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## **Oak barrel effects on Chardonnay wine composition**

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## **INTRODUCTION**

The influence of the addition of oak chips and barrel ageing had on basic wine parameters and volatile compounds of Chardonnay wines has been studied. Chardonnay wines were obtained by the traditional wines making process. Oak chips (4 g/l – untoasted and light toasted) were added at the final stage of winemaking process for ageing 1, 2 and 3 months, respectively. Also, the initial wines were aged in untoasted barrel for the same period of time. Following LLE/GC-MS analysis, alcohols, esters, fatty acids, lactones, phenolic compounds were identified and quantified. The light toasted wine was clearly separated by phenolic compounds (vanillin, p-vinyl guaiacol and acetovanillone). After 3 months, the volatile compounds of wine from untoasted medium (chips and barrels) were almost similar from the volatile profile point of view. This could have economic and vinification management interests since oak barrels are expensive and wine oak barrel aging is a long process. All wines studied in this research can provide a viable alternative to traditional Chardonnay wines

#### Table 1. Experimental variants - IW, N1M, N2M, N3M, L1M, L2M, L3M, BAR1M, BAR2M and BAR3M are the abbrevia-tions for 10 variants of Chardonnay (IW - initial wine, N- natural chips, L - light toasted chips, BAR untoasted barrel; 1M, 2M and 3M – number of months of ageing

## **MATERIALS AND METHODS**

Chardonnay grapes (Vitis vinifera L.) were manually harvested at maturity in Teaca winery (Lechinta, Bistrita Nasaud County, Romania) during the 2017 vintage (225 g/L sugar in grape juice, 8.95 g/L must total acidity expressed as tartric acid and 104 g weight of 100 berries). The Lechinta grapevine growth area is known for high acidity of wines, due to climatic conditions (oenoclimatic aptitude index of 4221) [23]. Grapes were crushed and destemmed (Enoitalia® WE223S, Italy) on the day of harvest. A pneumatic press (Vaslin-Bucher® RPS 50, France) was used filled at 75-80% of its capacity. Potassium metabisulphite (4.5±0.5 g/hL) was added during the transfer of must to stainless steel tank (5000 L capacity). Saccharomyces cerevisiae (Zymaflore VL1, Laffort®, France), a commercial active dry yeast was included at a rate of 20 g/hL to perform alcoholic fermentation at 16° C. Three sets of experiments were performed depending on the container where maturation took place.

#### Determination of Volatile Compounds

Analysis of volatile organic compounds from wine samples were performed using a Shimadzu QP 2010 PLUS Mass Spectrometer coupled with Gas Chromatograph (Shi-madzu, Japan) equipped with a Carbowax type column from Agilent (30 m x 0.32 mm ID and 0.50 µm film thicknesses). Helium (6.0) was used as carrier gas with a flow rate 1.7 mL/min. The injector, the detector and the interface temperature were set at 220°C. The column temperature program was conducted as follows: 40°C was the initial temperature for 5 min, increasing at a rate of 4°C/min to 220°C, and holding 220°C for 15. Quadrupole mass detector acquisition was carried out using the positive EI-mode at the 70 eV, with continuous scanning from 40 to 500 amu. Standard compounds in wines were identified by comparison of their relative retention times and mass fragmentation with those of computer matching against commercial library (NIST and Willey).

Time of maturation (months)	Type of vessel maturation	Oak chips (4g/L)	Codes		
Initial Wine – unaged wine	_	-	IW		
	Demijhons / Glass	Natural – untoasted	N1M		
1	Dennijnons / Olass	Light-toasted	L1M		
	Barrel	Untoasted	BAR1M		
	Domiihons/Glass	Natural – untoasted	N2M		
2	Demijhons/Glass	light-toasted	L2M		
	Barrel	Untoasted	BAR2M		
	Domiihons/Class	Natural – untoasted	N3M		
3	Demijhons/Glass	Light-toasted	L3M		
	Barrel	Untoasted	BAR3M		

