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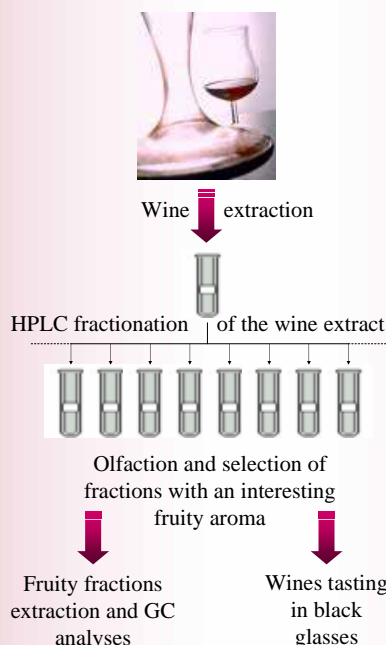
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Till now, more than 800 aromatic compounds have been identified in wines. Wine is so a very complex matrix, from which it is difficult to isolate, previously to analysis, a specific aromatic character. Some recent researches have suggested the use of High Pressure Liquid Chromatography (HPLC) preparative methods (1) as a help for the isolation of aromatic compounds. The main goal of our study was to determine how HPLC applied to wine extracts could be helpful for selection and characterization of red and black fruits aromas of Bordeaux red wines.

Preparation of wine extracts. 500 ml of wine was extracted using 100 ml, 50 ml, and 50 ml dichloromethane, with magnetic stirring (500 rpm) for 5 minutes. The three extracts were blended, dried over anhydrous sodium sulfate and concentrated to obtain a 1 ml wine extract.

HPLC assays. Normal Phase (NP) HPLC was performed with NH2 column, and Reversed Phase (RP) HPLC were performed with C8 and C18 columns. All columns were from Waters, 30 cm long and 3,9 mm i.d. Chromatographic conditions were optimized as follows: flow rate, 0,5 ml/min; injection volume: 256 µl of wine extract; program gradient: phase A, pentane (NP-HPLC) or water (RP-HPLC), phase B, dichloromethane (NP-HPLC) or ethanol (RP-HPLC) - minute 0-2, 100% A, linear programmed until 100% B in minute 50. The effluent was collected in 1 ml fractions. 25 fractions in hydroalcoholic solutions were so obtained and then directly evaluated by a panel of three trained judges, to only retain fruity fractions.

MATERIALS and METHODS



Fruity fractions extraction. Fraction 17 to 21, obtained with the C18 column, were blended and diluted with distilled water, before being extracted following the method used to prepare the wine extracts, but with 5 ml, 5 ml, and 5 ml of diethylether/pentane (1:1, v:v) and a 200 µl final volume of extract. The same method was applied to quantify esters in 50 ml of wine, with 20 µg of octan-3-ol, used as internal standard, added prior to extraction.

GC analyses. Chromatographic conditions were the same as used by Pineau *et al.* (3) GC-O analyses were carried out by a panel of 3 trained judges. Esters quantification were performed in GC-MS (SIM mode).

Tastings were performed in black glasses by a panel of 15 trained judges. A red wine was desaturated by evaporating using a Rotavapor®, until no more fruity aroma were perceptible. For each fraction, a triangular test was performed as presented below. Judges were asked to recognize the wine with fraction added and to describe its aroma.



MERLOT WINE FRACTIONATION RESULTS

This wine was characterized by very fruity aromas. With both C8 and C18 fractionations, red fruits aromas are globally concentrated into fractions 16 to 21, whereas NH2 column shows only 2 fractions with characteristic red fruits aroma, plus another one more chemical and sweet.

