

# Influence of Storage Time and Temperature on Absorption of Flavor Compounds from Solutions by Plastic Packaging Materials

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**ABSTRACT:** Linear low-density polyethylene (LLDPE), oriented polypropylene (OPP), polycarbonate (PC), polyethylene terephthalate (PET film and PET bottle), and polyethylene naphthalate (PEN) were stored in a model solution containing 10 flavor compounds at 4, 20, and 40 °C and flavor absorption by the plastic materials was followed in time. The absorption rate and/or total amount absorbed increased considerably with temperature from 4 to 40 °C. Depending on storage temperature, total flavor absorption by the polyolefins (LLDPE and OPP) was 3 to 2400 times higher than by the polyesters (PC, PET, and PEN). Therefore, in the factor of flavor absorption, polyesters are preferred over polyolefins as packaging material.

**Keywords:** flavor, absorption, plastic, storage, packaging

## Introduction

PACKAGING MATERIALS ARE USED EXTENSIVELY to protect and preserve food products in storage and distribution environments. Food products may undergo loss of quality due to failure of the package and/or product-package interactions. Product-package interactions can be defined as an interplay between product, package, and the environment, which produces an effect on the product and/or package (Hotchkiss 1997). Some decades ago, pioneering research about interactions between flavor compounds and polymer films was reported (Salame and Steingiser 1977; DeLassus and Hilker 1987; Salame 1989). As plastic packaging is increasingly used in direct contact with foods, absorption of flavor compounds is becoming an important product-package interaction aspect. Flavor absorption may alter the aroma and taste of a product (Kwapong and Hotchkiss 1987), or change the mechanical properties of polymers, such as tensile strength (Tawfik and others 1998) and permeability (Hirose and others 1988). Flavor absorption extent is influenced by the properties of the polymer, the flavor molecules, and also external conditions. The chemical composition, chain stiffness, morphology, polarity, and crystallinity of the polymer influences flavor absorption, as does chemical composition, concentration, and polarity of the flavor compounds, as well as the presence of other chemical compounds. External factors such as storage duration, relative humidity, temperature, and the presence of other food components can also affect solubility of aroma com-

pounds in a polymer (Nielsen and others 1992; Leufvén and Hermansson 1994; Fayoux and others 1997; Johansson and Leufvén 1997; Van Willige and others 2000a, b).

The most widely used polymers for food packaging applications are the polyolefins, such as polyethylene (PE) and polypropylene (PP). Polyolefins are used as an interior lining in box-type containers for beverages because of their good heat sealability and excellent moisture resistance. However, low-molecular-weight compounds (especially apolar compounds, as in most flavor substances) are readily absorbed (Johansson 1996). The use of plastic bottles, particularly polyethylene terephthalate (PET) bottles for carbonated beverages, is increasing steadily. PET is a relatively good barrier against permeation of gases and flavor compounds, due to the biaxial orientation of the molecules (Van Lune and others 1997). As a relatively new member of the polyester family, polyethylene naphthalate (PEN) has excellent performance characteristics due to its high glass-transition temperature ( $T_g$ ). In comparison to PET, PEN provides approximately 5 times the barrier for carbon dioxide, oxygen, or water vapor transmission. PEN also provides better performance at high temperatures than PET, allowing hot-fill, rewash, and reuse. However, the cost of PEN is about 3 to 4 times that of PET (Newton 1997). PEN likely would be used for niche markets such as beer (Goodrich 1997), where the superior barrier properties of PEN may win out over other choices, de-

spite PEN's higher cost. A few years ago, a reusable polycarbonate (PC) bottle was successfully introduced by the Dutch dairy industry. These bottles take advantage of their toughness (breakage resistance) and transparency (visibility of contents). The fact that PC is much lighter than glass provides fuel savings in rolling and carrying, as well as productivity improvements, since several bottles can be handled at once. The disadvantages of PC are its high cost and poor gas barrier properties (Mihalich and Baccaro 1997). Several investigations have shown that PE and PP can absorb considerable amounts of flavor compounds. However, less information is available in the literature about the amount of flavor absorption by PET, PEN, and PC. Our objective therefore was to investigate the influence of temperature and storage time on the amount of flavor absorption by LLDPE, PP, PC, PET, and PEN.

## Materials and Methods

### Materials

Polymer packaging films used were linear low-density polyethylene (LLDPE) Dowlex 5056E from Dow Benelux NV, Terneuzen, The Netherlands; oriented polypropylene (OPP) Bior® MB200 from Mobil Plastics Europe, Kerkrade, The Netherlands; polycarbonate (PC) Lexan® 8B35 from General Electric Plastics, Bergen op Zoom, The Netherlands; polyethylene terephthalate (PET) Melinex® 800 from DuPont Teijin Films, Luxembourg; polyethylene naphthalate (PEN) Kaladex® 1000 from DuPont Polyes-

ter Films, Wilton, Middlesbrough, U.K.). Oriented PET bottles, supplied by Schmalbach-Lubeca in Bierre, France, were also studied. Characteristics of the polymers used in this study are listed in Table 1.

Decanal, hexanal, 2-nonanone, octanol, and (R)-carvone were purchased from Merck Co., in Darmstadt, Germany; the hexyl acetate (HA) and myrcene from Aldrich Chemical Co. in Milwaukee, Wis., U.S.A.; the linalool and ethyl 2-methylbutyrate (E2MB) from Acros Organics (Fisher Scientific UK Ltd., Loughborough, U.K.), and (+)-limonene from Sigma Chemical Co. (St. Louis, Mo., U.S.A.). The aroma compounds were selected upon differences in functional groups, polarity, and absorption affinity in the different polymers. Characteristics of the flavor compounds are listed in Table 2. Log P represents the hydrophobicity of a flavor compound; a higher Log P means a more hydrophobic compound.