Table 2. Oenological analyses of white wines aged with oak chips and barrel for 1, 2 and 3 months; analysis of variance (ANOVA) taking as factors time and ageing method. All values are expressed as means  $\pm$  SD. Different letters in each row of the same cultivar are significantly different at the 0.05 level according to ANOVA by Tukey's test. \*: p<0.05; \*\*: p<0.01; \*\*\*: p<0.001; ns: not significant. IW, N1M, N2M, N3M, L1M, L2M, L3M, BAR1M, BAR2M and BAR3M are the abbreviations for 10 variants of Chardonnay (IW - initial wine, Nnatural/untoasted chips, L – light toasted chips, BAR – untoasted barrel; 1M, 2M and 3M – number of months of ageing).

Variants/ Oenologic parameters	IW	N1M	N2M	N3M	L1M	L2M	L3M	BAR1M	BAR2M	BAR3 M	Time	Agein g meth od
Ethanol (%v/v)	12.77 ±0.03a	12.75 ±0.04a	$12.69 \pm 0.08ab$	$12.66 \pm 0.06 bc$	12.75 ±0.04a	$12.69 \pm 0.08ab$	$12.66 \pm 0.06 bc$	$\begin{array}{c} 12.62 \\ \pm 0.02 bc \end{array}$	$12.54 \pm 0.04c$	$12.55 \pm 0.02c$	**	***
Volatile acidity (g/L acetic acid)	$\substack{0.22\\\pm0.02c}$	0.24 ±0.03c	$\begin{array}{c} 0.28 \\ \pm 0.02 bc \end{array}$	$\substack{0.31\\\pm0.01b}$	0.24 ±0.03c	$\substack{0.28\\\pm0.02bc}$	$\substack{0.31\\\pm0.01b}$	$\substack{0.32\\\pm0.02b}$	0.41 ±0.04a	0.46 ±0.02a	***	***
Total acidity (g/L tartric acid)	7.96 ±0.07a	7.86 ±0.04ab	7.74 ±0.04bc	$7.58 \\ \pm 0.08 c$	7.84 ±0.03ab	7.73 ±0.04bc	$\begin{array}{c} 7.58 \\ \pm 0.08 c \end{array}$	7.91 ±0.04a	7.87 ±0.04ab	$7.76 \pm 0.07b$ c	***	**
Dry extract (g/L)	23.60 ±0.23a	23.52 ±0.35a	23.41 ±0.39a	23.37 ±0.25a	23.52 ±0.35a	23.41 ±0.42a	23.36 ±0.33a	23.52 ±0.31a	23.41 ±0.40a	23.37 ±0.35a	ns	ns
Non-reducing dry extract (g/L)	22.03 ±0.03a	22.01 ±0.04a	22.05 ±0.04a	21.99 ±0.04a	22.06 ±0.05a	$\underset{\pm 0.03a}{20.00}$	21.92 ±0.04a	22.04 ±0.09a	22.07 ±0.12a	$\begin{array}{c} 20.05 \\ \pm 0.06a \end{array}$	ns	ns
Free SO <sub>2</sub> (mg/L)	$36 \pm 1.00a$	33 ±0.58ab	$32 \pm 0.58$ bc	$31 \pm 1.00c$	34 ±0.81ab	$33 \pm 0.72$ bc	$30 \pm 0.50c$	$32 \pm 1.42 bc$	$30 \pm 1.15c$	$\underset{\pm 1.05\text{d}}{27}$	**	***
Total SO <sub>2</sub> (mg/L)	$\begin{array}{c} 120 \\ \pm 0.76 bc \end{array}$	$123 \pm 2.00$ ab	124 ±1.53a	126 ±1.85a	$123 \pm 1.44ab$	$124 \pm 2.08a$	126 ±1.76a	$\begin{array}{c} 118 \\ \pm 0.76 \text{cd} \end{array}$	$\begin{array}{c} 116 \\ \pm 0.85 \text{cd} \end{array}$	$115 \pm 0.83d$	*	***
рН	$\substack{3.22\\\pm0.02b}$	$\substack{3.23\\\pm0.02b}$	$3.23 \pm 0.01 b$	3.24 ±0.01a	$\substack{3.22\\\pm0.02b}$	$3.23 \pm 0.01 b$	3.24 ±0.02a	$\substack{3.23\\\pm0.02a}$	$3.25 \pm 0.03a$	3.24 ±0.01a	**	ns

