Dynamic Head Space Analyses of Orange Juice Flavor Compounds and Their Absorption into Packaging Materials

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ABSTRACT: A simple, rapid, sensitive, and reproducible dynamic headspace gas chromatography (DH-GC) was developed to study the absorption of d-limonene, α -pinene, ethyl butyrate, and octanal into laminated polymeric packaging materials containing low-density polyethylene, polyethylene terephthalate, polyvinylidene chloride, and ethylene-vinyl alcohol copolymers. The linear regression lines showed that the flavor compound peak areas were directly proportional to the concentrations of the standard flavor compounds with $R^2 \geq 0.97$. The coefficients of variation for the DH-GC analyses were less than 4%. A test cell was designed to study orange juice flavor absorption into the packaging materials for 28 d at 25 °C. Ethyl butyrate or octanal absorption was not different among the 4 packaging materials (P > 0.05). Addition of a polyethylene terephthalate and a ethylene-vinyl alcohol copolymer layer to the packaging materials reduced the d-limonene and α -pinene absorption by 20% and 50%, respectively.

Keywords: flavor absorption, orange juice, packaging materials, dynamic headspace gas chromatography, storage

Introduction

Orange juice is the most popular fruit beverage in the United States, accounting for over 60% of juice market revenue (Foley and others 2002). It is the most popular fruit juice because of its delicate and well-balanced citrus flavors. The d-limonene, α -pinene, ethyl butyrate, and octanal are the "top-note" orange flavor compounds, representing more than 90% of total flavor compounds in single-strength orange juice.

Conventional isolation and separation methods for volatile compounds include solvent extractions (Shimoda and others 1988; Moshonas and Shaw 1989; Ikegami and others 1991; Pieper and others 1992) and static headspace analyses (Nisperos-Carriedo and Shaw 1990; Shaw and others 1993). They are either very time-consuming, laborious, have low reproducibility, or have low detection sensitivity. Tatum and others (1975) reported that 5 consecutive solvent extractions were needed in analysis of single-strength orange juice. The relatively low detection sensitivity of the static headspace analysis limited its application in analyzing low concentrations and high boiling point volatile compounds. Dynamic headspace analysis, also known as the purge and trap method, is a gas extraction technique and has been used with foods (Park and Goins 1992; Yang and Min 1994). The dynamic headspace analysis does not use any solvents to avoid damaging the integrity of the volatile compounds of samples. In addition, its high reproducibility and sensitivity to very low concentration cannot be achieved by other techniques.

The flavor of fresh orange juice in polymeric packaging materials changes during storage, which makes orange juice less acceptable or unacceptable to consumers. The absorption of flavor compounds

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into packaging materials occurs when flavor compounds are directly in contact with packaging materials. A small loss of flavor compounds will lead to serious changes in flavor quality because flavor compounds in food exist in extremely small quantities. Flavor absorption is an important concern in food/package compatibility. The flavor absorption of d-limonene by laminated polymeric packaging materials, such as low-density polyethylene has been extensively investigated (Kwapong and Hotchkiss 1987; Mannheim and others 1987; Imai and others 1990; Pieper and others 1992). Sadler and Braddock (1991) showed that limonene, α-pinene, and ethyl butyrate were more absorbed by low-density polyethylene than octanal. A rapid d-limonene reduction of 40% to 60% within a week was observed in a model flavor solution containing strips of lowdensity polyethylene (Miltz and Mannheim 1986). Approximately 30% to 40% of d-limonene was reported to be absorbed by lowdensity polyethylene in 25 d at 20 °C (Halek and Meyers 1989).

