American Journal of Essential Oils and Natural Products
Available online at www.essencejournal.com
AkiNik
AJEONP
American Journal of Essential Oils and Natural Products

ISSN: 2321 9114
AJEONP 2016; 4(2): 37-40

Received: 07-02-2016
Accepted: 08-03-2016

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Volatile components of oak and cherry wood chips used in aging of beer, wine, and sprits

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Abstract
The volatile oils from toasted American oak (*Quercus alba*), toasted French oak (*Quercus petraea*), and toasted and untoasted cherry (*Prunus avium*) wood chips have been obtained by hydrodistillation and analyzed by gas chromatography – mass spectrometry. The major volatile components in toasted oak wood were 2-furaldehyde, 5-methylfurfural, *cis*-whiskey lactone, and *trans*-whiskey lactone, with lesser amounts of guaiacol, *p*-creosol, eugenol, *cis*-isoeugenol, and vanillin. The major components in toasted cherry wood were benzaldehyde, benzyl alcohol, 2-furaldehyde, 5-methylfurfural, phenylacetaldehyde, methyl benzoate, massoia lactone, and guaiacol, along with the fatty acids palmitic acid and oleic acid. Untoasted cherry wood was rich in benzaldehyde, benzyl alcohol, and fatty acids.

Keywords: Cherry, oak, wood volatiles, chemical composition

1. Introduction
Wooden casks are often used to store and age beer, wine, and distilled spirits such as whiskey, brandy, rum and tequila. As an alternative to using wooden casks, wood chips can be added to the beverages, which are stored in stainless steel, glass, or other storage containers. The nature of the wooden cask or wood chips can have a profound effect on the taste and aroma of the resulting beverage. Oak (*Quercus* spp.) is, by far, the most popular wood for storage casks as well as wood chips for aging. Several alternative woods have been tried, however, including cherry (*Prunus avium*), false acacia (*Robinia pseudoacacia*), chestnut (*Castanea sativa*), mulberry (*Morus* spp.) [1], kapok (*Bombax* spp.), silk tree (*Albizia* spp.) [2, 3], amendoim (*Pterogyne nitens*), pereiro (*Aspidoespermapyrifolium*), and jatobá (*Hymenaea stigonocarpa*) [4]. Homebrewers and avid drinkers of craft beers and red wines generally have an interest in the volatile components that aging in wood can impart to beverages. In this work, the volatile components from toasted American oak (*Quercus alba*), toasted French oak (*Quercus petraea*), and toasted and untoasted cherry (*Prunus avium*) wood chips have been determined.

2. Materials and Methods
2.1 Wood Chips
Medium toasted American oak (*Quercus alba*) chips and medium toasted French oak (*Quercus petraea*) chips were obtained from MoreWine!™, Concord, California. Cherry (*Prunus avium*) wood chips were obtained from Amazon.com (Seattle, Washington). Cherry wood chips (untoasted) were toasted to a "medium toast" in the broiler of an oven. Each sample of wood chips was hydrodistilled using a Likens-Nickerson apparatus [5] for four hours with continuous extraction with dichloromethane to give yellow volatile oils (Table 1).

2.2 Gas Chromatography – Mass Spectrometry
The wood volatile oils were analyzed by GC-MS using an Agilent 6890 GC with Agilent 5973 mass selective detector [MSD, operated in the EI mode (electron energy = 70 eV), scan range = 45-400 amu, and scan rate = 3.99 scans/sec], and an Agilent ChemStation data system. The GC column was an HP-5ms fused silica capillary with a (5% phenyl)-polymethylsiloxane stationary phase, film thickness of 0.25 μm, a length of 30 m, and an internal diameter of 0.25 mm. The carrier gas was helium with a column head pressure of 48.7 k Pa and a flow rate of 1.0 mL/min. Inlet temperature was 200 °C and interface temperature was 280 °C. The GC oven temperature program was used as follows: 40 °C initial temperature, hold for 10 min; increased at 3 °C/min to 200 °C; increased 2°/min to 220 °C. A 1% w/v solution of each sample in dichloromethane was prepared and 1 μL was injected using a splitless injection technique.

Identification of the oil components was based on their retention indices determined by reference to a homologous series of *n*-alkanes, and by comparison of their mass spectral fragmentation patterns with those reported in the literature [6] and stored on the MS library [NIST database (G1036A, revision D.01.00)/ Chem Station data system (G1701CA, version C.00.01.080]. The percentages of each component are reported as raw percentages based on total ion current without standardization. The compositions of the wood volatile oils are summarized in Tables 2 and 3.

