# GC-MS IDENTIFICATION OF VOLATILE COMPOUNDS IN OAK CHIPS AND POWDER

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#### INTRODUCTION

Alternative products to oak barrels, such as chips and staves, are allowed by the OIV). The increased use of these alternatives are mainly related to low investments, similar sensorial results obtained in shorter time, simplicity of use and the possibility to avoid contamination and off flavours, which too-often related to aged or contaminated barrels. In order to use oak alternative products in winemaking, such as chips and powder, it is mandatory to know their chemical composition, namely volatile fraction.

The objectives of the present work were: (1) characterization of the volatile compounds in oak alternatives (chips and powder) of Macedonian *Q. Robur* and their possible relationship to the diversity of their form and (2) to investigate the influence of the toasting temperature on the content and changes of different volatile compounds in the oak samples.

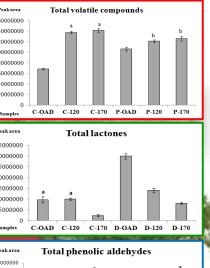
## MATERIALS AND METHODS

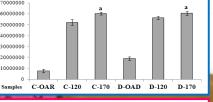
Oak samples	es Chips	Powder	SPME conditions: SPME fiber: DVB/Carboxen/PDMS 50/30, 2 cm stable flex
Open air dried	C-OAD	P-OAD	(Supelco, Bellfonte, USA)
Toasted at 120	°C C-120	P-120	
Toasted at 170 °		P-170	Separation – medium polar capillary column, HP5MS, 30 m * 0,25 mm * 1µm, Agilent Technologies
Instrumen	tation (	GC-MS)	Carrier gas - He with a flow rate of 1.5 mL/min.
			Working parameters: Injector temperature: 270 °C; MS source: 230 °C; MS Quad: 150 °C, Transfer line: 280 °C
		<u>.</u>	<ul> <li>10 °C for 1 min with a temperature ramp of 8 °C min<sup>-1</sup></li> </ul>
loacac	N Gas Chromate	ograph	up to 270 °C (holding time 1 min).  of the volatile compounds
Agilent 68901 Total ion detected in	N Gas Chromate	atogram	• up to 270 °C (holding time 1 min). of the volatile compounds ple toasted at 120 °C (C-120)
Agilent 6890	N Gas Chromate	atogram	of the volatile compounds
Agilent 68901 Total ion detected in	N Gas Chromate	atogram	of the volatile compounds
Agilent 68901 Total ion detected in	N Gas Chromate	atogram	of the volatile compounds
Agilent 68901 Total ion detected in Austrice 10000 10000 10000 10000 10000	N Gas Chromate	atogram	of the volatile compounds
Agilent 68901 Total ion detected in	N Gas Chromate	atogram	of the volatile compounds
Agilent 68901 Total ion detected in Auser 10000 10000 10000 10000 10000 10000 10000 10000 10000 10000 100	N Gas Chromate	atogram	of the volatile compounds

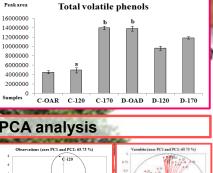
Acknowledgment. This work was supported by a grant from the CEEPUS, CII-HU-0010-03-0809 Network.