Even though separation is not as powerful as with C8 and C18 phases, the use of NH2 column could so be a way to improve results obtained with silica gel (2), by both controlled elution conditions (pressure, flow rate, etc.) and increased selectivity (more numerous fractions).

fraction	NP-HPLC – NH2 column	RP-HPLC – C8 column	RP-HPLC – C18 column
3	-	light fruity (pear, peach)	cream, milky
4	-	fatty	cream, milky
5	-	milky, butyric, light caramel	caramel, milky
6	-	intense caramel, milky	cheese, butyric
7	flowery	buttery, butyric	resinous, flowery
8	flowery, fatty	vegetables, cheese	solvent, green
9	flowery, cheese	solvent	alcohol, fresh
10	flowery, fatty	solvent	phenolic, fatty
11	flowery, light spicy	solvent	polish, banana
12	-	solvent, citrus fruits	solvent, flowery
13	-	green, cut grass	flowery, fresh strawberry
14	-	fatty, biscuit	fruity, roasted nut
15	light fresh red fruits	dusty, flowery	flowery, citrus
16	flowery, sweet, chemical	roasted, alcohol	mint, spicy, red fruits
17	raspberry, strawberry jams	roasted meat, citrus fruits	intense red fruits
18	roasted, burning	mint, spicy, red fruits	red fruits, citrus fruits
19	light, paper	red fruits, chemical, anise	raspberry, strawberry jams
20	light, meat	earl grey, fresh raspberry	light red fruits, burnt rubber
21	sweet	rubber, raspberry jam	roasted, burnt
22	-	alcohol, burnt	-(alcohol)
23	light biscuit, burnt	-(alcohol)	-(alcohol)

CHARACTERIZATION OF WINES FRUITY AROMAS

4 red fruity wines from each Merlot (M) and Cabernet Sauvignon (CS) varieties, were fractionated on C18-column.

Table shows a very good conservation of aroma characteristics between wines and fractions.

In triangular tests, more than 80 % of the judges succeed in recognizing the wine with fraction added and descriptors given were similar or close to the table ones.

Wine	Merlot	Cabernet Sauvignon
	caramel, fresh cherry	blackcurrant, black fruits liquor, spicy
F5	caramel	-
F7	caramel, fatty	fatty, cheese
F18	heavy, winy	black fruits, spicy
F19	chemical, strawberry	chemical, strawberry
F20	light black fruits	light red fruits
F21	fresh cherry, blackberry	intense blackcurrant
F22	light black fruits	fresh, spicy

GC analyses of fruity fractions extracts allowed identifying 12 ethyl esters and acetates, known for their red fruits aromas. From the results, blackberry and black-currant aromas, characteristics of wines M1, CS3 and CS4 might be linked to a higher content in ethyl propanoate, ethyl 2-methylpropanoate and ethyl 2-methylbutanoate. That result tends to be confirmed by sensorial analysis.

Wine	C2-2MeC3	C2C4	C2C6	C2C8	C2iC5	2MeC3C2	C4C2	C6C2	C8C2	3OHC4C2	C3C2	2MeC4C2
M1	44,9	-	0,7	-	240	45,6	171	225	220	679	75,6	5,0
M2	118,0	2,2	7,7	0,2	1463	15,1	411	707	699	911	11,8	2,5
M3	80,1	-	6,0	0,3	1121	14,4	270	452	419	626	17,1	2,4
M4	54,6	1,1	5,6	0,2	925	15,6	194	392	360	400	9,9	2,0
CS1	18,6	-	1,3	-	110	18,9	90	195	203	393	11,5	3,8
CS2	86,6	1,4	17,3	0,3	1758	15,5	190	422	402	596	14,5	2,3
CS3	57,8	1,2	8,7	0,1	845	24,0	166	321	255	350	34,2	3,8
CS4	58,8	-	11,5	0,2	1016	18,9	168	373	302	318	78,1	3,7

C2-2MeC3: 2-methylpropyl acetate – C2C4: butyl acetate – C2C6: hexyl acetate – C2C8: octyl acetate – C2iC5: isoamyl acetate – 2MeC3C2: ethyl 2-methylpropanoate – C4C2: ethyl butanoate – C6C2: ethyl hexanoate – C8C2: ethyl octanoate – 3OHC4C2: ethyl 3-hydroxybutanoate – C3C2: ethyl propanoate – 2MeC4C2: ethyl 2-methylbutanoate

The HPLC method pointed out allows a selective separation of aromatic compounds with a red fruits characteristic from the whole aromatic compounds initially presents in the wine extract. It is so a powerful way of purification, which makes easier the characterization of aromatic compounds responsible for the specific fruity aromas observed in Bordeaux red wines.

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