Analysis of Volatile Organics in Cooking Oils by Thermal Desorption–Gas Chromatography–Mass Spectrometry

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Volatile organic compounds (VOCs) were collected from samples of 14 commercial olive oils and cooking oils using a purge and trap technique (P&T) and analyzed by thermal desorption–gas chromatography–mass spectrometry (TD–GC–MS) to identify, compare, and quantify the volatile organics present. The P&T or dynamic headspace technique permits the analysis of a wider range of both volatile and semivolatile organic compounds and is more sensitive by a factor of at least 100 as compared to the static headspace technique. No solvent extraction is required, which eliminates the exposure of laboratory personnel to these compounds and also eliminates the disposal of these solvents. This analytical method can be utilized for the quality control of these oils during production, can be used to detect adulteration or contamination of the pure product, and may provide for a "fingerprint" chromatographic pattern for the comparison of oils or determination of origin.

Keywords: *Olive oil; flavors/aromas; thermal desorption; dynamic headspace*

INTRODUCTION

Volatile and semivolatile organic compounds present both in the matrix and in the headspace aroma are primarily responsible for the flavor/fragrance qualities of commercial cooking oils. There is a concern in the food industry over the quality in cooking oils due to plant origin, plant maturity, adulteration, or dilution of the final product. High-purity cooking oils such as olive, peanut, and soybean oils are 90% unsaturated with little or no residual solvents and are considered the cooking oils of choice. Olive oil, which is produced by crushing or pressing the fruits of the olive tree, *Olea europeae*, is unique among vegetable oils in that it can be consumed in this crude form, called virgin olive oil. Oils properly processed from fresh mature fruits of good quality provide a delicate and unique aroma. However, cheaper cooking oils such as cocoa, butter oils, and lower grade olive oils are less pure as the result of multiple pressings or water extractions and possibly are without a distinctive aroma or flavor components. In a previous study, Olias et al. (1980) observed from the relationship between fruit maturity and aroma components that the characteristic flavor is obtained by the balance between "green" and fruity notes.

In the determination of an oil's plant origin and in the assessment of its overall flavor quality, it would be extremely advantageous to have a reliable and efficient method for the detection, identification, and quantification of the volatile organic compounds responsible for the unique flavors and aromas in these oils. Because oils possess highly characteristic aromas due to specific volatile organic components, it can be anticipated that the chemical analysis of the aroma and flavor components of a given oil could give a "fingerprint" which could be dependent on the fruit or floral source. Montedoro et al. (1986) applied statistical methods to the analysis of 52 different Italian olive oils from Umbria covering the contents of hydrocarbons, sterols, phenols, volatile aroma compounds, and chlorophyll's and separated the oils into three groups characteristic of the northwest, central, and southern areas of the province. Soil, climatic conditions, and extraction methods were considered the reasons for these differences. Similar studies have been done for the identification of flavors in foods using GC headspace techniques. Bouseta et al. (1992) determined that in honeys some compounds appeared to be characteristic of the floral source such as hexanal and heptanal in lavender, acetone in fir, diketones, sulfur compounds, and alkanes in eucalyptus, and three unidentified compounds in dandelion and rape utilizing the dynamic headspace GC technique.

In previous studies of flavors and aromas in oils and food products, GC methods have been the method of choice. Static headspace techniques are limited to their level of detection and identification of many organic volatiles and especially the semivolatile organics. More sensitive analytical techniques are needed to profile and identify flavors, fragrances, off-flavors, off-odors, and potential contaminants that may be present as flavor and fragrance additives at lower concentration levels. The purpose of this investigation was to develop an analytical technique that could detect and identify a wide range of volatile and semivolatile organic compounds in commercial cooking oils. For this study, volatile organic compounds (VOCs) were collected from samples of cooking oils using a purge and trap technique (P&T), followed by trapping on Tenax TA adsorbent resin (Buchem bv, Apeldoom, Holland) and subsequent analysis by thermal desorption–gas chromatography–mass spectrometry (TD–GC–MS). When compared to static headspace, the P&T or dynamic headspace technique permits the analysis of a wider range of both volatile and semivolatile organics including higher molecular weight compounds and is more sensitive by a factor of at least 100. In addition, no solvent extraction is required, which eliminates the exposure of laboratory personnel to these potentially dangerous chemicals as well as eliminates the need for waste disposal for these solvents. Not only can the P&T technique be used to differentiate between the various grades of olive oil, it can also be used for both the qualitative and quantitative analyses of cooking oils and for the development of a quality control method for the food industry.

