Correlations of Flavor Score with Volatiles of Vegetable Oils[1]

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ABSTRACT

A simple, direct, gas chromatograph (GC) technique is described for eluting flavor-related volatile components from commercially produced vegetable oils. A sample of oil was placed onto glass wool contained in a GC liner, and the liner was inserted in the heated inlet of the GC. Volatiles from the oils were rapidly eluted by heat and carrier gas onto the GC column. Profiles of the volatiles were obtained by temperature-programmed gas chromatography. Flavor score was highly correlated with individual volatile components considered separately, and very highly correlated with multiple volatile components considered together, indicating that reliable flavor characteristics of vegetable oils may be obtained rapidly and efficiently by instrumentation.

INTRODUCTION

The process of evaluating flavor characteristics in edible products is a complex one (1). The physical requirements for sensory testing (2) and the selection and training of qualified sensory judges (3) requires much time, effort, and expense. Even at its best, sensory testing is limited by the taster's subjectivity. A recent study (4) showed that volatiles in salad oils and shortenings can be examined at a level of 10 ppb by direct GC, without prior enrichment. Profiles of the levels of volatile components in various oils indicated that there might be a correlation between the profile of volatiles and actual flavor score. The work described in this paper was undertaken to obtain profiles of volatiles for several commercially produced vegetable oils by GC and to compare instrumental data with taste panel flavor scores.

EXPERIMENTAL PROCEDURES

Sample Preparation and Analysis

A 3 3/8 in. length of 3/8 in. OD borosilicate glass tubing was packed with volatile-free glass wool, loose enough to permit diffusion of oil throughout the packing, yet tight enough to prevent seepage of the sample from the liner onto the GC column. Clearance of 1/4 in. was allowed at the bottom of the liner and 1/2 in. at the top. A 500 mg vegetable oil sample was added at the top. The septum nut, septum, and retainer nut of the GC were removed, and the liner containing the sample was inserted in the inlet of the GC on top of the silicone O-ring. When the retainer nut was tightened above the upper lip of the liner, a seal was formed between the base of the inlet and the lower lip of the liner. On closing the inlet system with the septum and septum nut, the carrier gas was forced to flow upward and then down through the sample, as shown in Figure 1. The integrator and programmer were turned on immediately. Volatiles were rapidly eluted from the sample as the carrier gas swept through the heated liner and were adsorbed on the top portion of the column, which was maintained at 40 C during the initial hold period of 20 min. Temperature programming was then begun; after 5 min the liner containing the spent sample was removed from the inlet. When temperature programming was complete, the temperature

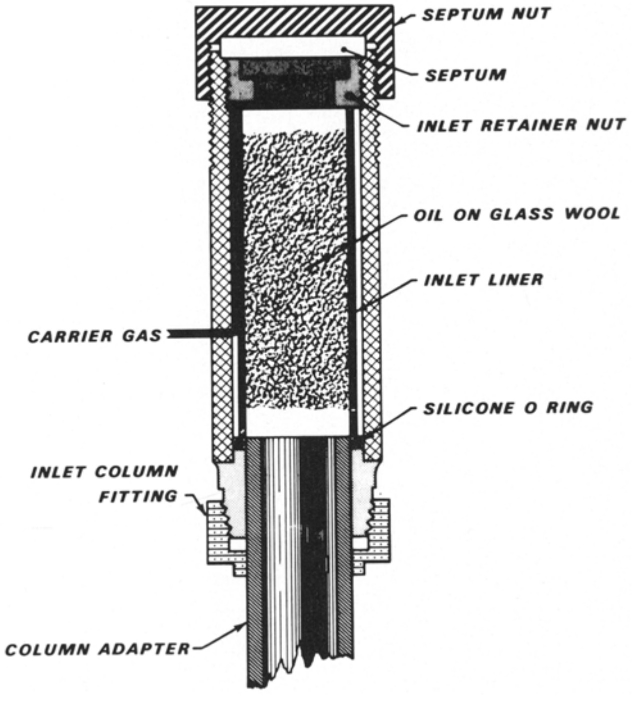

FIG. 1. Cross section of inlet of GC showing inlet liner with sample.

