Behavior of Some Solid Food Simulants in Contact with Several Plastics Used in Microwave Ovens

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The behavior of two synthetic adsorbents, Tenax and Porapak, as food simulants in contact with some plastic containers commercially available, recommended to be used for heating food in microwave ovens, has been studied. Containers of polycarbonate (PC), polypropylene (PP), polypropylene random (PP-random), polypropylene−20% talcum (PP%T), and the copolymer styrene−acrylonytrile (SAN) have been studied at 150 °C (PC) and 120 °C (the others). The volatile compounds released from the plastics and trapped into Tenax and Porapak have been analyzed by thermal desorption−gas chromatography−mass spectrometry (TD-GC-MS). The results obtained from these migration studies at high temperature are shown and discussed. Tenax performs better than Porapak at low temperature since the former desorbs the compounds at high temperature.

KEYWORDS: Migration; solid simulants; food analysis; volatile compounds; packaging.

INTRODUCTION

The presence of precooked food to heat in microwave oven and the food to boil in the same plastic package require a major knowledge of the packaging food interaction processes, mainly the migration process, at high temperature. It is well-known that migration increases at high temperatures, favoring the possible contamination of food and modifying the organoleptic characteristics of the product, which causes consumer rejection, this point being serious to the international market. The major and minor components of the plastic material can be also decomposed when these packaging materials increase in temperature, and the decomposition compounds (*1*) can have a more toxic character than their parent compounds. However, very little or none information is supplied about these substances (*2*).

The release of volatile chemical substances from the plastic materials and articles can occur in the high-temperature applications. There are studies about these uses, including plastics to cook in the oven (*3*), microwave susceptors, thermosealed polyester kitchen recipient (*4*), and paper and board as packaging materials to use in the oven (*5*). In all cases it was found that the migration of the volatile compounds to the heated food was an inefficient process, since only between 0.3 and 3% of the volatile substances released from the material was transferred to food. Nevertheless, the trapping efficiency of the food and food simulants used in these studies is under discussion, and a general conclusion about such behavior cannot be extrapolated.

Migration conducts the presence in the food of nondesired substances that cause the loss of quality to the content and can provoke toxicity in the food. The process is controlled by the diffusion of the compounds through the polymeric matrix, until they reach the interface (*6, 7*) with the simulant or with the food. As a thermodynamic process, the diffusion increases with the temperature; therefore, when the temperature increases, the migration will increase too and, consequently, the risk of food contamination (*8*) appears.

According to the established general procedures, migration tests determine the possible compounds which can be transferred to the food either in specific or global migration. These migration values have maximum limits, which are specified in the Directive 90/128/CEE of European Union and their amendments (*9−14*), relative to the materials and articles intended to be in contact with foodstuffs.

In the high-temperature applications, the food simulants should be solid adsorbents able to trap the volatile and nonvolatile compounds in a wide range of temperatures. Several simulants have been studied such as a mixture of zeolites and olive oil (*15*), but none of them has been accepted yet for this purpose, and no official food simulant has been established up to now. Tenax TA is a porous polymer resin based on 2,6-diphenylene. Modified polyphenylene oxide (MPPO) is indicated as a substitute test for fatty food simulants in the Council Directive 82/711/EEC (*16*). Its use is recommended at higher testing temperatures or if a single side test with solvents is impossible because of excessive penetration of the solvent through pinholes (*17*).

This paper shows the study carried out with two synthetic solid adsorbents, Tenax and Porapak, used as food simulants in the high-temperature applications. Other adsorbents such as Amberlite XAD-4 and XAD-7 were rejected because of their ability to trap water and, consequently, their change in adsorption properties. The behavior of these solid food simulants in contact with several plastic containers used in microwave ovens is shown and discussed.

