

Artificial aging of wines using oak chips

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Abstract

Wine treated with oak chips was analysed for its contents of furfural, vanillin, guaiacol, oak lactone, eugenol and syringaldehyde. These wood-originated volatiles were released into the wine and their levels were measured by gas chromatography, at different time intervals, for fourteen days. The amounts measured were compared to those found in barrel-aged wine. Statistical analysis of the data indicates that syringaldehyde, primarily, and then vanillin, guaiacol and furfural can be used to discriminate artificially aged from barrel-aged wine. Two different sizes of oak chips were used for this study.

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1. Introduction

Oak wood is mainly composed of three large insoluble polymers – cellulose, hemicellulose and lignin. It also contains other compounds of lower molecular weight, such as volatile and non-volatile acids, sugars, steroids, terpenes, volatile phenols, and lactones which can be extracted in wine or hydroalcoholic solvents (Maga, 1984; Nykanen, 1983). Although aging conditions, such as the wine cellar temperature and humidity and the length of time in barrel, affect the characteristics of wine (Dubois, 1989; Towey & Waterhouse, 1996a, 1996b), the most important factor is the raw material of the barrel – oak – and its treatment (Towey & Waterhouse, 1996b; Miller & Howell, 1992), as these two factors determine the wood compounds extracted in wine.

Studies carried out on the contributions of oak to the olfactory characteristics of wine have shown that these are mainly influenced by compounds such as furfural, guaiacol, whisky lactone, eugenol, vanillin, and syringaldehyde (Pollnitz, Jones, & Sefton, 1999; Mosedale, Puech, & Feuillat, 1999; Perez-Coello, Sanz, & Cab-

ezudo, 1999; Spillman, Iland, & Sefton, 1998; Spillman, 1997; Chatonnet, Cutzach, Pons, & Dubourdiou, 1999).

More specifically, furfural (2-furancarboxaldehyde) originates from degradation of monosaccharides produced by partial hydrolysis of hemicellulose. It contributes the character of dried fruits, and particularly that of burned almonds (Sauvageot & Feuillat, 1999; Spillman et al., 1998; Chatonnet et al., 1999).

Guaiacol (*o*-methoxyphenol) is produced by the lignin's breakdown during wood toasting and it is responsible for the burn overtones of wine aroma (Aiken & Noble, 1984; Guymon & Crowell, 1972; Hale, McCafferty, Larmie, Newton, & Swan, 1999; Chatonnet, 1998).

Oak lactone (*cis* and *trans* isomers of β -methyl- γ -octalactone), which is often referred to as whisky lactone, since it was discovered in bourbon (Suomalainen & Nykanen, 1970), originates from oak lipids and directly influences the character of wine (Mosedale et al., 1999). That is why the concentration of this compound in wine significantly determines quality, as well as, acceptance by the consumer (Spillman, 1997). Whisky lactone mainly attributes a woody and coconut character (Pollnitz et al., 1999; Chatonnet, 1995; Boidron, Chatonnet, & Pons, 1988; Perez-Coello et al., 1999; Spillman et al., 1998; Spillman, 1997), while some studies have shown that high concentrations of this

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compound are associated with wine that has an intense perfume of vanilla (Boidron et al., 1988; Feuillat, Keller, Sauvageot, & Puech, 1999; Miller & Howell, 1992; Sauvageot & Feuillat, 1999; Spillman et al., 1998).

Eugenol (2-methoxy-4-(2-propenyl) phenol), a volatile phenol, is produced by the lignin's breakdown during wood toasting and contributes the character of spices, cloves and smoke (Aiken & Noble, 1984; Feuillat et al., 1999).

Vanillin (4-hydroxy-3-methoxybenzaldehyde) emanates from lignin degradation and can be synthetically produced from eugenol or guaiacol. It influences wine aroma directly and pleasantly by attributing a character of vanilla (Puech, 1987).

Syringaldehyde (hydroxy-3,4-dimethoxybenzaldehyde) is formed by lignin breakdown during wood toasting and is related to the vanilla character of wine (Chatonnet et al., 1999; Chatonnet, 1998).