		1.00					
	t <sub>e</sub> /min	[HP5]	2	Peakarea	Т	otal vola	tilo
			<b>*</b>			1 ai v 012	une
Formic acid t	7.08	-		160000000	1		
Acetic acid	7.85	703		140000000	_	a	_
Propanoic acid	8.96	719				-	
2-Propenoic acid	9.03 10.45	720 741		120000000	-		- 11
Butanoic acid Butanoic acid, 3-methyl-	10.45	741 811		100000000	-		- 11
Pentanoic acid	12.46	821		80000000			- 11
Hexanoic acid	14.64	902		80000000			
Octanoic acid	18.39	1007		60000000	- 1		- 11
Nonanoic acid	20.22	1037		40000000			
Decanoic acid Vanillic acid	21.80	1111	100				- 11
Vanillic acid Dodecanoic acid	23.08	1133 1212		20000000	-		- 11
Tetradecanoic acid	27.59	1311		0			
Pentadecanoic acid	28.88	1336		Samples	C-OAD	C-120	C-
Hexadecanoic acid	30.15	1416	1	Samples		_	_
Furfuryl alcohol	12.05	815	100	Peak area		Tota	l la
1-Hexanol, 2-ethyl- Benzyl alcohol	15.43 15.60	914 916					
Phenethyl alcohol	15.60	916		35000000	1		
Phenethyl alconol	17.20	340		30000000	-		
(E) 2-Butenal, 2-methyl-	9.72	731					
Hexanal	10.31	739		25000000	1		
3-Furaldehyde	10.65	744		20000000	-		
Furfural (2-Furaldehyde)	11.12	802		15000000			
n-Heptanal	12.68	824 844			а	а	
5-Methyl furfural 1H-Pyrrole-2-carboxaldehyde	14.07 15.08	844 908		10000000	- <b>-</b>	<b>F</b>	
n-Nonanal	16.96	908		5000000	-		
Maltol	17.20	940		3000000			_
(E) 2-Nonenal	18.04	1001		0			
Decanal	18.89	1015		Samples	C-OAD	C-120	C-1
Undecanal	20.70	1044				-	_
Octadecanal	30.86	1430					
(E)15-Heptadecenal	31.60	1444		Peakarea	Tot	al phei	noli
						- P	
1-Methoxy-2-propyl ester of acetic acid	12.03	815		70000000			
Butanoic acid, butyl ester	21.90	1113		60000000 -			
Benzoic acid, 4-hydroxy-3-methoxy-,	24.21	1200				Ŧ	
methyl ester		1200		50000000 -		т	
Hexadecanoic acid, methyl ester (Palmitic acid, methyl ester)	29.68	1407		40000000 -			
Hexadecanoic acid, ethyl ester	30.52	1423		30000000 -			
nexadecation acid, entit ester	30.32	1423		3000000 -			
2-Propanone, 1-hydroxy-	8.41	712		20000000 -			
2-Butanone (Methyl ethyl ketone)	11.91	813		10000000 -			
2-Acetylfuran (Ethanone, 1-(2-furanyl))	12.91	827			(Th		
				o +		_	
2-Pentanone, 4-hydroxy-4-methyl- Propiovanillone	13.44 25.21	835 1220		Samples (	C-OAR	C-120	C-1'
Propiovaniione	25.21	1220					
Butyrolactone	13.06	829					
2(5H)-Furanone (gamma	13.09	830		<b>Peakarea</b>	Т	otal vo	lati
Crotonolactone)	13.09	830		16000000 -			
cis-Methyl-hydroxyoctanoic acid lactone	20.50	1041					1
(cis-whisky lactone)				14000000 -			
trans-4-Hydroxy-3-methyloctanoic acid lactone (trans-whisky lactone)	21.08	1050		12000000 -			
lactorie (dans whisty lactorie)				10000000 -			
				8000000 -			
Benzaldehyde	14.00	843					
Benzaldehyde, 2-hydroxy-	15.79	919	2	6000000 -		a 	
Benzaldehyde, 4-methoxy-	19.86	1031	5	4000000 -		1	
Benzaldehyde, 4-methoxy- Vanillin	22.45	1122	8			T	
Benzaldehyde, 4-methoxy- Vanillin Benzaldehyde, 4-hydroxy-3,5-dimethoxy			8	4000000 - 2000000 -		1	
Benzaldehyde, 4-methoxy- Vanillin	22.45	1122	8	2000000 -			
Benzaldehyde, 4-methoxy- Vanillin Benzaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde)	22.45 26.37	1122 1243			C-OAR	C-120	C-I
Benzaldehyde, 4-methoxy- Vanillin Benzaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol	22.45 26.37 14.48	1122 1243 850		2000000 -	C-OAR	C-120	C-1
Bernzidehyde, 4-methoxy- Vanillin Bernzidehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol Phenol, 2-methoxy (Guaiacol)	22.45 26.37	1122 1243		2000000 - Samples <sup>0 -</sup>			60