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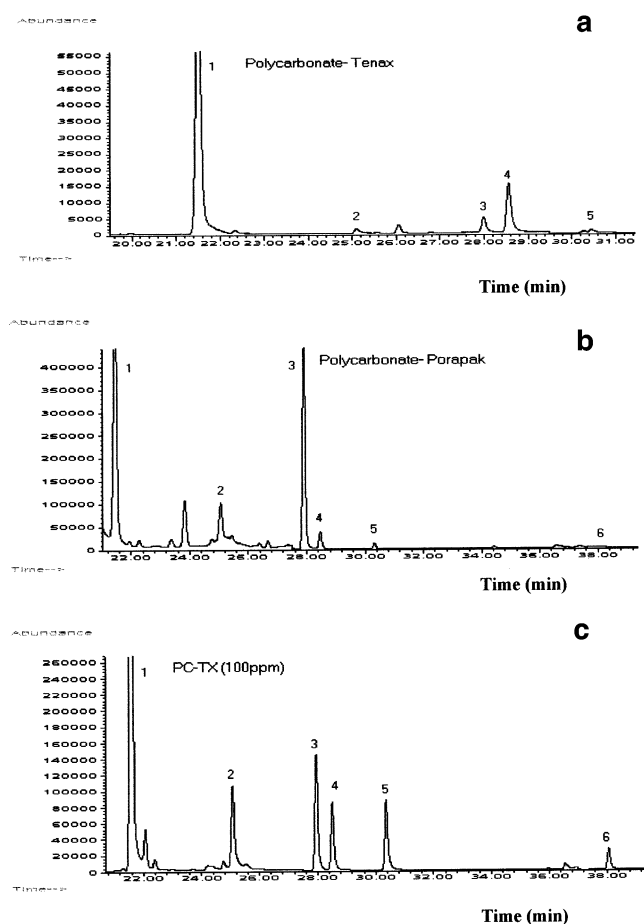
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Figure 1. (a) Chromatogram of volatiles released from Tenax after the migration test from a PC container. (1) Methylbenzene (RT = 21.48 min); (2) 1-octene (RT = 25.07 min); (3) ethylbenzene (RT = 28.00 min); (4) xylene (RT = 28.57 min); (5) styrene (RT = 30.42 min). (b) Chromatogram of volatiles released from Porapak after the migration test from a PC container. (1) Methylbenzene (RT = 21.43 min); (2) 1-octene (RT = 24.97 min); (3) ethylbenzene (RT = 27.85 min); (4) xylene (RT = 28.45 min); (5) styrene (RT = 30.33 min). (c) Chromatogram of volatiles released from Tenax in the calibration plot of 100 μg/g. (1) Methylbenzene (RT = 21.60 min); (2) 1-octene (RT = 25.08 min); (3) ethylbenzene (RT = 27.92 min); (4) xylene (RT = 28.49 min); (5) styrene (RT = 30.29 min).

Table 1. Recovery Values (%) of Volatile Compounds from the Two Different Solid Adsorbents

	recovery, %	
compounds	Tenax	Porapak
methylbenzene	93.56 ± 6.3	94.25 ± 4.5
1-octene	91.53 ± 4.6	96.27 ± 5.1
ethylbenzene	93.67 ± 8.3	83.16 ± 6.1
xylene	90.60 ± 7.6	80.77 ± 10.3
styrene	96.51 ± 7.0	93.37 ± 10.1
1,4-dichlorobenzene	84.54 ± 4.1	91.10 ± 9.4

MATERIALS AND METHODS

Apparatus and Reagents. A Tekmar 2000 purge and trap system connected in line with a HP-5890A Series II gas Chromatograph and a HP 5971 mass detector was used.

An empty trap of stainless steel which was filled each run with the solid simulants was placed in the P&T system instead of the solid trap supplied with the equipment. This way, the P&T system was working as a thermal desorption system.