Recently, many new techniques have been introduced in winemaking. One of these involves putting new pieces of wood (oak chips or inner staves) into inert containers. It offers some distinct and previously unavailable flavour advantages, as well as new options in wine handling. Since wood is being put into wine and not wine into wood, the entire surface area is usable and not just 40% of it. The result is a compelling application that has been adopted by many (Stutz, Lin, & Herdman, 1999).

The main aim of this work was to quantify the wood-released aromatic compounds (furfural, guaiacol, whisky lactone, eugenol, vanillin and syringaldehyde) in arti-

cially-aged and barrel-aged wine. Our second objective was to evaluate the effect of the size of wood chips, and of contact time, on the aromatic compound levels in artificially-aged wine. Finally, the possibility of discriminating artificially-aged from barrel-aged wine (of the same grape variety), using PCA (principal component analysis) and DA (discrimination analysis), was explored, based on the concentration of the above mentioned aromatic compounds.

2. Materials and methods

2.1. Samples

Samples of bottled wine (10 bottles of Asyrtiko variety and 7 bottles of other varieties) aged in oak barrels were purchased from the local market. The wine (only of Asyrtiko variety) used for treatment with oak chips was purchased from a vinery of Santorini. All Asyrtiko wine samples used for this study were from the same V.Q.P.R.D. region, Santorini, apart from the sample '5'.

Wine samples were treated as follows: 20 g of NaCl were added to 100 ml of the sample and 1 ml of 3-octanol solution (70 mg/100 ml) was added as internal standard. This mixture was extracted twice using 200 ml of an *n*-pentane and diethyl ether (1:1, v/v) mixture each time. The obtained organic phase was washed three times with 30 ml of saturated sodium bicarbonate solution and once with 30 ml of H₂O. The organic layer was dried over Na₂SO₄.

Table 1
The uptake of aroma compounds by the wine during artificial aging with *Small* oak chips

Name	Time	Furfural	Guaiacol	Oak lactone	Eugenol	Vanillin	Sy/gal
w1	1	0.0327	0.0137	0.299	0.005	2.77	0.194
w2	2	0.157	0.0154	0.315	0.0052	2.68	0.204
w3	3	0.715	0.0178	0.338	0.0085	2.62	0.224
w4	4	1.37	0.0308	0.406	0.0078	3.09	0.366
w5	5	1.46	0.0400	0.409	0.0066	3.09	0.3722
w6	6	2.723	0.0394	0.280	0.0098	3.05	0.225
w7	7	3.89	0.0532	0.361	0.0076	2.89	0.302
w14	14	6.99	0.0772	0.377	0.0097	3.00	0.293
w1'	1	0.0985	0.0203	0.266	0.0009	1.61	0.186
w2'	2	0.459	0.0177	0.265	0.0058	1.74	0.219
w3'	3	0.522	0.0203	0.290	0.0050	2.07	0.229
w4'	4	1.57	0.0330	0.282	0.0063	2.20	0.280
w5'	5	1.14	0.0298	0.363	0.0057	2.69	0.290
w6'	6	3.00	0.0279	0.395	0.0044	2.24	0.300
w7'	7	4.01	0.0568	0.401	0.0063	2.02	0.299
w14'	14	7.26	0.0615	0.433	0.0086	2.97	0.300
w1''	1	0.964	0.0138	0.282	0.0056	2.55	0.194
w2''	2	1.569	0.0146	0.290	0.0056	2.59	0.190
w3''	3	1.23	0.0195	0.314	0.006	2.72	0.246
w4''	4	1.57	0.029	0.344	0.006	2.84	0.261
w5''	5	2.37	0.0352	0.386	0.0074	2.58	0.284
w6''	6	2.99	0.0492	0.338	0.006	2.24	0.292
w7''	7	3.52	0.0536	0.38	0.0074	3.00	0.301
w14''	14	7.53	0.0695	0.405	0.0095	3.12	0.31

The time is expressed in days, the amounts in mg/l, and w1, w1', w1'' indicate replicates of the same day.

Finally, it was placed in a pear-shaped flask equipped with a Vigreux column of 20–25 cm length and it was slowly concentrated to 0.2 g by heating in a water bath at 45–55 °C (Schneider, Baumes, Bayonove, & Razungles, 1998).

