

Aroma Compound Sorption by Oak Wood in a Model Wine

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Oak wood used for wine barrels was immersed into a model wine containing eight aroma compounds (e.g., aromatic and terpene alcohols, ethyl esters, and aldehyde), for which activity coefficients in water and model wine were determined using the mutual solubility measurement. A mass balance of these volatiles considering their reactivity in model wine was established. For most of the studied aroma compounds, and mainly for linalool and ethyl octanoate, a sorption behavior into wood was reported for the first time. This phenomenon was selective and could not be related to the solubilities in model wine and hydrophobicities of the studied aroma compounds, suggesting that acid–base and polar characteristics of wood were more involved in this sorption mechanism. This study has also shown that the level of sorption is a function of the ratio of wood surface area/solution volume.

Keywords: Sorption; aroma compounds; solubility; oak wood; wine

INTRODUCTION

Aging of wines in oak barrels has many advantages with regard to wood extractives. Wood volatiles, such as *cis*- and *trans*- β -methyl- γ -octalactone, the odor of which has been described as oak or coconut, and aldehydic phenols, play a major role in the organoleptic quality of premium quality wine (1). The importance of the ellagitannins to the flavor of wine and brandy is also well-known (2–4). However, oak wood may influence the concentration of wine components playing a role in the flavor of wine, such as aroma compounds, through a sorption mechanism. In addition to its mechanical, chemical, and nutritive properties, wood is also known for its sorptive property, particularly in regard to water (5).

Wood is a heterogeneous material that is formed of three major components, cellulose, hemicellulose, lignin, and two subcomponents that are organic extractives and inorganic minerals. It has been established that major tree wood components are capable of donating or accepting electrons, consequently giving wood an acid–base character (6), and that lignin consists of hydrophobic sites. According to Gardner et al. (7), wood extractives such as fatty acids and terpenes may also influence the wood's chemical properties. Thus, the wood by its structure and chemical composition may interact with wine, giving rise to sorption of wine aroma compounds along with other physicochemical events. Sorption phenomena in foods packaged with polymeric

and glass materials are well-known and were recently reviewed by Gnanasekharan and Floros (8).

The purpose of this work was to investigate the sorption of selected aroma substances by oak wood used for aging wine. After determination of the hydrophobicity and solubility parameters of volatiles, this sorption phenomenon was evaluated by monitoring the decrease in the concentration of aroma compounds in a model wine put in contact with a piece of oak wood in a closed system.

MATERIALS AND METHODS

Materials. The volatile compounds linalool, 2-phenylethanol, benzaldehyde, ethyl hexanoate, ethyl octanoate, ethyl decanoate, and β -ionone were purchased from Fluka (Buchs, Switzerland), and isoamyl acetate was from Sigma-Aldrich (St. Louis, MO). Their purities were >95%. Ethanol, 99.9% purity, L-malic acid (>99%), acetic acid (>99.8%), potassium sulfate (99%), and magnesium sulfate (99%) were obtained from Merck (Darmstadt, Germany). Solutions were made with ultrapure water, obtained from a Milli-Q system (Millipore, Bedford, MA). The ethyl acetate and diethyl ether (99.8%) obtained from Carlo Erba Reactives (Rodano, Italy) were used as solvents of extraction. The stave wood samples studied were taken from trees of sessile oak (*Quercus petraea* Liebl.) from the Citeaux forest and were kindly donated by Office National des Forêts (ONF), which measured all of the structural wood and chemical characteristics (9, 10). For our study, small plates of wood (2 × 10 × 20 mm; 0.24 ± 0.05 g) were cut from stave wood samples and were not toasted.