3. Results and Discussion

3.1 Oak

The major components found in the volatiles derived from toasted oak chips were 2-furaldehyde (65.6% and 84.0% in American oak and French oak, respectively), 5-methylfurfural (17.5% and 10.0%, respectively), and the two isomers of whiskey lactone (5-butyldihydro-4-methyl-2(3*H*)-furanone, oak lactone), *cis*-whiskey lactone (11.3% and 1.5%, respectively) and *trans*-whiskey lactone (2.4% and 1.0%, respectively). American oak had more whiskey lactones and yielded more volatile oil than the French oak. These results are qualitatively similar to those reported by Cadahía and co-workers [7], Díaz-Maroto and co-workers [8], and Fernández de Simón and co-workers [9]. Generally, toasted oak volatiles are dominated by 2-furaldehyde, 5-methylfurfural, and *cis*-whiskey lactone, with lesser amounts of *trans*-whiskey lactone, guaiacol, eugenol, *trans*-isoeugenol, and *p*-creosol. Cadahía *et al.* [7] and Fernández de Simón *et al.* [10] did report high concentrations of 5-hydroxymethylfurfural in both American and French oaks, which were not detected in this current work.

Table 1: Hydrodistillation of wood chips.

Wood chips	Mass of wood (g)	Yield of oil (g)	Percent Yield
American oak	201.39	3.2076	1.593%
French oak	182.31	2.1745	1.193%
Untoasted cherry	187.73	0.1122	0.060%
Toasted cherry	176.13	0.9658	0.548%

Assyrtiko wine (Santorini), treated with oak chips (origin of oak not specified), was found to extract furfural, vanillin, guaiacol, whiskey lactone, eugenol, and syringaldehyde, from the oak chips [11]. Chardonnay wine, macerated with toasted American oak chips, extracted 2-furaldehyde, *trans*-whiskey lactone, *cis*-whiskey lactone, eugenol, guaiacol, 4-vinylguaiacol, 4-methylguaiacol, syringol, and vanillin [12]. In a similar experiment, wine vinegars aged with American oak chips were found to extract 2-furaldehyde, 5-methylfurfural, *trans*-whiskey lactone, *cis*-whiskey lactone, eugenol, and vanillin [13].

Table 2: Volatile oil compositions of toasted oak wood chips.

RI	Compound	Percent Composition	
		American Oak	French Oak
800	Hexanal	0.2	0.3
813	3-Furaldehyde	---	0.2
836	2-Furaldehyde	65.6	84.0
855	2-Furanmethanol	0.1	tr
872	α-Angelica lactone	---	0.1
909	2-Acetylfuran	0.4	0.3
963	Benzaldehyde	tr	0.1
968	5-Methylfurfural	17.5	10.0
978	Methyl 2-furoate	0.2	0.1
1041	Salicylaldehyde	0.3	0.2
1063	1-(5-Methyl-2-furyl)-2-propanone	0.2	0.2
1088	Guaiacol	0.4	0.3
1111	2-Phenylethanol	---	0.2
1159	(2*E*)-Nonenal	tr	tr
1191	2-Methoxy-*p*-cresol (= *p*-Creosol)	0.5	0.6
1281	α-Terpinen-7-al	0.2	tr
1291	*trans*-Whiskey lactone	2.4	1.0
1321	*cis*-Whiskey lactone	11.3	1.5
1349	Syringol	tr	tr
1356	Eugenol	0.6	0.5
1366	Dihydroeugenol	tr	tr
1373	Methyl *p*-anisate	tr	---
1396	Vanillin	tr	0.2
1448	*cis*-Isoeugenol	tr	0.2
	Total Identified	99.9%	100%

Table 3: Volatile oil compositions of untoasted and toasted cherry wood chips.

RI	Compound	Percent Composition	
		Untoasted	Toasted
800	Hexanal	0.8	1.2
822	Methylpyrazine	---	1.1
833	2-Furaldehyde	0.4	15.0
868	1-Hexanol	0.2	---
963	Benzaldehyde	29.7	35.5
966	5-Methylfurfural	---	4.6
988	6-Methyl-5-hepten-2-one	tr	---
992	2-Pentylfuran	tr	---
1033	Benzyl alcohol	19.3	21.1
1043	Phenylacetaldehyde	0.4	2.6
1054	*o*-Cresol	0.3	tr
1057	(2*E*)-Octenal	0.2	tr
1070	1-Octanol	tr	---
1072	*cis*-Linalool oxide (furanoid)	0.3	tr
1074	*p*-Cresol	---	tr
1076	Benzyl formate	---	tr
1088	Guaiacol	0.6	1.1
1094	Methyl benzoate	tr	2.7
1104	Nonanal	0.5	tr
1111	2-Phenylethyl alcohol	0.6	tr
1159	(2*E*)-Nonenal	0.3	tr
1168	*cis*-Linalool oxide (pyranoid)	0.7	---
1175	Octanoic acid	0.5	tr
1205	Decanal	0.2	tr
1252	Chavicol	0.6	tr
1269	Nonanoic acid	0.9	tr
1314	(2*E*,4*E*)-Decadienal	0.3	tr
1355	Eugenol	0.4	tr
1367	Decanoic acid	0.3	tr
1477	Massoia lactone	2.3	2.3
1564	Dodecanoic acid	0.6	tr
1765	Tetradecanoic acid	0.7	---
1861	Pentadecanoic acid	0.2	---
1934	Hexadecenoic acid	0.7	---
1959	Palmitic acid	11.6	6.1
2131	Linoleic acid	20.7	4.6
2133	Oleic acid	---	2.2
	Total Identified	94.3%	100%