Bernaldehyde, 4-methoxy- Vanillin Bernaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol Phenol, 2-methoxy (Guaiacol) 2-Methoxy-4-ethylphenol (4-	22.45 26.37 14.48	1122 1243 850	000 U 1000	2000000 - Samples <sup>0 -</sup>			60
Bernaldehyde, 4-methoxy- Vanillin Bernaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol, Phenol, 2-methoxy (Gualacol) 2-Methoxy-4-ethylphenol (4- Ethylguaisol)	22.45 26.37 14.48 16.72 18.71	1122 1243 850 933 1012	COMPANY OF MARK	2000000 -			60
Benzaldehyde, 4-methoxy- Vanilin Benzaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol Phenol, Phenol, Gualiacol) 2-Methoxy-4-ethylphenol (4- Ethylguaiscol) 2-Methoxy-4-winylphenol (4- Vinniguaiscol)	22.45 26.37 14.48 16.72 18.71 20.90	1122 1243 850 933 1012 1047	COMPANY NO.	2000000 - Samples <sup>0 -</sup>			60
Benaldelyde, 4-methoxy- Vanilin Benaldelyde, 4-hydroxy-3,5-dimethoxy (syringaldelyde) Phonol Phonol Phonol, 2-methoxy (Gualacol) Phonol (4- Ethylgalacol) 2-Methoxy-4-winylphenol (4- Vinglqalacol) Vinglqalacol)	22.45 26.37 14.48 16.72 18.71	1122 1243 850 933 1012	COMPANY AND A	2000000 - Samples <sup>0 -</sup>		lysi	5
Benzaldehyde, 4-methoxy- Vamilin Benzaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol Phenol, 2-methoxy (Gualiacol) 4-Methoxy-4-ethylphenol (4- tthylguaiacol) 4-Methoxy-4-Unrylphenol (4- Vinriguaiacol) Phenol, 2-dimethoxy (52yringol) Phenol, 2-dimethoxy (42,propenyi)-	22.45 26.37 14.48 16.72 18.71 20.90 21.51	1122 1243 850 933 1012 1047	COMMENT OF THE OWNER	2000000 - Samples <sup>0 -</sup>	ana as (axes PC1 an	<b>lysi</b> 1952: 65.73 %	5
Benaldehyde, 4-methoxy- Vanilin Benaldehyde, 4-hydroxy, 3,-dimethoxy (Syringaldehyde) Phonol Phonol, 2-methoxy (Gualaco) 2-Methoxy 4-khyphenol (4- 2-Methoxy 4-kinyphenol (4- 2-Methoxy 4-kinyphenol (4- Vin(gualaco)) Phenol, 2-methoxy (5yringo) Phenol, 2-methoxy (5yringo)	22.45 26.37 14.48 16.72 18.71 20.90	1122 1243 850 933 1012 1047 1106	COMPANY AND INCOME	2000000 - Samples <sup>0 -</sup>	ana as (axes PC1 an	lysi	5
Benzaldehyde, 4-methoxy- Vamilin Benzaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol Phenol, Penethoxy (Gualiacol) 2-Methoxy-4-ethylphenol (4- tthylguaicol) 2-Methoxy-4-Unrylphenol (4- Vinriguaicol) Phenol, 2-sedimethoxy (Syringol) Phenol, 2-sedimethoxy (42-propenyl)-1 (Eugenol) Acetoxanilone (Ethanone, 1-(4-	22.45 26.37 14.48 16.72 18.71 20.90 21.51	1122 1243 850 933 1012 1047 1106	CONTRACTOR OF THE OWNER OF	2000000 - Samples 0 - PCA Observation	ana as (axes PC1 an	<b>Iysi</b> 1952: 65.73 %	5
Benaldehyde, 4-methoxy- Vamilin Benaldehyde, 4-hydroxy 3,5-dimethoxy (yringaldehyde) Phenol, 2-methoxy (Gualacol) 2-Methoxy - 4ethylphenol (4- Vinig(galacol) Phenol, 2-dimethoxy (Syringol) Phenol, 2-dimethoxy (Syringol) Phenol, 2-dimethoxy (Syringol) Phenol, 2-dimethoxy (Syringol) Acetoxanillone (Ethanone, 1-(4- hydroxy-3-methoxyhenyl-)	22.45 26.37 14.48 16.72 18.71 20.90 21.51 21.64 23.77	1122 1243 850 933 1012 1047 1106 1108 1145	CONTRACTOR OF A LOCAL	2000000 - Samples 0 - PCA Observation	ana as (axes PC1 an	<b>lysi</b> 1952: 65.73 %	5
Benzaldehyde, 4-methoxy- Vamilin Benzaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol Phenol, Penethoxy (Gualiacol) 2-Methoxy-4-ethylphenol (4- tthylguaicol) 2-Methoxy-4-Unrylphenol (4- Vinriguaicol) Phenol, 2-sedimethoxy (Syringol) Phenol, 2-sedimethoxy (42-propenyl)-1 (Eugenol) Acetoxanilone (Ethanone, 1-(4-	22.45 26.37 14.48 16.72 18.71 20.90 21.51 21.64	1122 1243 850 933 1012 1047 1106 1108	CONTRACTOR OF A DESCRIPTION OF A DESCRIP	2000000 - Samples 0 - PCA Observation	ana ans (axes PC1 and	<b>Iysi</b> 1952: 65.73 %	5