The chromatographic conditions used were as follows: an HP-624 capillary column of 25 m × 0.32 mm i.d. and 1.5-μm film thickness

was used. Injection temperature 250 °C, det. temperature 280 °C. Oven program: initial temperature 40 °C, then 3 °C/min up to 145 °C, and then 8 °C/min up to 200 °C and hold 3.12 min. SIM mode was used in MS

The desorption conditions were as follows: desorption time 8.00 min, desorption temperature 180 °C, cryofocusing temperature −20 °C, injecton time 1.00 min, injection temp 250 °C, bake time 10.00 min, bake temp 225 °C, valve lines and mount temperature 250, 260, and 120 °C.

Tenax TA 60−80 mesh (Supelco) and Porapak Q 80−100 mesh (Supelco) were used as food simulants. Both adsorbents were previously cleaned with methanol in a Soxhlet for 8 h and then with hexane for an additional period of 8 h. Finally, they were dried at 160 °C under a nitrogen current. Every set of adsorbent was checked by GC-MS before the use.

Samples. Several plastic containers comercially available and all of them recommended for being used in microwave ovens, were supplied by two different companies. The plastic materials were polypropylene (PP) random, polypropylene−20% talcum (PP%T), polypropylene copolymer (PP-copo), polycarbonate (PC), low density polyethylene (LDPE) (this polymer was only used in the covers), and styrene−acrilonitrile copolymer (SAN).

Standards. Pure chromatographic standards of methylbenzene, ethylbenzene, styrene, xylene, 1,2-dichlorobenzene, and 1-octene from Aldrich were used to prepare the calibration plots as well as for recovery studies and quantitative analysis.

Procedure. Tenax (0.30 g) and Porapak (0.60 g) were poured over 8 cm² of each plastic container, covering the surface. Then, the sample (plastic + adsorbent) was placed in a glass Petri dish in an oven for 30 min at 150 °C in the case of PC samples and at 120 °C for all types of PPs and SAN samples.

Immediately after the test, the plastic was removed and the solid adsorbent filled the empty trap of the P&T system, which consisted of a stainless steel tube of 1/8 in. (0.2 cm) internal diameter and 12 in. (30 cm) length. The filling procedure applied was in sandwich mode, which means placing glass wool on the bottom and on the top of the trap. This trap was placed in the P&T system to be used as the thermal desorption system.

A calibration graph was obtained as follows: 2 μL of an hexane standard solution containing the compounds to be quantified were placed in a glass Petri dish, and the same amount of adsorbent as that used with the plastic sample was poured in the Petri dish, taking care that the adsorbent and the standard solution were not in contact. Increasing concentrations of the standard solution were used in the same way to obtain the calibration plot. Each Petri dish was placed in an oven at 150 °C (PC) or 120 °C (PP, SAN and LDPE) for 30 min. Then, the empty trap was filled with the solid adsorbent following the same procedure described above.

Several portions of 0.30 g of Tenax and 0.60 g of Porapak were spiked with a standard solution of the compounds under study, and then the thermal desorption-gas chromatography−mass spectrometry (TD-GC-MS) was applied. The obtained signals were analyzed versus the calibration plot described above. This way, the recovery values of both simulants in the TD-GC-MS procedure were obtained.

RESULTS AND DISCUSSION

Quantitative Analysis. To analyze the volatile compounds released from the plastic samples when they are heated in an oven, the compounds are first trapped on a solid adsorbents, and then several procedures can be applied for both qualitative and quantitative analysis as follows: (a) static headspace (HS) coupled in line to a GC-MS; (b) dynamic headspace (purge & trap); (c) thermal desorption (TD).

In HS only a fraction of the vapor in equilibrium with the sample at a fixed temperature is injected into the GC column. Consequently, the sensitivity is not as high as it can be in other procedures.