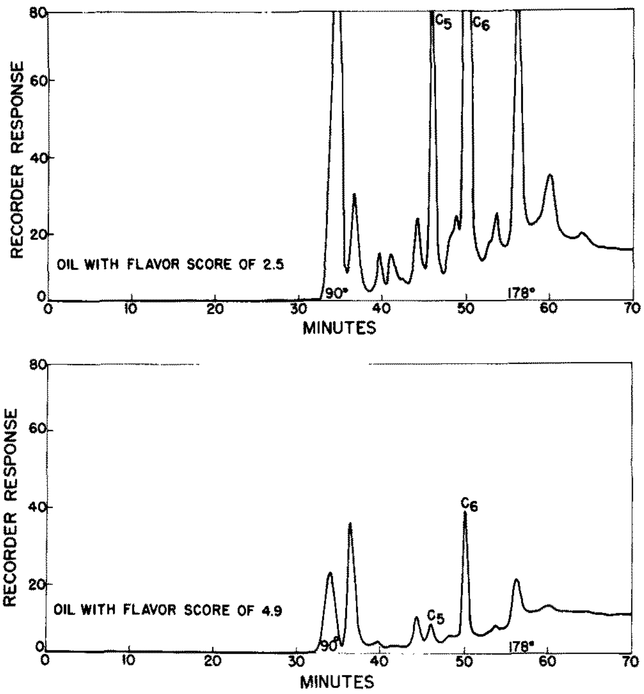

FIG. 2. GC profile of volatiles for low quality and higher quality experimental corn oils.

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[1] Presented at the AOCS Spring Meeting, Mexico City, April 1974.

was maintained on final hold to resolve all the volatiles and elute them from the column. The oven was then cooled to 40 C in preparation for the next sample.

Materials

Porapak P, 80-100 mesh, was obtained from Waters Associates, Framingham, MA. Silicone O-rings (heated at 200 C for 2 hr to remove volatiles) were obtained from Applied Science Laboratories, State College, PA. Pyrex glass wool (heated at 200 C for 16 hr to remove volatiles) came from Corning Glass Works, Corning, NY; refined, bleached, and deodorized corn, soybean, and blended salad oils were obtained from various commercial sources. The samples of soybean oil were light treated by industry to accelerate deterioration.

Gas Chromatography

The following conditions were used to obtain profiles of volatiles from refined vegetable oils.

Instrument:	Tracor MT-220 gas chromatograph with dual independent hydrogen flame detectors, a Westronics MT22 recorder, and a Hewlett-Packard Integrator model 3370B.
Columns:	Stainless steel U-tubes, 1/8 in. OD, 9 ft long, packed with Porapak P.
Flow rates:	Helium carrier gas, 60 ml/min in each column; hydrogen 60 ml/min to each flame; air, 1.2 ft^3/hr (fuel and scavenger gas for both flames).
Temperatures:	Inlet temperature was 120 C for corn oils and 170 C for soybean and blended oils. Detector was at 250 C. Column oven was programmed between 40 and 180 C, initial hold at 40 C for 20 min, programmed at 4/C min for 35 min, inlet liner removed after 5 min of programming; final hold at 180 C for 30 min.
Special seal:	A silicone O-ring was positioned around the 1/4 in. stainless steel adapter, which projected into the bottom of the inlet of the GC.
Attenuation:	10 x 4.
Chart speed:	15 in./hr.

Mass Spectrometry

Instrument: Hewlett Packard Model No 5930 A (Quadrapole).
Ionization potential: 70 eV.
Sample introduction: Interfaced with (GC).

RESULTS AND DISCUSSION

In obtaining profiles of volatiles of vegetable oils by direct GC there appears to be a relationship between instrumental data and subjective taste panel flavor scores. The number and concentration of peaks obtained tend to vary with the flavor quality of the oil. In general, poor quality oils with low flavor scores will produce GC profiles of volatiles with many peaks of high concentration, whereas the profiles of good quality oils with high flavor scores will have fewer peaks of lower concentration. Among the peaks that were found to be most significant in this relationship were: a peak eluting at 90 C, the pentanal (C_5) and hexanal (C_6) peaks, and a peak eluting at 178 C. The C_5 and C_6 peaks were identified by GC retention times and mass spectrometry. The mass spectrometry data for C_5 was: m/e 58, 57, 44, 41, 72, 86 and that for C_6 m/e 56, 57, 43, 72, 82, 67. It was also observed that the summation of total volatiles (TV) gave good correlation with the flavor scores of the various oils evaluated.

