidity, volatile acidity, pH, ash) were made using standard methods proposed by OIV. Alcohol strength was done by measurement of the density of the distillate with a pycnometer, reducing sugars were determined by titrametric-rebelein method, total acidity was determined by potentiometric titration, volatile acidity by titration of the volatile acids separated from the wine by steam distillation, ash content by ignition at a temperature between 500 and 550 °C until complete combustion of organic material was achieved (OIV, 2007). The oak volatile compounds were analysed by the GC–MS (furfural, 5-methylfurfural, phenol, guaiacol, 4-ethylphenol, 4-ethylguaiacol, 4-vinylphenol, cis- and trans-oak lactone, eugenol, cis- and trans-isoeugenol) and HPLC (vanillin, syringaldehyde) method (Brandes, Wendelin, & Eder, 2002).

2.3. GC–MS analyses

2.3.1. Materials

Reference standards of furfural, 5-methylfurural, guaiacol, whisky lactone (cis- and trans-oak lactone), 4-ethylguaiacol, *o*-cresol, *p*-cresol, *m*-cresol, ethylphenol, 4-vinylphenol, eugenol and methyl-4-chlorobenzoate were purchased from Sigma–Aldrich (USA). Methyl-4 (10 mg) was dissolved in 100 ml pure methanol. This solution was used as internal standard.

2.3.2. Sample preparation

To wine (200 ml), 0.5 ml internal standard, 200 μl conc. H_2SO_4 and a spatula tip $K_2S_2O_5$ were added and steam distilled in water until approx. 200 ml of distillate was collected. Conc. H_2SO_4 (200 μl) was added to the distillate and in a funnel was extracted two times with dichloromethane (2 × 25 ml). The combined organic extract was brought to dryness with a Na_2SO_4 on a Rotavapor and dissolved in 0.5 ml chloroform and then injected into the GC–MS.

2.3.3. GC–MS analyses

Analyses were performed on a Hewlett–Packard 5890 gas chromatograph (GC) coupled to a 5970 mass selective detector (MSD). The GC was fitted with a BP-20 capillary GC column of dimensions (60 m × 0.25 mm ID, thickness 0.25 μm). The carrier gas was helium (5.6) and flow rate was 1.8 ml/min. Injected volume was 1 μl in splitless mode at an injector temperature of 255 °C. Oven temperature programme was: starting oven temperature was 50 °C, 20 °C/min to 110 °C, 2 °C/min to 226 °C and 50 °C/min to a final temperature of 255 °C. The mass detector was in positive ion electron impact single ion monitoring mode, at 70 eV.

2.4. HPLC analyses

2.4.1. Materials

Vanillin and syringaldehyde reference standards were purchased from Roth, Germany. These analytes were determined by high performance liquid chromatography with diode array detection (HPLC–DAD) with a 510 Waters system. Vanillin and syringaldehyde (10 mg) were dissolved in 100 ml pure methanol. 3-Hydroxybenzaldehyde (internal standard) was purchased from Roth, Germany. 3-Hydroxybenzaldehyde (32 mg) was dissolved in 100 ml methanol and this solution was used as internal standard.

2.4.2. Sample preparation

To 10 ml sample, 0.5 ml internal standard was added and the alcohol was removed on the Rotavapor for 3 min. The residue was made up to 20 ml with 1 M HCl and then transferred onto an activated RP-18 solid phase extraction cartridge (BondElut, Varian). The phenols were eluted from the SPE cartridge with 5 ml ethyl acetate and the organic solvent was removed on a Rotavapor at 30 °C. The residue was dissolved in 1 ml methanol, filtered (0.45 μm) and then used for HPLC analyses.

2.4.3. HPLC analyses

The column used was a Lichrospher RP-18, 240 × 4.5 μm with precolumn 4 × 4.5 μm (Merck, Germany). Column temperature was 40 °C. Flow was 0.8 ml/min and injection volume was 30 μl. The mobile phases consisted of solutions A (0.5% (v/v) formic acid in water) and B (methanol). The gradient elution conditions were as follows: 0 min 100% A, 20 min 90% A, 75 min 80% A, 80 min 20% A, 95 min 20% A, 100 min 100% A and finally 115 min 100% A.

Detection wavelengths were 280 and 310 nm. Quantification of peak area was performed at 310 nm.

2.5. Sensory analysis

The wines were subjected to sensory evaluation by the 100-point OIV/UIOE method (Crettenand, 1999), and by descriptive analyses (Lindblom, 1999), with a panel of 12 experienced judges.

2.6. Statistical analysis

One-way analysis of variance (ANOVA) and least significant difference (LSD) comparison test using Statistical Modelling with SAS/STAT software (SAS Institute) were done to interpret differences in means, if any, at the 95% confidence level.

