Analysis of Aromatic Volatile Compounds in ‘Miyabi’ Melon (*Cucumis melo* L.) using the Porapak Q Column

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Summary

To evaluate the effectiveness of the Porapak Q column concentration method (PQM) of analyzing the volatile aromatic compounds in ‘Miyabi’ melon, we compared it with the direct headspace sampling method (DHSM) and simultaneous distillation extraction method (SDEM) in regard to quantitative value, recovery rate, and reproducibility of the compounds. A total of 103 volatile compounds were quantified by PQM, 66 by SDEM, and 8 by DHSM, respectively. The widest range of volatile compounds, ranging from low-boiling-point to high-boiling-point, were quantified by using the PQM rather than by the other two methods. A total of 76 compounds were identified by the GC-MS analysis and the Kovats Index. Among the alcohols, 2-methyl-1-propanol, 2-methyl-1-butanol, 1-hexanol, 1-nonanol, (Z)-3-nonen-1-ol, (Z)-6-nonen-1-ol, (Z,Z)-3,6-nonadien-1-ol, benzyl alcohol, and 2-phenethyl alcohol also occurred in relatively large quantities. Among the aldehydes, 3-methylbutanal, nonanal, and (E,Z)-2,6-nonadienal were identified in large quantities by PQM. Among the esters, propyl acetate, 2-methylpropyl acetate, butyl acetate, 2-methylbutyl acetate, and benzyl acetate were detectable in relatively large quantities by PQM and SDEM. The recovery rates of most low boiling-point compounds, such as methyl butyrate and ethyl propionate, were same in both methods, whereas the recovery rates of medium and high boiling-point compounds were higher with the PQM. Among sulfur-containing compounds, ethyl (methylthio)acetate and 2-(methylthio)ethyl acetate were identified by the PQM and SDEM, while ethyl 3-(methylthio)propionate and 3-(methylthio)propyl acetate were only detected by PQM. The average coefficient of variation of the compounds was 11.90% using the PQM, with stable reproducibility compared with that of SDEM (37.13%). With respect to flavor, the odor concentrates from the PQM were similar to the natural aroma of muskmelon, but the odor concentrates from the SDEM were degraded to a thick, off-flavor odor. Therefore, we concluded that PQM is an excellent method to extract volatile aromatic compounds of melon because it is quick, easy to operate, and capable of recovering a wide range of boiling-point volatile compounds without degrading their flavor. Furthermore, the results are highly reproducible.

Key Words: headspace, melon, Porapak Q column, simultaneous distillation extraction (SDE), volatile analysis.

Introduction

The price of melon, a popular fruit in Japan, often fluctuates greatly, depending on its variety and quality. Numerous studies have focused on the qualities of melon especially shape, sugars, and flesh (Hayata et al., 2000), but there have been very few studies on its aroma, which is an important aspect of quality. The quantitative and qualitative differences in volatile compounds among varieties have not been examined although few studies have been devoted to the influences

that volatile compounds impart to aroma (Iwabuchi and Ohsaki, 1990; Wyllie and Leach, 1992). This is partly due to the difficulty in analyzing volatile aromatic compounds, compared with other facets of quality. The direct headspace sampling method (DHSM) (Yabumoto and Jennings, 1977; Hashinaga et al., 1984) and simultaneous distillation extraction method (SDEM) (Buttery et al., 1982; Horvat and Senter, 1987) have been used to extract volatile compounds from melon. Although the components and concentration of gas samples can be analyzed by the former method, the recovery volume is small and the compounds extracted are limited to low-boiling-point ones (Sugisawa, 1983). The latter method, a modified Likens-Nickerson apparatus developed by Schultz et al. (1977), is widely used to extract volatile aromatic compounds, although it degrades the flavor

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because of the heating process (Shimoda et al., 1995).