2.2. Wine treatment with oak chips

Two different types of oak chips, toasted at 200 °C for 2 h, were used for the artificial aging of wines: a

Small one, sized 1 cm × 1 cm × 0.1 cm and a *Big* one, sized 3.4 cm × 2 cm × 1 cm. White wine, of Asyrtiko variety, was used (11.5 vol.% of alcohol, pH 3.0).

The procedure followed was: 1g of the *Small* oak chips or ~8 g of the *Big* oak chips were added to 200 ml of wine sample in order to have about the same surface of contact in both cases. The wine samples were then stored in closed flasks for 1, 2, 3, 4, 5, 6, 7 or 14 days. Each experiment was run three times (annotated as w,

Table 2
The uptake of aroma compounds by the wine during artificial aging with *Big* oak chips

Name	Time	Furfural	Guaiacol	Oak lactone	Eugenol	Vanillin	Sy/gal
n1	1	0.854	0.0661	0.238	0.0069	2.64	0.191
n2	2	1.27	0.0617	0.259	0.0068	2.81	0.141
n3	3	1.92	0.0721	0.308	0.0087	2.47	0.257
n4	4	2.98	0.0927	0.270	0.0094	2.78	0.301
n5	5	3.01	0.123	0.260	0.0055	2.76	0.278
n6	6	3.40	0.104	0.396	0.0120	2.52	0.315
n7	7	6.62	0.148	0.353	0.0111	3.11	0.357
n14	14	7.58	0.161	0.416	0.0112	3.19	0.238
n1'	1	0.496	0.0386	0.235	0.0052	1.91	0.204
n2'	2	1.28	0.0332	0.287	0.0040	1.26	0.236
n3'	3	1.58	0.0375	0.297	0.0051	1.64	0.256
n4'	4	2.02	0.0503	0.300	0.0078	2.10	0.236
n5'	5	3.69	0.101	0.299	0.0078	1.93	0.274
n6'	6	4.24	0.114	0.316	0.0110	3.35	0.301
n7'	7	5.97	0.100	0.350	0.0073	2.17	0.316
n14'	14	6.01	0.137	0.357	0.0104	2.47	0.346
n1''	1	1.83	0.0501	0.278	0.0056	2.30	0.246
n2''	2	1.65	0.0601	0.278	0.0068	2.47	0.252
n3''	3	2.24	0.0952	0.257	0.0081	2.40	0.249
n4''	4	3.10	0.0985	0.286	0.0101	2.59	0.266
n5''	5	3.903	0.109	0.300	0.0110	2.56	0.278
n6''	6	5.00	0.115	0.313	0.0099	2.93	0.280
n7''	7	6.24	0.128	0.339	0.0114	2.90	0.299
n14''	14	7.13	0.156	0.429	0.0126	3.00	0.316

The time is expressed in days, the amounts in mg/l, and n1, n1', n1'' indicate replicates of the same day.

Table 3
The quantities of the six aroma compounds in barrel-aged wine

Name	Furfural	Guaiacol	Oak lactone	Eugenol	Vanillin	Sy/de	Variety
1	1.41	0.029	0.0083	0.0211	2.949	0.0379	Asyrtiko
2	1.97	0.0494	0.0076	0.0289	1.8200	0.0281	Asyrtiko
3	1.80	0.0294	0.0023	0.006	2.0391	0.0116	Asyrtiko
4	1.62	0.0322	0.0030	0.0099	1.9691	0.0327	Asyrtiko
5	1.84	0.0424	0.0027	0.0099	1.4650	0.0287	Asyrtiko
6	1.77	0.0338	0.0027	0.0078	1.4339	0.0124	Asyrtiko
7	2.36	0.0101	0.3497	0.0098	2.5482	0.0293	Asyrtiko
8	1.48	0.0173	0.868	0.0107	2.3062	0.0430	Asyrtiko
9	2.30	0.0333	0.907	0.0087	3.0289	0.0479	Asyrtiko
10	2.62	0.0242	1.145	0.0139	1.4803	0.0580	Asyrtiko
11	2.11	0.0888	0.0851	0.0094	2.0831	0.0887	Non-Asyrtiko
12	2.02	0.0938	0.099	0.0072	1.3534	0.136	Non-Asyrtiko
13	1.32	0.1216	0.112	0.0068	1.4913	0.163	Non-Asyrtiko
14	2.42	0.2201	0.888	0.0211	3.0622	0.488	Non-Asyrtiko
15	2.01	0.1354	1.316	0.0174	2.6038	0.455	Non-Asyrtiko
16	1.44	0.0218	0.763	0.0197	1.7197	0.338	Non-Asyrtiko
17	1.88	0.0893	0.460	0.0196	1.0807	0.326	Non-Asyrtiko
Average	1.91	0.0301	0.30	0.0127	2.1040	0.033	Asyrtiko

