

CHANGES IN VOLATILE COMPOSITION AND SENSORY PROPERTIES OF VUGAVA WINES AGED IN CROATIA OAK BARRELS

PROMJENE U KEMIJSKOM SASTAVU I SENZORNIM SVOJSTVIMA VINA VUGAVA DOZRIJEVANOG U BARIK BAČVICAMA OD HRVATSKE HRASTOVINE

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ABSTRACT

Vugava musts were fermented in medium-toasted Croatian barrique barrels (225 L) made from *Quercus petrea* and *Q. robur* oak wood. The oak species used in this research influenced the specific change of the aroma structure of Vugava wines. During the age period the increase in the concentration of cis and trans oaklactons, guaiacol, eugenol, furfural and 5-methylfurfural was noted. Wines fermented and aged in *Q. petrea* barrels have higher concentrations of most volatile phenols compared to wines from *Q. robur* oak wood. From the organoleptic point of view this study suggested that fermentation and on the lees ageing production method in Croatian oak barrels positively influenced the quality of Vugava wines where best results were achieved by use of *Q. petrea* oak wood.

Keywords: Vugava, croatian barrique, volatile components

SAŽETAK

Moštovi od grožđa Vugave fermentirali su u srednje paljenim hrvatskim barik bačvicama od hrasta kitnjaka i hrasta lužnjaka. Kemijska analiza hlapivih spojeva provedena je odmah po završetku alkoholne fermentacije te nakon 12 mjeseci dozrijevanja. Vrsta hrastovog drveta korištena u istraživanju značajno je utjecala na aromastski sastav vina Vugava. Posebice je uočena razlika u koncentracijama cis i trans hrastovog laktona, guajakola, eugenola i furfurala. Tijekom perioda starenja vina došlo je do promjena u sastavu većine hlapivih komponenti posebice furfurala, 5-metilfurfurala te hrastovih laktona. Dobiveni rezultati istraživanja ukazuju na pozitivan utjecaj hrvatske hrastovine na kakvoću vina Vugave.

Ključne riječi: Vugava, hrvatski barik, hlapive komponente

DETALJNI SAŽETAK

Fermentacija moštova Vugave provedena je u hrastovim barik bačvicama od hrasta kitnjaka te hrasta lužnjaka srednjeg intenziteta paljenja. Po završetku fermentacije prvi puta je provedena kemijska analiza vina koja je obuhvatila osnovnu kemijsku analizu te analizu hlapivih komponenti drveta primjenom tekućinske i plinske kromatografije. Isti postupak ponovljen je nakon 12 mjeseci dozrijevanja vina u barik bačvicama. Uz kemijsku analizu provedena je i senzorna analiza dobivenih vina metodom 100 pozitivnih bodova te deskriptivnom metodom. Dobiveni rezultati istraživanja ukazuju na pozitivan utjecaj hrastovine i vremena dozrijevanja na kakvoću vina Vugave. Kemijskom analizom utvrđene su razlike u odnosu cis i trans hrastovog laktona između hrasta kitnjaka i hrasta lužnjaka. Uočeno je i značajno povećanje sadržaja furfurala, 5 metilfurfurala i guajakola kod svih analiziranih vina. Tijekom senzornog ocjenjivanja najbolje su ocjenjena vina fermentirana i čuvana u bačvicama od hrasta kitnjaka. Prema deskriptivnoj metodi ocjenjivanja uočena je razlika u intenzitetu arome hrastovine, začina, i dimljenog u odnosu na vrstu drveta u kojoj je vino fermentirano i čuvano. Puno izraženije i dugotrajnije arome dobivene izdvajanjem pojedinih hlapivih komponenti iz drveta uočene su kod vina iz bačvica od hrasta kitnjaka dok su vina iz bačvica od hrasta lužnjaka imala naglašeniju voćnu, sortnu aromu.