Porapak Q is a substance used to pack the column in GC which was adapted recently for PQM to concentrate volatile compounds (Shimoda et al., 1987). The PQM is an easy, speedy method that is capable of trapping, extracting and concentrating volatile compounds efficiently in a solvent, under room temperature. Although the PQM has been used for analyzing volatile compounds in green tea (Shimoda et al., 1995) and *sake* (Sakamoto et al., 1993), no study has used the PQM for analysis of volatile compounds in vegetables or fruits except for *Perilla frutescens* (Sakamoto et al., 1995).

In this study, we performed a comparative investigation on the number of identifiable peaks, the recovery rate, and the reproducibility of volatile compounds using the PQM, DHSM, and SDEM, and evaluated the effectiveness of the PQM for the analysis of volatile aromatic compounds in muskmelon.

Materials and Methods

Plant Materials

Fresh, mature 'Miyabi' muskmelons (Yokohama Ueki Corp Co., Ltd., Yokohama, Japan) were obtained from JA Kochi-Haruno, Kochi Prefecture. Where the plants were cultured in a plastic-film greenhouse at Haruno field from 27 August through 31 November 2000. Melons were harvested 55 days after anthesis and held at 25 °C for one week to ripen. The firmness and Brix of the mesocarp were measured by a rheo meter (Fudoh kogyo Co., Tokyo) and a hand refractometer (N 1; Atago Co., Tokyo) averaged $0.64 \text{ kg} \cdot \text{cm}^{-2}$ and 14.53, respectively. The mesocarp (100 g) from each of three fruits was cut vertically along the polar diameter, mixed and then homogenized using a blender (Sanyo SM-KW 50, Sanyo Electronic Co., Ltd., Osaka, Japan). The mixture was centrifuged at $10,000 \times g$ for 20 min and the supernatant subjected to each extraction method.

Porapak Q Column Concentration Method (PQM)

The sample was passed through a glass column ($2 \times 10 \text{ cm}$) packed with Porapak Q (polydivinylbenzene, 50–80 mesh, Waters Co., Ltd., Milford, MA, USA). The column was washed with 100 ml of deionized water, followed by 100 ml (1 time) or 60 ml (3 times) of diethyl ether to elute the adsorbed compounds according to Shimoda et al. (1987). After adding the internal standard of 10 μl 0.1 % cyclohexanol, the elute was dried over anhydrous sodium sulfate for six hours. The solvent was evaporated to about 50 μl for GC analysis.

Simultaneous Distillation Extraction Method (SDEM)

The supernatant derived from 300 g of mesocarp as above which was diluted with an equal volume of deionized water was applied to the SDEM apparatus according to Shimoda et al. (1987). Diethyl ether (100 ml) was added to the sample to distil the volatile

compounds at 75 mm Hg and 65 °C for 60 min. The distillate was evaporated, and the residue was analyzed as in the PQM for GC.

Direct Headspace Sampling Method (DHSM)

A sample (300 ml) of melon fruit was placed in a triangle-bottom flask and tightly sealed. After holding at 25 °C for 2 hr, a 2-ml sample was withdrawn from the headspace and injected into the GC.

Gas Chromatography (GC)

A Shimadzu GC-17A (Shimadzu Co., Ltd., Kyoto, Japan), equipped with a 60 m $\times$ 0.25 mm i.d. DB-Wax, a column film thickness of 0.25 μm (J&W Scientific Inc., Folsom, USA), and a flame ionization detector (FID) was used to identify the volatile compounds. The injector and detector were kept 230 °C and 250 °C, respectively. The column was maintained at 40 °C for 10 min, then raised to 220 °C at a rate of $3 \text{ }^{\circ}\text{C} \cdot \text{min}^{-1}$, and maintained at 220 °C for 10 min. The flow rate of the helium carrier gas was $30 \text{ cm} \cdot \text{sec}^{-1}$ with a split ratio of 1:20. For quantitative analysis, the amount of volatile compound was calculated based on the ratio between the peak area of the volatile and that of the internal standard.