INSTRUMENTATION

Samples were collected using a purge and trap system (Scientific Instrument Services, Ringoes, NJ). This apparatus

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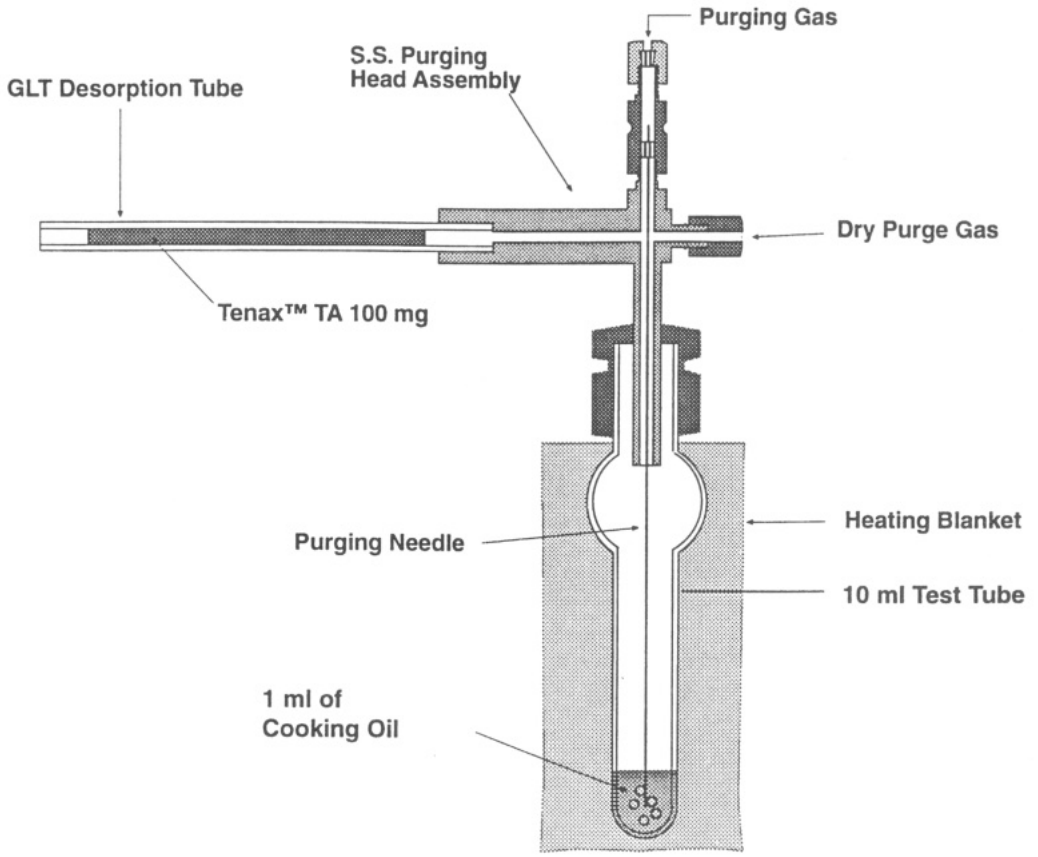

Figure 1. Purge and trap apparatus; theory of operation.

(Figure 1) consists of a purge gas inlet connected to a stainless steel purging needle that is inserted through an adaptor fitting into a 10 mL glass test tube. A dry purge gas inlet is located at a right angle to the purge gas inlet at the top of the apparatus. The purpose of the dry purge is to reduce the water vapor condensation on the adsorbent trap. This problem can be especially troublesome when volatiles are isolated from aqueous solutions at high temperatures. Although the adsorbent traps packed with Tenax have a low affinity for water, it is inevitable that some condensation will occur in the trap due to the high relative humidity of the purge gas as it exits the apparatus. When moisture condenses on the adsorbent, it can block the pores of the resin matrix and thereby drastically reduce the diffusion of volatile organics into the trapping agents. This will result in reduced trapping efficiency. Opposite the dry purge inlet is the connector for the glass-lined stainless steel (GLT) desorption tube containing 100 mg of Tenax TA adsorbent resin. The liquid purging system also contains two ball rotameters with adjustable needle valves mounted on a stationary base and permits the visual indication and independent adjustment of the carrier gas flow to each of the gas inlets.

The thermal desorption of the Tenax TA traps into the GC was conducted using a Short path thermal desorption system, Model TD-2 (Scientific Instrument Services). The theory of operation of this system is described elsewhere (Manura et al., 1990; Manura and Hartman, 1992). The thermal desorption system heater blocks were preheated to 220 °C. The thermal desorption system flow rate was set to 5.0 mL/min and the sampling time was set to 5.0 min for all samples to be analyzed.