Some researchers used model flavor solutions to study flavor absorption into food packaging materials (Mohney and others 1988; Baner and others 1991; Nielsen and others 1992). Orange juice is a complex food matrix system. Flavor absorption could be affected by pH, vitamins, minerals, as well as the concentration of flavor compounds in orange juice. Previous experiments of flavor absorption were generally designed with the direct exposure of 2 sides of packaging material surfaces to orange juice instead of 1 surface (Schwartz 1985; Kwapong and Hotchkiss 1987). The 1-side exposure may simulate flavor absorption into food packages more practically than the 2-side exposure. A better understanding of flavor absorption will be helpful in predicting what food flavor compounds may be absorbed into polymeric packaging materials and in selecting appropriate packaging materials for specific foods. The objectives of this study were (1) to develop a simple and reliable dynamic headspace gas chromatography method for qualitative and quantitative analyses of orange juice flavor compounds, (2) to study the flavor absorption into the packaging materials using the dynamic headspace gas chromatography method, and (3) to evaluate the effects of flavor

compounds, packaging materials, and thickness of packaging materials on the flavor absorption.

Materials and Methods

Samples and reagents

A premium pasteurized single-strength orange juice was purchased from a grocery store (Kroger, Columbus, Ohio, U.S.A.). The standard flavor compounds, d-limonene, α-pinene, ethyl butyrate, and octanal, were purchased from Sigma Chemical Co. (St. Louis, Mo., U.S.A.). Packaging materials were provided by Combibloc Inc. (Columbus, Ohio, U.S.A.). The compositions for packaging materials are listed in Table 1.

Dynamic headspace gas chromatography analysis

The isolation and separation of orange juice headspace flavor compounds by dynamic headspace gas chromatography (DH-GC) were essentially the same as the method of Yang and Min (1994). One milliliter of orange juice sample was transferred into a 20-mL serum vial containing a 5-mm magnetic spin bar. The sample vial was sealed with a Teflon-coated septum and aluminum cap (Supelco, Bellefonte, Pa., U.S.A.). The vial was magnetically stirred in a 30 °C water bath for 5 min and then purged with nitrogen gas for 0.5 min to remove the headspace volatile compounds from orange juice. The volatile compounds were absorbed on a 12-inch × 1/8inch inner dia Tenax TrapTM column in the LSC-2000 Tekmar Dynamic Headspace Analyzer (Tekmar Co., Cincinnati, Ohio, U.S.A.). Flow rates of nitrogen purge gas and carrier gas were 10 mL/min and 1 mL/min, respectively. The absorbed volatile compounds were dry-purged for 4 min to remove excessive moisture, thermally desorbed at 180 °C for 8 min, and then concentrated in the capillary interface, which was cryogenically cooled down to -150 °C by liquid nitrogen. The condensed volatile compounds were automatically injected at 180 °C onto a capillary column (DB-WAX, 30-mm × 0.25mm inner dia, 0.25-mm film thickness) in the gas chromatograph (HP 5890, Hewlett Packard, Wilmington, Del., U.S.A.) with a flame ionization detector. Temperature of the GC was held at 40 °C for 3 min, increased at 3 °C/min to 60 °C, and then increased at a rate of 8 °C/min to a final temperature of 200 °C and held for 10 min. The gas chromatographic peaks of orange flavor compounds were quantitatively determined by an integrator (HP 3390A, Hewlett Packard, Wilmington, Del., U.S.A.).

The flavor compounds were identified by comparing the retention times of authentic orange juice samples and authentic juice samples mixed with standard flavor compounds. The flavor compounds were quantified from calibration lines obtained by plotting the peak areas against the different concentrations of each standard compound in the deodorized orange juice. The reproducibility of flavor compound analyses by DH-GC was determined by analyzing the quantities of d-limonene, α -pinene, ethyl butyrate, and octanal in orange juice in quadruplicate.

Effect of purge time on the analysis of flavor compounds

The 20-mL serum vial containing 1-mL strength orange juice and a 5-mm magnetic spin bar was purged with nitrogen gas for 0.5, 1.0, or 2.0 min for the dynamic headspace gas chromatography analysis.