Gas Chromatography-Mass Spectrometry (GC-MS)

A Shimadzu GC-MS-QP5050 (Shimadzu Co., Ltd., Kyoto, Japan) was used to identify the volatile compounds. The injector and interface were held at 230 °C; the column and oven conditions were the same as those for GC analysis. The volatile compounds resulting from the GC-MS were identified by comparing them with those in the NIST mass spectra database using a computer; the identities were confirmed with the Kovats Index of standard compounds.

Results and Discussions

Chromatograms of the volatile extracts in 'Miyabi' melon fruit obtained by the PQM, SDEM, and DHSM (Fig. 1) reveal that a total of 103 volatile compounds were isolated by PQM, 66 by SDEM, and 8 by the DHSM. Previously, Iwabuchi and Ohsaki (1990) identified about 100 volatile compounds in 'Yubari melon' (11 kg) using the SDEM, and Hashinaga et al. (1984) identified 22 in 'Prince melon' using the DHSM. The PQM recovered high-boiling-point compounds that were not recovered by the SDEM because Porapak Q, consists of porous polymer beads that efficiently trap micropolar and nonpolar compounds independent of their boiling points, whereas they are not readily distilled with diethyl ether. The total numbers of volatile compounds identified by both the SDEM and DHSM in this study were fewer than those found by Iwabuchi and Ohsaki (1990) and Hashinaga et al. (1984). Although the difference could be attributed to the variations in volatile compounds among cultivars, it might have been the sample sizes as our SDEM size that was almost 1/37

Fig. 1. Gas chromatograms of volatile compounds in 'Miyabi' melon obtained by direct headspace method (A), simultaneous distillation extraction method (B) and Porapak Q column concentration method (C). Identified peaks are listed in Table 1. I.S.: internal standard (0.1% cyclohexanol). BHT: dibutylated hydroxytoluene.

smaller than that of Iwabuchi and Ohsaki (1990). In the DHSM, the fewer volatile compounds might have resulted because of the shorter incubation time and lower temperature. Considering that the total number of volatile compounds identified by the DHSM was lower than that of the other two methods, we will discuss the results

from PQM and SDEM.

Only 76 volatile compounds were identified (Table 1) because the high-boiling-point ones after peak no. 84 were impossible to identify; there are no reported values to compare them, based on the conventional methods of extraction. Almost the same number of volatile com-

Table 1. Identification and comparative ; relative level of volatile compounds separated from melon fruit (*Cucumis melo* L. cv. Miyabi) using Porapak Q column concentration method (PQM) and simultaneous distillation extraction method (SDEM).

Peak ^z No.	KI ^y	Compound	Peak area ratio ^x	
			PQM	SDEM
1	901	Isopropyl acetate	0.232	0.171
2	906	Methyl propionate	0.360	0.273
3	910	2-Methylbutanal	0.039	0.039
4	913	3-Methylbutanal	0.039	0.026
5	919	Methyl 2-methylpropionate	0.112	0.097
6	923	2-Propanol	0.080	nd ^v
7	934	Ethanol	0.900	11.610
8	945	Unknown	0.036	0.283
9	952	Ethyl propionate	0.537	0.489
10	961	Ethyl 2-methylpropionate	0.076	0.068
11	971	Propyl acetate	0.942	0.720
12	983	Methyl butyrate	0.318	0.261
13	1010	Methyl 2-methylbutyrate	0.939	0.886
14	1014	2-Methylpropyl acetate	3.480	3.338
15	1019	Methyl 3-methylbutyrate	0.018	0.015
16	1036	Ethyl butyrate	0.250	0.231
17	1043	Propyl propionate	0.048	0.068
18	1052	Ethyl 2-methylbutyrate	0.199	0.177
19	1072	Butyl acetate	1.647	1.500
20	1079	2-Methylpropyl propionate	0.025	0.035
21	1095	2-Methyl-1-propanol	1.257	0.769
22	1121	2-Methylbutyl acetate	2.661	2.925
23	1148	1-Butanol	0.405	0.180
24	1172	Pentyl acetate	0.023	0.021
25	1175	Unknown	0.031	nd
26	1177	Unknown	0.035	0.930
27	1192	Methyl hexanoate	0.039	0.025
28	1209	2-Methyl-1-butanol	8.550	3.937
29	1223	Unknown	0.039	nd
30	1228	2-Pentylfuran	0.025	nd
31	1233	Ethyl hexanoate	0.014	0.008
32	1245	2-Hexanol	0.023	nd
33	1249	3-Methyl-2-butenyl acetate	0.026	nd
34	1253	1-Pentanol	0.081	0.037
35	1271	Hexyl acetate	0.438	0.345
36	1281	Cyclohexanone	0.468	0.021
37	1285	4-Heptanol	0.069	0.016
38	1295	Unknown	0.029	nd
39	1306	(E)-3-Hexen-1-yl acetate	0.028	0.013
40	1314	(Z)-3-Hexen-1-yl acetate	0.193	0.150
41	1321	(E)-2-Hexen-1-yl acetate	0.045	0.016
42	1354	1-Hexanol	0.915	0.403
43	1364	Heptyl acetate	0.022	0.014
44	1378	2-Acetoxy-3-butanone	0.122	0.073
45	1384	(Z)-3-Hexen-1-ol	0.315	nd
46	1389	Nonanal	0.042	0.029
47	1409	(E)-3-Hepten-1-yl acetate (T) ^w	0.057	0.005
48	1447	Ethyl (methylthio) acetate	0.081	0.042
49	1455	1-Heptanol	0.183	0.108
50	1484	Butene-2,3-diol diacetate (T)	2.523	0.207