INTRODUCTION

The use of oak wood in the aging of wines has a great influence on their composition, affecting their organoleptic properties [28]. During this oxidative aging, physical, chemical and physical-chemical complex processes take place, in which compounds from the wood, like volatile and polyphenol substances have great influence. The oak species most commonly used in barrel making are *Quercus alba*, also known as American oak, *Quercus petraea* and *Quercus robur* which grow in Europe, the most popular being French oak [17]. Croatian oak is traditionally exported in other European countries for years, but its influence on wine has not been much explored. Because of that in the period from 2000 to 2002 year the influence of fermentation on the quality of wine from French and Croatian oak barrels was investigated and the results suggested no significant differences between the tested oak barrels [15]. Also Herjavec et al. [16] noted that fermentation in Croatian oak barrels positively influenced the quality of Chardonnay and Sauvignon wines. During ageing in oak barrels new volatile compounds appear in wine, mainly extracted

from the wood. These compounds play an important role in the wine's aroma profile. However, wines aged in oak barrels are less rich in floral and fruity notes, due to their evolution and decrease during the process of ageing [13]. Until now, more than 200 volatile compounds extracted from oak have been identified. For instance, oak lactones are already present in natural wood and their concentrations increased due to seasoning and toasting, the volatile phenols and phenolic aldehydes are formed by degradation of lignine, the furfural compounds are formed by thermolysis of the cellulose and hemicellulose together with Maillard reactions which take place during the process of elaboration of the barrels [25]. The accumulation of oak volatile compounds in wine during storage in oak barrels can depend on many factors. Although hundreds of volatile compounds have been identified in untoasted oakwood, relatively few volatiles, including vanillin, eugenol, and cis- and trans-oaklactones are present in significant amounts [33]. The degradation of oakwood lignin generates a variety of volatile phenols, which can be extracted from the wood into the wine. The most abundant of these compounds are vanillin and syringaldehyde. Vanillin, present in all kinds of wood, is the most important because of its characteristic scent of vanilla. It can be used as an indicator of fermentation and aging in oak barrels [30]. In the white wines this apparently occurred only during barrel fermentation and maturation on lees [27]. Oak lactones occur in their highest levels in bourbon. The oaklactones possess a strong woody character and contribute to the unique aroma and flavor of bourbon. Although they occur in oak woods used for cooperage the cis isomer occurs in much higher levels in American white oak compared to other species. The cis isomer has a more intense character than the trans isomer, but generally both isomers are described as woody and coconut like. 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 [6,11]. However the concentration of these compounds in wine does not always reflect the amount that has been extracted from wood because they can be reduced to the corresponding alcohols by wine microorganisms [32]. In addition, wine compounds can be sorbed by wood and by wine lees, so that this factor can also have an influence on wine volatile composition [4]. The objective of this study was to define the influence of Croatian oak barrels made from *Q. petraea* and *Q. robur* on the chemical composition and sensory properties of the Vugava wines after alcoholic fermentation and after 12 months on the lees aging period.

MATERIAL AND METHODS

Must and fermentation

Vugava wine grape obtained from the vineyard of Vis island was harvested during 2004. season. The free-run juice was treated with 50 mg/l SO₂ and allowed to settle overnight. The juice was racked and the must distributed into used, one year old barrique barrels [2 Quercus petraea medium-toasted and 2 Quercus robur, medium-toasted]. Q. petraea and Q. robur wood used for barrel making was handled in the same way from the time of cutting, seasoning, heating until the moment wine entered the barrels. Also all the barrels were made by the same cooper. Must alcoholic fermentation was carried out with selected Saccharomyces paradoxus RO 54 strain obtained from the Department of Microbiology, Faculty of Agriculture, University of Zagreb. Yeast strain culture was preincubated in sterilized grape must for 48 h at 25°C and finally inoculated at 8x10⁶ CFU/ mL. Fermentation temperature was between 18-20 °C. Sugar degradation in all wines was completed in 30 days. At the end of fermentation aging on the lees started for a period of 12 months. Wines were regularly stirred, with greater at the beginning and lesser frequency toward the end, to facilitate the transfer of compounds from lees

to the wine. The Vugava wine samples from all barrels were chemically and sensory analyzed after the end of fermentation and 12 months later when aging on the lees was finished.