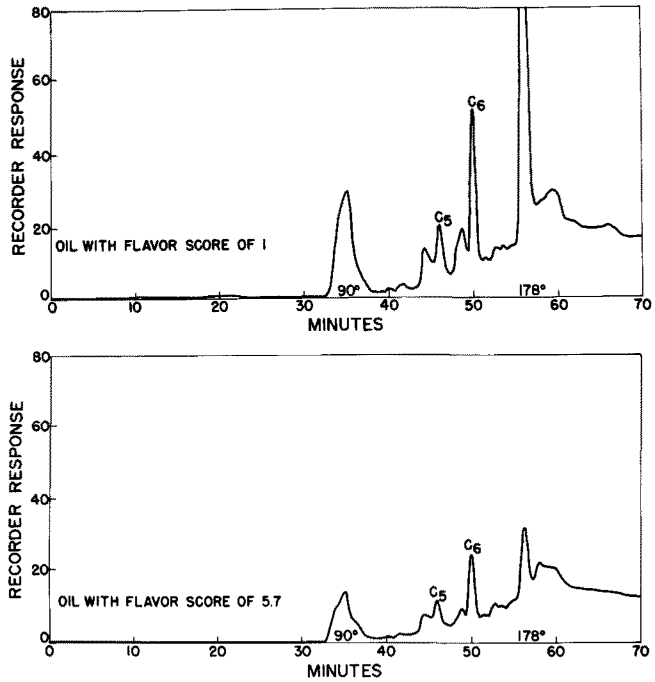

FIG. 3. GC profile of volatiles for low quality and higher quality experimental soybean oils.

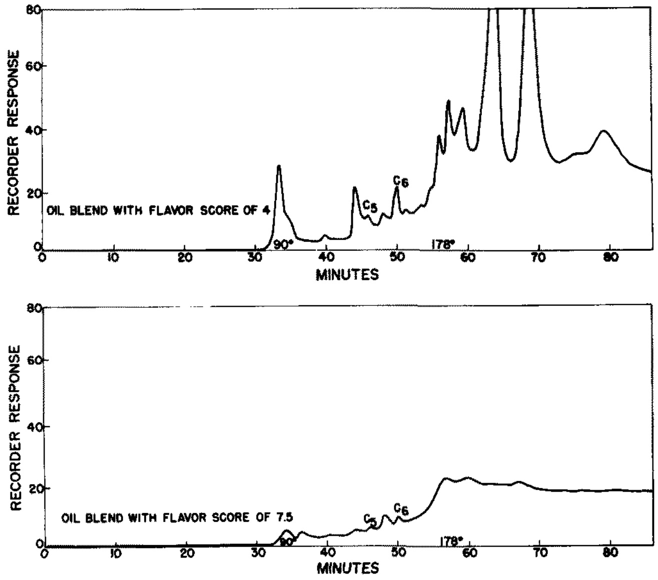

FIG. 4. GC profile of volatiles for low quality and higher quality experimental blended oils.

Figure 2 shows the profiles of volatiles obtained by GC for two of a series of corn oils which were rated on a 1 to 7 flavor scale. The differences in the GC chromatograms for these oils are readily apparent. The higher quality oil (flavor score of 4.9) has a low total volatile content, while the poor quality oil (flavor score of 2.5) has a large concentration of peaks as the 90 C, C_5, C_6, and 178 C peaks.

This relationship is also demonstrated by the GC chromatograms for two of a series of light treated soybean oils shown in Figure 3, which were scored on a 1 to 9 flavor scale. The higher quality soybean oil, scored at 5.7, shows moderate 90 C, C_5, C_6, and 178 C peaks, whereas the lower quality oil, with a projected score of 1, shows approximately twice the concentration of 90 C, C_5, C_6 peaks, with an even greater increase in the 178 C peak.