#### Multivariate analysis

Figure shows the chemical analysis results of the Chardonnay wines overlaid over the studied variants, with the wines projected on to that space. According to PLSR analy-sis, the distance between the variable and the center of the circle shows the interpretive degree of the principal components to the variable. The PLSR was established to deter-mine the influence of methods and duration of ageing on volatile compounds.

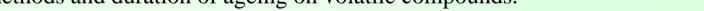
## **RESULTS AND DISSCUSION**

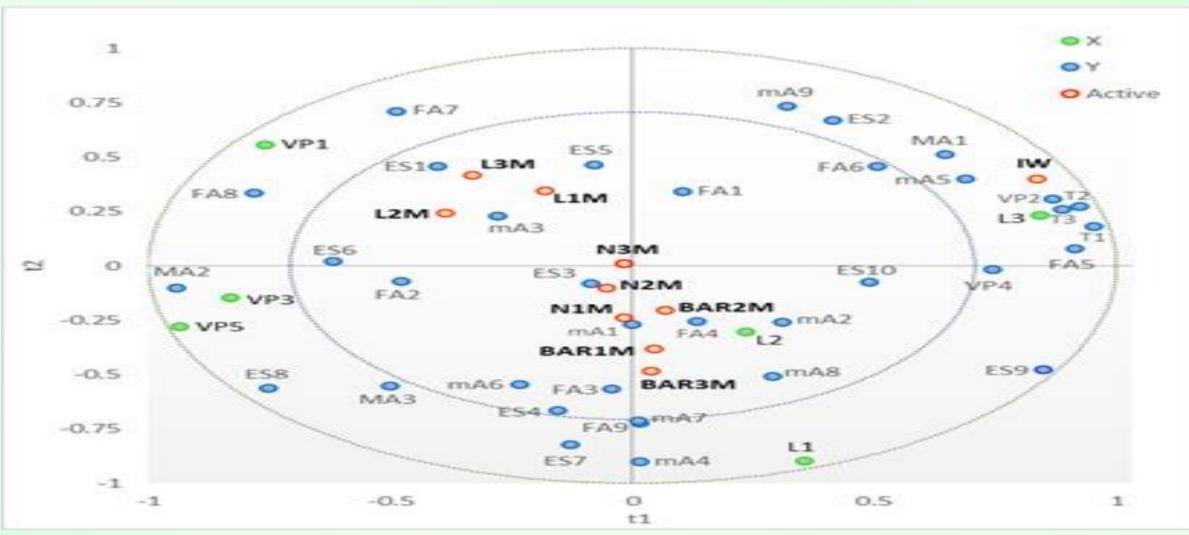
#### **Statistical analysis**

Univariate analysis was performed using ANOVA, applying the Tukeys multiple range test. Partial least squares regression (PLSR) analysis was also carried out, using the XLSTAT Addinsoft 2014.5.03 version (Addinsoft Inc., USA).

#### Table 3. Odor activity values of compounds reaching a concentration above the odor threshold (OAV>1) in, at least, one variant. IW - initial wine, N- natural chips, L - light toasted chips, BAR - untoasted barrel; 1M, 2M and 3M - number of months of ageing); OT- Odor Threshold