### Preparation of model flavor solutions

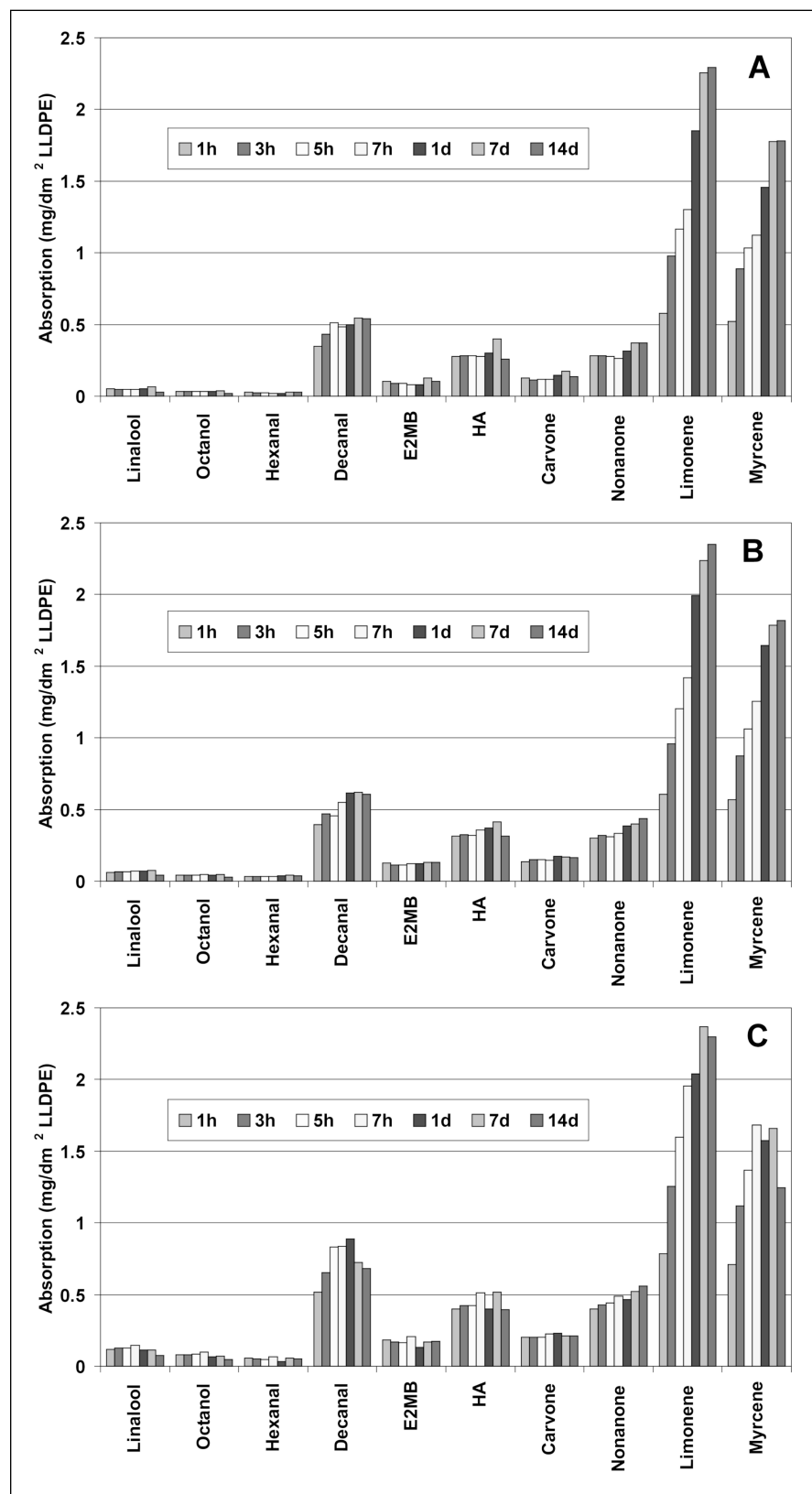
At  $t = 0$ , mixtures of the 10 flavor compounds were freshly prepared by dissolving the flavor compounds (each 100  $\mu\text{L/L}$ ) in 6 g/L aqueous Tween 80® (pH =  $4.2 \pm 0.2$ ) from Merck. The Tween 80® was used as an emulsifier to disperse the flavor compounds in the aqueous phase. Sodium azide (Merck) was added at a concentration of 0.2 g/L to prevent microbial growth. Flavor compounds were added using a micropipette which was equipped with a glass capillary tube (Socorex, Lausanne, Switzerland). An Ultra Turrax T25 (IKA-Labortechnik, Staufen, Germany) was used for homogenization for 2 min at 9500 rpm.

### Exposure conditions

Strips of LLDPE (1.5 x 2.0 cm), OPP (1.5 x 2.0 cm), PC (1.5 x 10.0 cm), PET (1.5 x 20.0 cm), PEN (1.5 x 20.0 cm), and PET bottle (1.0 x 10.0 cm, cut from the middle part of the bottle) were individually placed into 15-mL Teflon screw-cap vials (Supelco, Bellefonte, Penn., U.S.A.), then fully immersed in the model solution (15 mL). Due to the low absorption values of PC, PET, and PEN, it was necessary to increase the strip size of these polymers. Samples and model solution without strips (control) were stored in the dark at 4, 20, and 40 °C. LLDPE and OPP strips were in contact with the model solution for 1, 3, 5, 7 h and 1, 7, and 14 d. Due to their low absorption rate, PC, PEN, and PET were exposed to the model solution for 7, 14, 21, and 28 d. In preliminary experiments no significant edge absorption effect was found for the investigated flavor com-

pounds. Strips and model solutions were analyzed using Large Volume Injection Gas

Chromatography (LVI-GC) and static headspace GC, respectively.