Model Wine. The model wine was a hydroalcoholic solution with 3 g/L L-(+)-malic acid, 0.111 g/L acetic acid, 0.1 g/L potassium sulfate, and 0.025 g/L magnesium sulfate (11, 12). The pH of the model wine was adjusted to 3.5 with 1 M sodium hydroxide. Alcohol strength chosen was 12.6% (v/v) in order to compare experimental values determined with those of the literature. In the case of sorption experiments, a 10% (v/v) ethanol solution was retained to have lower solubility conditions for aroma compounds in model wine. Aroma compounds were added to model wine at concentrations between 5 and 8

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Table 1. Physicochemical Characteristics of the Aroma Compounds Used in the Model Wine Solutions

aroma compound	solubility (g L ⁻¹)		log <i>P</i> ^a	activity coefficient γ^∞			
	water	model wine		water exptl value	water lit. exptl value	model wine exptl value	model wine lit. exptl value
isoamyl acetate	2.40 ± 0.5	4.15 ± 0.56	2.13	3027 ± 662 ^b	5016 ^c	1593 ± 219 ^b	
ethyl hexanoate	0.51 ± 0.11	1.03 ± 0.2	2.79	15710 ± 3554 ^b	18954 ^d	7105 ± 1434 ^b	9424 ^d
ethyl octanoate	0.04 ± 0.01	0.076 ± 0.01	3.97	239265 ± 63804 ^b	599500 ^d	115016 ± 15400 ^b	300233 ^d
ethyl decanoate	0.0048 ± 0.0011	0.0052 ± 0.0009	5.02	2318404 ± 560750		1954606 ± 352862	
linalool	1.23 ± 0.14	2.05 ± 0.51	2.43	6968 ± 803 ^b	8543, ^c 8631 ^c	3819 ± 1012 ^b	
2-phenylethanol	15.96 ± 3.25	19.19 ± 3.60	1.33	426 ± 90		324 ± 63	
benzaldehyde	4.48 ± 0.98	7.98 ± 1.94	1.50	1317 ± 302 ^b	1816, ^c 1436, ^c 1456 ^e	676 ± 174 ^b	
β -ionone	0.1078 ± 0.02	0.19 ± 0.02	3.78	99104 ± 19042 ^b	1210000 ^f	51356 ± 5466 ^b	-

^a Values of logarithm of the partition between water and *n*-octanol (log *P*) and molecular surface calculated by a chemical software Windows Molecular Pro. ^b The values of activity coefficient in model wine solution were significantly different from that in water (*P* < 0.05). ^c Espinosa et al., (22). ^d Lubbers et al., (11). ^e Farès et al., (23). ^f Sadafian and Crouzet (24) at 10 mmHg.

ppm. These are typical of wine aroma composition (13), and they were chosen because they exhibit a broad range of chemical structures (Table 1). Ethyl esters constitute a homologous series of volatile compounds; log *P* (hydrophobicity) increases with the number of carbon atoms.

Estimation of Hydrophobicity. The hydrophobicity (log *P*) of aroma compounds is often defined as the logarithm of the partition coefficient of solute between *n*-octanol and water (*P*_{o/w}). This constant was estimated by the chemical software Windows Molecular Pro. The estimations were made by molecular fragmentation in atoms (14).

Determination of Solubility. Aroma compound (0.5 mL) was solubilized alone in 1 mL of water or model wine solution with ethanol 12.6% (v/v) in glass vials fitted with PTFE-lined silicone septa in plastic screw caps. These solutions were equilibrated for at least 24 h under regular agitation at 25 °C and centrifuged at 5000g for 20 min at the same temperature. Then, 2 μ L of the aqueous phase was sampled with a syringe and injected into a Chrompack model CP 9000 gas chromatograph (Middleburg, The Netherlands). This apparatus was equipped with a flame ionization detector, a direct injector, and a stainless steel column (3 m \times 2 mm) packed with Chromosorb W-AW 100–200 mesh Carbowax 20 M-10%. The gas chromatograph was operated under isothermal conditions at temperatures of 60 °C for isoamyl acetate, 100 °C for ethyl hexanoate, 150 °C for linalool, 160 °C for benzaldehyde, 180 °C for ethyl octanoate and ethyl decanoate, and 190 °C for β -ionone and 2-phenylethanol. The injector and detector temperatures were maintained, respectively, at 190 and 200 °C. The carrier gas was nitrogen with a flow rate 20 mL min⁻¹.