3. Results and discussion

3.1. Chemical composition

The results presented in Table 1 show the basic chemical composition of Malvazija wine before the barrel ageing period. As it can be seen the wine was dry with high alcohol content and relatively high total acidity, which is a typical characteristic of Malvazija wine. Tables 2 and 3 contain the results of volatile phenol compound concentration during the ageing period. "Smoke" aromas result from breakdown of wood lignin when the wood is toasted. This is mainly attributed to the simple volatile phenols and in this study four of these compounds were found: phenol, guaiacol, *p*-cresol and *o*-cresol. Phenol itself has little flavour impact (threshold around 40 ppm) while guaiacol has a sweet smoke aroma and a threshold as low as 0.05 ppm. *o*-Cresol and *p*-cresol have medicinal, tarry aromas, the former has a threshold of 0.3 ppm, *p*-cresol is lower at 0.06 ppm (Swan & Burtles, 1978). As it can be seen in acacia barrel ageing wines simple volatile phenols were found. On the contrary, in all oak barrels there were no *o*-cresol and *p*-cresol detected while wines from acacia barrels contained relatively low phenol concentrations. Guaiacol was found in the highest concentration in acacia from Medjimurje barrels (Table 2). Ordinarily, amongst European species, *Q. petraea* contains the highest quantities of oak lactones, furfurals and phenolic aldehydes, though there is high diversity according to geographical origin (Herjavec et al., 2007; Prida & Puech, 2006). Chemical analysis of barrel-aged wines pointed out a higher concentration of oak lactone in wines from French barrels while acacia barrels and also Croatian oak barrels yield wines with very low concentrations of these compounds. Previous investigations by Herjavec et al. (2007) also pointed out low concentrations of oak lactone in wines aged in *Q. petraea* barrels from Croatia. Vanillin and syringaldehyde were the two phenolic aldehydes studied in this work. These compounds are formed during toasting of the barrels through the thermal degradation of lignin (Chatonnet et al., 1989). Vanillin, which can be found in many kinds of wood, is important because of its characteristic scent of vanilla. It can be used as an indicator of fermentation and ageing in oak barrels (Sefton, Francis, & Williams, 1989). Vanillin concentration is known to vary significantly with variation in coopering heat (Chatonnet et al., 1989). Spillman, Pollnitz, Liacopoulos, Skouroumounis, and Sefton (1997) established that there was no significant difference in final vanillin concentration amongst the oak samples they investigated. All Malvazija wines had very low vanillin concentrations after 3 and 12 months ageing period but in spite of that all wines were sensory described as having vanilla character, which could be attributed to the levels of oak lactone (Pollnitz, Jones, & Sefton, 1999). Syringaldehyde has a higher level of organoleptic perception of 15 mg/l. This content, however, never occurred in the wines tested and is unlikely to play a role in organoleptic perception, especially because of its lack of characteristic odour (Puech, 1987). According to Arapitsas, Antonopoulos, Stefanou, and Dourtoglou (2004) syringaldehyde is the most important compound for the discrimination of barrel-aged wine from wine treated with oak chips. But, it is possible that the wood-imparted compounds, while occurring below their detection threshold, interact synergistically with flavours in the wine, thus yielding a detectable change when none would be predicted based solely on concentration data (Miller et al., 1992). Vinylphenols (4-vinylphenol and 4-vinylguaiacol) in white wines and ethylphenols (4-ethylphenol and 4-ethylguaiacol) in red wines are quantitatively the most significant volatile phenols identified as classic components of French wine aroma (Chatonnet,

Table 1
Chemical composition of Malvasia from Istria wines, vintage 2003.

Compound	Malvasia from Istria
Alcohol (vol.%)	13.5
Reduc. sugar (g/l)	2.0
Total acidity[a] (g/l)	6.8
Volatile acidity[b] (g/l)	0.4
pH	3.1
Ash (g/l)	2.2

[a] Tartaric acid.
[b] Acetic acid.

Table 2
Volatile phenol concentrations in Malvazija wines after 3 and 12 months of barrel ageing (mean values of four acacia barrique barrels).