**Figure 1—Absorption of flavor compounds by LLDPE ( $\mu\text{g}/\text{dm}^2$ ) after different storage times at (A) 4 °C, (B) 20 °C and (C) 40 °C**

### LVI-GC 'in vial' extraction of the polymer strips

After exposure the strips were removed

from the model solution, rinsed with ethanol for 10 s, and thoroughly wiped with paper tissue to remove any model solution ex-

cess. The strips were cut in small pieces and immediately placed into 10-mL vials containing 5 mL n-hexane (Enviroscan®; Labscan, Dublin, Ireland), or 5 mL of a 2:1 mixture of n-pentane: dichloromethane (Labscan). The choice of extraction solvent was based on extraction time and efficiency. The vials were tightly closed with a Teflon®-silicone seal and an aluminum crimp cap. In-vial extraction was carried out for 60 min in an ultrasonic bath (Ultrawave, Cardiff, U.K.). Longer ultrasonic treatment did not achieve better extraction. Recovery values (polymer + solution) of all flavor compounds were in the range of 95 to 102% after a day's exposure. GC analysis was performed using a LVI-GC system (Ultra Trace™ from Interscience, Breda, The Netherlands), as described in a previous paper (Van Willige and others 2000a). The LVI-GC conditions and extraction solvents that were used are listed in Table 3. Helium was used as carrier gas at a constant flow of 2.3 mL/min. Calibration curves ( $r^2 > 0.997$ ) were established for each component with the external standard method. A relative standard deviation (RSD) of less than 10% was found between triplicate determinations. To enable a direct comparison of results between the polymer samples having a difference in thickness and exposed area, concentrations of flavor compounds found in the extracts were converted to surface-related values ( $\text{mg}/\text{dm}^2$  or  $\mu\text{g}/\text{dm}^2$ ), taking double-sided exposure of the strips into account.

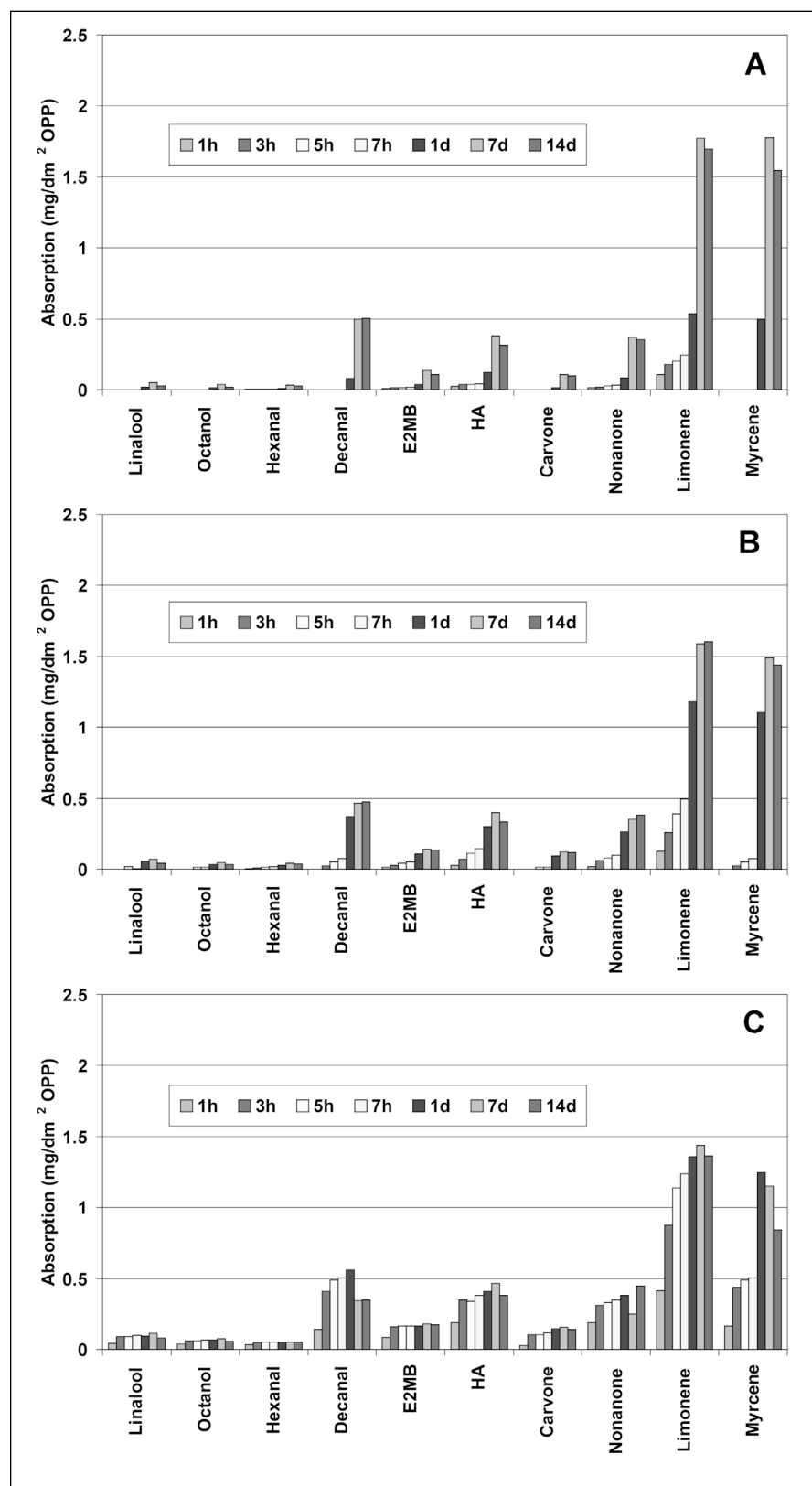
### Static headspace GC extraction of the model solutions

Besides absorption of flavor compounds by packaging materials, flavor changes in the model solution can also be induced by other factors as well (for example, degradation of flavor compounds due to storage or higher temperatures). Because such reactions can influence the absorption behavior, it was necessary to determine the remaining quantity of flavor compounds in the model solutions. The concentration of flavor compounds in 100  $\mu\text{L}$  of model solution was calculated from the partition coefficients (headspace/model solution) of each flavor compound. These were determined at  $t = 0$  and after each exposure period, using static headspace GC. The calculation method, equipment, and GC conditions which were used have been described in a previous paper (Van Willige and others 2000a).