Seven replicates of each experiment were conducted, and the quantities of aroma compounds obtained were averaged.

Sorption Conditions. Experimental samples were prepared by immersing 1, 5, or 10 plates of wood into 37 mL glass flasks filled with the model solution containing ethanol 10% (v/v) and all aroma compounds (5–8 ppm) and tightly stoppered with Teflon caps to prevent losses of the volatile compounds. When one plate of wood was immersed, the ratio of wood surface area to volume of model solution was 140 cm² L⁻¹ compared to 118 cm² L⁻¹ for a 228-L Bourgogne wine barrel. Experimental samples and model solution without wood (control sample) were stored at 10 °C in a dark room without stirring. After different contact times (15, 30, 45, and 60 days), experimental samples (two for each time) and control sample were analyzed according to liquid–liquid extraction and gas chromatography (GC) methods. The amount sorbed was determined by the difference method.

Desorption Conditions. After an initial equilibration period of 30 days as described above, the solutions were analyzed and the plates of wood were directly transferred into 37 mL glass flasks filled with the model wine solution (ethanol 10%, v/v) without aroma compound. Samples were then stored at 10 °C in a dark room without stirring for 30 days before their analysis.

Aroma Compounds Analysis. Aliquots of 6 g of standard aroma compound solutions and sample solutions from sorption and desorption experiments were extracted two times with 1

mL of ethyl acetate/diethyl ether mixture (3:1 v/v) under magnetic stirring in a 15 mL flask. Before extraction, 30 μ g of 5-nonanol was added as internal standard. The extracts of aroma compounds were then dried over anhydrous sodium sulfate and analyzed by GC. The tests of recovery with this extraction procedure gave the following results: 98% isoamyl acetate, 92% ethyl hexanoate, 93% ethyl octanoate, 87% ethyl decanoate, 93% linalool, 96% 2-phenylethanol, 99% benzaldehyde, and 75% β -ionone.

A Chrompack CP 9001 gas chromatograph fitted with a split injector (1/10), a flame ionization detector, and an HP-Wax fused silica capillary column (30 m \times 0.25 mm i.d.; 0.25 μ m bonded phase, Hewlett-Packard) were used. The column temperature was programmed at 3 °C min⁻¹ from 40 to 160 °C and then increased at 8 °C min⁻¹ to 200 °C. Flow rates were 2 mL min⁻¹ of N₂ for the carrier gas and 30 mL min⁻¹ of H₂ and 300 mL min⁻¹ of air for the detector gases. The detector and injector temperatures were maintained at 200 °C. Data acquisition and treatment were carried out under Winilab II software (Perichrom Inc., version 2.06E).

GC-MS Analysis. Mass spectra were recorded by coupling a Hewlett-Packard (HP) 6890 gas chromatograph equipped with a DB-1701 fused capillary column (30 m \times 0.32 mm i.d., 1 μ m bonded phase) and an splitless injector (purge time of 18 s) to an HP 5973 mass spectrometer. The transfer line was heated at 230 °C and the injector at 250 °C. The column temperature was programmed at 4 °C min⁻¹ from 40 to 220 °C with helium as carrier gas at 1 mL min⁻¹. The source was heated at 230 °C, and mass spectra were scanned at 70 eV in the *m/z* range from 20 to 300 mass units.

RESULTS AND DISCUSSION

Physicochemical Characteristics of the Aroma Compounds. Solubilities of the selected aroma compounds in water and model wine solution with ethanol 12.6% (v/v) were determined by a GC method involving direct injection of aqueous or hydroalcoholic solutions. Activity coefficients reported in Table 1 were calculated as described previously by Voilley and Loncin (15) and Le Thanh et al. (16). According to these authors, the activity coefficient (γ^∞) of compounds sparingly soluble (under 10 g L⁻¹), approximately equal to the reverse of their solubility, is expressed as a molar fraction (*X*). Most of the experimental values of activity coefficients in water are in accordance with data from the literature, which were obtained by other methods. The activity coefficients of ethyl hexanoate in model wine and water are close to those previously obtained by headspace analysis (11). These results confirmed the relationship between the solubility and the activity coefficient for an aroma compound described by Le Thanh et al. (16). The solubility of aroma compounds in the model wine significantly increases by ~50% for isoamyl acetate, ethyl hexanoate, ethyl octanoate, linalool, benzaldehyde,