Preparation of deodorized orange juice by vacuum rotary evaporator

The flavor compounds of single-strength orange juice with 11.8°Brix were removed by a vacuum rotary evaporator. Five hundred grams of single-strength orange juice was concentrated from

Package	S/V ratio ^a (1/cm)	Structure (from outside to inside)
LDPEb 1	1.04	LDPE/Paper/PE ^c /Aluminum foil/
LDPE 2	0.60	Adhesive/Inner LDPE (1.85 mil) LDPE/Paper/PE/Aluminum foil/
PVdC ^d	1.04	Adhesive/Inner LDPE (2.45 mil) LDPE/Paper/PE/Aluminum foil/
1 VuO	1.04	Adhesive/Inner LDPE
PET ^e 1	0.60	(1.85 mil)/PVdC (0.17 mil) LDPE/Paper/PE/Aluminum foil/
PEI'I	0.60	Adhesive/Inner LDPE (2.45 mil)/
		PET (0.10 mil)
PET 2	0.60	LDPE/Paper/PE/Aluminum foil/ Adhesive/Inner LDPE (2.45 mil)/
		PET (0.17 mil)
EVOH	0.60	LDPE/Paper/PE/EVOHf (0.50 mil)/
		Adhesive/Inner LDPE (2.45 mil)

aSurface area of a packaging material to orange juice volume ratio. bLow-density polyethylene.

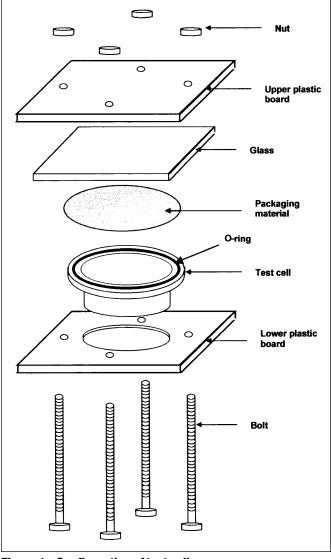


Figure 1 - Configuration of test cell

^cPolyethylene.

dPolyvinylidene chloride.

ePolyethylene terephthalate.

fEthylene-vinyl alcohol.

11.8° to 45°Brix by a vacuum Buchi R 110 rotary evaporator (Brink Mann Instrumental, Inc., Westbury, N.Y., U.S.A.). The concentrated juice with 45°Brix was designated as deodorized concentrated orange juice. The DH-GC analysis showed that the 45°Brix deodorized concentrated orange juice was practically volatile free.

Calibration lines of flavor compounds in orange juice

The d-limonene, α-pinene, ethyl butyrate, and octanal were selected to study the absorption of flavor compounds into packaging materials because of their important flavor contribution or quantity in orange juice. They also represent the families of hydrocarbons, esters, and aldehydes. The concentrations of d-limonene, α -pinene, ethyl butyrate, and octanal in the single-strength orange juice were measured in a preliminary study and they were 154, 1.12, 0.78, and 0.42 ppm, respectively. Different levels of d-limonene, α -pinene, ethyl butyrate, and octanal flavor standards were spiked into the 45°Brix deodorized juice. The d-limonene was added to deodorized orange juice to obtain 0, 50, 100, and 200 ppm. Similarly, 0, 0.5, 1.0, and 2.0 ppm α -pinene, 0, 0.25, 0.50, and 1.0 ppm ethyl butyrate 0, 0.25, 0.50, and 1.00 ppm octanal orange juices were prepared. The samples were mixed, sealed, and refrigerated. After 24 h of refrigeration, the 45°Brix juice containing d-limonene, α-pinene, ethyl butyrate, and octanal was diluted to a 11.8°Brix juice with distilled water. The 11.8°Brix is the value of single-strength orange juice. Calibration lines of d-limonene, α pinene, ethyl butyrate, and octanal were obtained by plotting the peak areas against the different concentrations of each standard compound in the deodorized orange juice.