Compound (μg/l)	Acacia from Medjimurje (Croatia)		LSD	Acacia from Istria (Croatia)		LSD
	3 months barrel ageing	12 months barrel ageing		3 months barrel ageing	12 months barrel ageing	
o-Cresol	1.3 ± 0.1	1.3 ± 0.1	n.s.	0.3 ± 0.0	0.3 ± 0.1	n.s.
p-Cresol	0.3 ± 0.2	0.3 ± 0.1	n.s.	0.3 ± 0.1	0.3 ± 0.2	n.s.
m-Cresol	0.9 ± 0.2	0.9 ± 0.0	n.s.	0.5 ± 0.1	0.5 ± 0.1	n.s.
Furfural	405.8 ± 5.2	1236.3 ± 8.9	5% = 90.2	94.2 ± 8.3	82.8 ± 5.8	5% = 5.5
5-Methylfurfural	107.9 ± 2.7	250.6 ± 4.1	5% = 20.7	3.4 ± 1.1	4.3 ± 1.5	5% = 0.20
Phenol	3.0 ± 1.1	3.1 ± 0.9	n.s.	1.2 ± 0.4	1.8 ± 0.7	n.s.
Guaiacol	25.2 ± 1.5	31.4 ± 1.2	5% = 0.22	2.2 ± 0.5	2.7 ± 0.9	n.s.
4-Ethylphenol	0.5 ± 0.1	0.7 ± 0.2	n.s.	0.3 ± 0.1	0.4 ± 0.1	n.s.
4-Ethylguajacol	2.5 ± 0.8	2.6 ± 0.6	n.s.	0.4 ± 0.1	0.7 ± 0.2	n.s.
4-Vinylphenol	32 ± 1.9	31.8 ± 1.1	n.s.	31.7 ± 6.1	37.2 ± 8.0	n.s.
Cis-oak lactone	0.7 ± 0.2	0.5 ± 0.1	n.s.	0.3 ± 0.1	0.4 ± 0.1	n.s.
Trans-oak lactone	0.3 ± 0.1	0.3 ± 0.2	n.s.	0.2 ± 0.1	0.2 ± 0.0	n.s.
Eugenol	7.9 ± 1.8	8 ± 2.0	n.s.	1.9 ± 0.3	2.5 ± 0.8	n.s.
Cis-isoeugenol	0.9 ± 0.1	1.6 ± 0.4	n.s.	0.2 ± 0.1	0.5 ± 0.0	n.s.
Trans-isoeugenol	18.2 ± 3.1	33.1 ± 2.5	5% = 0.77	5.6 ± 1.2	8.6 ± 1.4	5% = 0.67
Vanillin	<0.02 ± 0.01	0.03 ± 0.01	n.s.	0.02 ± 0.01	<0.02 ± 0.0	n.s.
Syringaldehyde	0.17 ± 0.1	0.29 ± 0.1	n.s.	0.16 ± 0.2	0.25 ± 0.1	n.s.

n.s., not significant.

Table 3
Volatile phenol concentrations in Malvazija wines after 3 and 12 months of barrel ageing (mean values of four oak barrique barrels).

Compound (μg/l)	*Q. petraea* from France		LSD	*Q. petraea* from Croatia		LSD
	3 months barrel ageing	12 months barrel ageing		3 months barrel ageing	12 months barrel ageing	
o-Cresol	–	–		–	–	
p-Cresol	–	–		–	–	
m-Cresol	–	–		–	–	
Furfural	405.3 ± 10.1	1795.8 ± 12.1	5% = 99.3	232.9 ± 6.3	740.2 ± 5.1	5% = 11.2
5-Methylfurfural	64.3 ± 5.7	173.3 ± 8.9	5% = 18.7	40.2 ± 2.1	93 ± 2.9	5% = 5.0
Phenol	1.6 ± 0.2	1.6 ± 0.1	n.s.	2.2 ± 0.2	2.1 ± 0.1	n.s.
Guaiacol	3.9 ± 0.4	4.9 ± 0.2	5% = 0.22	7.1 ± 0.3	9.2 ± 0.2	5% = 0.77
4-Ethylphenol	–	–		–	–	
4-Ethylguajacol	0.5 ± 0.1	0.6 ± 0.2	n.s.	1.2 ± 0.1	1.3 ± 0.2	n.s.
4-Vinylphenol	38.1 ± 1.1	43.7 ± 2.1	n.s.	35.2 ± 1.2	35.5 ± 1.3	n.s.
Cis-oak lactone	33.6 ± 3.3	42.7 ± 2.9	n.s.	0.6 ± 0.1	1.2 ± 0.0	5% = 0.21
Trans-oak lactone	30.3 ± 1.8	38.7 ± 1.5	5% = 1.57	0.3 ± 0.1	0.5 ± 0.1	n.s.
Eugenol	5.6 ± 0.6	6.6 ± 0.8	5% = 0.11	3.9 ± 0.3	4.4 ± 0.5	n.s.
Cis-isoeugenol	–	–		–	0.6 ± 0.1	
Trans-isoeugenol	2.1 ± 0.3	3.6 ± 0.2	5% = 0.47	4.7 ± 0.2	7.9 ± 0.9	5% = 0.67
Vanillin	<0.02 ± 0.01	<0.05 ± 0.0	n.s.	<0.02 ± 0.0	0.02 ± 0.0	n.s.
Syringaldehyde	0.05 ± 0.01	0.12 ± 0.01	n.s.	0.05 ± 0.01	0.13 ± 0.0	n.s.

n.s., not significant.