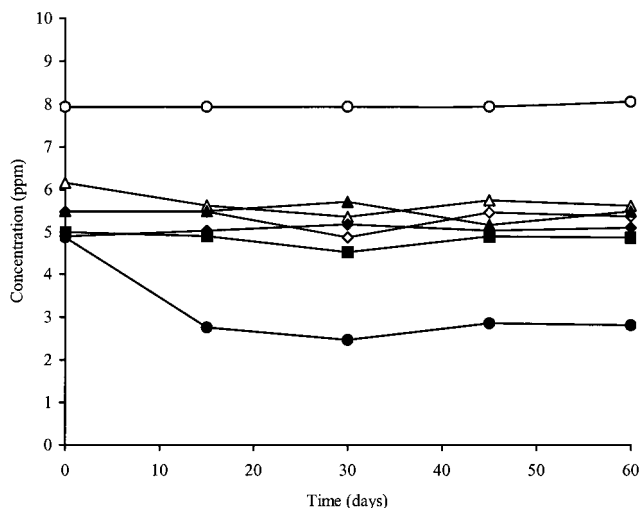


Figure 1. Evolution of concentration of isoamyl acetate (◇), ethyl hexanoate (■), ethyl octanoate (△), ethyl decanoate (▲), linalool (●), 2-phenylethanol (◆), and benzaldehyde (○) in model wine (ethanol 10% v/v) stored for 60 days at 10 °C.

and β -ionone in comparison with values obtained in water. The lowest values of solubility of ethyl esters in water is likely due to their amphiphilic character: they have a polar headgroup and hydrocarbon chain tail. The results support a previous study (11) in which the activity coefficients of volatile compounds in the model wine were half those obtained in water. For 2-phenylethanol and ethyl decanoate, the presence of ethanol does not significantly affect their solubility and therefore does not influence their activity coefficient.

Reactivity of Aroma Compounds in Model Wine.

Before sorption experiments were conducted, we needed more knowledge about the reactivity of aroma compounds in model wine in our experimental conditions. Figure 1 shows results obtained from the analysis of a control sample stored for 2 months at 10 °C. After 15 days, around 44% of the linalool and 9% of the ethyl octanoate were lost. Other volatile compounds were stable during the experimental time period. Values obtained for β -ionone were not reported because they were highly variable. GC-MS analysis of a control sample stored for 30 days at 10 °C showed numerous hydroxylated monoterpenols in addition linalool, namely, nerol, geraniol, α -terpineol, and 6,7-dihydroxylinalool, and suggested the presence of several C13 norisoprenoids. The hydroxylated monoterpenols cited above are chemical degradation products of linalool in acid media (17, 18). As that of monoterpenols, the reactivity of norisoprenoids in acid medium is well-known (19, 20); the detected megastigmane structures were likely formed by the chemical degradation of β -ionone.

Kinetics of Sorption into Oak Wood. Figure 2 shows the kinetics of sorption by the wood of aroma compounds in a mixture of wine model with ethanol 10% and a ratio of wood surface area to volume of model solution of 140 cm² L⁻¹, close to that of a 228-L Bourgogne wine barrel. The percent of sorption was calculated by mass balance for each compound and contact time in the presence and absence of wood. For most aroma compounds, sorption behavior into wood was observed, and >80% of the final sorption was reached within the first 15 days. Values obtained for β -ionone were highly variable, and no conclusion on its behavior could be established. For other volatiles, the amount sorbed by oak wood at equilibrium ranged