The amounts are expressed in mg/l.

w', w'' or n', n'' for the small and big oak chips, respectively) and the samples were analyzed.

2.3. Gas chromatography analysis

Each wine sample's extract was subjected to GC analysis, using a Hewlett Packard 6890 GC, equipped with an HP-1, Hewlett Packard, fused silica capillary column, cross-linked, 100% methyl siloxane (30 m

length, 0.32 mm i.d., 0.25 μm film thickness). Samples (0.5 μl) were injected using split mode with a split ratio of 1:44. The nitrogen carrier gas flow rate was set at 2.84 ml/min. The injector temperature was 220 $^{\circ}\text{C}$.

The column temperature programme was as follows: 50 $^{\circ}\text{C}$ (held for 3 min) to 80 $^{\circ}\text{C}$ at a rate of 2.00 $^{\circ}\text{C}/\text{min}$ and then to 240 $^{\circ}\text{C}$ at a rate of 3.50 $^{\circ}\text{C}/\text{min}$ with a 5-min final isotherm. Detection was carried out by flame ionization maintained at 280 $^{\circ}\text{C}$.

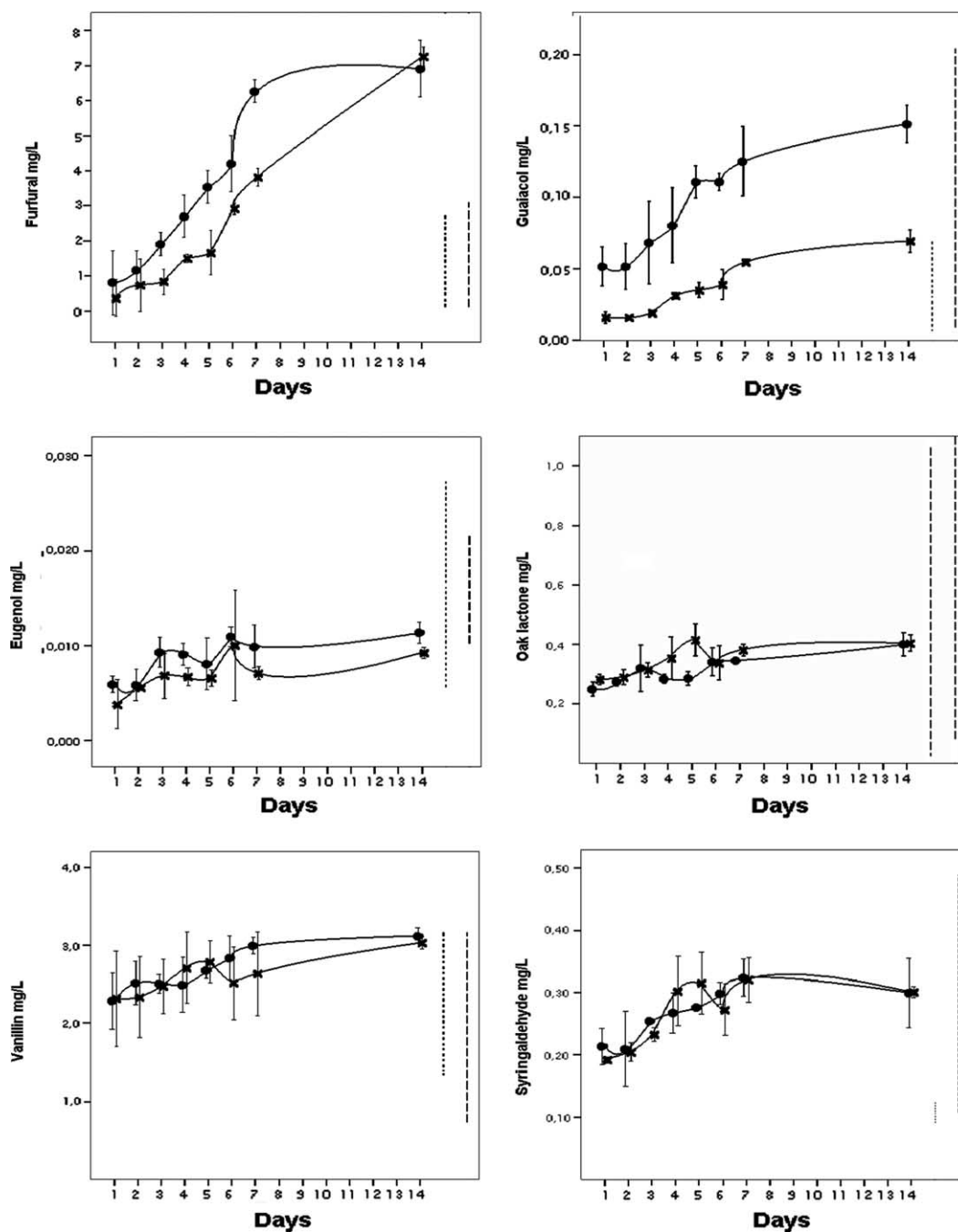


Fig. 1. The uptake of furfural, guaiacol, oak lactone, eugenol, vanillin, syringaldehyde by the Asyrtiko wine during treatment with oak chips (—●— big and —×— small). Inside each figure the vertical lines (dot for Asyrtiko and dashed for non-Asyrtiko) indicate the concentration of each compound in barrel-aged wines.

