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

Oak samples	Chips	Powder
Open air dried	C-OAD	P-OAD
Toasted at 120 °C	C-120	P-120
Toasted at 170 °C	C-170	P-170

Instrumentation (GC-MS)

Agilent 5975 Mass Spectrometer coupled to an Agilent 6890N Gas Chromatograph

SPME conditions: SPME fiber: DVB/Carboxen/PDMS 50/30, 2 cm stable flex (Supelco, Bellfonte, USA)

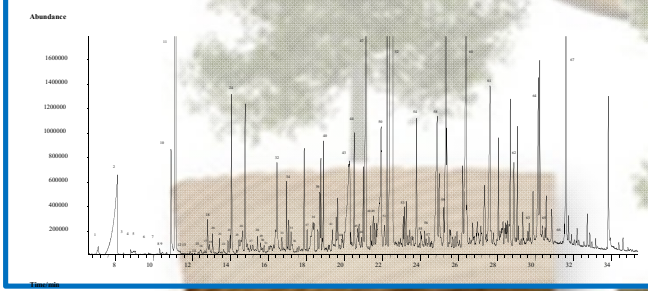
Separation – *medium* polar capillary column, HP5MS, 30 m * 0,25 mm * 1µm, Agilent Technologies

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

- 10 °C for 1 min with a temperature ramp of 8 °C min^{-1}
- up to 270 °C (holding time 1 min).

Total ion chromatogram of the volatile compounds detected in oak chip sample toasted at 120 °C (C-120)



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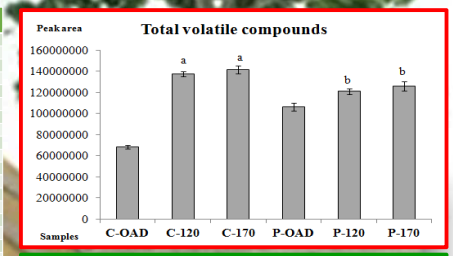
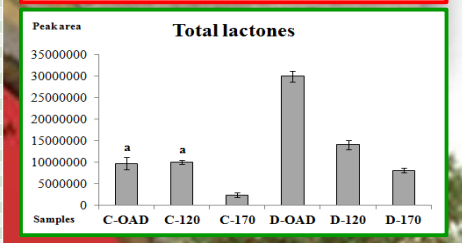
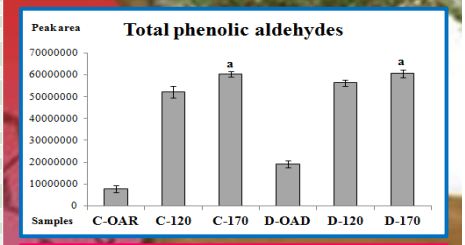
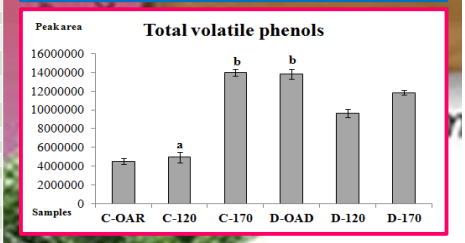
RESULTS AND DISSCUSION