## Results and Discussion

### Flavor absorption by LLDPE and OPP

Summarized in Figure 1 and 2 are the values of the 10 flavor compounds absorbed



**Figure 2—Absorption of flavor compounds by OPP ( $\mu\text{g}/\text{dm}^2$ ) after different storage times at (A) 4 °C, (B) 20 °C and (C) 40 °C**

by LLDPE and OPP film during 14 d of storage at 4, 20, and 40 °C. In general, more than 75% of the total amounts absorbed after 14 d were in fact absorbed during the 1st

day of storage. OPP stored at 4 °C absorbed only 30% of the total amounts absorbed after 14 d during the 1st day of storage. This exception was probably due to the small dif-

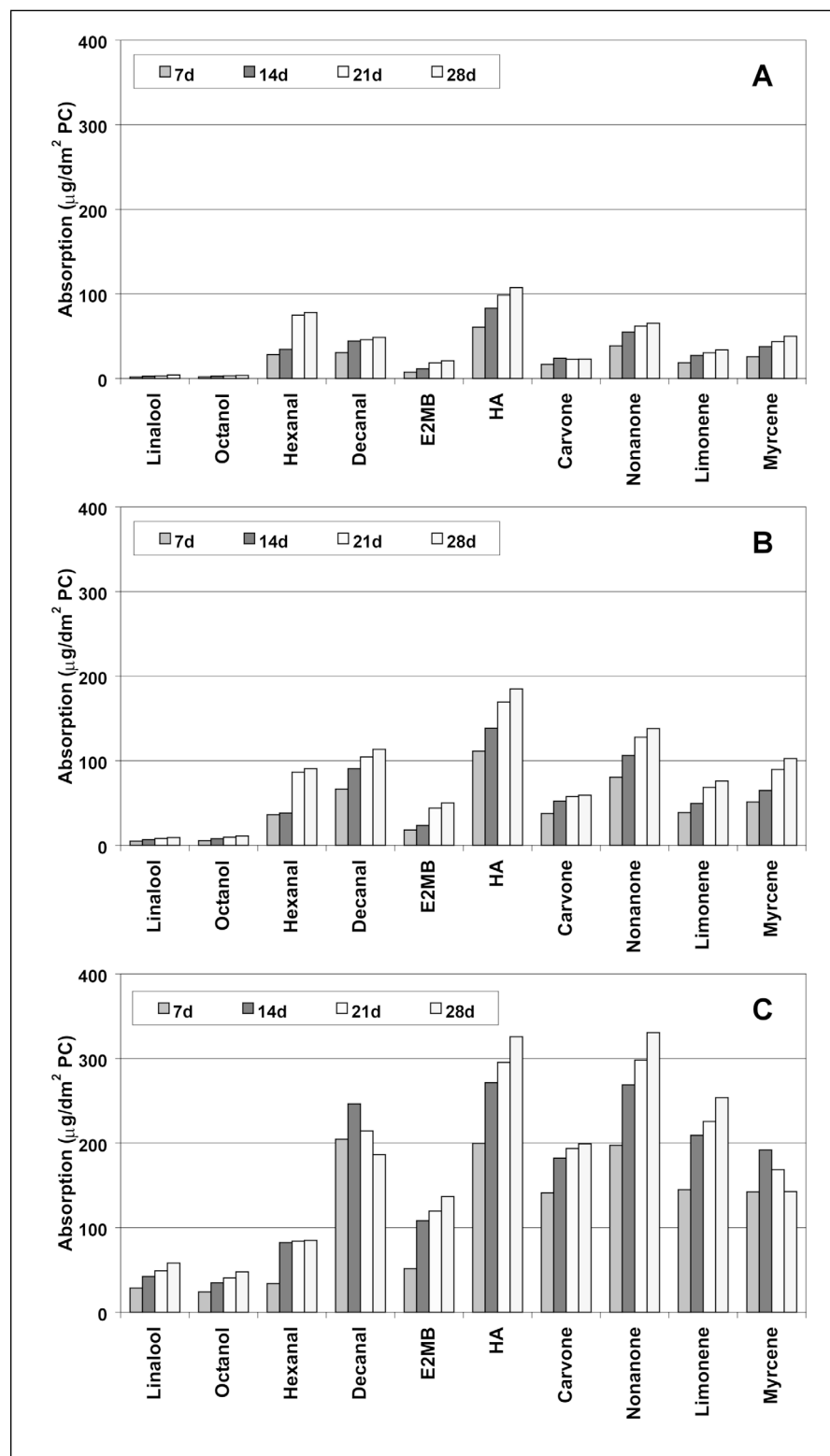
ference between the storage temperature and the glass transition temperature of OPP (further discussed in the paragraph about storage temperature's influence).

Flavor absorption by LLDPE and OPP reached equilibrium on d 7 for most of the flavor compounds. LLDPE and OPP easily absorbed limonene (2.37 and 1.77 mg/dm<sup>2</sup>) and myrcene (1.82 and 1.78 mg/dm<sup>2</sup>), followed by decanal, hexyl acetate, and nonanone. E2MB, carvone, linalool, octanol, and hexanal were absorbed in the smallest quantities. The absorption behavior of different classes of flavor compounds depends to a great extent on their polarity. Different plastic materials have different polarities; hence their affinities toward flavor compounds may differ from each other (Gremli 1996). In the present study, observed differences in absorption by LLDPE and OPP (both apolar) follow the inverse order of the flavor compounds' polarity (Table 1), according to the rule that "like dissolves like". A similar trend was reported for OPP by Lebossé and others (1997). Exceptions to this rule were the 2 alcohols (linalool and octanol), which were absorbed in smaller quantities than the more polar flavor compounds E2MB, HA, and carvone. This was probably due to structural differences or to the capability of alcohols to form hydrogen bonds in the aqueous phase. The effect of polarity was also observed by comparing the absorption behavior of limonene and carvone. These flavor molecules have similar structures, but limonene is an apolar terpene while carvone is an oxygenated polar terpene. Due to this difference in polarity, limonene was absorbed in larger quantities than carvone.