The thermal desorption system was connected to the injection port of an HP 5890 Series II GC (Hewlett-Packard Co., Palo Alto, CA) interfaced to an HP 5971 mass selective detector (Hewlett-Packard). A short 20 cm × 0.53 mm diameter fused silica precolumn was attached to the injection port end of a 30 m × 0.25 mm i.d. DB-5MS capillary column containing a 0.25 μm film thickness (J&W Scientific, Folsom, CA). The GC injection port was set to 250 °C and a 5:1 split was used. The head of the column was maintained at −70 °C using a GC Cryo-Trap Model 951 (Scientific Instrument Services) during the desorption and extraction process and then ballistically heated to 200 °C, after which the GC oven was temperature programmed from 35 °C (hold for 5 min) to 80 °C at 10 °C/min, then to 200 °C at 4 °C/min, and finally to 260 °C at a rate of 10 °C/min.

The mass spectrometer was operated in the electron impact mode (EI) at 70 eV and scanned from 35 to 400 Da during the GC run for the total ion chromatogram. The total ion signal was integrated using Hewlett-Packard ChemStation software, and each of the chromatogram peaks was library searched utilizing the NBS reference library to identify the organic compounds. All library matches had a quality match of at least 90. This identification is "tentative" since no additional analytical techniques were used to identify the compounds.

EXPERIMENTAL PROCEDURES

Sample sizes of 1 mL of the various oils were pipetted into a 10 mL test tube and heated to 80 °C for 30 min. Samples were purged with high-purity helium at 20 mL/min with an additional 25 mL/min dry purge using the SIS purge and trap system (Figure 1). Volatile analytes were purged from the liquid matrix and carried to a preconditioned 4.0 mm i.d. glass-lined stainless steel desorption tube packed with 100 mg of Tenax TA. Once the samples were collected in the desorption tubes, they were spiked with a mixture of 100 ng of toluene-d_8 (MSD Isotopes, Merck Chemical Division, St. Louis, MO), 200 ng of cymene-d_{14} (MSD Isotopes), and 50 ng of naphthalene-d_8 (MSD Isotopes) internal standard by injecting 1 μL of the stock solution in methanol by syringe injection into the Tenax matrix. An additional purging of 120 mL of purge gas was required to purge the methanol from the Tenax trap.

Five brands of olive oil and one brand each of corn, vegetable, canola, and canola and vegetable oil were analyzed to identify, compare, and quantify the volatile organics of different manufacturers' brands. In four of the olive oils the extra virgin olive oil was compared to lesser grade oils of the same manufacturer's brand. For quantification, an internal standard was spiked into the adsorbent traps after the sample

Table 1. Relative Amounts (Nanograms per Milliliter) of Volatile Organics in Cooking Oils

		brand A	brand B			brand C		brand D		brand E	
peak no.		extra virgin	extra virgin	regular	extra light	extra virgin	regular	extra virgin	regular	extra virgin	extra mild
1	butanal		3.3		3.9	15.5			2.0	4.1	4.5
2	hexane	53.0	18.4					95.3		19.1	
3	ethyl acetate	87.4	165.1	15.9	1.3	82.6	6.3	133.8	15.4	76.2	
4	3-methylbutanal		11.8			21.7		18.6	3.1	27.9	
5	2-methylbutanal		11.2			22.7		20.0		17.6	
6	3-pentanone		62.5				5.0			134.9	
7	heptane	144.0						390.3			
8	pentanal		48.7	50.8	68.1	295.1			52.4		46.0
9	toluene-d_8					(internal standard)					
10	toluene	115.1	28.3	19.1	9.1	22.7	8.8	60.7	11.3	28.6	15.1
11	1-octene	50.5		13.8	14.5	24.8	10.6	63.2	13.0	24.9	
12	2-octene		17.8								
13	octane	2310.1		228.1			121.4	3107.8		244.9	
14	hexanal	208.6	516.4		319.5	1903.0	24.2		255.9	366.6	151.8
15	(*E*)-2-octene							147.5			
16	(*Z*)-2-octene							79.3			
17	(*E*)-2-hexenal	72.6		119.6	8.8	689.4	153.3		254.4	1758.1	
18	(*Z*)-3-hexenal	32.6	869.1					1330.9			
19	(*Z*)-3-hexen-1-ol		88.2								
20	(*E*)-2-hexen-1-ol		436.2	8.9		183.7		249.1	36.2	286.7	
21	4-hexen-1-ol					117.6		26.0			
22	2-heptanone	22.2	48.0	17.5	17.6		16.7	62.0	21.7	51.3	25.5
23	styrene	113.8				175.4					
24	heptanal	25.8	46.1	15.9	58.4	227.1	13.6	128.9	18.5	52.1	40.1
25	(*E*)-2-heptenal	210.7	23.1	20.9			8.5	200.6		24.6	8.6
26	(*Z*)-2-heptenal					124.3					
27	2-pentylfuran				35.4	149.5	12.0		38.1		
28	octanal				43.5	174.2			30.3		28.1
29	(*Z*)-3-hexen-1-ol	324.8						227.4			
30	(*Z*)-3-hexen-1-ol acetate		246.6							193.4	
31	(*Z*)-4-hexen-1-ol			18.0			23.3		22.9		
32	acetic acid, hexyl ester	45.6	80.7			14.9	6.7	64.3	4.4	78.2	
33	cymene-d_{14}					(internal standard)					
34	limonene	7.6	11.8	10.7	2.0			286.4	418.4	6.9	
35	(*E*)-2-octenal	40.3			9.2	138.1		49.8			
36	nonanal	274.6	84.7	18.9	68.6	334.6	19.2	307.0	186.0	63.5	36.9
37	naphthalene-d_8					(internal standard)					
38	methylcycloheptane		38.8			17.8				17.8	
39	(*Z*)-2-decanal	42.2	7.6				1.7			2.8	
40	2-cyclohexen-1-ol				19.9	28.8		34.8			
41	2-undecenal		8.2		16.8	11.4				1.2	