The P&T system is much more sensitive, since the total mass of volatile compounds contained in the sample is purged by

Table 2. Migration of Volatile Compounds Released from the Plastic Packaging Using Tenax as Food Simulant

		methylbenzene	1-octene	ethylbenzene	Xylene	styrene	1,4-dichlorobenzene
PC	mg A/kg of Tenax	1.79×10^{-2}	8.92×10^{-4}	4.36×10^{-3}	1.84×10^{-2}	5.39×10^{-3}	$< 1.31 \times 10^{-4}$
	mg/dm^2 of food	6.55×10^{-5}	3.28×10^{-6}	1.60×10^{-5}	6.77×10^{-5}	1.98×10^{-5}	
	mg/kg of food	1.09×10^{-5}	5.47×10^{-7}	2.66×10^{-6}	1.13×10^{-5}	3.31×10^{-6}	
PPR	mg A/kg of Tenax	3.28×10^{-2}	8.42×10^{-4}	1.24×10^{-3}	2.68×10^{-2}	2.90×10^{-3}	$<1.31 \times 10^{-4}$
	mg/dm^2 of food	1.22×10^{-4}	3.13×10^{-6}	4.59×10^{-6}	9.98×10^{-5}	1.08×10^{-5}	
	mg/kg of food	2.03×10^{-5}	5.22×10^{-7}	7.64×10^{-7}	1.66×10^{-5}	1.81×10^{-6}	
PP%T	mg A/kg of Tenax	1.62×10^{-1}	2.30×10^{-3}	2.92×10^{-2}	8.91×10^{-2}	2.96×10^{-3}	$<1.31 \times 10^{-4}$
	mg/dm^2 of food	6.08×10^{-4}	8.59×10^{-6}	1.10×10^{-4}	3.34×10^{-4}	1.10×10^{-5}	
	mg/kg of food	1.01×10^{-4}	1.43×10^{-6}	1.83×10^{-5}	5.57×10^{-5}	1.84×10^{-6}	
PPCo	mg A/kg of Tenax	2.48×10^{-2}	7.49×10^{-4}	4.65×10^{-4}	2.30×10^{-2}	2.88×10^{-3}	$<1.31 \times 10^{-4}$
	mg/dm^2 of food	9.32×10^{-5}	2.82×10^{-6}	1.74×10^{-6}	8.66×10^{-5}	1.08×10^{-5}	
	mg/kg of food	1.55×10^{-5}	4.70×10^{-7}	2.90×10^{-7}	1.44×10^{-5}	1.80×10^{-6}	
SAN	mg A/kg of Tenax	4.71×10^{-2}	7.75×10^{-4}	6.78×10^{-3}	3.00×10^{-2}	1.06×10^{-2}	$<1.31 \times 10^{-4}$
	mg/dm^2 of food	1.75×10^{-4}	2.87×10^{-6}	2.52×10^{-5}	1.12×10^{-4}	3.96×10^{-5}	
	mg/kg of food	2.92×10^{-5}	4.78×10^{-7}	4.20×10^{-6}	1.86×10^{-5}	6.60×10^{-6}	
LDPE	mg A/kg of Tenax	2.13×10^{-1}	7.19×10^{-3}	5.09×10^{-2}	2.62×10^{-1}	3.90×10^{-3}	$<1.31 \times 10^{-4}$
	mg/dm^2 of food	8.10×10^{-4}	2.73×10^{-5}	1.94×10^{-4}	9.94×10^{-4}	1.46×10^{-5}	
	mg/kg of food	1.35×10^{-4}	4.55×10^{-6}	3.23×10^{-5}	1.66×10^{-4}	2.44×10^{-6}	

Table 3. Migration of Volatile Compounds Released from the Plastic Packaging Using Porapak as Food Simulant

