VOLATILE COMPONENTS OF GRAPE LEAVES

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SUMMARY: The volatile constituents in different grape leaves variaties (Gazaz, Banati, Baladi, Biz El-Khanza) have been investigated by gas liquid chromatography. On polar and non-polar phase. Extraction of aroma concentrate was done by steam distillation method. Clear quantitative differences exist between the amounts of hexenal, trans-2-hexenal and cis-3-hexenal of grape leaves of the four varieties.

Key words: Liquid chromatography.

INTRODUCTION

Grape leaves find wide acceptance in Egypt as boiled edible commodity cooked with meat and rice known as (Mahshi). It has an attractive taste and aroma. To the best of our knowledge, the literature appears to be devoid of references to work done on the aroma of boiled grape leaves.

Extensive studies have been carried out concerning the volatile constituents in grape brandies, wines and whiskies. Beter and co-workers (1) identified about 139 neutral volatile compounds by gas chromatography, they are mainly ethyl esters of C3, C5, C6, C7 and C14.

The present paper deals with the separation and identification of the volatile components of the aroma of boiled grape leaves. The material used is divided into two parts and is subjected to the same treatment for collection of the aroma concentrate and for analysis by gas liquid chromatography. Uses of two different phases, polar and non-polar was achieved to obtain accurate results.

MATERIALS AND METHODS

The leaves of Vitis vinifera cv. four local varieties namely; Baladi, Banati, Gazaz and Biz El-Khanza, were obtained from a farm in Giza. The authentic reference compounds and solvent were extra pure or analytical grade.

Lipid extraction

Fresh grape leaves were soaked in diethyl ether for 24 hours then filtered. The extracte was transferred into a 50ml flask and solvent was evaporated in vacuo.

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Preparation of the aroma concentrate

Clean grape leaves (0.5 kg) were boiled by steam distillation method for about one hour under reduced pressure (about 30 mm Hg) yielding 0.00234 kg of aroma concentrate (2).

Gas liquid chromatographic analysis

Gas liquid chromatographic analysis was conducted using Varian 3700 equipped with dual flame ionization detectors. The analysis was carried out using polar and non-polar phase to achieve good separation. The column was packed with 20% diethylene glycol succinate (DEGS) on chromosorb W (60-80 mesh), carrier gas flow rate 30 ml/min (N2), column temperature 100 - 195°C with programming rate 5°C/min. Injection port temperature was 200°C. The chart speed was set at 1 cm/min. The other column was packed with 10% OV - 101 on chromsorb G11 (80-100 mesh), column temperature was 100-295°C with programming rate 9°C/min.

Identification of the components was done tentatively using authentic reference compounds under the two different phases. Also GC-MS was used for elucidation of the structure of the volatiles.

RESULTS AND DISCUSSION

Fatty acid composition

The gas chromatograms of the fatty acid composition of grape leaves of four varieties are shown in Table 1. The components together with their concentrations as obtained from gas liquid chromatographic analysis are illustrated in the table.

Over 50 volatile components were isolated by gas liquid chromatographic analysis, in the aroma of boiled

Table 1: Fatty acid composition of grape leaves.

Carbon No	Concentration %			
	Baladi	Gazaz	Biz El-Khanza	Banati
C14	19.20	19.00	9.00	10.00
iso C16	4.01	4.50	4.54	5.50
C16	46.00	37.00	38.50	49.34
C16:1	7.21	6.60	7.81	8.02
C16:2	0.41	0.23	0.45	0.46
C18:0	3.60	5.04	5.85	6.49
C18:1	10.00	19.50	20.51	14.57
C18:2	4.00	2.12	5.34	2.22
C18:3	6.45	5.91	8.00	8.90

grape leaves. About 27 compounds were identified. The compounds together with their concentrations are given in Table 2.

Considering the aroma substances of the different chemical classes in relation to their concentrations in the various varieties of grape leaves, the following conclusion may be drawn.

Aldehydes are forming the major part of flavor. There is a clear difference in the quantitative distribution of aldehydes between the various varieties. Trans-2-hexenal was found in high quantity (30.24%) in Gazaz compared to 1.84% in Baladi, 5.04% in Biz El-Khanza and 3.32% in Banati. While hexenal was found in minimum quantity, 0.08, trace, 1.04 and 1.00 for Gazaz, Baladi, Biz El-Khanza and Banati respectively. Hexenal and trans-3-hexenal are important flavor component of fruits, vegetables and green leaf products, along with the corresponding alcohols. Cis-2-hexenal was found in maximum amount (52.8%) in Baladi and (54.23%) in Banati. Precursors of the saturated and the unsaturated C6 compounds are linoleic acid (LA) and linolenic acid (LNA) respectively. Lipoxygenase (LPO) and hydroperoxide lyase (HPO lyase) are responsible for C6 aldehyde formation from C18 fatty acids (3). It was found that the ratio of trans-2-hexenal to hexenal is approximately 30 in Gazaz variety, while the ratio of cis-2-hexenal to hexenal is approximately 53 in Baladi and 54 in Banati varietes. Drawert *et al.*, (4) found that the ratio of trans-2-hexenal to hexenal is 30 in grape leaves.