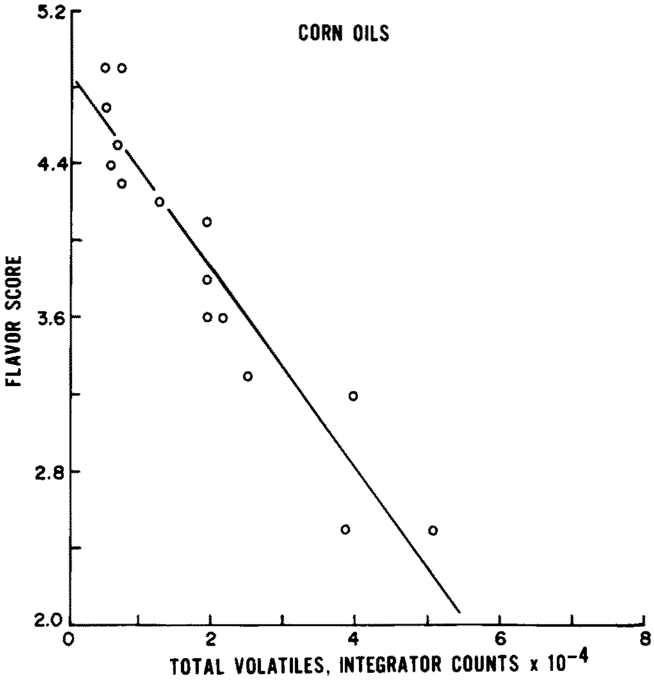

FIG. 5. Linear regression line plots of total volatiles in corn oils and taste panel flavor scores.

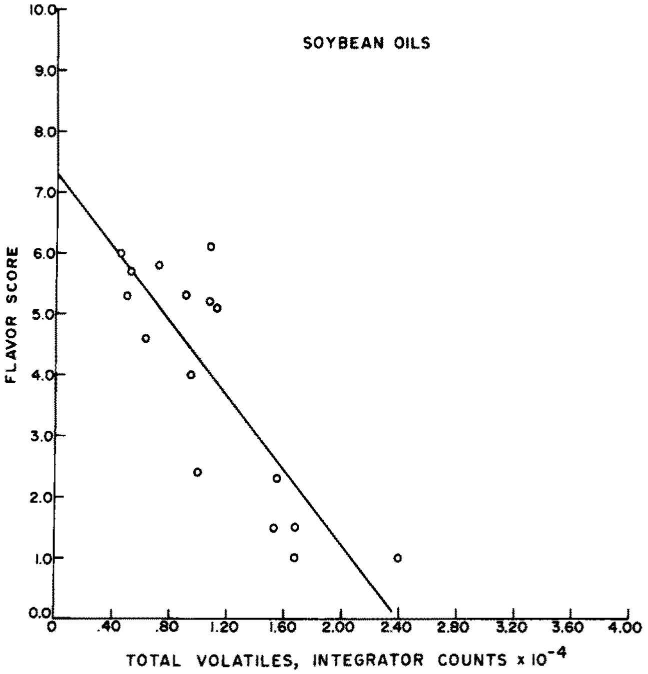

FIG. 6. Linear regression line plots of total volatiles in soybean oils and taste panel flavor scores.

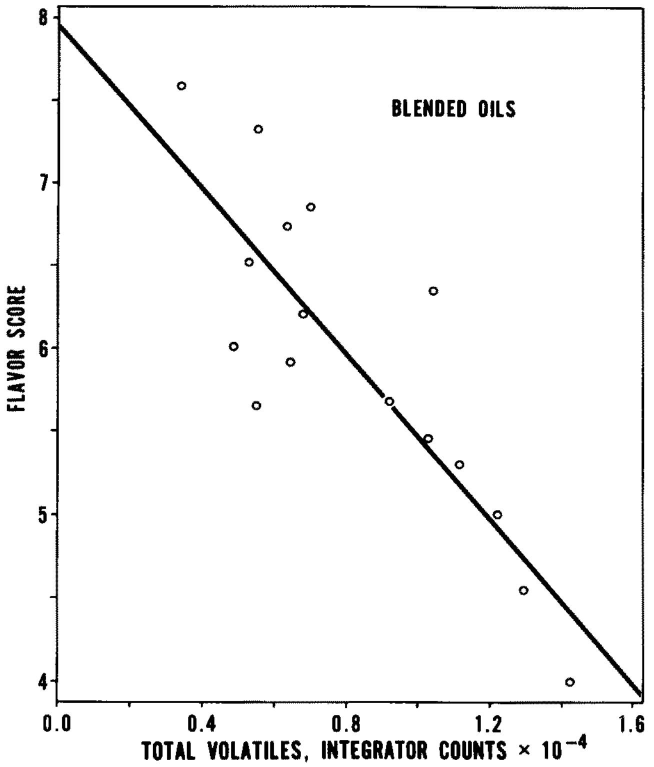

FIG. 7. Linear regression line plots of total volatiles in blended oils and taste panel flavor scores.