Chemical analyses

Routine analyses of basic components (alcohol, reduce. sugar, total acidity, volatile acidity, pH, ash) were made using standard methods [19]. The oak volatile compounds were analyzed by the GC-MS (furfural, 5-methylfurfural, phenol, guajacol, 4-ethylphenol, 4-ethylguajacol, 4-vinylphenol, cis and trans oaklactons, eugenol, cis and trans isoeugenol) and HPLC (vanillin, syringaldehyde) method [3] in Höhere Bundeslehranstalt und Bundesamt für Wein und Obstbau, Klosterneuburg. For the GC-MS analyses the aroma substances are isolated from the wine by means of steam distillation and manual liquid-liquid extraction. Due to its purity the concentrated extract can be used directly. Less volatile substances are isolated by means of solid phase extraction and then determined by HPLC and UV-detection.

Sensory analysis

The wines were subjected to sensory evaluation by the 100-point O.I.V. / U.I.O.E method, and by descriptive

Table 1 Chemical composition of Vugava wines, after fermentation and 12 months ageing on the lees (average values of two barrels)

Tablica 1 Kemijski sastav vina Vugava nakon alkoholne fermentacije i 12 mjeseci čuvanja na kvascu (srednja vrijednost dviju bačvica)

Compound Sastojak	<i>Q. robur</i>		LSD	<i>Q. petraea</i>		LSD
	after fermentation nakon fermentacije	12 months on the lees 12 mjeseci na kvascu		after fermentation nakon fermentacije	12 months on the lees 12 mjeseci na kvascu	
Alcohol vol.% Alkohol	12,90	12,96	n.s.	12,78	12,80	n.s.
Reduc. sugar g/L Reduc. šećeri	2,5	2,0	n.s.	2,0	2,0	n.s.
Total acidity* g/L Ukupna kiselost	4,9	4,5	n.s.	4,6	4,3	n.s.
Volatile acidity** g/L Hlapiva kiselost	0,65	0,68	n.s.	0,60	0,68	n.s.
pH	3,38	3,42	n.s.	3,40	3,42	n.s.
Ash g/L Pepeo	2,10	2,13	n.s.	2,13	2,12	n.s.

* as tartaric acid /kao vinska kiselina ** as acetic acid/kao octena kiselina n.s. not significant/nesignifikantno

analyses [18], with a panel of 13 judges.

Statistical analyses

One-way analysis of variance and Least Significant Difference (LSD) comparison test were used to statistically interpret mean differences in mean values if any, at 95 % and 99% accuracy level.

RESULTS AND DISCUSSION

Chemical composition

The results presented in Table 1 show that *Saccharomyces paradoxus* strain 54 metabolized the total must sugar content, confirming its good fermentation abilities, as reported in earlier experiments [24]. At the end of alcoholic fermentation degradation of malic acid was also completed [data not shown] so that slight difference in total acidity and pH values after 12 month aging period on the lees was due to salt precipitation. In Table 2, 3 initial results (the mean-average of two duplicates for each *Quercus*) about oak volatiles in the wines fermented and aged on the lees in Croatian barrique barrels are reported. Level of the oak lactones in oakwood appear to vary widely and this has been attributed to variations in oak origin. The ratio of cis- and trans-oak lactone in fermented wine is characteristic of the wood's genetic source [35]. For all the European wood the ratio is between 1 and 1,5 while for the American wood it range between 5 to 8. Our results show that for Croatian oak wood this ratio is 0,5. In a comparison of French oak samples from Tronçais [*Q. petraea*] and Limousin [*Q. robur*] the former contained higher levels of oak lactones. An identical species comparison for oak sourced from a German forest showed the similar trend [22,23]. Our results also show that wines fermented in *Quercus petraea* barrels contained higher concentration of both oak lactones, compared to *Quercus robur* barrels. However, in comparison with literature data, the ratio of cis- and trans-oak lactone was different. Namely, Vugava wines after fermentation and after 12 months on the lees in both types of barrique barrel contained higher concentration of trans- compared to cis- lactone (Table 2). The compounds extracted from wood have different origins. Some are already present in natural wood like oak lactones and vanillin, but their concentrations increases with seasoning and toasting. Guajacol, furfural and 5-methylfurfural have negligible quantities in green wood and are mainly formed during toasting. The volatile phenols [4-ethylguaiacol and 4-ethylphenol] have a microbiological origin. During oak maturation the concentration of some of these compounds in wine increases with oak contact time whereas other may be consumed by chemical or biological transformations [26]. In our results significant