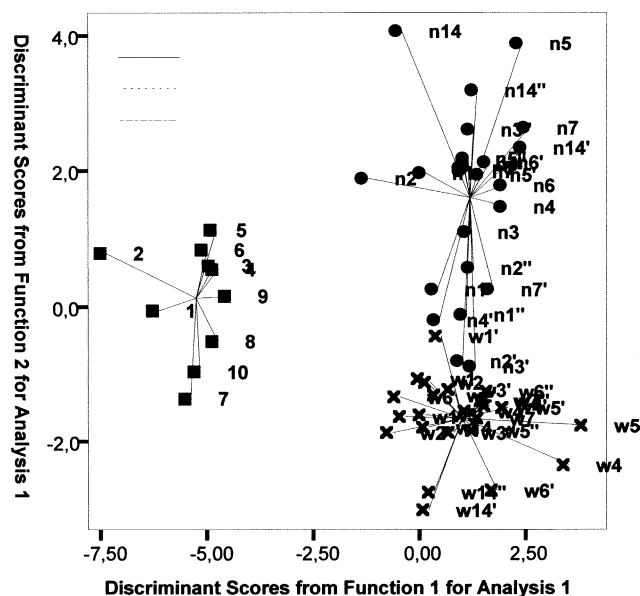


Fig. 2. Projection of a scatter diagram from the Discrimination analysis of the reference data used to discriminate the wines treated with oak chips (—●— big and —×— small) from the barrel-aged wines (□) of Asyrtiko variety. Function 1 is strongest absolute correlated with syringaldehyde and eugenol; Function 2 with guaiacol and furfural. Oak lactone and vanillin are not used in the analysis (see also in Table 4).

For the calibration table, a model wine (hydro-alcoholic solution at 10% (vol) alcohol, 5.5 g/l tartaric acid and pH adjusted to 3.5 with NaOH solution) was prepared. Two calibration standard solutions were prepared twice, using the model wine. The first one contained 1 mg/l of furfural, 10^{-2} mg/l of guaiacol, 10^{-2} mg/l of whisky lactone, 10^{-2} mg/l of eugenol, 7.5×10^{-2} mg/l of vanillin and 10^{-2} mg/l of syringaldehyde and the second one contained 7 mg/l of furfural, 1 mg/l of guaiacol, 1 mg/l of whisky lactone, 1 mg/l of eugenol, 1

mg/l of vanillin and 1 mg/l of syringaldehyde. Each standard solution was treated the same as the wine samples.