had been isolated. No correction for extraction efficiency of recovery is achieved using this technique; however, it serves as a useful means of quantifying the levels of components present on the adsorbent traps (SIS, 1991). Alternatively, the internal standard could have been spiked into the oil sample matrix to allow for the extraction efficiency of the purge and trap technique.

The desorption tube with sample and internal standard were then attached to the short path thermal desorption system, and a syringe needle was attached. The desorption tube was injected into the GC injection port at a desorption block temperature of 220 °C for 5 min and a flow rate of 5 mL/min. The GC and mass spectrometer were operated as described above.

To compare the static headspace technique versus the P&T–TD technique, 5 mL of olive oil was placed in a 10 mL headspace vial, which was sealed with a Teflon-coated silicon septum. The vial with sample was heated to 80 °C for 30 min. After an equilibrium period of 30 min, 2 mL of headspace vapors was withdrawn from the headspace vial via a syringe and injected into the GC injection port. The GC and mass spectrometer were operated as previously described.

The total ion signal from the mass spectrometer was integrated using Hewlett-Packard ChemStation software to determine the relative amounts of each of the volatile organics detected and identified in the chromatogram of each of the oils studied. The integrated signal from each of the organic volatiles detected was then divided by the integrated signal from the closest internal standard to obtain the relative signal strength of each of the components. This value was then multiplied by the amount of internal standard added to the sample to determine the semiquantitative amount of each of the volatiles detected in nanograms per milliliter of oil.

$$X = (AC)/S$$

Here X is the concentration of volatile compound (ng/mL), A is the integrated area of volatile compound (counts/mL of oil), S is the integrated peak area of internal standard (counts), and C is the amount of internal standard (ng).

Since samples of each of the organic volatiles were not actually used to establish calibration curves for each volatile compound present in the oil samples, these calculated values are not absolute in their accuracy. The calculation assumes unity response from each of the compounds. Unity response from the mass spectrometer detector is not realistic, nor is it achievable. However, for this study of the relative comparison of oil samples, these semiquantitative results are acceptable.

RESULTS AND DISCUSSION

Fourteen cooking oils including 5 brands of olive oil (brands A–E) and 1 brand each of corn, vegetable, canola, and canola and vegetable oil were analyzed to identify, compare, and quantify the volatile organics present. Table 1 shows the most relevant VOCs detected and the relative amounts of each of these compounds in each cooking oil analyzed and calculated as described above. Over 200 volatile organics were identified in the various oils studied. Most of the cooking oils studied produced 50 or more volatile organ-