Absorption of the aldehydes also was related to their structures (that is, the length of the carbon chain). The shorter chain C-6 aldehyde hexanal was absorbed less than the C-10 aldehyde decanal. With increasing carbon chain length the polarity decreases and consequently, the absorption increases. Shimoda and others (1988) reported that in a homologous series of saturated aldehydes (hexanal through dodecanal), the partition coefficient (plastic/solution) increased with the molecular weight, indicating an increase in absorption. The difference in absorption behavior of the esters E2MB and hexyl acetate also suggests an influence of the carbon chain length and thus, polarity. Strandburg and others (1990) showed that this was the case for absorption of linear esters by different polymer films.

#### Flavor absorption by PC, PET, and PEN

Figure 3 through 6 show the absorption



**Figure 3—Absorption of flavor compounds by PC ( $\mu\text{g/dm}^2$ ) after different storage times at (A) 4 °C, (B) 20 °C and (C) 40 °C**

values of the flavor compounds by PC, PET (film and bottle), and PEN during 28 d of storage at the 3 test temperatures. These 4 polymer samples showed a different ab-

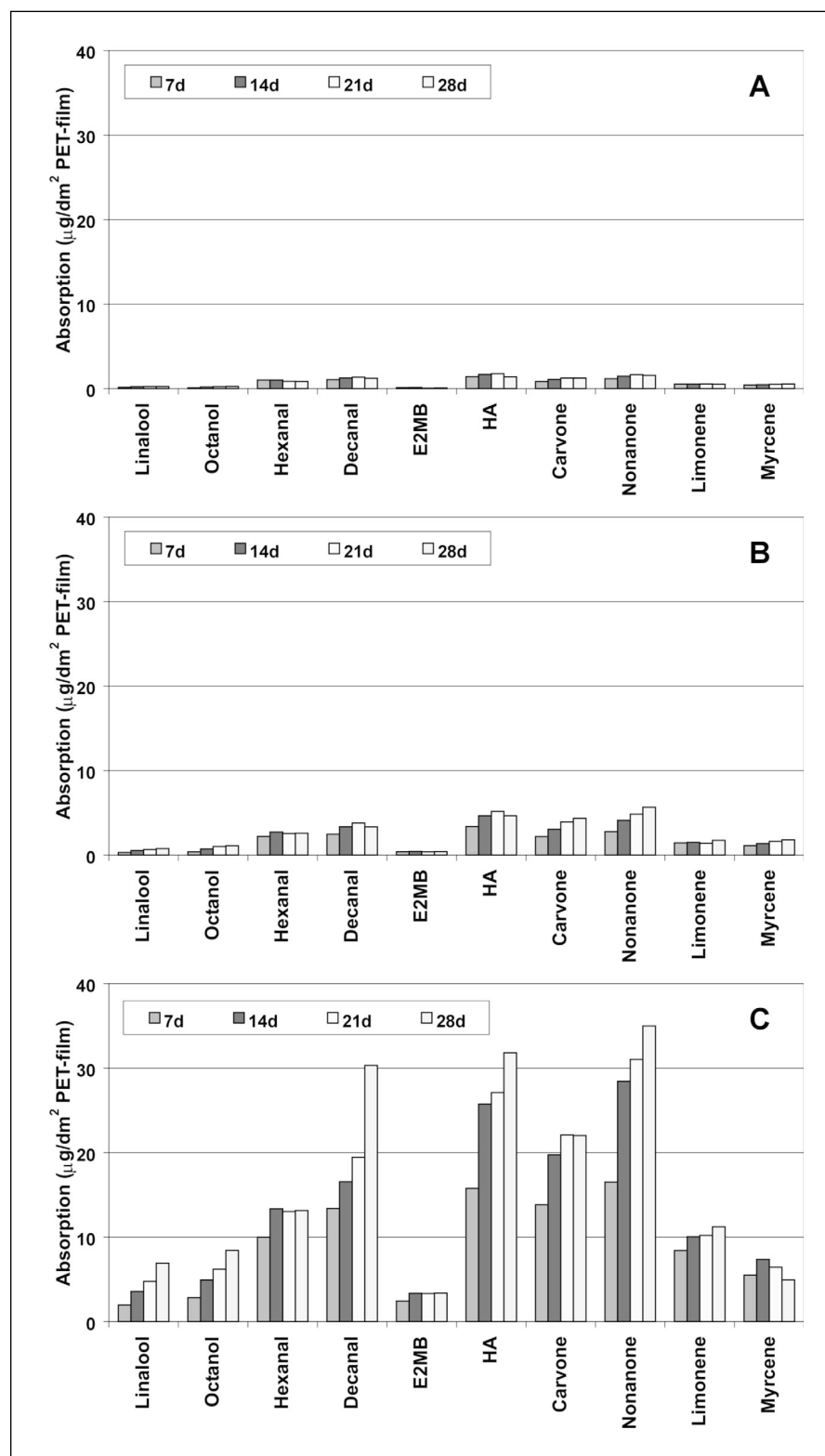
sorption behavior for the 10 flavor compounds, compared with LLDPE and OPP. Hexyl acetate and nonanone were the most readily absorbed, followed by decanal and

carvone. Due to structural differences and the more polar character of PC, PET, and PEN, the apolar terpenes limonene and myrcene were absorbed in smaller quantities than the aforementioned flavor compounds. For most of the flavor compounds, absorption continued during the entire period of storage. The thickness of the polymers and/or the slow absorption rate might explain why a stable value was not reached as rapidly as for LLDPE and OPP. Nielsen (1994) showed that the absorption equilibrium of limonene and myrcene by PET was not achieved even after 12 wk of storage at 25 °C. Major differences were also found between amounts of flavor compounds absorbed by the different polymers. Depending on storage temperature, total flavor absorption by LLDPE and OPP was 3 to 2400 times higher than by the polymers PC, PET, and PEN. This difference was considered attributable to the difference in  $T_g$  between the materials (Table 1). LLDPE and OPP were in the rubbery state at the investigated temperatures, consequently having high diffusion coefficients for flavor compounds. The time to reach steady-state is established quickly in such structures. The glass transition temperatures of PC, PET, and PEN are much higher than the test temperatures, meaning that these polymers were in the glassy state. These glassy polymers have very low diffusion coefficients for flavor compounds (Yamada and others 1992; DeLassus 1996; Giacini and Hernandez 1997). Yamada and others (1992) concluded from this result that absorption of flavor compounds could be reduced if the glass transition temperature of the polymer is much higher than the storage temperature. Difference in glass transition temperature might also explain the difference in absorbed flavor quantities between PET and PEN.