increase of trans-oak lactone in both types of barrique and cis-oak lactone in wines from *Q. petraea* barrique was noted. According to Spillman et al. [24] eugenol concentrations are mainly affected by oak origin and are highly correlated with the concentrations of cis-oak lactone. Our results show some difference in eugenol concentrations between wine fermented in *Q. robur* and *Q. petraea* barrels but the correlation with cis-oak lactone was not confirmed. During aging period on the lees the concentration of eugenol significantly increased even thou Moreno et al. [21], stated that the concentration of oak volatiles decrease due to their binding by the lees. According to them eugenol, 4-propylguaiacol, 4-methylguaiacol, furfural, 5-methylfurfural have the highest affinity for the lees binding but our results did not confirm that. The concentration of aromatic aldehydes furfural and 5-methylfurfural is considered to reflect the intensity of toasting process. However, their concentration in wine does not always reflect the amount that has been extracted from wood because they can be reduced to the corresponding alcohols by wine microorganisms [11]. Lower concentration of furfural and 5-methylfurfural in all Vugava wines just after fermentation is probably connected with barrel fermentation and biological reduction of these components to the corresponding [less sensorial potent] alcohols, what is in agreement with data published by Chatonnet et al. [16]. However, analytical data of Vugava wines after 12 months on the lees pointed out significant increase in furfural and 5-methylfurfural content. Vanillin concentration is known to vary significantly with variation in coopering heat [11]. Spillman et al. [33] established that there was no significant difference in final vanillin concentration among the oak samples. It can be extracted in significant quantity from unheated oakwood and in much higher amounts from strongly heated wood. Chatonnet et al. [16] and Dubois [12] concluded, that vanillin can influence the flavor of some wines matured in new oak barrels, although this role is much diminished when wines are fermented on lees. Depletion of vanillin as a result of fermentation appears to be a general phenomenon, and the degree to which this occurs can depend on the strain of yeast employed in fermentation [17]. It seems that also other component presence in wine probably influence the perception of the vanilla aroma. All Vugava wines had very low vanillin concentrations after fermentation and no changes were noted after 12 months period but in spite of that all wines were sensory described as having vanilla character. 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

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Table 2 Volatile phenol concentrations in Vugava wines after fermentation and 12 months ageing on the lees (mean values of two barrique barrels)

Tablica 2 Koncentracija hlapivih fenola u vinima Vugave nakon alkoholne fermentacije i 12 mjeseci čuvanja na kvascu (srednja vrijednost dviju barik bačvica)