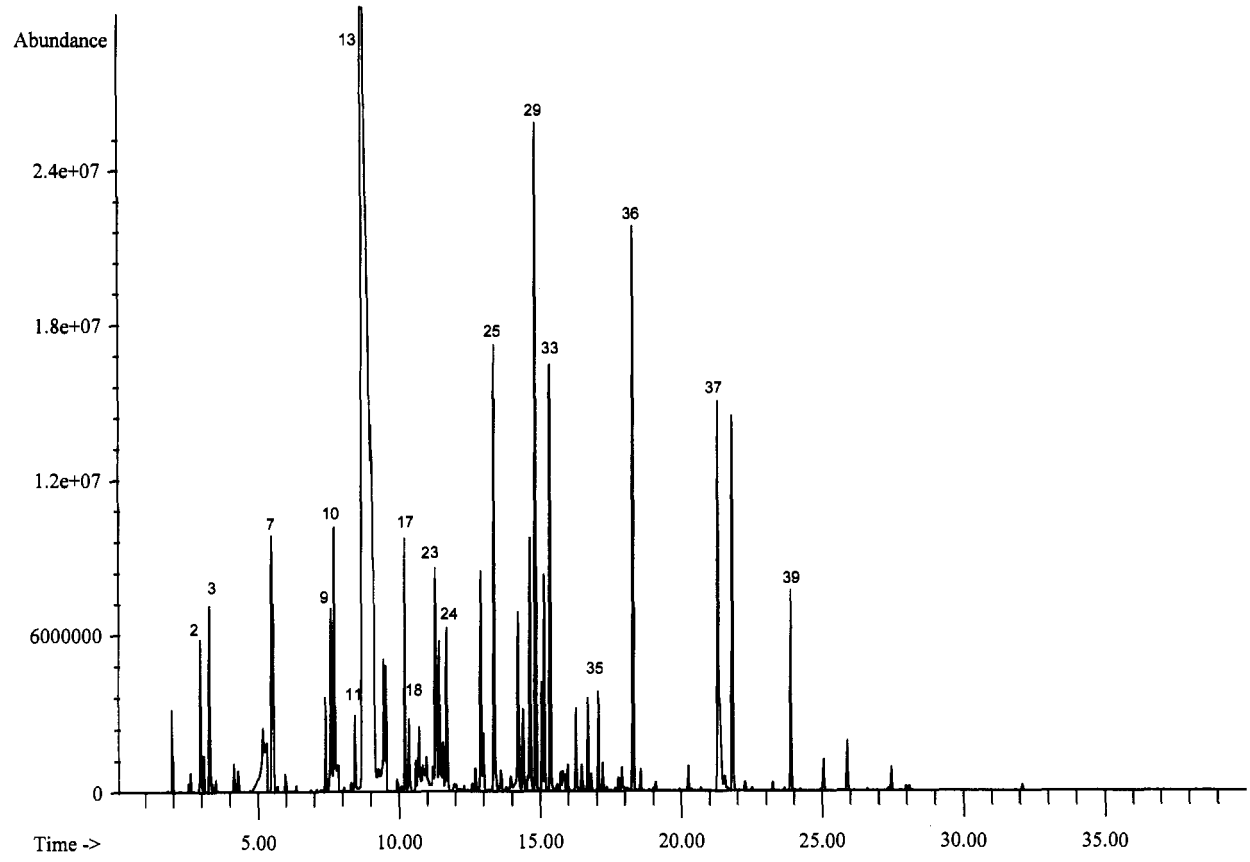

Figure 2. Chromatogram of extra virgin olive oil, brand A.

ics, which were identified in addition to many more that were either too weak to identify or for which a good NBS library match was not achievable.

When compared to the static headspace technique, the P&T or dynamic headspace technique permitted the analysis of a wider range of both volatile and semivolatile organics including higher molecular weight compounds and was more sensitive by a factor of at least 100 (1000× when compared to the most sensitive peak). This significant increase in sensitivity was because the sample was purged using 600 mL of gas in the dynamic headspace technique (1 mL of olive oil purged at 20 ml/min for 30 min), while with static headspace only 2 mL of headspace volume was injected from 5 mL of olive oil.

Five different manufacturers of olive oils (brands A–E) and several different grades (extra virgin, virgin, regular, light, and extra mild) of these olive oils were studied, and the data are presented in Table 1. A typical profile of an extra virgin olive oil is shown in Figure 2. The cooking oils were found to contain numerous straight and branched chain hydrocarbons, aldehydes, alcohols, ketones, esters, and furans in addition to many benzene derivatives. Significant differences in the volatiles present from different manufacturers as well as different grades occur in these olive oils. The extra virgin olive oils in brands A and D and the extra light olive oil in brand B contain the aromatic compound benzene.

Figure 3 demonstrates the differences between the different grades of olive oil from the same manufacturer. Manufacturers have established their own criteria for the classification of the different grades of olive oils. There appears to be no industrywide standard. Typically the virgin or extra virgin olive oils are the oils extracted from the first presses of the oils. The regular and other grades are the result of second pressings and are classified as being of lower quality. As can be seen in Figure 3, the extra virgin oil has a higher concentration of the lower molecular weight volatiles including ethyl acetate, 2-methylbutanal, and hexanal. Toluene was also detected in each of the cooking oils, with the highest levels in the extra virgin olive oils.

Extra virgin olive oils from three different manufacturers are compared in Figure 4. Although many of the same volatiles are present in all of these oils, there is significant variation in the quantities as well as the variety of volatile organics present. This variation could be due to the origin and maturity of the harvested olive fruits as well as differences in the manufacturing processes.