Although the  $T_g$  of PC was much higher than the  $T_g$  of PET and PEN, absorption of flavor compounds by PC was much higher than by PET and PEN. This was attributed to the lack of crystalline regions in PC, which is a totally amorphous polymer. Letinski and Halek (1992) showed that amorphous regions in a polymer have a higher affinity for flavor compounds than crystalline regions. Therefore, PC will exhibit more flavor absorption than the semi-crystallines PET and PEN. The difference in thickness between the PET bottle and PET film samples was probably responsible for the larger flavor absorption values found in the PET bottle strips.

#### Influence of storage temperature on flavor absorption

All investigated polymers showed an in-



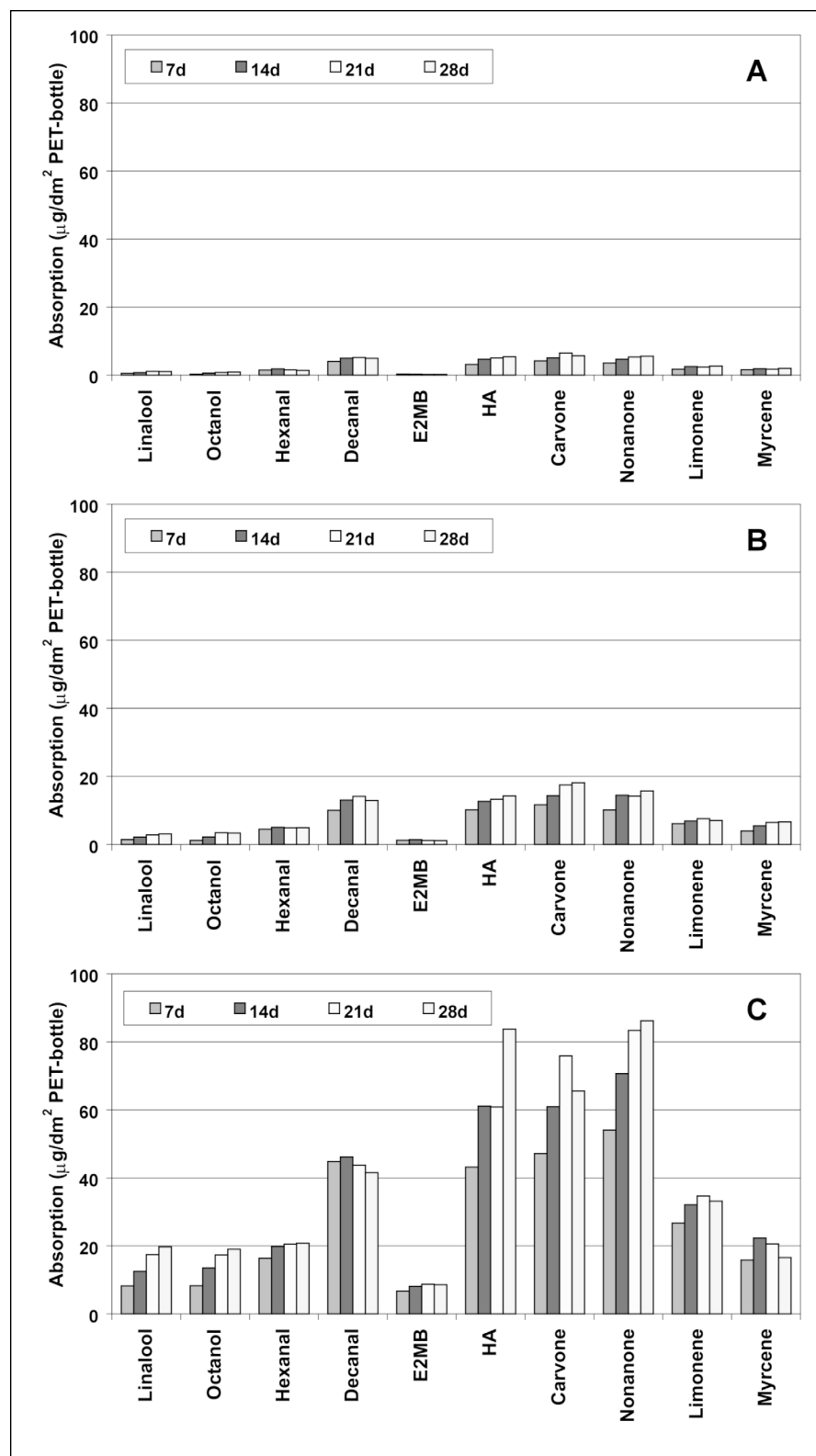
**Figure 4—Absorption of flavor compounds by PET-film ( $\mu\text{g}/\text{dm}^2$ ) after different storage times at (A) 4 °C, (B) 20 °C and (C) 40 °C**

creased absorption rate at higher storage temperatures. LLDPE and OPP absorption equilibriums were reached quicker due to a

more rapid diffusion process at higher temperatures. Nielsen and others (1992) also reported that temperature affected the ab-

sorption of flavor compounds by LDPE significantly. They too found a higher flavor absorption level; approximately twice as much was absorbed at 75 °C compared with 5 °C. They suggested that this increase was from the greater mobility of the molecules, or the swelling of the polymer at higher temperatures, creating more space for solvation of the flavor molecules. In the present study, both higher flavor absorption levels and rates associated with increasing storage temperatures were only found for PC, PET, and PEN. A combination of faster diffusion process and higher equilibrium constant (polymer/solution) at the higher storage temperatures resulted in a higher amount of flavor absorption. Van Lune and others (1997) showed that absorption of toluene and methanol by PET and PEN bottles increased with rising temperatures, partly due to an increase in the diffusion coefficient of the contaminants with increasing temperature. They also suggested that the crystallinity of PET decreased while the free volume increased at higher temperatures, resulting in molecules' easier absorption. In the study of Tawfik and others (1998), it was reported that PET stored for 15 d at 37 °C in a model solution containing 320 ppm limonene absorbed 7 times more limonene than when stored at 5 °C, but only 4 times more after 45 d. They concluded that the diffusion process was temperature-dependent, as could be expected from the slower rate at a lower temperature.