The identification and quantification of the above-mentioned compounds were achieved by using the calibration curve of each compound and of the Calibration table programme of HP GC ChemStation Rev. 06.03 software.

2.4. Statistical analysis of results

The data set, composed of values obtained from GC analysis, was subjected to principal component analysis (PCA) and stepwise discrimination analysis (DA). The statistical programme SPSS for Windows (SPSS Inc., v10.0.7) was used to calculate and plot the data from PCA and DA. In addition, in order to evaluate the predictive accuracy of the discrimination model, a classification matrix was calculated. The results were cross-validated, meaning that each case was classified by the functions derived from all other cases. The original results may provide overly optimistic estimates and cross-validation attempts to remedy this problem. The classification results were compared to the ones that could be classified correctly by chance, taking into account the group sizes.

3. Results and discussion

The results of the GC analysis are listed in Table 1 for wine treated with *Small* oak chips, in Table 2 for wine treated with *Big* oak chips, and in Table 3 for barrel-aged wine. The concentrations of furfural, guaiacol, oak lactone, eugenol, vanillin and syringaldehyde in wine are plotted versus the time of contact of oak chips with wine

Table 4
Classification results of each sample

		Groups	Small	Big	Asyrtiko	Total
Original	Count	Small	24	0	0	24
		Big	4	20	0	24
		Asyrtiko	0	0	10	10
	%	Small	100	0	10	100
		Big	16.7	83.3	0	100
		Asyrtiko	0	0	100	100
Cross-validated ^a	Count	Small	24	0	0	24
		Big	4	20	0	24
		Asyrtiko	0	0	10	24
	%	Small	100	0	10	100
		Big	16.7	83.3	0	100
		Asyrtiko	0	0	100	100

93.1% of the original grouped cases correctly classified. 93.1% of the cross validated grouped cases correctly classified and numbers of samples classified.

^a In cross validation, each case is classified by the functions derived from all cases other than the case.

(Fig. 1). Each figure depicts two different plots for the different oak chips (*Small* and *Big*). Each concentration is presented with an error bar of standard deviation ± 0.1 . The concentration plots for each compound are compared to the concentration levels of that compound in Asyrtiko and non-Asyrtiko barrel-aged wine (vertical dotted and dashed lines).

In Fig. 1, furfural appears to have a very fast and progressive extraction rate. Guaiacol's extraction rate is dependent on oak chip size (double the rate for the *Big* in comparison to the *Small* ones). This means that the size of wood is critical for the concentration of guaiacol.

Table 5

D.A. structure Matrix that contains within-group correlation of each predictor variable with the canonical function

Variable	Function 1	Function 2
Furfural	0.084	0.167 ^a
Guaiacol	0.206	0.634 ^a
Oak Lactone ^b	0.206 ^a	-0.204
Eugenol	-0.224 ^a	0.191
Vanillin ^b	0.059	0.273 ^a
Syringaldehyde	0.777 ^a	-0.024

^a Largest absolute correlation of variable with function.

^b This variable is not used in the analysis.