Table 1.

(Continued)

Peak No.	KI	Compound	Peak area ratio	
			PQM	SDEM
51	1490	2-(Methylthio) ethyl acetate	0.071	0.008
52	1501	Butane-2,3-diol diacetate 2(T)	0.156	0.023
53	1509	Benzaldehyde	0.216	nd
54	1520	2-Methylthioethanol	1.257	0.098
55	1525	Furfuryl acetate	0.036	nd
56	1541	1-Octanol	0.184	nd
57	1560	Butane-2,3-diol monoacetate 1(T)	0.687	nd
58	1570	Ethyl 3-(methylthio) propionate	0.149	nd
59	1572	Butane-2,3-diol monoacetate 2(T)	2.094	nd
60	1582	(E,Z)-2,6-Nonadienal	0.318	0.130
61	1594	Unknown	0.124	nd
62	1613	Unknown	0.255	nd
63	1619	(Z)-6-Nonen-1-yl acetate	0.232	0.066
64	1627	3-(Methylthio) propyl acetate	0.429	nd
65	1639	p-Mentha-(cis)-2,8-dien-1-ol	0.150	nd
66	1644	Unknown	0.214	0.014
67	1650	Unknown	0.315	nd
68	1658	1-Nonanol	0.834	0.534
69	1676	3,6-Nonadienyl acetate	0.190	nd
70	1682	(Z)-3-Nonen-1-ol	0.537	0.256
71	1714	(Z)-6-Nonen-1-ol	4.860	1.395
72	1721	3-Methylthiopropanol	0.384	nd
73	1726	Benzyl acetate	0.999	0.243
74	1737	Ethyl undecanoate	0.402	nd
75	1748	(Z,Z)-3,6-Nonadien-1-ol	3.001	nd
76	1752	Unknown	0.795	0.513
77	1764	Unknown	0.109	nd
78	1774	Butane-1,4-diol	0.078	nd
79	1813	2-Phenethyl acetate	0.226	0.046
80	1830	Ethyl dodecanoate	0.144	nd
81	1851	Unknown	0.149	0.032
82	1874	Benzyl alcohol	14.460	0.061
83	1910	2-Phenethyl alcohol	3.330	nd
84	1924	Unknown	0.222	0.037
85	1971	3-Phenylpropyl acetate	1.707	nd
86	1978	Unknown	0.205	nd
87	1986	Unknown	0.951	0.070
88	1996	Unknown	0.372	nd
89	2045	3-Phenylpropyl alcohol	0.257	nd
90	2050	Unknown	0.408	nd
91	2090	5-Ethyl-4-hydroxy-2-methyl-3(2H)-furanone	2.367	0.402
92	2117	Unknown	22.083	nd
93	2169	Unknown	0.342	nd
94	2234	Unknown	1.328	nd
95	2245	Unknown	1.281	nd
96	2280	Unknown	0.291	nd
97	2289	Cinnamyl alcohol	0.381	nd
98	2316	Unknown	0.279	0.075
99	2345	Unknown	0.127	nd
100	2382	Dihydroactinidiolide	1.707	0.043
101	2447	Unknown	0.292	nd
102	2460	Unknown	0.733	nd
103	2495	Unknown	0.477	0.236