Benzidehyde, 4-methoxy- Vanilin Benzidehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde) Phenol Phenol, Penethoxy (Gualiacol) 2-Methoxy-4-ethylphenol (4- tthylguaicol) 2-Methoxy-4-unylphenol (4- Vinriguaicol) Phenol, 2-sedimethoxy (51/ringol) Phenol, 2-sedimethoxy (42-propenyl)- (Eugenol) Acetoxanilone (Ethanore, 1-(4- hydroxy-3-methoxyhenyl)-1)	22.45 26.37 14.48 16.72 18.71 20.90 21.51 21.64 23.77	1122 1243 850 933 1012 1047 1106 1108 1145	CONTRACTOR OF A CONTRACT	2000000 - Samples 0 -	ana ms (axes PC1 am	<b>Iysi</b> 1952: 65.73 % :-120 C-0AD	5
Benaldelyde, 4-methoxy- Vanilin Benaldelyde, 4-hydroxy-3,5-dimethoxy (Syringaldelyde) Phenol, 2-methoxy (Gualacol) Phenol, 2-methoxy (Gualacol) 4-thylgalacol) 7-thylgalacol Viny(gualacol) Phenol, 2-dimethoxy (Syringol) Phenol, 2-dimethoxy (Syringol) Phenol, 2-dimethoxy-4-(2-propeny)- (Eugenol) Acetoranillone (Ethanone, 1-(4- hydroxy-3-methoxyheny)-) Phenol, 2.6-bis(1,1-dimethylethyl)-4- methyl-	22.45 26.37 14.48 16.72 18.71 20.90 21.51 21.64 23.77	1122 1243 850 933 1012 1047 1106 1108 1145	COMPANY OF COMPANY OF COMPANY	2000000 - Samples 0 - PCA Observatie g 4 g 2 g 2	ana ans (axes PC1 and	<b>Iysi</b> 1952: 65.73 % :-120 C-0AD	5
Benzaldehyde, 4-methoxy- Vanilin Benzaldehyde, 4-hydroxy-3,5-dimethoxy (syringaldehyde) Phenol Phenol, Penchoxy (Gualacol) 2-Methoxy-4-ethylphenol (4- tthylgualcol) Phenol, 2-sethoxy-4-2;propenyl)- (Eugenol) Acetoxanilone (Ethanore, 1-(4- hydroxy-3-methoxy/benyl)-) Phenol, 2-failtoxy-12-gropenyl)- (Eugenol) Acetoxanilone (Ethanore, 1-(4- hydroxy-3-methoxybenyl)-) Phenol, 2-fail(1,1-dimethyl)-4- methyl-	22.45 26.37 14.48 16.72 18.71 20.90 21.51 21.64 23.77 24.14 13.65	1122 1243 933 1012 1047 1106 1108 1145 1151	CONTRACTOR OF THE OWNER OWNE	2000000 - Samples 0 -	ana ms (axes PC1 am	Iysi: aPC2: 65.73 % 120 C-0AD P-120	<b>5</b>
Benaldehyde, 4-methoxy- Vanilin Benaldehyde, 4-hydroxy 3,5-dimethoxy (Syringaldehyde) Phenol, 2-methoxy (Gualacol) Phenol, 2-methoxy (Gualacol) 4-bithisaircol Viniy(gualacol) Phenol, 2-dimethoxy (Syringol) Phenol, 2-dimethoxy (Syringol) Acetoranillone (Ethanone, 1-(4- hydroxy-3-methoxyhenyl-)) Phenol, 2-bit(1,1-dimethylethyl)-4- methyl- 2,5-Furandione, 3-dimethyl-	22.45 26.37 14.48 16.72 18.71 20.90 21.51 21.64 23.77 24.14 13.65 15.69	1122 1243 850 933 1012 1047 1106 1108 1145 1151 838 918	CONTRACTOR OF A CONTRACTOR OF A CONTRACT OF	2000000 - Samples 0 -	ana 3ms (axes PC1 am • C-170 • P-170	Iysi: APC2: 65.73 % -120 C-QAD P-120	) OAD
Benzaldehyde, 4-methoxy- Vanilin Benzaldehyde, 4-hydroxy-3,5-dimethoxy (syringaldehyde) Phenol Phenol, Penchoxy (Gualacol) 2-Methoxy-4-ethylphenol (4- tthylgualcol) Phenol, 2-sethoxy-4-2;propenyl)- (Eugenol) Acetoxanilone (Ethanore, 1-(4- hydroxy-3-methoxy/benyl)-) Phenol, 2-failtoxy-12-gropenyl)- (Eugenol) Acetoxanilone (Ethanore, 1-(4- hydroxy-3-methoxybenyl)-) Phenol, 2-fail(1,1-dimethyl)-4- methyl-	22.45 26.37 14.48 16.72 18.71 20.90 21.51 21.64 23.77 24.14 13.65	1122 1243 933 1012 1047 1106 1108 1145 1151	CONTRACTOR OF A CONTRACT OF A	2000000 - Samples 0 -	ana ms (axes PC1 am	Lysis PC2: 65.73 % C-QAD P-120	<b>5</b>

RESULTS AND DISSCUSION







### CONCLUSION

 All oak samples showed very complex volatile profiles rich in different families of aroma compounds such as acids, alcohols, aldehydes, esters, ketones, lactones, phenolic aldehydes, volatile phenols and other compounds.

Heating temperature significantly affects the composition of the volatiles and content of oak samples.

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