No	Compound name	t_R/min	LRI$_{exp.}$ [HP5]
Acids			
1	Formic acid [1]	7.08	-
2	Acetic acid	7.85	703
4	Propanoic acid	8.96	719
5	2-Propenoic acid	9.03	720
8	Butanoic acid	10.45	741
11	Butanoic acid, 3-methyl-	11.76	811
15	Pentanoic acid	12.46	821
26	Hexanoic acid	14.64	902
38	Octanoic acid	18.39	1007
43	Nonanoic acid	20.22	1037
50	Decanoic acid	21.80	1111
53	Vanillic acid	23.08	1133
58	Dodecanoic acid	24.82	1212
61	Tetradecanoic acid	27.59	1311
62	Pentadecanoic acid	28.88	1336
64	Hexadecanoic acid	30.15	1416
Alcohols			
14	Furfuryl alcohol	12.05	815
28	1-Hexanol, 2-ethyl-	15.43	914
29	Benzyl alcohol	15.60	916
36	Phenethyl alcohol	17.20	940
Aldehydes			
6	(E) 2-Butenal, 2-methyl-	9.72	731
7	Hexanal	10.31	739
9	3-Furaldehyde	10.65	744
10	Furfural (2-Furaldehyde)	11.12	802
16	n-Heptanal	12.68	824
24	5-Methyl furfural	14.07	844
27	1H-Pyrrole-2-carboxaldehyde	15.08	908
34	n-Nonanal	16.96	937
35	Maltol	17.20	940
37	(E) 2-Nonenal	18.04	1001
40	Decanal	18.89	1015
45	Undecanal	20.70	1044
66	Octadecanal	30.86	1430
67	(E)15-Heptadecenal	31.60	1444
Esters			
13	1-Methoxy-2-propyl ester of acetic acid	12.03	815
51	Butanoic acid, butyl ester	21.90	1113
56	Benzoic acid, 4-hydroxy-3-methoxy-, methyl ester	24.21	1200
63	Hexadecanoic acid, methyl ester (Palmitic acid, methyl ester)	29.68	1407
65	Hexadecanoic acid, ethyl ester	30.52	1423
Ketones			
3	2-Propanone, 1-hydroxy-	8.41	712
12	2-Butanone (Methyl ethyl ketone)	11.91	813
18	2-Acetylfuran (Ethanone, 1-(2-furanyl))	12.91	827
21	2-Pentanone, 4-hydroxy-4-methyl-	13.44	835
59	Propiovanillone	25.21	1220
Lactones			
19	Butyrolactone	13.06	829
20	2(5H)-Furanone (gamma.-Crotonolactone)	13.09	830
44	cis-Methyl-hydroxyoctanoic acid lactone (cis-whisky lactone)	20.50	1041
47	trans-4-Hydroxy-3-methyloctanoic acid lactone (trans-whisky lactone)	21.08	1050
Phenolic aldehydes			
23	Benzaldehyde	14.00	843
31	Benzaldehyde, 2-hydroxy-	15.79	919
42	Benzaldehyde, 4-methoxy-	19.86	1031
52	Vanillin	22.45	1122
60	Benzaldehyde, 4-hydroxy-3,5-dimethoxy (Syringaldehyde)	26.37	1243
Volatile phenols			
25	Phenol	14.48	850
33	Phenol, 2-methoxy (Guaiacol)	16.72	933
39	2-Methoxy-4-ethylphenol (4-Ethylguaiacol)	18.71	1012
46	2-Methoxy-4-vinylphenol (4-Vinylguaiacol)	20.90	1047
48	Phenol, 2,6-dimethoxy (Syringol)	21.51	1106
49	Phenol, 2-methoxy-4-(2-propenyl)- (Eugenol)	21.64	1108
54	Acetovanillone (Ethanone, 1-(4-hydroxy-3-methoxyphenyl)-)	23.77	1145
55	Phenol, 2,6-bis[1,1-dimethylethyl)-4-methyl-	24.14	1151
Other compounds			
22	2,5-Furandione, 3-methyl-	13.65	838
30	2,5-Furandione, 3,4-dimethyl-	15.69	918
32	2-Methoxy-6-methylpyrazine	16.46	929
41	Benzothiazole	19.36	1023
57	Dibenzofuran	24.28	1202

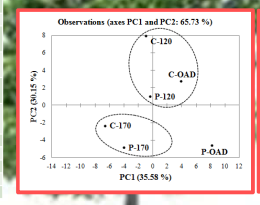


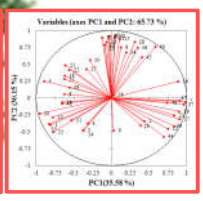






PCA analysis





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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