Aliphatic C_6 compounds hexanal, (*E*)-2-hexenal, and (*Z*)-3-hexenal, which contribute greatly to the "green" notes of the aroma, were present in significant quantities in the cooking oils, especially in the extra virgin olive oils. These compounds and corresponding hexyl esters have previously been reported in great quantities in the volatile components of olive oils (Montedoro et al., 1978; Olias et al., 1978; Guth and Grosch, 1991). The formation of C_6 aldehydes and alcohols in the plant is related to cell destruction. During processing of olive oil, milling and malaxation of the olive fruits prepare the paste for its extraction by pressing and centrifugation. Disruption of intact cells results in the formation of these compounds due to enzymatic reactions by releasing lipid-degrading enzymes that degrade the membranes or stored lipids in the presence of oxygen such as by homogenization of plant material (Schreier, 1981; Tressel et al., 1983). Hexenal, (*Z*)3-,(*E*)2-hexenal, and their analogous reduced products have been previously reported in disrupted tissues of apples, grapes (Drawert et al., 1966), and tomatoes (Kazeniac and Hall,

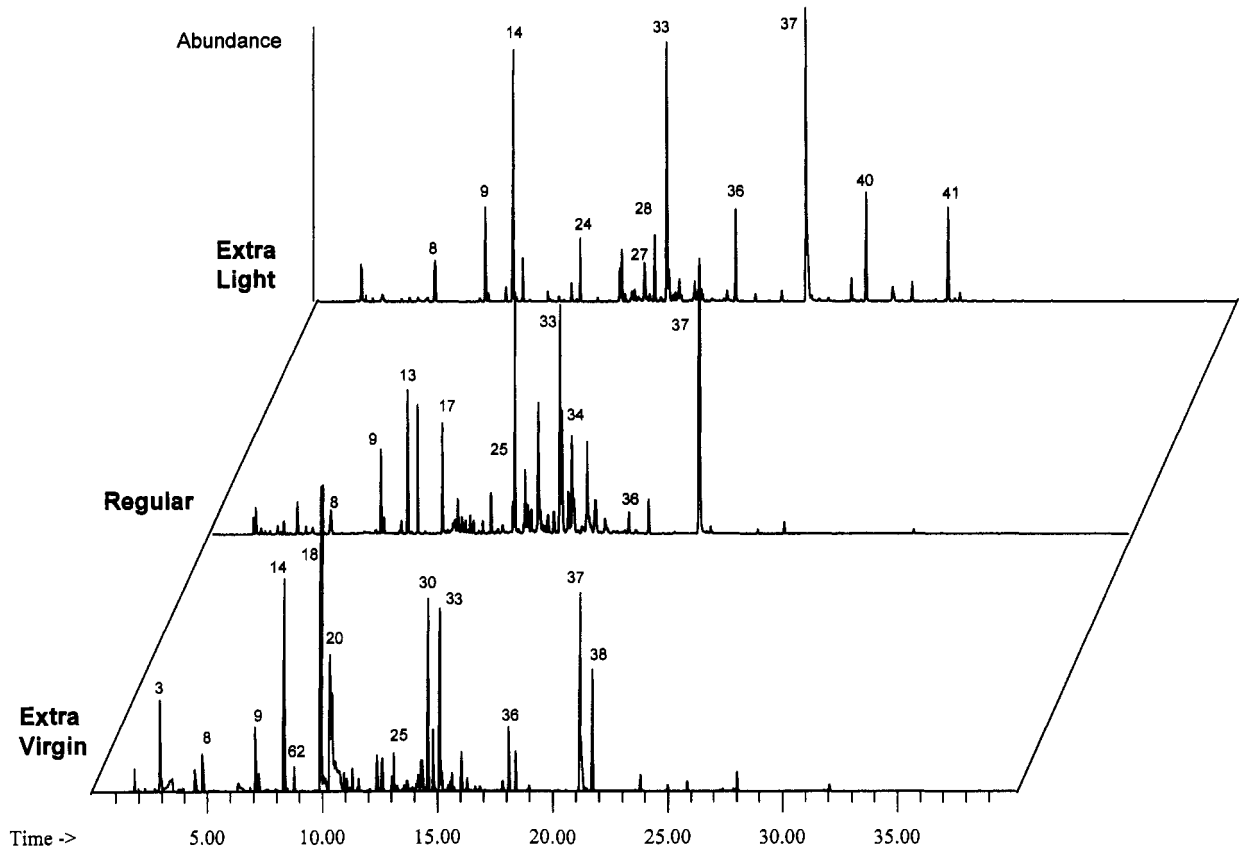

Figure 3. Comparison of volatile organics in different grades of olive oil from one manufacturer (brand B).