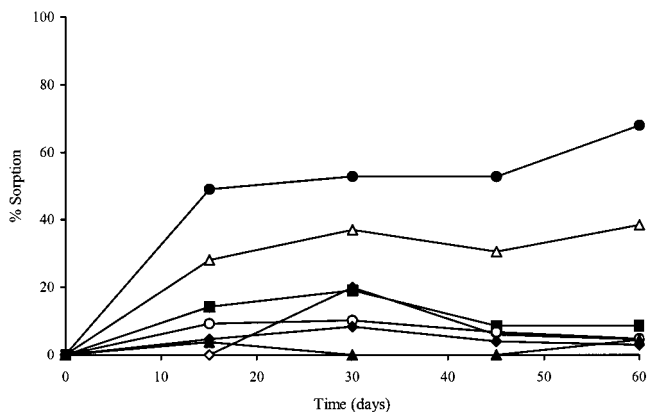


Figure 2. Sorption of isoamyl acetate (◇), ethyl hexanoate (■), ethyl octanoate (△), ethyl decanoate (▲), linalool (●), 2-phenylethanol (◆), and benzaldehyde (○) by wood (ratio = 140 cm² L⁻¹) in model wine (ethanol 10% v/v) stored for 60 days at 10 °C.

Table 2. Model Wine–Wood Partition Coefficients after 30 Days of Contact

compound	model wine– wood partition coeff (<i>K</i>)	compound	model wine– wood partition coeff (<i>K</i>)
isoamyl acetate	18.30	linalool	111.21
ethyl hexanoate	22.82	2-phenylethanol	13.77
ethyl octanoate	40.14	benzaldehyde	11.37
ethyl decanoate	<i>a</i>		

^a No significant differences were found by ANOVA test for concentrations with and without wood.

between 3 and 50%, depending on the nature of the volatile compound. Linalool and ethyl octanoate were sorbed to a greater extent than other volatiles studied, and for longer contact times (60 days) their sorption was slightly emphasized. For the homologous series of esters, no correlation was observed between the chain length of the lipophilic portion and sorption. Indeed, small sorptions were detected for ethyl hexanoate and ethyl decanoate at equilibrium, whereas a highly significant sorption was exhibited by ethyl octanoate. Consequently, log *P* did not appear to have any influence on the sorption level of aroma compounds into wood. In addition, the ethyl esters presented a lower sorption at equilibrium than linalool, although they are ~3–250 times less soluble in a model wine. Thus, observed differences in sorption followed neither differences of hydrophobicity nor those of solubility (Table 1). Little sorption of benzaldehyde and 2-phenylethanol was detected, which might be due to their high model wine solubility of >7 g L⁻¹ in comparison with that of other studied aroma compounds.

Sorption of aroma compounds of model solution in contact with sorbent is usually governed by the partition coefficient (*K*) between the solution and the sorbent, in this case, oak wood. *K* was obtained from

$$K = C_{\text{wood}} / C_{\text{solution}}$$

where C_{wood} and C_{solution} are the concentrations expressed in mg kg⁻¹ in the wood and the solution, respectively, at equilibrium. Partition coefficients at 30 days, when the steady state is reached, are listed in Table 2. For ethyl decanoate, *K* at 30 days was not calculated because no significant differences were found by ANOVA test for concentrations in solution for experimental ($p < 0.05$) and control samples, indicating