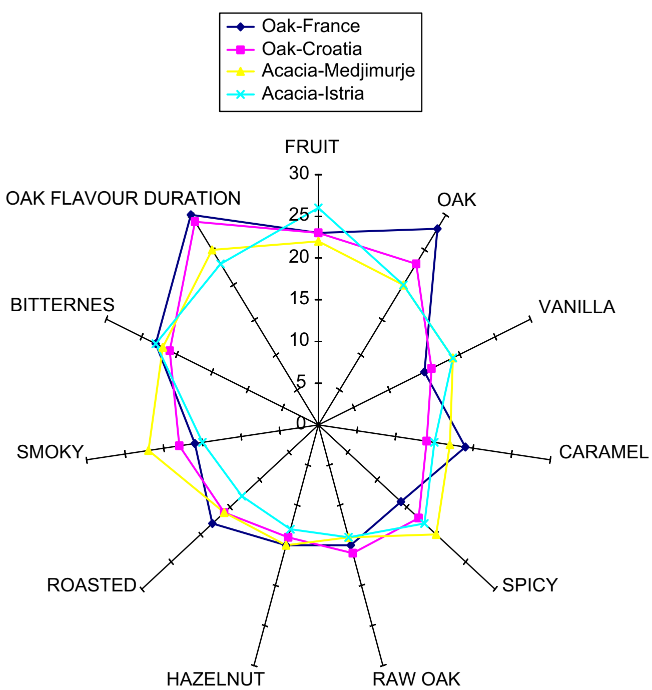

Fig. 1. Malvazija from Istria wine aroma descriptive profiles after 12 months of barrel ageing. Mean values of the results from 12 judges.

Viala, & Dubourdieu, 1997). All oak barrels aged Malvazija wines contained no 4-ethylphenol, while 4-ethylguaiacol concentrations were very low. In wines from acacia barrel very low concentrations of 4-ethylphenol were noted while 4-ethylguaiacol concentrations were similar to wines from oak barrels (Tables 2 and 3). 4-Vinylphenol concentration in all wines was under the threshold value (770 μg/l) according to Chatonnet, Dubourdieu, Boidron, and Pons (1992) and no changes after 12 months ageing period was noted. The aromatic aldehydes (furfural and 5-methylfurfural) are primarily formed in wood during the toasting process, and the concentration of these compounds in wood is considered to reflect the intensity of the toasting process (Chatonnet et al., 1989, 1991). Analytical data of Malvazija wines after 12 months ageing period pointed out a significant increase in furfural and 5-methylfurfural content in all samples with the exception of wines aged in acacia from Istria barrels.

3.2. Sensory properties of wines

Wines extract from oak wood volatile flavour components, which enhance the intensity and complexity of the wine flavour (Silva, Mazzoleni, & Parodi, 1999). Our results indicate that the sensorial characteristics of oak and acacia barrel-aged wines were modified, probably due to the wood-derived compounds. These wines manifested roundness in taste with a complex retronasal aroma. The results of the 100-point method show the similar quality of wines produced in French and Croatian oak barrels used in this experiment. Barrel ageing had a varying influence on the varietal aroma of investigated cultivar. The results of the sensory analysis (Fig. 1) pointed out differences between almost all the descriptors when wines from different oak and acacia barrels were compared. Oak flavour duration, caramel, roasted and hazelnut notes were higher in wine from oak barrels of French origin while raw oak, smoky and spicy notes were higher in wines from oak barrels of Croatian origin. The acacia barrels are less 'aggressive' than oak and add less wood character to the wines. Ageing the wines in acacia barrels made the wines suppler and finer textured, and enhances the natural sweetness of the fruit with more pronounced vanilla and spicy character. The wines from acacia barrels had more sweetness and honey taste with less pronounced oak flavour duration. These wines were also the best evaluated from the panellists (Table 4).

4. Conclusion

Results of this study indicate that ageing in oak or acacia barrels positively influence the quality of Malvasia from Istria wines.

Table 4
Results of Malvasia wine testing by 100-point OIV/UIOE method.

Wine types	Oak barrels (France)	Oak barrels (Croatia)	Acacia barrels (Medjimurje)	Acacia barrels (Istria)
Total score	82.75	80.79	84.30	85.25

These wines were characterised by complex flavour, aroma intensity and a potential for bottle ageing. Particularly, the best rated wines were made in acacia barrels. The results pointed out a significant increase in furfural, 5-methylfurfural, guaiacol and trans-isoeugenol content in all Malvazija wines after 12 months ageing period. Acacia barrel-aged wines were more abundant in simple volatile phenol compounds while oak barrel-aged wines had more oak lactones. These preliminary results show the complexity of the interactions involved and the need for further research, including more detailed chemical analysis. This research has also shown the complementary nature and an unexpected link between Malvazija wine and oak/acacia-derived volatiles.

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