^z The peak numbers correspond to the numbers in Fig. 1.^y Kovats index on DB-WAX.^x Values are GC peak area of compound / GC peak area of internal standard.^w Tentatively identified.^v Not determined.

pounds up to peak no. 28, a medium-boiling-point compound was recorded by SDEM as with PQM, but thereafter, the SDEM could hardly detect the high-boiling-point volatile compounds. High-boiling-point volatile compounds are considered to be associated not only with aroma but also with flavor (Osajima, 1992). Therefore, PQM seems to be a very useful method for investigating the roles of volatile compounds in melon fruit flavor.

Among alcohols, PQM and SDEM identified 2-methyl-1-propanol (no. 21), 2-methyl-1-butanol (no. 28), 1-hexanol (no. 42), 1-nonanol (no. 68), (Z)-3-nonen-1-ol (no. 70), and (Z)-6-nonen-1-ol (no. 71) in large quantities. The peak area of ethanol (no. 7) was 0.900 by the PQM, whereas it was 11.610 by the SDEM. Other alcohols, such as 1-butanol (no. 23), 1-hexanol (no. 42) and 1-heptanol (no. 49) were detected by PQM than by SDEM. (Z)-3-nonen-1-ol (no. 70) and (Z)-6-nonen-1-ol (no. 71), which are speculated to be the principal aroma volatile compounds in melon fruit (Kemp et al., 1972, 1974; Akahoshi, 1983), were, likewise, 2 to 3.5 times higher in the PQM; furthermore, benzyl alcohol (no. 82) was about 240 times more in PQM. 2-Phenethyl alcohol (no. 83), which is a volatile compound in *sake*, was also detected in large quantities by PQM. The reason for the reduction of ethanol in the PQM as compared with the SDEM is attributed to the low affinity of Porapak Q to relatively high polar compounds, such as ethanol, and high affinity to hydrophobic compounds. The concentration of benzyl alcohol (no. 82) was very high (14.460) in the PQM, but only a trace was recorded by SDEM. These results are similar to those obtained by green tea analyzed by PQM and SDEM by Shimoda et al. (1995).

Among aldehydes, 2-methylbutanal (no. 3), 3-methylbutanal (no. 4), nonanal (no. 46), and (E, Z)-2,6-nonadienal (no. 60) were identified by PQM and SDEM. (E, Z)-2,6 nonadienal (no. 60), a principal volatile compound of cucumber (Schieberle et al., 1990), was found to be about 2.5 times more concentrated with PQM than with SDEM. Among esters, propyl acetate (no. 11), 2-methylpropyl acetate (no. 14), butyl acetate (no. 19), and benzyl acetate (no. 73) were identified in great quantities by both methods. Most low-boiling-point volatile compounds, such as ethyl propionate (no. 9) and methyl butyrate (no. 12), were identified in similar quantities by both methods, although a medium-boiling-point compound, was detected in greater quantity by the PQM. 2-Methylbutyl acetate (no. 22), however, was found in greater quantity by the SDEM than by PQM. Among sulfur-containing compounds, ethyl (methylthio)acetate (no. 48) and 2-(methylthio)ethyl acetate (no. 51) were identified by both methods, although ethyl 3-(methylthio)propionate (no. 58) and 3-(methylthio)propyl acetate (no. 64) were identified only by the PQM.