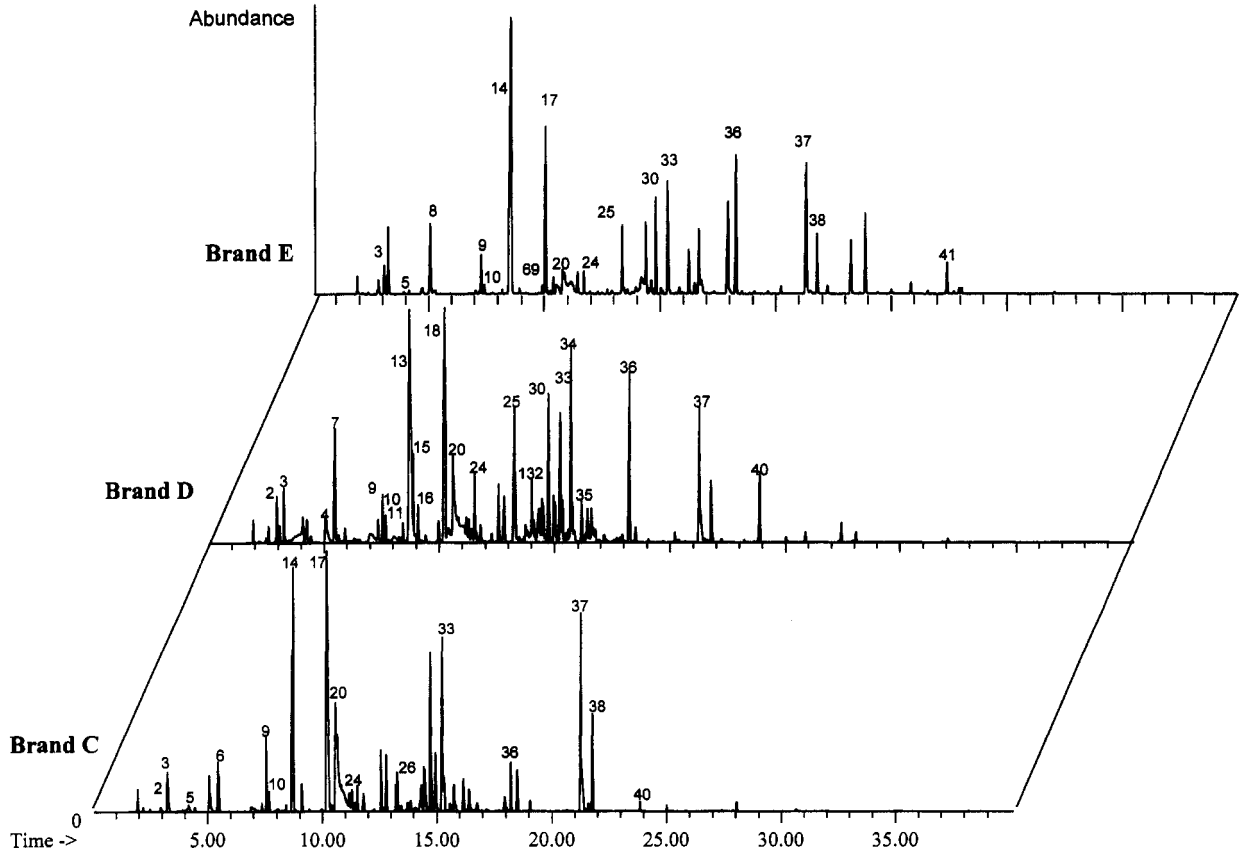

Figure 4. Comparison of volatile organics in extra virgin olive oil from different manufacturers.

1970). It has been assumed for a long time that unsaturated fatty acids are the precursors of these volatile compounds. A pathway involving lipoxygenase and hydroperoxide lyase has previously been demonstrated in banana and tomato (Tressel and Drawert, 1973; Stone et al., 1975). Olias et al. (1993) determined that in virgin olive oil fatty acid hydroperoxide lyase cleaved the 13-hydroperoxides of linoleic and linolenic

acids to form the volatile aldehydes hexanal and (*Z*)-3-hexenal, respectively. The aliphatic compounds heptanal, octanal, and nonanal were also identified in each of the cooking oils. Solinas et al. (1985) suggested that several aldehydes and ketones formed by oxidation of fatty acids in virgin olive oil, especially linoleic and linolenic acids, may be of importance for the development of rancid flavor. The volatiles were collected by an N_2 stripping activated C absorption system and analyzed by HPLC and GLC. The concentration of aldehydes initially decreased slightly and then increased as oxidation progressed. Solinas et al. (1987) later revealed a direct relation between rancidity and 2-pentenal, hexanal, 2-heptenal, 2-octenal, octanal, and nonanal, with 2-pentenal and 2-heptenal being the main indicators of rancidity.