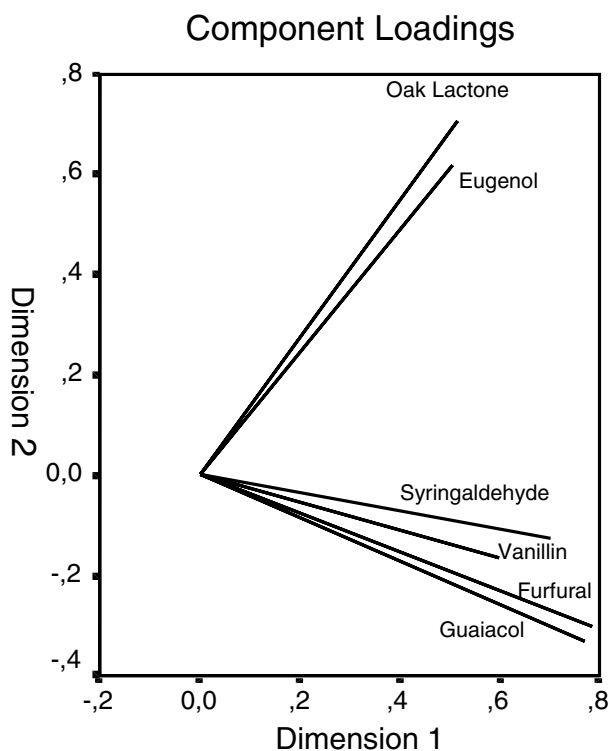


Fig. 3. The component loadings that show the relationships between the variables (furfural, guaiacol, oak lactone, eugenol, vanillin, syringaldehyde) and the Dimensions of the principal components analysis of Fig. 4.

Syringaldehyde shows a low extraction rate, especially after the third day.

The relationship between the concentration of each compound in wine treated with oak chips and that in barrel-aged wine (Asyrtiko and non-Asyrtiko) led to the following conclusions: due to the fast and progressive extraction rate of furfural, 5–6 days of wine treatment with oak chips were required so that this compound could reach the same level as that of barrel-aged wine while, at the end of the second week, its concentration in oak chips-treated wine was more than double that of barrel-aged wine.

In its average guaiacol concentration, the artificially-aged wine does not differ from the non-Asyrtiko barrel-aged wine, while it differs slightly from the Asyrtiko barrel-aged wine. This difference becomes particularly intense if the artificially-aged wine is treated with *Big* oak chips. Finally, the syringaldehyde concentration is much higher in wine treated with oak chips than that of Asyrtiko barrel-aged wine; therefore it is the most crucial compound of our study, as shown by the results of DA and PCA below.

DA was carried out in order to identify and evaluate the extractable oak compounds (furfural, guaiacol, oak lactone, eugenol, vanillin, syringaldehyde) which could be used for the distinction between wine treated with oak chips and barrel-aged wine of the same variety (Asyrtiko, in this case), as well as between wine treated with different oak chips (*Big* and *Small*). Using DA with the Mahalanobis Distance Stepwise method and setting the reference data of Tables 1–3 as variables, the projections of the scatter diagram of Fig. 2 were obtained. In this figure, Function 1 of DA is more highly correlated with syringaldehyde and eugenol; Function 2 of DA is more highly correlated with guaiacol and furfural; oak lactone and vanillin were not used for this analysis (Tables 4 and 5). In the same figure, the group of barrel-aged wine can be clearly discriminated from the groups of wine of the same variety treated with oak chips, mainly on the basis of syringaldehyde and eugenol, while the two groups of wine treated with different oak chips (*Small* and *Big*) are discriminated mainly on the basis of guaiacol. These DA results show acceptable levels of precision and repeatability (93.1% correct classification of cases, even after cross validation (Table 4)). Classification of four samples of the *Big* oak chips group into the *Small* one can be explained by the fact that this area is mainly composed of samples with a short extraction period which do not differ so much, because only small quantities of the substances have been extracted into the wine samples in both cases (*Small* and *Big* oak chips). As the time of extraction increases, this phenomenon is no longer observed and the proclivity of contraposition becomes intense.