Glass test cells for flavor absorption into packaging materials

Two special glass test cells were designed and manufactured to

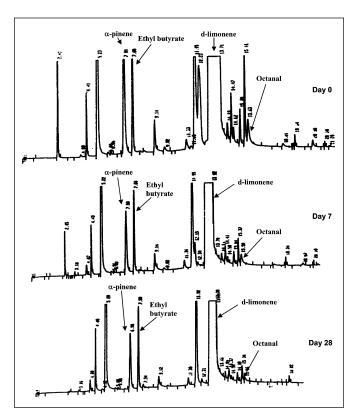


Figure 2 - Chromatograms of orange juice stored in low-density polyethylene (LDPE) 2 in day 0, day 7, and day 28

Table 2 - Reproducibility of essential orange flavor compounds from dynamic headspace gas chromatography (DH-GC)

Repli-	Total peak area (× 10 ⁷)	d- limonene (× 10 ⁷)	α- pinene (× 10 ⁵)	Ethyl butyrate (× 10 ⁵)	Octanal (× 10 ⁴)
1	3.94	3.94	3.10	1.24	4.83
2	4.01	3.72	3.15	1.17	4.62
3	4.11	3.80	3.34	1.21	4.69
4	4.12	3.82	3.29	1.18	4.56
Mean	4.04	3.82	3.22	1.18	4.67
SDa	0.09	0.09	0.12	0.03	0.11
CV (%)b	2.18	2.38	3.61	2.40	2.45

aStandard deviation bCoefficient of variation.

test the flavor absorption into packaging materials as shown in Figure 1. The test cells were designed to expose 1 side of a packaging material surface with orange juice. The small test cell had a surface/ volume ratio of 1.04, simulating a 250-mL package, whereas the large test cell had a ratio of 0.60, simulating a 1360-mL commercial package. Test cells were made of glass. A packaging material and a Viton O-ring (American Packing & Gasket Co., Houston, Tex., U.S.A.) were mounted on the top of the cell to seal the unit hermetically. A thick layer of glass was placed on the top of the packaging film to block the permeation of flavor compounds from the top. The glass test cell was tightly clamped by 2 plastic boards and sealed tightly with bolts and nuts.

Analysis of flavor compounds absorptions into packaging materials

The cell filled with orange juice was turned upside down to allow the packaging material to contact the orange juice sample. All test cells were shaken periodically and stored in the dark at 25 °C for 0, 1, 2, 4, 7, 14, and 28 d. The percentage of flavor absorption was calculated by the following equation:

% absorption = (Concentration of a flavor compound in the control glass cell -Concentration of the flavor compound in the test cell with a packaging material)/ (Concentration of a flavor compound in the control glass cell) × 100

where the control glass cell is the test cell without any packaging materials and covered with a glass cover.

Statistical analysis

Analysis of variance and Tukey's multiple comparisons method at the 5% significance level were conducted to determine significant differences. The entire analyses were duplicated with 4 measurements. Minitab 13.31 (Minitab, Inc., State College, Pa., U.S.A.) was used for all statistical analyses.

Results and Discussion

Dynamic headspace gas chromatography analysis

A typical gas chromatogram of orange juice is shown in Figure 2. DH-GC did not require long sample extraction before GC analysis. The DH-GC analysis took about 1 h for each run. The reproducibility for the DH-GC analysis of orange juice flavor compounds is shown in Table 2. The coefficients of variation (CV) for d-limonene, α -pinene, ethyl butyrate, and octanal were 2.38, 3.61, 2.40, and 2.45, respectively.

Sample size is one of the critical parameters to control optimum condition. Too much sample may contaminate and damage the capillary column, whereas too little sample may not have sensitive and reliable results. The low CV indicated that a 1-mL juice sample was enough for the quantitative analysis by DH-GC and the results were highly reproducible.