		IW	N1M	N2M	N3M	L1M	L2M	L3M	BAR1M	BAR2M	BAR3M	Time	Ageing method
•	Isobutanol	47±0.03a	39±2.7d	40±3.8 c	42±3.5 b	39±1.7 d	41±2.3 bc	41±1.9 bc	38±2.8 e	38±2.6 e	41±2.4 bc	**	***
•	Isopentyl alcohol	76±2.1e	83±4.5c	84±5.2 bc	83±5.7 c	85±4.2 ab	84±3.2 bc	86±5.8 a	82±2.1 d	83± 3.0 c	84±2.7 bc	*	***
,	2-phenylethanol	15±0.3e	16±2.4 d	17±2.0 c	17±2.8c	16±1.9d	18±2.5b	17±2.9c	19±2.6a	17±2.1c	18±2.8 b	*	***
j	Total major alcohols (mg/L)	138	138	141	142	140	145	144	139	138	143		
	Hexanol	564±5.7de	438±3.2e	398±7.8f	865±6.5b	375±2 4f	342±7.8f	$1057\pm50$ a	760±42bc	627±8.7c	1145±12.1a	*	***
	4-methyl-1-pentanol	22±0.6b	25±2.6ab	11±3cd	12±0.9cd	31±1.5a	10±1.8d	9.7±0.5d	33±5.7a	22±7.8bc	18±2.8bc	**	**
	E-3-hexenol	30±1.2b	30±3.5b	16±3.1c	17±1.5c	40±6.6a	41±3.5a	41±3.3a	27±1.2b	24±3.5 bc	45±3.4a	*	**
	Z-3-hexenol	112±0.3de	119±0.4bc	117±3.7 c	121±0.2b	111±2.6e	114±4.5b	113±0.3d	122±1.3b	124±2.8a	123±1.1b	*	*
	2-nonanol	173±9.6a	100±11b	47±4.5de	58±6.8de	115±3.5b	37±5.2e	59±6.8de	98±2.1c	78±7.8cd	13±0.6f	**	***
	1-heptanol	124±1.2e	230±9.4a	201±4.9bc	174±3.1d	240±35a	150±22d	136±6.0c	230±5.0a	211±2.6b	182±3.7c	***	***
	2,3-butanediol	301±35e	507±24de	1137±12.9 cd	1417±21c	330±27e	605±9.8d	769±45d e	1686±14.7 c	2797±35.2 b	4973±52a	***	***
	3-methylthio-1-	278±15bc	248±15bc	250±25bc	206±19d	176±15d	166±31d	222±17b	553±49a	290±17b	244±75bc	***	***
	propanol Benzylalcohol	58±3.5a	44±2.4c	45±1.90c	47±2.2bc	42±2.6d	49±2.4b	48±3.1b	32±2.4f	34±2.1e	39±3.5de	***	***
	Total minor alchols	1662	1741	2222	2917	1460	1514	2454	3541	4207	6782		
	(µg/L) Linalool	323±7.8a	59±4.9c	49±4.6cd	33±2.8e	Trace	trace	trace	83±7.3b	45±2.3de	36±2.3e	***	***
	Terpineol	127±8.7a	11±2.6c	trace	ND	Trace	trace	trace	23±3.6b	10±0.3c	11±1.2c	***	****
	Trans-geraniol	52±2.4a	11±0.5b	ND	ND	ND	ND	ND	14±2.5b	ND	ND	***	***
	Total terpenes	502	81	49	33	trace	trace	trace	120	55	47		
	((µg/L) Isobutyric acid	83±3.7b	81±2.5bc	51±4.6de	21±2.1f	108±10a	83±3.1b	39±4.0e	75±3.3bc	64±5.6cd	24±2.8f	***	***
		945±17e	1267±21d	1643±68a	1395±45c	1640±96a	1227±21	1355±40	1574±96b	1501±65b	1016±10d	***	***
	Isovaleric acid	132±2.1e	156±9.6d	219±82bc	ND	163±9.5d	e 189±15b	c 190±15b	229±7.4bc	с 296±19b	538±4.4a	**	***
	Lactic acid	54±4.7e	132±21d	228±19e	417±67c	141±5.8d	с 192±32d	с 108±10е	431±17c	808±75b	1317±145a	***	***
	Octanoic acid	2459±58c	2377±165b	2448±39c	2653±72b	2054±89d	2409±39	2442±54	2069±153	2447±154	2866±75a	**	***
	Decanoic acid	957±26a	c 449±56b	227±25d	125±9.8e	306±19cd	с 117±10е	с 124±13е	d 441±41b	c 274±30cd	324±21c	***	***
	Malic acid	571±62a	193±68c	176±2.5e	155±12d	576±42a	208±51c	184±7.2e	372±356b	205±24c	165±59d	**	***