With the exception of decanal absorption by PET film, absorbed quantities of decanal and myrcene decreased during prolonged storage, after reaching a maximum absorption level at 40 °C (Figure 1c to 6c). Apparently, decanal and myrcene desorbed from the polymers to the model solution. Figure 7 shows the influence of storage time and temperature on decanal's and myrcene's concentrations in a model solution without a polymer sample. At 40 °C the concentration of decanal and myrcene in the model solution (determined with static headspace analysis) decreased by 64% and 71%, respectively, due to degradation of decanal and myrcene. This degradation process caused a desorption of decanal and myrcene from the polymer samples in order to re-establish the equilibrium between polymer and solution. With increasing storage time and temperature, degradation of aldehydes (octanal, decanal, and citral) was also observed in orange juice by other researchers (Dürr and others 1981; Moshonas and Shaw 1989). However, no explanation for this process was given. All the other investigated fla-

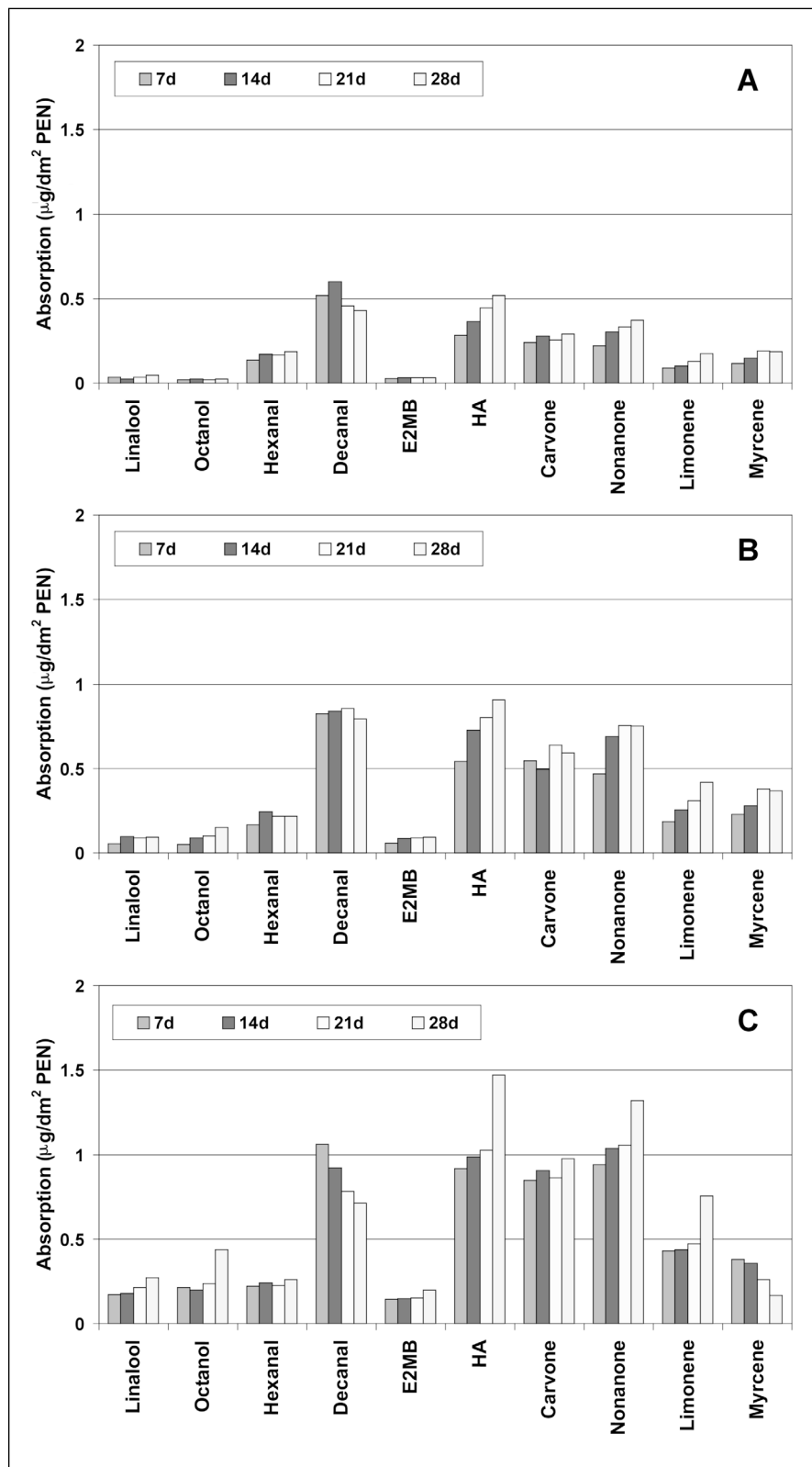


**Figure 5—Absorption of flavor compounds by PET-bottle ( $\mu\text{g}/\text{dm}^2$ ) after different storage times at (A) 4 °C, (B) 20 °C and (C) 40 °C**

vor compounds were quite stable in the model solution at all 3 storage temperatures.

Table 4 shows the influence of storage temperature on the total amount of flavor absorption by all investigated polymer sam-

ples after 14 or 28 d of storage. An increase in temperature had no remarkable effect on flavor absorption by LLDPE. At 40 °C a slight decrease of the total flavor absorption by OPP was measured, due to the degradation of mainly decanal and myrcene. A more pronounced effect of storage temperature on flavor absorption was found for the glassy polymers, PC, PET, and PEN. After 28 d of storage at 40 °C, total flavor absorption by PET film and PET bottle increased by a factor of 21.3 and 13.3, respectively (compared to storage at 4 °C). Total flavor absorption by PC and PEN increased by a factor of 4.1 and 2.9, respectively, when increasing the storage temperature from 4 to 40 °C. Temperature seemed to have a more pronounced effect on flavor absorption by PET than that of PC and PEN. With the increase of temperature, the difference with the  $T_g$  of PET became smaller and smaller, which probably caused a relaxation (that is, an increased free volume) of the polymer network. PC and PEN, having a higher  $T_g$  than PET, were obviously less affected.