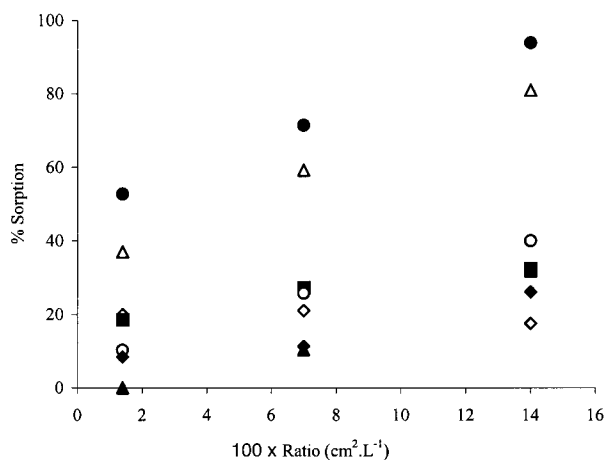


Figure 3. Sorption curves of isoamyl acetate (◇), ethyl hexanoate (■), ethyl octanoate (△), ethyl decanoate (▲), linalool (●), 2-phenylethanol (◆), and benzaldehyde (○) related to wood surface in a model wine (ethanol 10% v/v) at 10 °C during 30 days of equilibrium.

that this compound is not influenced by wood in our study conditions. The affinity of linalool for the tested wood is nearly 3 times more important than that of ethyl octanoate. However, it is quite possible that the determined value for linalool was overestimated because of its chemical degradation in experimental conditions. With a polyethylene film as sorbent, Lebossé et al. (21) obtained a value of the partition coefficient of linalool close to that found in our study. Isoamyl acetate and ethyl hexanoate were sorbed with a similar ratio, and the same trend was observed for benzaldehyde and 2-phenylethanol.

According to these results, it seems that hydrophobic interactions were not the dominating mechanism of sorption of aroma compounds by oak wood. The polar nature of the wood surface (θ) and that of the terpene alcohol showing a high sorption behavior may also lead to other specific interactions such as H-bonding.

Effect of the Ratio of Wood Surface Area to Solution Volume on Sorption. Sorption at an equilibrium time of 30 days was measured for all compounds in the mixture in the presence of wood with three different ratios of wood surface area to solution volume (Figure 3). Except for isoamyl acetate, a good linear correlation was found between the sorption of aroma compounds and the ratio of wood surface area to solution volume. Sorptions of linalool and ethyl octanoate were almost total with the higher ratio, which is ~ 10 times more than that of a 228-L Bourgogne wine barrel. Above $140 \text{ cm}^2 \text{ L}^{-1}$, the sorption level of isoamyl acetate was not more greatly influenced, although that of ethyl decanoate was significantly affected. The absence of change of behavior of isoamyl acetate could be due to a competition with other volatiles for the same sorption sites.

These last results suggested that the use of wood chips for which the ratio of wood surface area to solution volume is high, as an alternative to the aging of wine in oak barrels, might significantly influence sorption behavior of wine aroma compounds such as linalool and ethyl octanoate.

Release of Sorbed Compounds by Model Wine. Table 3 shows the concentrations of aroma compounds sorbed by wood before and after desorption by the model wine (ethanol 10% v/v). As anticipated, for all sorbed compounds the concentration in the wood decreased.

Table 3. Amount of Aroma Compounds Sorbed by Wood before and after Desorption by a Model Wine (Ethanol 10% v/v) after Storage of 30 Days at 10 °C

compound	initial sorbed quantity ^a (mg kg ⁻¹ of wood)	final sorbed quantity ^b (mg kg ⁻¹ of wood)	yield of desorption
isoamyl acetate	147	122	18
ethyl hexanoate	84	41	51
ethyl octanoate	153	79	49
linalool	166	101	39
2-phenylethanol	65	36	45
benzaldehyde	68	11	84

^a Quantity calculated by difference of mass after sorption of aroma compounds in mixture for 30 days. ^b Quantity calculated by difference of mass after desorption of aroma compounds by wine model (ethanol 10% v/v) for 30 days.

After a 30 day equilibrium period, the quantity of aroma compounds desorbed ranged from 18 to 84%. These results were analogous to those obtained for sorption except for isoamyl acetate and 2-phenylethanol, which seem to have in this case a partition more in favor of the wood. For these changes of sorption behavior, it can be postulated that equilibrium was not reached after 30 days as observed for sorption experiments and was consequently dependent on initial conditions (i.e., aromas in the solution or in the wood). Thus, the partition between wood and model wine may be affected by an excess of aroma. Linalool, which is reactive in acidic solution, presented a low yield of desorption.

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