compound [µg/L] sastojak	<i>Q. robur</i>		LSD	<i>Q. petraea</i>		LSD
	after fermentation nakon fermentacije	12 months on the lees 12 mjeseci na kvascu		after fermentation nakon fermentacije	12 months on the lees 12 mjeseci na kvascu	
Furfural	76,7	147,1	5%=5,5 1%=10,1	221,4	583,4	5%=99,3 1%=182,5
5-Methylfurfural	6,5	23,3	5%=5,0 1%=9,1	24,8	132,9	5%=11,2 1%=20,6
Phenol	3,8	4,2	n.s.	4,1	5,3	n.s.
Guajacol	0,9	1,4	5%=0,22 1%=0,41	4,1	8,4	5%=1,9 1%=3,5
4-Ethylphenol	-	-		-	-	
4-Ethylguajacol	0,1	0,2	n.s.	0,6	0,7	n.s.
4-Vinylphenol	169,3	155,3	n.s.	125,4	112,1	n.s.
Cis-oaklacton	0,4	0,5	n.s.	13,5	28,5	5%=2,9 1%=5,3
Trans-oaklacton	0,7	3,5	5%=0,22 1%=0,41	22,1	55,4	5%=20,7 1%=38,1
Eugenol	5,2	7,7	5%=0,67 1%=1,23	9,8	18,2	5%=1,57 1%=2,89
Cis-isoeugenol	-	-		-	-	
Trans-isoeugenol	0,9	2,0	5%=0,77 1%=1,43	2,3	6,9	5%=2,9 1%=5,3
Vanillin	20	20	n.s.	20	20	n.s.
Syringaldehyde	50	40	n.s.	60	20	5%=20,7 1%=38,1

n.s. not significant/nesignifikantno

of its lack of characteristic odor [27]. It is formed by lignin breakdown during wood toasting and is according to Chatonnet et al., [5] and Chatonnet, [10] related to vanilla character of wine. According to Arapitsas et al., [1] syringaldehyde is the most important compound for the discrimination of the barrel-aged wine from the wine

treated with oak-chips. But, it is possible that the wood – imparted compounds, while occurring below their detection threshold, interact synergistically with flavors in the wine, thus yielding a detectable change when none would be predicted based solely on concentration data [20]. Vinylphenols [4-vinylphenol and 4-vinylguaiacol]

Table 3 Comparison of the volatile phenol concentrations in Vugava wines between *Q. robur* and *Q. petraea* barrels (mean values of two barrique barrels) after fermentation and 12 months on the lees**Tablica 3** Koncentracija hlapivih fenola izmeđ vina Vugave iz *Q. robur* i *Q. petraea* bačvica (srednja vrijednost dviju barik bačvica) nakon alkoholne fermentacije i 12 mjeseci čuvanja na kvascu

compound [$\mu\text{g/L}$] sastojak	<i>Q. robur</i>	<i>Q. petraea</i>	LSD	<i>Q. robur</i>	<i>Q. petraea</i>	LSD
	after fermentation nakon fermentacije	after fermentation nakon fermentacije		12 months on the lees 12 mjeseci na kvascu	12 months on the lees 12 mjeseci na kvascu	
Furfural	76,7	221,4	5%=11,2 1%=20,6	147,1	583,4	5%=90,2 1%=176,5
5-Methylfurfural	6,5	24,8	5%=5,5 1%=10,1	23,3	132,9	5%=20,7 1%=38,1
Phenol	3,8	4,1	n.s.	4,2	5,3	n.s.
Guajacol	0,9	4,1	5%=0,67 1%=1,23	1,4	8,4	5%=5,7 1%=8,89
4-Ethylphenol	-	-		-	-	
4-Ethylguajacol	0,1	0,6	5%=0,22 1%=0,41	0,2	0,7	5%=0,22 1%=0,41
4-Vinylphenol	169,3	125,4	n.s.	155,3	112,1	n.s.
Cis-oaklacton	0,4	13,5	5%=0,77 1%=1,43	0,5	28,5	5%=0,67 1%=1,23
Trans-oaklacton	0,7	22,1	5%=5,0 1%=9,1	3,5	55,4	5%=11,2 1%=20,6
Eugenol	5,2	9,8	5%=0,67 1%=1,23	7,7	18,2	5%=1,57 1%=2,89
Cis-isoeugenol	-	-		-	-	
Trans-isoeugenol	0,9	2,3	5%=0,77 1%=1,43	2,0	6,9	5%=2,9 1%=5,3
Vanillin	20	20	n.s.	20	20	n.s.
Syringaldehyde	50	60	n.s.	40	20	5%=5,7 1%=8,89

n.s. not significant/nesignifikantno

Table 4 Results of Vugava wine testing by 100-point O.I.V/U.I.O.E. method (mean value results of 13 judges)
Tablica 4 Rezultati senzornog ocjenjivanja metodom 100 bodova (srednja vrijednost ocjena 13 degustatora)