Effect of purge time

The effects of different purge time on the gas chromatographic peak area of d-limonene, α -pinene, ethyl butyrate, and octanal compounds are shown in Figure 3. The high regression coefficients of flavor compounds indicated that the peak area responses were linearly correlated to the purge time up to 2 min. The 0.5-min purge time per sample was proven to be good to analyze d-limonene, α -pinene, ethyl butyrate, and octanal flavor compounds. With longer purge time, larger amount of flavor compounds would cause cross-

contamination of samples and carryover problems within the instrument. Reducing the purge time for each sample eliminated the presence of interferences and reduced the amount water introduced to the chromatograph.

Deodorized orange juice

The primary factor of the headspace analysis of flavor compounds by dynamic headspace analysis is vapor pressures of volatile flavor compounds. The vapor pressure of volatile compounds is greatly influenced by sample matrices. In a preliminary study, dlimonene was added to a 10-mL deodorized orange juice or 10 mL of distilled water to determine the effects of the orange juice matrix on the dynamic headspace analysis. The 20 ppm d-limonene of deodorized orange juice or distilled water was analyzed by DH-GC. The GC peak areas of 20 ppm d-limonene from deodorized orange juice and distilled water by DH-GC were quite different. The result

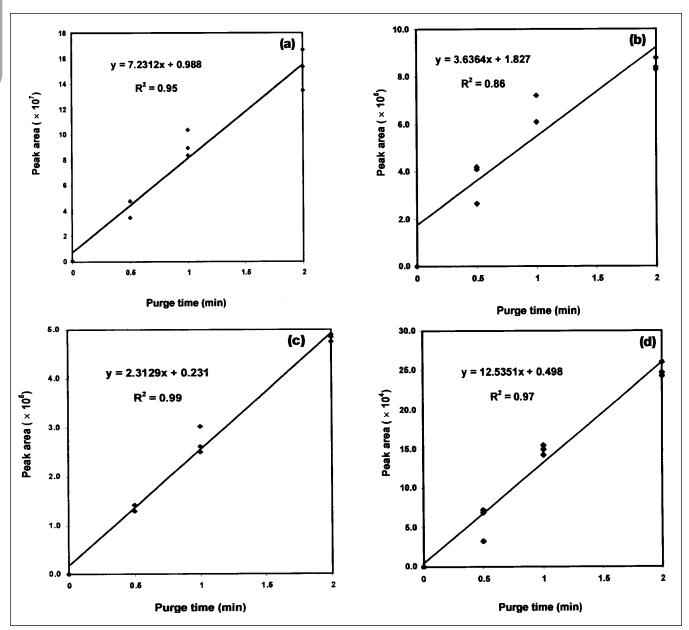


Figure 3 – Effects of different purge times on the gas chromatographic peak areas of (a) d-limonene, (b) α -pinene, (c) ethyl butyrate, and (d) octanal

Table 3—Regression equations between the gas chromatography (GC) peak area (electronic counts^a) and the concentration of the flavor compounds (ppm)

Compound	Regression equation	R²	Concentration range (ppm)
d-limonene	$Y = 0.0204X_1 + 0.372$	0.97	0 to 200
α -pinene	$Y = 0.1897X_{2}^{'} + 1.540$	0.99	0 to 2.0
Ethyl butyrate	$Y = 1.2812X_{3}^{2} - 1.098$	0.98	0 to 1.0
Óctanal	$Y = 0.1041X_1^3 - 0.211$	0.99	0 to 1.0

aX₁ = electronic counts of GC peak area (x 10⁻⁷); X₂ = electronic counts of GC peak area (x 10⁻⁴); X₃ = electronic counts of GC peak area (x 10⁻³); Y = compound in parts per million.

from this preliminary study showed that the sample matrix affected the vapor pressure of volatile compounds and the sensitivity of headspace analysis by DH-GC. For the quantitative analyses of dlimonene, α-pinene, ethyl butyrate, and octanal in orange juice by DH-GC, the calibration lines of these compounds were obtained by adding different contents of standard compounds to deodorized orange juice instead of adding to distilled water. The deodorized orange juice prepared by rotary evaporator was practically volatile free according to the DH-GC analysis.