In addition, the presence of the branched aldehydes 2-methylbutyraldehyde and 3-methylbutyraldehyde, which contribute to the fruity flavor notes in the extra virgin olive oils (Figures 3 and 4), reflects a microbial quality and thermal treatment of the olive oil. Bouseta et al. (1992) also reported the presence of the branched aldehydes and alcohols in unifloral honeys and suggested production mechanisms for these aldehydes such as possible fermentation and/or amino acid metabolism by contaminating yeasts. Another possible synthetic pathway may be the Maillard reactions (Strecker pathway) occurring during extraction and/or storage of the oil. This reflects product quality. In a study by Colakoglou et al. (1982), the concentrations of some volatile compounds fluctuated during storage. Hexanal varied most consistently, rising under all storage conditions. Montedoro et al. (1985) also demonstrated that the infestation of olives by the fruit fly, *Dacus oleae*, adversely affected fruit quality, resulting in the classification of the oil being reduced from extra virgin to superfine. Volatile compound concentrations of hexanal, isopentanol, hexenal, hexanol, and hexenol increased while sensory quality dropped. In addition, lipase production, lipolysis, and the formation of volatile compounds by the lipolytic bacterium, *Pseudomonas fluorescens,* detected in the headspace gas over the fat containing media supplemented with either olive, sunflower, or soy oil have previously been reported (Andersson, 1980). The volatile fraction was found to contain alcohols, aldehydes, ketones, esters, furans, sulfur compounds, and hydrocarbons.

Linear alkenes such as 1- and 2-octene from lipid oxidation decomposition products were also identified in the cooking oils. Very high concentrations of octane were found in the extra virgin olive oils in brands A and D. Barrio Perez-Cerezal et al. (1981) discovered that octane concentration in the headspace of olive oil containers increased with time during storage. The greatest concentrations of the flavor compounds ethyl and hexyl acetates were present in the extra virgin olive oils compared to the lesser grade oils of the same manufacturer (Table 1). These flavor compounds were not identified in the corn, vegetable, canola, and canola and vegetable oils. This suggests that the extra virgin olive oils are the richest in flavor/aroma compared to their lesser grade counterparts and the other vegetable oils. Chloroform, most likely from water treatment during malaxation of the olive fruits, was detected in the extra virgin olive oil in brand C.

Styrene, which may have leached from the plastic cap liners, was found in the extra virgin olive oils in brands A and C. This is possible when polymers such as polystyrene, acrylonitrile–butadiene–styrene (ABS), are used as packaging material with foods. Adverse effects to human health have previously been reported (Nerin et al., 1993), and styrene concentration in food has already been limited in European countries. Monoterpenes such as 1-phellandrene, α- and β-pinenes, cymene, Δ-3-carene, *trans*-ocimene, and a significant amount of limonene were identified in both the extra virgin and the lesser grade olive oil in brand D. Additional compounds included a series of sesquiterpenoid compounds such as α-cubebene and α-copaene. A series of straight chain hydrocarbons and the linear alkenes 1- and 2-octene from lipid oxidation decomposition products were identified in the corn, vegetable, canola, and canola and vegetable oils. However, these cooking oils were not as rich in flavor or aroma compared to any of the olive oils. It is apparent from this study that the purest and highest quality cooking oils were the extra virgin olive oils.

CONCLUSION

The purge and trap or dynamic headspace technique followed by thermal desorption has been utilized for the identification and quantification of the volatile organic, aroma, and flavor components in olive oils and other cooking oils. This analytical method can be utilized for quality control during the production of the cooking oils as well as a method for the detection of adulteration or dilution of these oils. The chromatograms produced can provide for a chromatographic "fingerprint" for the comparison of the cooking oils to determine origin, to compare different manufacturers, or for quality control. Many kinds of flavors are used in the food industry, and there is a demand for new and improved ones, especially natural ones. Such a source are oils which can produce a variety of flavors. The short path thermal desorption system used in conjunction with a dynamic headspace technique permits the identification and accurate quantification of trace volatile organic compounds in cooking oils. The P&T technique offers several unique advantages over other techniques such as solvent extraction, static headspace sampling, and microdistillation including greater sensitivity and the detection of a wider range of volatile organics including higher molecular weight compounds and is more sensitive by a factor of at least 100 as compared to the static headspace technique. No solvent extraction is required, which eliminates the exposure of laboratory staff to these compounds and also eliminates the disposal of these solvents. This technique can easily be incorporated into troubleshooting techniques to detect problems in a wide variety of commercial food products and to compare various competing manufacturers' products as well as into a quality control program. This technique has been applied to other applications such as quantification of the aromatic compounds benzene and toluene in food products (SIS, 1991) and of flavors and fragrances in food products (Manura, 1993; Hartman et al., 1992), commercial products (SIS, 1992), and plant material (Patt et al., 1992). It has also been used in the analysis of volatile organics in indoor air pollution.

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