	Time of testing Vrijeme ocjenjivanja	<i>Q. robur</i>	<i>Q. petraea</i>
Total score Ukupni zbroj	after fermentation nakon fermentacije	80,22	84,55
	12 months on the lees 12 mjeseci na kvascu	82,55	86,79

in white wines and ethylphenols [4-ethylphenol and 4-ethylguaiaicol] in red wines are quantitatively the most significant volatile phenols identified as classic components of wine aroma [9]. Their biosynthesis is a result of microbiological transformation of trans ferulic and trans-p-coumaric acids, nonvolatile, odorless precursors present in all wines. Volatile phenol synthesis by yeast depends on the nature of the strain and on the presence of certain polyphenolic inhibitors and it is strictly limited to alcoholic fermentation [7]. No vinylphenol can therefore be synthesized in a dry wine stored in the presence of lees [14]. All Vugava wines contained no 4-ethylphenol, while 4-ethylguaiaicol concentrations were very low. 4-vinylphenol concentration in all wines was under threshold value [770 µg/l according to Chatonnet et al. [8] and small decrease after 12 months aging period was noted. According to Chassagne et al. [4] wine lees have sorption affinity of volatile phenols and our results are in line with that statement.

Sensory properties of wines

“On the lees or sur lie” technology is one of many options available to the winemaker in adding another dimension to wine and it is usual technology in Chablis for production of high quality Chardonnay wines. Wines extract from oakwood volatile flavor components, which enhance the intensity and complexity of the wine flavor [31]. Vugava is autochthonous cultivar from Vis island whose wine is characterized with very pleasant and fruity, honey like aroma. Barrel fermentation of Vugava must - and its influence on the wine quality has never been explored. The results of our experiment indicate that the sensorial characteristics of Vugava wines were modified, due to the wood-derived compounds. Generally, these wines manifested roundness in taste with a complex retro nasal aroma. The results of the 100-point method show the marked differences in quality of the wines produced in barrels made from different oak species. The wines fermented in *Q. petraea* barrels were better quality compared to wines from *Q. robur*. Sensory analyses of

the wines after 12 month ageing period confirmed these results. However, it is important to notice that ageing on the lees had positive effect on all tested Vugava wines what is shown by higher total scores [Table 4]. According to the descriptive analysis, marked differences were observed in the oak, spice, smoky and oak flavor duration among *Q. robur* and *Q. petraea* wines. These characteristics were more intensive in *Q. petraea* wines, while *Q. robur* wines contained fruitier, varietals aromas. Descriptive analysis of the Vugava wines after 12 months ageing period resulted in the similar descriptive definition of tasted wines, but marked differences were noted in the intensity of oak, smoky and spicy flavor between *Q. robur* and *Q. petraea* wines. Reazin [29] noted that furfural has an important modifying effect on the perception of the oak lactones in wine. Probably this was the reason why Vugava wines after 12 months ageing period had more pronounced oak flavor. According to Boidron et al. [2] eugenol contributes to the spicy and clove like character of the wines and our descriptive analysis confirmed that. Significant difference in the eugenol content between wines after fermentation and after 12 months ageing period pointed out differences in sensory perception of spicy character in Vugava wines especially from *Q. petraea* barrique barrels. In both type of wines the intensity of fruit and vanilla perception decreased while varietals aroma totally disappeared [Fig. 1].