Calibration lines of the flavor compounds

Calibration linear regressions of d-limonene, α-pinene, ethyl butyrate, and octanal are shown in Table 3. Linear relationships between GC peak areas and the concentrations of standard compounds had $R^2 \ge 0.97$ for 4 compounds. The linear regression lines showed the GC peak area responses were directly proportional and highly correlated to the concentration of the flavor compounds. The GC peak areas could be used to obtain the concentration of flavor compounds using the calibration lines. The high correlations between the GC peak areas and the compound concentrations also indicated the deodorized orange juice was capable of forming a

stable matrix with the standard compounds to produce a reproducible DH-GC analysis.

Flavor compounds absorptions into packaging materials during storage

The concentrations of d-limonene in the control glass test cell and the packaging test cell of low-density polyethylene (LDPE) 2 during storage for 28 d are shown in Figure 4. The concentration of d-limonene in the control glass cell did not significantly change for 28 d (P > 0.05). The concentration of d-limonene in the test cell of LDPE 2 decreased continuously for up to 20 d and leveled off there-

The flavor compounds would have been lost from juice products in 3 ways: absorption, permeation, and degradation. Permeation was minimized in this research by addition of a thick glass piece over the packaging material. Degradation and permeation effects were accounted for by comparing the concentrations of the flavor compounds from the test cell with the packaging material and the control glass cell without the packaging material. The concentration

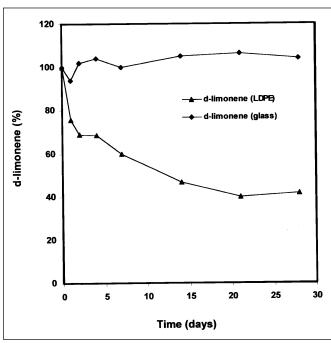


Figure 4-Relative % of d-limonene in the control glass test cell and the test cell of low-density polyethylene (LDPE) 2 during storage for 28 d at 25 °C

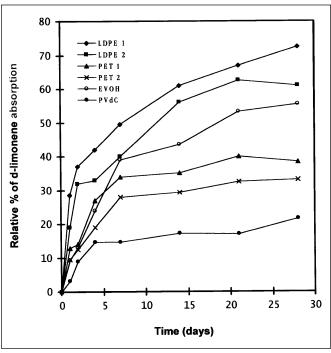


Figure 5 - Relative % of d-limonene absorption in low-density polyethylene (LDPE) 1 (♦), LDPE 2 (■), polyethylene terephthalate (PET) 1 (▲), PET 2 (×), ethylene-vinyl alcohol (EVOH) (○), and polyvinylidene chloride (PVdC) (●) packaging materials during storage for 28 d at 25 °C

Table 4—Mean values of relative % absorption of the orange juice flavor compounds in the packaging materials after 28 d storage at 25 °C^a

	Mean % absorption Flavor compounds ^a				
_					
Package	d-limonene	α-pinene	Ethyl butyrate	Octanal	
LDPE 1	$70.4 \pm 4.3 \text{ c}$	70.4 ± 2.6 d	18.2 ± 2.8 a	29.4 ± 6.4 a	
LDPE 2	65.5 ± 3.8 c	65.2 ± 2.9 d	16.1 ± 2.1 a	28.8 ± 2.7 a	
PET 1	41.1 ± 3.5 b	35.1 ± 4.5 b	$20.0 \pm 4.2 a$	$30.4 \pm 2.6 a$	
PET 2	$32.7 \pm 4.4 \text{ ab}$	$30.6 \pm 0.6 b$	16.8 ± 1.0 a	27.6 ± 3.9 a	
EVOH	$55.6 \pm 2.7 \text{ c}$	$56.5 \pm 0.7 \text{ c}$	16.4 ± 2.3 a	25.5 ± 3.4 a	
PVdC	20.2 ± 3.9 a	19.7 ± 1.8 a	$20.5 \pm 2.4 a$	$27.0 \pm 2.2 a$	

aMeans with the same letter within the same column are not significantly different (*P* > 0.05). EVOH = ethylene-vinyl alcohol; LDPE = low-density polyethylene; PET = polyethylene terephthalate; PVdC = polyvinylidene chloride.