	5- oxotetrahydrofuran -2-carboxilic acid	71±1.9 de	61±6.3e	83±3.6 d	91±6.8cd	103±14bc	116±9.8b	126±32a	36±4.0f	28±0.9f	71±5.2de	**	***
	2-oxoapidic	8±0.7f	12±1.5de	17±1.9e	14±1.7cd	22±1.6a	19±1.4ab	17±1.2b	15±1.5c	11±1.8e	12±1.6de	**	**
	Total fatty acids (µg/L)	5280	4728	5071	4792	4645	4336	4977	5035	5634	6540		
	N-amyl acetate	2483±12c d	2433±12d	2487±40c d	2471±8 cd	2581±42b c	2514±11 bc	2603±31 a	2462±41c d	2508±36b c	2548±52bc	**	***
	Hexylacetate	536±13a	474±31bc	463±35c	423±21e	486±23b	479±13b c	465±28c	452±19cd	416±13e	421±22de	*	*
	Ethyl hexanoate	3151±76c	3165±96b	3213±43d	3120±27b c	3193±36b	3160±62 b	3141±58 c	3240±22a	3103±93d	3128±71bc	***	***
	Ethyl lactate	395±15f	1031±59bc	766±38cd	849±75 cd	1179±54b	616±58e	$659{\pm}78e$	1010±64b c	954±87bc	2122±72a	***	***
	Ethyl octanoate	2271±12b	2148±34bc	2322±32a	2297±46b	2177±54b c	2362±59 a	2214±26 c	2122±27b c	2131±12c	2184±68b	***	ns
	Phenethyl acetate	334±15d	487±15bc	530±30b	586±21a	476±19bc	490±23b c	517±45b	374±38c	357±41cd	506±11bc	ns	***
	Diethyl malate	142±23g	222±29c	286±29a	219±20c	169±32f	162±9.8f	202±18e	256±54b	299±32a	282±49a	**	***
	Diethyl succinate	115±21d	283±32ab	270±38b	244±25c	285±41ab	265±21b	240±28c	269±29b	286±21ab	294±72a	**	***
	Trimethylene acetate	431±25a	363±12e	341±50f	358±6.2e	276±36c	287±24c d	273±5de	369±35e	360±42e	399±28b	***	***
	Ethyl-4- hydroxybutanoate	275±14b	296±25a	149±13d	171±14c	281±26ab	117±12e	150±14d	285±46a	224±20cd	154±32d	**	***
	Total esters (µg/L)	10133 344+41b	10902 443+37ab	10827 369+27ab	10738 342+29ab	11103 228+21c	10452 260±14.5	10464 208+14c	10839 532+18a	10638 466±12ab	12038 461+10ab	**	*
	Butyrolactone	344±41b	443±37ab	369±27ab	342±29ab	228±21c	bc	208±14c	532±18a	466±12ab	461±10ab	*	*
	Pantolactone 3,4-dimethyl-2(5)-	12±0.3b	trace	trace	trace	11±3.2b	trace $33\pm3$ 2de	trace $42+9.2$ cd	trace $71\pm2$ lbc	trace	44±3.6a	***	***
	furanone Total lactones	122±12a	65±6.7bc	43±3.5cd	52±4.2cd	84±9.6b	33±3.2de	42±9.2cd	71±2.1bc	69±7.0bc	51 ±5.7cd	· · · · · · · · · · · · · · · · · · ·	
	(µg/L)	478	508 26+3.6h	412 28+3.2h	394	323	293	250	603	535	556	*	***
	p-vinyl guiacol Methyl-	18±2c	26±3.6b	28±3.2b	31±2.4b	58±12a	61±12a	65±13a	26±6.4b	29±3.1b	33±4.2b	*	***
	hydroxycinamate	77±6.3a	32±4.5b	16±1.2d	12±1.3e	21±4.7c	14±1.1de	11±0.9e	$26\pm 2.4bc$	$13\pm1.4e$	12±1.2e	***	***
	Acetovanillone 2,3-	61±0.8e	157±21b	122±24d	119±12d	171±35a	167±19a	143±15c	162±32ab	139±21cd	121±11d		ጥ ጥ
	hydroxybenzofuran e	156±5.4a	114±1.6c	63±5.9e		116±3.1c			127±6.3b	99±8.5d	56±2.1ef	***	*
		39±3.5e	134±2.1c	136±1.9bc	133±1.7c	139±2.1a b	138±1.8b	141±2.3a	127±1.4d	126±1.1d	128±1.9d	*	**
	Total volatile phenols (µg/L)	351	463	365	374	505	424	393	468	406	350		