It has been reported that the principal volatile aromatic compounds of melon fruit are C-9 alcohol and

such aldehydes as (Z)-3-nonen-1-ol (no. 70), (Z)-6-nonen-1-ol (no. 71), 1-nonanol (no. 68), and nonanal (no. 46) (Kemp et al., 1971, 1974; Yabumoto and Jennings, 1977); these compounds were also identified at high concentration levels in this study. Methyl 2-methylpropionate (no. 5) and 2-methylbutyl acetate (no. 22), which were responsible for the characteristic melon-like aroma according to Grosch et al. (1993), were identified and speculated to influence the aroma of the 'Miyabi' melon. Ethyl 2-methylbutyrate (no. 18), which is the key volatile aromatic compound of the 'Delicious' apple, was also identified (Flath et al., 1967). Thus, it is clear that the principal volatile aromatic compounds of other fruits also contribute to the aroma of melon fruit. (E, Z)-2,6-Nonadienal (no. 60), a principal volatile compound of the cucumber, was identified by both methods. Although the concentration level was low, this volatile compound appears to impart the characteristic grassy odor of the Cucurbitaceae family to melon. Willie and Leach (1992) reported the presence of 2-(methylthio)ethyl acetate and 3-(methylthio)propyl acetate, sulfur-containing compounds, which greatly influence aroma despite its low concentration, were also detected in Melon. This presumably resulted from the use of improper FID (this detector is very low sensitive for sulfur-containing compounds) to identify sulfur-containing compounds during GC analysis, not from the use of a different cultivar or inappropriate extraction method.

The PQM and SDEM were repeated five times and their reproducibilities were compared (Table 2). The coefficient of variation of the ten volatile compounds randomly selected by the PQM reveal that for peak no. 94, it was relatively high (20.37%), others were low,

Table 2. Comparison of reproducibility by Porapak Q column concentration extraction method (PQM) and simultaneous distillation extraction method (SDEM).

Peak ^z No.	C.V. (%) ^y	
	Porapak Q	SDE
9	16.37	40.68
19	15.59	21.37
23	5.42	37.15
42	10.01	37.06
45	11.27	38.82
58	11.94	53.41
71	5.29	48.78
69	8.58	25.26
83	14.19	37.77
94	20.37	31.02
Average	11.90	37.13

^z The peak numbers correspond to the numbers in Fig. 1.

^y Coefficient of variation.

Fig. 2. Relationship between sample volume and recovery of volatile compounds in 'Miyabi' melon by Porapak Q column concentration method. a) The peak numbers correspond to those in Fig. 1.

ranging from 5.29% to 16.37%, with an average of 11.90%. In SDEM, peak no. 19 was the lowest (21.37%); others were higher than 30% except peak no. 69; the average was 37.13%. Thus, the reproducibility of all ten volatile compounds by PQM was approximately three-fold greater than that of SDEM. It was difficult to establish the same extraction conditions using the SDEM because the reduced pressure and temperature could not be controlled within narrow limits. In the PQM, volatile compounds are extracted quickly at room temperature, resulting in higher reproducibility. The coefficient of variation was 5.7% in sake (Sakamoto et al., 1993), 14.0% in beer (Shimoda et al., 1987), and 8.2% in *Perilla frutescens* (Sakamoto et al., 1995). A comparative study of reproducibility among PQM, solvent extraction method, and SDEM, demonstrated that PQM was the most effective. Although the results of this study were similar to those of other researchers, the coefficient of variation (11.90%) was slightly higher. The difference is attributed to fewer volatile aromatic compounds and the presence of more sugars and amino acid.

The concentrates, that are isolated by the PQM possessed a characteristic, natural melon-like odor, whereas the concentrates from the SDEM had a degraded, thick, over-cooked odor, similar to that reported for green tea (Shimoda et al., 1995). Thus, PQM has been proven to be more efficient and favorable for the extraction of their natural aromatic constituents.