of d-limonene in the control glass cell was constant under the identical storage conditions. Therefore, the decrease of d-limonene in the test cell of LDPE 2 during storage is considered to be the absorption of d-limonene into LDPE 2.

The absorptions of d-limonene, α -pinene, ethyl butyrate, and octanal absorption into LDPE 1, LDPE 2, PET 1, PET 2, EVOH, and PVdC are shown in Figure 5 through 8, respectively. A rapid loss of volatile flavor compounds was shown at the early storage period of 1 to 7 d for all packaging materials. Table 4 lists mean values of the flavor compound absorption in the packaging materials after 28 d storage.

Ethyl butyrate and octanal

The statistical results showed no significant differences (P > 0.05) in ethyl butyrate absorption and octanal absorption among all packaging materials in this study. None of the packaging materials used in this experiment significantly affected the extent of ethyl butyrate and octanal absorption. Ethyl butyrate showed the lowest

absorption rate into the packaging materials. The results agreed with the conclusion of Pieper and others (1992) that absorption generally decreased from hydrocarbons, ketones, and aldehydes to alcohols and esters. Greater absorption rates were found if flavor compounds have the similar chemical structure, or similar polarity as the functional group of packaging materials (Landois-Garza and Hotchkiss 1987).

Only d-limonene and α -pinene absorption will be considered in the following discussion.

d-Limonene and α-pinene

Significant amounts (P<0.01) of d-limonene and α -pinene were absorbed by LDPE compared with the other materials. This may be expected because the greater the similarity of structure, functionality, and polarity of the flavor compounds to the packaging materials, the greater the extent of flavor absorption (Landois-Garza and

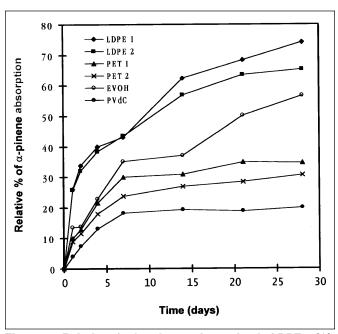


Figure 6—Relative % of α -pinene absorption in LDPE 1 (\blacklozenge), LDPE 2 (\blacksquare), PET 1 (\blacktriangle), PET 2 (x), EVOH (\bigcirc), and PVdC (\spadesuit) packaging materials during storage for 28 d at 25 °C

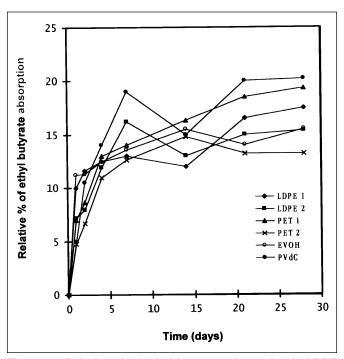


Figure 7—Relative % of ethyl butyrate absorption in LDPE 1 (♦), LDPE 2 (■), PET 1 (▲), PET 2 (×), EVOH (○), and PVdC (●) packaging materials during storage for 28 d at 25 °C

Hotchkiss 1987). LDPE absorbed more d-limonene and α -pinene probably because terpene hydrocarbons are nonpolar compounds and their lipophilic nature enables them to possess a stronger affinity for the nonpolar hydrocarbons of the low-density polyethylene layer. On the other hand, the greater absorption of d-limonene into LDPE may be because of low-density polyethylene layer's amorphous nature and lower crystallinity.