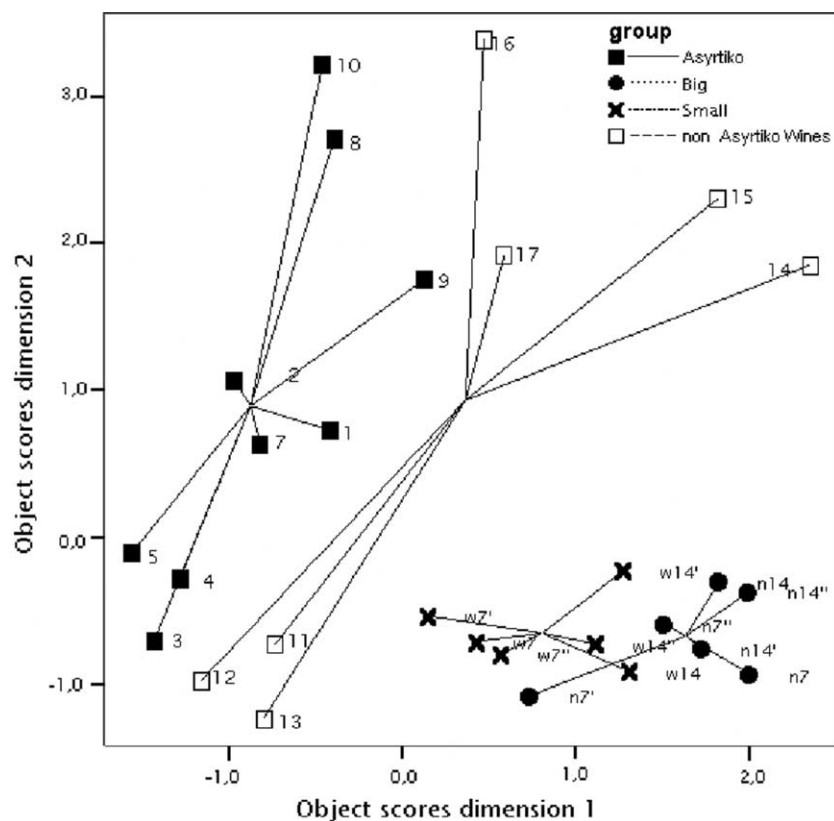


Fig. 4. Projection of a scatter diagram from the principal components analysis of the reference data used to discriminate the wines treated with oak chips of Asyrtiko variety from the barrel-aged wines of Asyrtiko variety and the barrel-aged wines of non-Asyrtiko variety. The component loadings of Fig. 3 are associated with the Dimensions of this matrix.

Consequently, according to the results of our experiment, syringaldehyde is the most important compound for the discrimination of the barrel-aged wine from wine of the same variety (Asyrtiko in this case) treated with oak chips, as is clearly shown in Figs. 1 and 2. Moreover, the combination of Figs. 1 and 2 show a distinction between wine treated with *Big* and *Small* oak chips, which means that the distinction of wines treated with different sized oak chips is possible only on the basis of guaiacol.

Use of PCA also permits the distinction between the barrel-aged wine and wine of the Asyrtiko variety treated with oak chips. The distinction showed in Fig. 4, was achieved considering the axis created by the loadings of *syringaldehyde-vanillin-furfural-guaiacol* (Fig. 3). In this analysis, 7 barrel-aged wine samples of non-Asyrtiko variety have been added, while only the samples of 7 and 14 days of artificial aging with oak chips were examined. Fig. 4 shows a distinction between the non-Asyrtiko wine, and the *Small* and *Big* oak chips wine groups and a weak distinction between the non-Asyrtiko and Asyrtiko wine, but in both cases the discrimination is clear. It is also very important to note that the '16' non-Asyrtiko barrel aged wine sample, which is the only white wine (Chardonnay) apart from Asyrtiko wine, is close to the group of Asyrtiko.

4. Conclusions

The main aim of the present work was to study the extraction rate of the most important aromatic components – vanillin, guaiacol, whisky lactone, eugenol, furfural, and syringaldehyde – from toasted oak wood into wine. As far as these compounds are concerned, furfural exhibits the fastest extraction rate. On the other hand, syringaldehyde presents a slow extraction rate during the first three days of extraction, which becomes minimal afterwards. The values obtained from GC analysis were used for principal component analysis and stepwise discrimination analysis. The results of this analysis could be used for the distinction between barrel-aged wine and wine of the same variety (Asyrtiko) treated with oak chips. Furthermore, the discrimination of wine samples treated with two different types of oak chips (*Big* and *Small*), which have almost the same surface of contact but not the same weight, is based on the different velocities of guaiacol extraction in these two cases. The concentration of syringaldehyde is higher in Asyrtiko wine treated with oak chips than in barrel-aged wine and seems to be the most important factor for the distinction between these two categories. This distinction is also influenced by the concentrations of vanillin, guaiacol and furfural.

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