Partial least squares regression (PLSR) analysis, chemical data and the correlation of Chardonnay wines between ageing method and time (t1, chemical components of initial wine; t2, chemical components released from wood). IW, N1M, N2M, N3M, L1M, L2M, L3M, BAR1M, BAR2M and BAR3M are the abbreviations for 10 variants of Chardonnay (IW – initial wine, N- natural chips, L – light toasted chips, BAR – untoasted barrel; 1M, 2M and 3M – number of months of ageing). MA1 (iso-butanol), MA2 (isopenthyl), MA3 (2-phenylethanol), mA1 (1-hexanol), mA2 (4-methy-1pentanol), mA3 (E-3-hexenol), mA4 (Z-3hexenol), mA5 (2-nonanol), mA6 (1-heptanol), mA7 (2,3-butanediol), mA8 (3-methylthio-1-propanol), mA9 (benzyl-alcohol), T1 (linalool), T2 (terpineol), T3 (trans-geraniol), FA1 (iso-butyric acid), FA2 (hexanoic acid), FA3 (isovaleric acid), FA4 (octanoic acid), FA5 (decanoic acid), FA6 (malic acid), FA7 (5-oxotetrahydrofuran-2-carboxilic acid), FA8 (2-oxoapidic), FA9 (lactic acid), ES1 (N-amyl acetate), ES2 (hexyl-acetate), ES3 (ethyl hexanoate), ES4 (ethyl lactate), ES5 (ethyl octanoate), ES6 (phenethyl acetate), ES7 (di-ethyl malate), ES78 (diethyl succinate), ES9 (tri-methylene acetate), ES10 (ethyl-4hydroxybutanoate), L1 (butyrolactone), L2 (pantolactone), L3 (3,4-dimethyl-2(5)-furanone), VP1 (p-vinyl guaiacol), VP2 (methyl-hydroxycinamate), VP3 (acetovanillone), VP4 (2,3-hydroxybenzofurane), VP5 (vanillin).

#### **CONCLUSIONS**

This work is the first study on volatile compounds of young and short matured Chardonnay wines from Romania. The method and time of ageing have a significant influence on basic wine parameters and on the volatile composition. The untoasted medium (chips and barrel) ageing is correlated with lactones, esters (ethyl lactate and ethyl caproate) and fatty acids (isovaleric and lactic acids). The majority of volatile phenols were quantified in wines aged with light toast. The oak chip method of ageing of Chardonnay wines could be a useful tool to obtain wines as a viable alternative to the traditionally made Chardonnay wines. The amount of chips used in this study (4 g/l) was selected in order to avoid an excessive impact of the wood character in wines that could produce a negative effect on tasting. For short periods of maturation, Chardonnay wines can obtain almost the same wine aged with untoasted chips and untoasted barrel, from the chemical point of view, but with more efficient cost, from

#### **Acknowledgements**

financial point of view. For white wines, the use of oak chips could avoid the oxidation of aromatic volatile

compounds that could be produced during barrel aging, and impart oak notes to wines without decreasing the

fresh and fruity characteristics. This approach would enable diversification on the market and increase the range of

products on offer to the consumer.

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