Individual comparison of packaging materials according to their composition structures is divided into the following 4 groups:

Comparison of LDPE 1 and LDPE 2

Data were normalized as the relative percentage of absorption by LDPE 1 and LDPE 2 per unit volume of LDPE and per unit volume of orange juice for direct comparison because LDPE 1 and LDPE 2 had different area/volume ratios and film thickness (normalized data not shown). Statistical results showed that the absorptions of d-limonene and α -pinene into LDPE 1 and LDPE 2 were not significantly different from each other (P > 0.05).

Comparisons of LDPE 2 with PET 1 and PET 2

The polyethylene terephthalate layers in PET 1 and PET 2 significantly reduced (P < 0.01) the absorption of d-limonene and α pinene, which could be noticed when those absorption values are compared with the value of LDPE 2. However, no statistical difference was found between the differences between PET 1 and PET 2 and between PET 2 and LDPE 2 (P > 0.05). There was also no significant difference of d-limonene and α-pinene absorption between PET 1 and 2 (P > 0.05). The difference in thickness (0.07 mil) between the 2 PET packaging materials did not have significant effects on the d-limonene and α -pinene absorption.

Comparison of PVdC and LDPE 1

PVdC had the same structure as LDPE 1 except for an additional polyvinylidene chloride (0.17 mil) layer in the inner side. Experi-

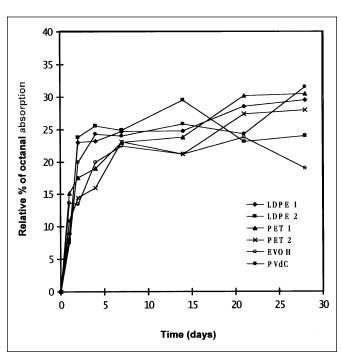


Figure 8-Relative % of octanal absorption in LDPE 1 (♦), LDPE 2 (■), PET 1 (▲), PET 2 (×), EVOH (○), and PVdC packaging materials during storage for 28 d at 25 °C

mental data showed that the additional film significantly reduced the absorption of d-limonene and α -pinene by over 50% (P < 0.05). PVdC was proved to be a superior barrier to the flavor compounds of orange juice.

Comparison of EVOH and LDPE 2

LDPE had a barrier layer of aluminum foil, whereas EVOH has a barrier layer of 0.5-mil-thick ethylene-vinyl alcohol copolymers (Table 1). The statistical analysis result showed that d-limonene and α -pinene absorption in EVOH were not significantly different from that in LDPE 2 (P > 0.05). This may be because of the fact that both LDPE and EVOH had a low-density polyethylene layer as the innermost contact layer.

Conclusions

simple, rapid, sensitive, and reproducible dynamic headspace Agas chromatography technique has been developed to determine the absorption of orange juice flavor. There was a high linear regression relation between the flavor response and the purge time. The 0.5-min purge time and 1-mL sample size were used for the DH-GC analysis. The standard calibration curves for d-limonene, α pinene, ethyl butyrate, and octanal could be used to determine the concentration of flavor compounds by analyzing their gas chromatographic peak areas.

The d-limonene and α -pinene were more absorbed by LDPE and EVOH than other packaging materials because of the nonpolar nature of the flavor compounds and the packaging materials. There were no significant differences between the 4 packaging materials with respect to ethyl butyrate or octanal absorption. An additional polyethylene terephthalate layer or polyvinylidene chloride layer added to LDPE significantly reduced the d-limonene and α -pinene absorption by at least 20% and 50%, respectively. The thickness decrease in the polyethylene terephthalate layer from 0.17 mil to $0.10\ mil\ did\ not\ affect\ d\text{-limonene}$ and $\alpha\text{-pinene}$ absorption.

Acknowledgments

Research support provided by Combibloc Inc., Columbus, Ohio, is gratefully acknowledged. References to commercial products and trade names are made with the understanding that no discrimination or endorsement by The Ohio State Univ. is implied.

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