CONCLUSIONS

Results of this study indicate that fermentation and aging on the lees in Croatian oak barrels positively influence the quality of Vugava wines and confirmed the compatibility of on the lees production method with this grape variety. The ratio of cis and trans-oaklactons from Croatian *Q. petraea* and *Q. robur* oak wood was different in comparison with literature data. Concentration of trans- compared to cis- lactone was higher in all analyzed Vugava wines after fermentation and also

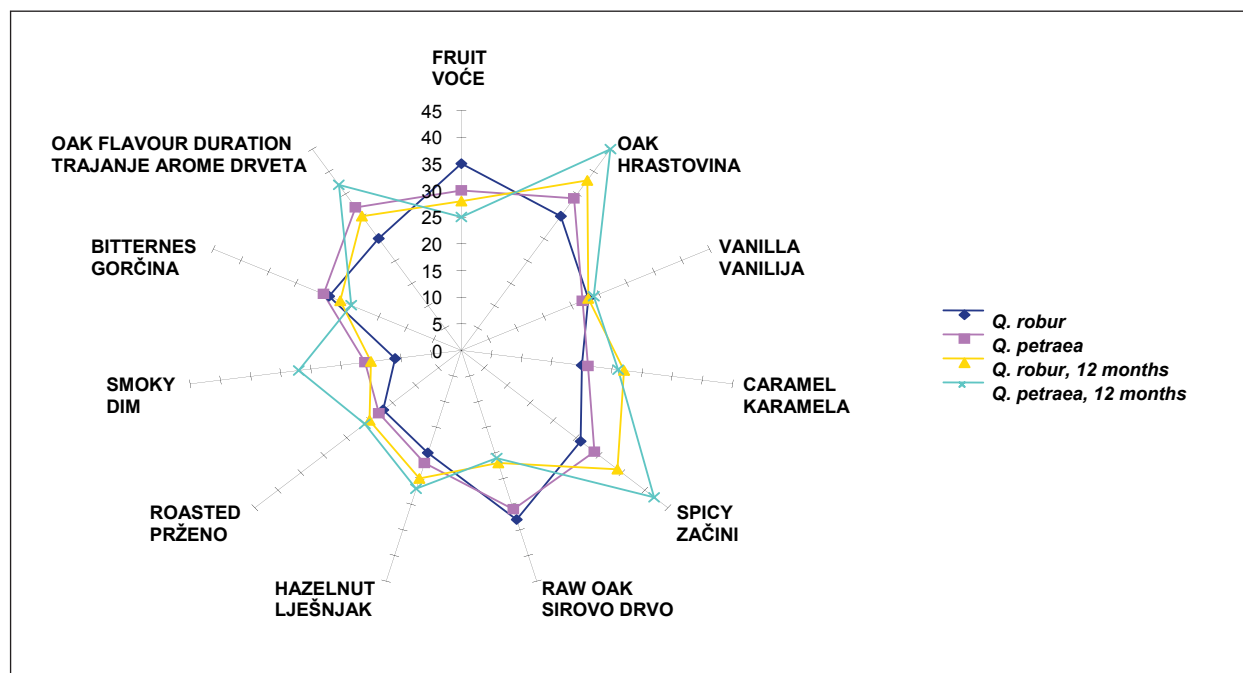


Figure 1 Vugava aroma descriptive profiles after fermentation and 12 months ageing on the lees. Mean values of the results from 13 judge
 Figura 1 Vugava deskriptivni aromatski profil nakon alkoholne fermentacije i 12 mjeseci čuvanja na kvascu. Srednja vrijednost rezultata ocjena 13 degustatora.

after 12 months ageing on the lees period (Table 2). The results pointed out significant increase in furfural, 5-methylfurfural, guajacol, cis and trans oaklactons content in Vugava wines after on the lees ageing period. Better organoleptic quality of Vugava wines was obtained by fermentation and ageing in *Q. petraea* barrels, probably because of higher concentration of cis oaklactone and some other volatile compounds. Lower overall intensity of flavor in the *Q. robur* wines was in line with lower concentrations of some oak volatile components. At the end this research has also shown the complementary nature and an unexpected link between Vugava wine and oak-derivate volatiles.

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