Pérez-Coello and co-workers [14] carried out fermentation of Airén grape must with both American (*Q. alba*) and French (*Q. petraea*) oak chips. These workers found that the resultant wine with American oak had higher concentrations of *cis*-whiskey lactone and 2-furaldehyde than that fermented with French oak chips. The French oak chips, on the other hand, imparted higher concentrations of *trans*-whiskey lactone. Concentrations of extracted guaiacol, eugenol, and vanillin were comparable for the two oaks. Similarly, Rodríguez-Bencomo and co-workers [15] macerated Spanish red wine ("Tinta del País") with both American oak (*Q. alba*) and French oak (mixture of *Q. petraea* and *Q. robur*) chips, and concentrations of 2-furaldehyde, *cis*-whiskey lactone, and vanillin, were extracted in higher concentrations from American oak, while *trans*-whiskey lactone was extracted in slightly higher concentrations from French oak. Guaiacol and eugenol concentrations were comparable.

Spanish red wine (Tempranillo, Rioja), aged in barrels of French oak (*Q. petraea*) and American oak (*Q. alba*) revealed higher concentrations of 5-methylfurfural, *cis*-whiskey lactone, guaiacol, and eugenol in the wine stored in American oak barrels, while red wine stored in French oak had higher concentrations of *trans*-whiskey lactone [10]. Very similar results were observed with Spanish red wine (63% Tempranillo, 17% Cabernet Sauvignon, 17% Merlot) aged in American oak (*Q. alba*) and French oak (*Q. sessilis*) [16]. 2-Furaldehyde, *cis*-whiskey lactone, guaiacol, and eugenol were found in higher concentrations in the wine stored in American oak, while *trans*-whiskey lactone was more concentrated in that stored in French oak.

3.1 Cherry

The major components in cherry wood were 2-furaldehyde (15.0% in toasted cherry wood, but only 0.4% in untoasted), benzaldehyde (29.7% and 35.5% in untoasted and toasted wood, respectively), benzyl alcohol (19.3% and 21.1% in untoasted and toasted, respectively), and several fatty acids in the untoasted wood (20.7% linoleic acid, 11.6% palmitic acid). Toasting cherry wood increased the yield of volatiles, increased furaldehydes, and decreased fatty acids. The cherry wood volatiles in this study are remarkably different from those reported by Fernández de Simón and co-workers [17, 18]. These investigators prepared hydroalcoholic extracts of the wood rather than using a hydrodistillation approach. Thus, the concentrations of 2-furaldehyde (1.0% in toasted cherry), benzaldehyde (0.16% and 0.34% in untoasted and toasted cherry wood, respectively), and benzyl alcohol (1.1% and 0.11%, respectively), were much lower. Palmitic acid was the only fatty acid reported, and it was detected in only low concentrations in untoasted cherry wood. Consistent with these results, Italian red wine (Raboso Piave) [1] or Spanish red wine (D. O. Somontano) [19], stored in cherry wood barrels did extract 2-furaldehyde, 5-methylfurfural, guaiacol, eugenol, benzaldehyde, benzoic acid, and benzyl alcohol.

4. Conclusions

The major volatile components in toasted oak wood were 2-furaldehyde, 5-methylfurfural, *cis*-whiskey lactone, and *trans*-whiskey lactone, with lesser amounts of guaiacol, *p*-creosol, eugenol, *cis*-isoeugenol, and vanillin. The major components in toasted cherry wood were benzaldehyde, benzyl alcohol, 2-furaldehyde, 5-methylfurfural, phenylacetaldehyde, methyl benzoate, massoia lactone, and guaiacol, along with the fatty acids palmitic acid and oleic acid. The volatile components from toasted woods used to store and/or age beverages impart certain important aromas (see Table 4).

Table 4: Olfactory descriptions of important wood volatiles [8, 20, 21].

Compound	Odorant description
2-Furaldehyde	slightly toasty, caramel
Benzaldehyde	almonds, sweet
5-Methylfurfural	spicy, toasty, sweet
2-Acetylfuran	toasty, toasted grain
Benzyl alcohol	floral, rose
Guaiacol	spicy, toasty, smoky/burnt
2-Phenylethanol	floral, rose
p-Creosol	spicy, lightly green, phenolic
trans-Whisky lactone	vanilla, oaky, clove, coconut
cis-Whiskey lactone	vanilla, oaky, clove, coconut
Eugenol	spicy, clove, cinnamon
Vanillin	sweet, vanilla
trans-Isoeugenol	spicy, clove, woody/oak

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