**Figure 6—Absorption of flavor compounds by PEN ( $\mu\text{g}/\text{dm}^2$ ) after different storage times at (A) 4 °C, (B) 20 °C and (C) 40 °C**

## Conclusions

ALL PACKAGING MATERIALS SHOW A CERTAIN absorption capacity for flavor compounds. Rate and quantity of flavor absorption are related to differences in polymer characteristics (such as polarity,  $T_g$ , and crystallinity) as well as to the structure and polarity of the different flavor compounds. Absorption of flavor compounds by PC, PET, and PEN is much less than by the polyolefins LLDPE and OPP. Therefore, from the standpoint of flavor absorption and loss of flavor compounds, PC, PET, and PEN should be preferred over LLDPE and OPP. On the other hand, storage temperature does not seem to influence the total amount of flavor absorption by the rubbery polymers LLDPE and OPP, although temperature rises do seem to affect flavor absorption rate and quantity in the glassy polymers PC, PET, and PEN.

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**Table 1—Characteristics of the polymers used in this study<sup>a</sup>**

Polymer	Polarity	T <sub>g</sub> <sup>b</sup> (°C)	Crystallinity (%)	Thickness (μm)	Density (g/cm <sup>3</sup> )
LLDPE film	Apolar	-75	45	50	0.921
OPP film	Apolar	-5 to 0	80	30	0.916
PC film	Polar	+145	0	75	1.20
PET film	Polar	+78	45	50	1.40
PET bottle	Polar	+78	22 to 25	300	1.37
PEN film	Polar	+120	45	75	1.36

<sup>a</sup> Specifications from manufacturers<sup>b</sup> Glass transition temperature**Table 2—Characteristics of the flavor compounds used in the model solutions**

Flavor compound <sup>a</sup>	bp (°C)	Log P <sup>b</sup>	Solubility <sup>c</sup> (g/L)	Density (g/mL)	MW (g/mol)
Linalool	195	3.28	0.11	0.87	154.3
Octanol	177	3.00	0.21	0.82	130.2
Hexanal	130	1.78	2.89	0.81	100.2
Decanal	208	4.09	0.012	0.83	156.3
E2MB	133	2.12	2.47	0.87	130.1
HA	168	2.83	0.37	0.88	144.2
(R)-Carvone	230	2.23	2.11	0.96	150.2
2-Nonanone	192	3.30	0.21	0.82	142.1
(+)-Limonene	178	4.58	0.0027	0.84	136.1
Myrcene	167	4.58	0.0026	0.80	136.1

<sup>a</sup>E2MB = ethyl 2-methylbutyrate; HA = hexyl acetate<sup>b</sup>Measure of hydrophobicity, calculated with ACD/Log P v3.6 (ACD 1999)<sup>c</sup>Solubility at 25 °C in water, calculated with ACD/Aqueous Solubility v4.0 (ACD 1999)**Table 3—Large-volume injection GC conditions**

Extraction solvent	Hexane		Pentane: Dichloromethane (2:1)
Conditions	LLDPE, PP, PC	PEN	PET
Injection volume	30 μL	200 μL	200 μL
Injection speed	5 μL/s	2 μL/s	3 μL/s
Sec. cooling time	10 s	30 s	5 s
SVE delay time	10 s	30 s	5 s
SVE temperature	200 °C	200 °C	200 °C
FID temperature	290 °C	290 °C	290 °C
Oven program	50 °C (10') => 5 °C/min =>190 °C => 30 °C/min => 280 °C (5')		40 °C (10') => 5 °C/ min => 190 °C => 30°C/min => 280 °C (5')

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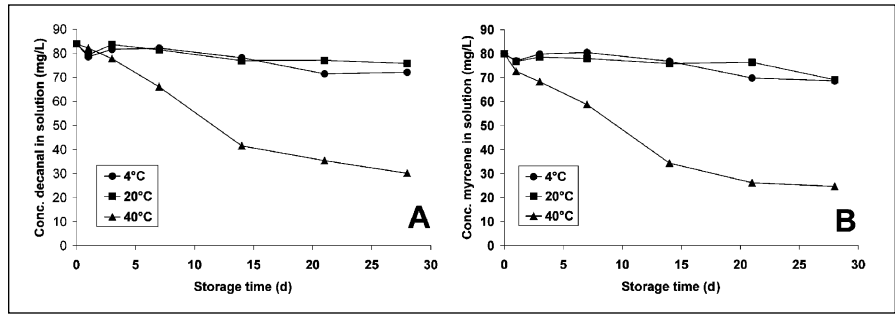
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**Figure 7—Influence of storage time and temperature on the concentration of decanal (A) and myrcene (B) in a Tween 80 model solution without polymer sample**

**Table 4—Temperature effect on the total amount of flavor absorption by different polymers after 14 or 28 d of storage at 4, 20, and 40 °C.**

Polymer	Temp (°C)	Total absorption at day 14 (mg/dm <sup>2</sup> )	Temperature effect		
			4-20 °C	20-40 °C	4-40 °C
LLDPE film	4	5.6	1.1	1.0	1.0
	20	5.9			
	40	5.7			
PP film	4	4.7	1.0	0.8	0.8
	20	4.6			
	40	3.9			

Polymer	Temp (°C)	Total absorption at day 28 (µg/dm <sup>2</sup> )	Temperature effect		
			4-20 °C	20-40 °C	4-40 °C
PC film	4	434.7	1.9	2.1	4.1
	20	834.5			
	40	1765.4			
PET film	4	7.8	3.4	6.3	21.3
	20	26.3			
	40	167.2			
PET bottle	4	29.6	3.0	4.5	13.3
	20	87.6			
	40	394.8			
PEN film	4	2.3	1.9	1.5	2.9
	20	4.4			
	40	6.6			

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