In Table 3, it is obvious that the ratio of C18:3 to C18:2 is approximately 1.6, 2.8, 1.5 and 4.0 for Baladi, Gazaz, Biz El-Khanza and Banati respectively. This result assured the obtained data mentioned above. Similar findings were obtained by Saijyo and Takeo (5) who calculated the ratio of LNA and LA in tea leaves and found it to be 3.

Table 2: The volatile components of different varieties of grape leaves.

Peak No	Rt	Concentration %				
		Gazaz	Baladi	Biz El-Khanza	Banati	Compounds
1	0.93	2.45	0.84	0.34	-	Ethanol
2	1.21	0.31	0.17	1.07	0.11	2-Methyl butanal
3	1.39	0.78	3.53	2.30	4.80	3-Methyl butanal
4	1.65	Trace	1.92	4.00	9.75	1-Propanal
5	1.80	4.77	Trace	14.0	0.26	2-Methyl propanal
6	2.20	0.94	1.91	1.30	0.13	2-Methyl pentanal
7	2.71	-	-	0.13	0.28	
8	3.27	0.37	2.00	2.44	0.76	1-Pentanal
9	3.42	1.24	0.09	1.25	1.90	1-Butanol
10	3.65	-	-	-	0.52	
11	3.95	1.17	-	0.24	1.88	
12	4.23	-	-	0.58	-	
13	4.73	0.12	2.10	2.11	2.97	1-Pentanol
14	4.90	0.89	-	-	-	
15	5.16	0.26	-	-	0.56	
16	5.37	-	-	0.55	-	
17	5.80	-	0.21	0.30	0.63	
18	6.09	-	-	-	0.52	
19	6.44	4.86	0.16	0.74	0.62	2-Pentenal
20	6.74	2.66	-	2.24	0.45	
21	7.42	0.03	-	0.35	-	2-Hexanone
22	7.81	0.11	0.16	1.25	0.62	2-Pentenol
23	7.97	1.03	-	0.42	-	
24	8.21	0.29	-	0.07	0.04	
25	8.69	0.08	Trace	1.04	1.00	
26	9.49	1.09	0.18	0.33	0.63	
27	10.41	7.26	-	1.47	0.81	
28	10.98	30.24	1.84	5.04	3.32	Trans-2-hexenal
29	11.29	-	-	-	0.26	
30	11.70	-	-	-	0.61	
31	11.81	0.79	-	0.83	0.56	2-Heptanone
32	12.36	2.15	2.44	4.00	4.60	1-Hexenol
33	13.24	0.40	-	6.19	2.00	
34	13.40	-	-	-	0.70	
35	15.73	3.69	6.04	1.36	1.07	2-Hexenol
36	14.71	-	-	0.03	1.00	
37	15.00	0.17	-	0.11	1.17	Heptanal
38	15.22	-	-	0.05	-	
39	15.54	0.11	-	0.15	-	
40	15.77	-	-	0.08	-	
41	16.25	0.07	-	0.11	-	
42	16.95	0.23	52.81	0.23	54.23	Cis-3-hexenal
43	17.61	0.93	-	0.78	-	Octanal
44	18.17	-	5.74	-	-	
45	18.42	0.45	1.68	1.05	0.14	2-Heptenol
46	19.75	-	-	0.26	0.11	
47	20.35	0.04	-	0.23	0.06	1-Octanol
48	21.25	Trace	1.65	0.05	0.07	1-Nonanal
49	24.13	-	0.65	-	0.86	
50	25.08	-	0.58	0.10	-	
51	26.24	29.84	5.96	43.10	-	Trans-2-nonenal
52	32.81	-	1.85	0.23	-	
53	34.02	-	0.92	-	-	
54	36.23	-	2.27	-	-	
55	37.61	-	1.03	-	-	
56	39.01	-	1.27	-	-	

A similar relation of saturated C9 and the unsaturated C9 (trans-2-nonenal) was observed in Gazaz and Biz El-Khanza. The ratio of trans-2-nonenal to nonenal was about 30 in Gazaz and 43 in Biz El-Khanza.

Among the alcohols arising from the grape leaves aroma, 1-butanol, 1-pentanol, 2-pentanol, 1-hexenol, 2-hexenol and 2-heptenol were detected. Concerning the amounts of the last mentioned compound differences between the various grape varieties could be found.

Only two ketones were identified, namely, 2-hexanone and 2-heptanone. Concerning the amount of both ketones, differences between the various varieties were observed.

The so far determined amount of volatile aroma substances lead to the conclusion that the quantitative differences between the individual varieties of grape leaves occur especially among hexenal trans-2-hexenal and cis-3-hexenal which can probably influence the aroma of grape leaves.

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