The relationship between sample volume and the recovery of volatile compounds in muskmelon fruit are shown in (Fig. 2). The recovery of ten randomly selected compounds which increased relative to sample volume, with a negligible change in ratio, indicates that Porapak Q was not saturated by a 300-g sample of melon extract.

A preliminary test on the volume of diethyl ether, necessary to elute the absorbed volatile compounds in

Table 3. Recoveries of volatile compounds from Porapak Q column concentration method.

Peak No.	Recovery (%) ^y		
	1st	2nd	3rd
9	99.1	1.79	0
19	98.2	1.79	0
23	96.9	3.06	0
42	94.1	5.86	0
45	98.8	1.25	0
58	98.8	1.19	0
71	98.2	1.76	0
69	97.3	2.66	0
83	98.7	1.33	0
94	96.1	3.86	0
Average	97.6	2.45	0

^z The peak numbers correspond to the numbers in Fig. 1.

^y The volatile compounds were eluted with 60 ml of diethyl ether and this procedure was repeated three times.

Porapak Q (Table 3), showed that 60 ml of that eluted 99.1% of compound no. 9 in the first wash and more than 94% of nine others as well. The remaining ones were almost completely eluted by the second wash. Thus, slightly more than 60 ml of diethyl ether was required to elute the absorbed volatile compounds from 300 g of melon mesocarp with PQM; that is, the necessary volume of diethyl ether was about 8 to 10 times the volume of Porapak Q.

Therefore, we conclude that the PQM is an excellent technique for extracting volatile aromatic compounds in melon fruit. It is an easy, quick, efficient, and a highly reproducible one capable of recovering volatile compounds with a wide range of boiling points without degrading them. Only PQM can recover high-boiling-point compounds, which are strongly associated with aroma and flavor.

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Porapak Q カラム濃縮法を用いた ‘ミヤビ’ メロン (*Cucumis melo* L.) の香気分析早田保義・坂本隆行・河塚 寛¹・坂本宏司²・箴島 豊*

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摘 要

メロンの香気成分分析において Porapak Q カラム濃縮法 (PQM) の有効性を明らかにするため、従来の抽出方法である直接ヘッドスペースガス採取法 (DHSM) および減圧連続蒸留抽出法 (SDEM) と成分の回収量や回収率および再現性等について比較・検討した。検出された揮発成分数は PQM が 103 成分で最も多く、次いで SDEM が 66 成分、DHSM が 8 成分であった。同定された揮発性成分は 76 成分であり、アルコール類では、2-Methyl-1-propanol, 2-Methyl butanol, 1-Hexanol, 1-Heptanol, 1-Nonanol, (Z)-3-Nonen-1-ol, (Z)-6-Nonen-1-ol, (Z, Z)-3,6-Nonadien-1-ol, Benzyl alcohol および 2-Phenethyl alcohol などが PQM で比較的多量に検出された。アルデヒド類では 2-Methyl butanal, 3-Methyl butanal, Nonanal, (E, Z)-2,6-Nonadienal および Dodecanal が、PQM で多く回収された。エステル類では両抽出法で Propyl acetate, 2-Methylpropyl acetate, Butyl acetate および Benzyl acetate が比較的多量に回収されたが、回収効率

は Ethyl propionate および Methyl butyrate など低沸点成分の多くがほぼ同程度であり、中高沸点成分は PQM が良好であった。含硫化合物は Ethyl (methylthio)acetate および 2-(Methylthio)ethyl acetate が PQM, SDEM 共に、Ethyl 3-(methylthio)propionate, 3-(Methylthio)propyl acetate が PQM でのみ検出された。成分の抽出変動係数は PQM では平均 11.90% であったが、SDEM では平均 37.13% と PQM の安定的な再現性が得られた。また、PQM で得られた香気濃縮物そのものの香りはメロン特有の自然な香りであったが、SDE では煮え立ったような重い匂いに変性した。PQM は従来の抽出方法に比べ操作が簡便・迅速に行え、更に幅広い沸点範囲の香気成分を変性させることなく回収でき、再現性が高いことからメロンの香気抽出法として優れた方法であると判断された。

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