TABLE I

Correlation Coefficients of Oil Flavor Scores with GC Integrator Counts of Volatile Peaks

Number and type of oils	Volatile peaks	Correlation coefficient	Standard error
	90 C peak	0.87	0.40
	C_5 peak	0.94	0.28
15 Corn oils[a]	C_6 peak	0.83	0.45
	178 C peak	0.74	0.54
	TV[b] peaks	0.94	0.27
	90 C peak	0.54	1.63
	C_5 peak	0.71	1.33
16 Soybean oils[c]	C_6 peak	0.72	1.39
	178 C peak	0.85	1.03
	TV peaks	0.79	1.19
	90 C peak	0.72	0.69
	C_5 peak	0.34	0.93
16 Blended oils[c]	C_6 peak	0.44	0.89
	178 C peak	0.45	0.89
	TV peaks	0.84	0.54

[a] Standard error of mean for taste panel = 0.37.
[b] TV = total volatiles.
[c] Standard error of mean for taste panel not available for these oils.

The consistency of this effect is further seen in the GC chromatograms shown in Figure 4 for two of a series of blended oils which were rated on a 1 to 9 flavor scale. The higher quality oil, flavor scored at 7.5, shows small 90 C, C_5, and C_6 peaks, whereas the poorer quality oil, flavor scored at 4, shows moderate increases in the intensity of these three peaks and a substantial increase in the TV peaks.

These observations are substantiated by a linear regression analysis of flavor score on GC results. Table I lists the correlation coefficients and standard errors of regression for the relationship of flavor scores to the GC integrator counts for the chromatographic peaks of volatiles in 15 corn oils, 16 soybean oils, and 16 blended oils. From the correlation coefficients shown for the three types of oils, a high degree of certainty (99%) exists for the relationship between the GC results and the taste panel flavor scores.

The specific peaks which have the greatest effect on flavor will vary from one type of oil to another; thus, no single peak can be used to predict the flavor scores of all three types of oils evaluated. For corn oil, the best correlation with flavor score was obtained with the C_5 peak. The soybean oil flavor score correlated best with the 178 C peak. The blended oil (a blend of different oils) was best correlated with the TV peaks, rather than with a single peak. The volatile content is the basic factor which characterizes oil flavor quality; since these volatiles can be detected by both taste panelists and the GC instrument, the TV peaks in the GC method apparently is the independent variable which was most consistent in giving high correlation with taste panel flavor scores for all three types of oil.

The linear regression plot of total volatiles in corn oils and taste panel flavor scores is shown in Figure 5. The standard error of the regression is an indication of how accurate a flavor score prediction can be made from the regression equation. The flavor scores used in the regression calculations were the taste panel means which were calculated from the sample being tasted by several different persons. The standard error of regression for the corn oil in this plot is 0.26, and the standard error of the mean score for the taste panel is 0.37. There is no statistical difference between these two values, indicating little likelihood that any large bias or lack of fit is left in the regression equation. Regression plots of total volatiles and taste panel flavor scores for soybean and blended oils are shown in Figures 6 and 7. The standard error of the regression for soybean oils is 1.19 and 0.54 for the blended oils. These values are higher than those obtained for corn oil. However, an estimate of standard error of the taste panels for these oils was not available; therefore, no conclusions on the adequacy of the regression equation can be drawn. Using the GC profile of volatiles as a basis for calculation, preliminary work indicates that the development of multiple linear regression equations will greatly enhance the statistical description of all oils examined.

Although more investigations with other oils are indicated, this GC technique appears to have excellent potential for flavor scoring vegetable oils, especially because objectivity, accuracy, and economy are inherent in instrumental analysis.

ACKNOWLEDGMENT

Gordon S. Fisher and Michael G. Legendre provided the mass spectral data.

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[Received December 5, 1975]