		methylbenzene	1-octene	ethylbenzene	xylene	styrene	1,4-dichlorobenzene
PC	mg A/kg of Porapak	1.05×10^{-1}	5.38×10^{-2}	1.40×10^{-1}	3.04×10^{-2}	7.81×10^{-3}	1.65×10^{-2}
	mg/dm^2 of food	7.87×10^{-4}	4.01×10^{-4}	1.06×10^{-3}	2.29×10^{-4}	5.87×10^{-5}	1.23×10^{-4}
	mg/kg of food	1.31×10^{-4}	6.68×10^{-5}	1.76×10^{-4}	3.82×10^{-5}	9.78×10^{-6}	2.04×10^{-5}
PPR	mg A/kg of Porapak	2.55×10^{-3}	$<1.31 \times 10^{-4}$	5.16×10^{-2}	1.13×10^{-1}	3.15×10^{-1}	8.16×10^{-2}
	mg/dm^2 of food	1.90×10^{-5}		3.83×10^{-4}	8.10×10^{-4}	2.29×10^{-3}	6.04×10^{-4}
	mg/kg of food	3.17×10^{-6}		6.38×10^{-5}	1.35×10^{-4}	3.81×10^{-4}	1.01×10^{-4}
PP%T	mg A/k of Porapak	2.83×10^{-3}	$<1.31 \times 10^{-4}$	4.99×10^{-2}	8.11×10^{-2}	2.45×10^{-1}	1.06×10^{-1}
	mg/dm^2 of food	2.12×10^{-5}		3.73×10^{-4}	6.08×10^{-4}	1.84×10^{-3}	7.94×10^{-4}
	mg/kg of food	3.53×10^{-6}		6.22×10^{-5}	1.01×10^{-4}	3.06×10^{-4}	1.32×10^{-4}
PPCo	mg A/kg of Porapak	2.14×10^{-3}	$<1.31 \times 10^{-4}$	4.16×10^{-2}	8.59×10^{-2}	2.35×10^{-1}	4.48×10^{-2}
	mg/dm^2 of food	1.61×10^{-5}		3.14×10^{-4}	6.21×10^{-4}	1.73×10^{-3}	3.40×10^{-4}
	mg/kg of food	2.68×10^{-6}		5.24×10^{-5}	1.04×10^{-4}	2.89×10^{-4}	5.67×10^{-5}
SAN	mg A/kg of Porapak	3.55×10^{-2}	7.29×10^{-3}	1.15×10^{-1}	3.06×10^{-1}	5.35×10^{-1}	3.77×10^{-2}
	mg/dm^2 of food	2.67×10^{-4}	5.51×10^{-5}	8.67×10^{-4}	2.30×10^{-3}	4.02×10^{-3}	2.84×10^{-4}
	mg/kg of food	4.45×10^{-5}	9.18×10^{-6}	1.45×10^{-4}	3.83×10^{-4}	6.70×10^{-4}	4.73×10^{-5}
LDPE	mg A/kg of Porapak	1.11×10^{-1}	6.58×10^{-2}	1.04×10^{-1}	1.24×10^{-1}	9.17×10^{-2}	1.39×10^{-2}
	mg/dm^2 of food	8.23×10^{-4}	4.89×10^{-4}	7.75×10^{-4}	9.36×10^{-4}	6.95×10^{-4}	1.04×10^{-4}
	mg/kg of food	1.37×10^{-4}	8.15×10^{-5}	1.29×10^{-4}	1.56×10^{-4}	1.16×10^{-4}	1.73×10^{-5}

the inert gas used in the system and transferred into the solid trap. This trap is then thermally desorbed and the compounds introduced into the GC column coupled in line to the P&T. Without doubt, this is the most sensitive procedure, since no equilibrium is reached and the total mass of the compounds initially contained in the sample reaches the detector.

Finally, TD is similar to P&T but without the purge step. In this paper the later has been applied but using P&T equipment without the purge step, so that the system is working as TD. To avoid the further width of the chromatographic peaks, a cryogenic module was placed between the trap and the GC column.

Figure 1 shows a chromatogram obtained when the plastic containers were heated in contact with a solid adsorbent such as Tenax. As was previously investigated (*18*), the most important compounds released by the plastics were methylbenzene, ethylbenzene, styrene, xylene, 1,2-dichlorobenzene, and 1-octene and most of them have limited restrictions in the European Legislation. For this reason, it was decided to study their migration ability to solid food simulants. Analytical characteristics of these compounds once they were trapped on both Tenax and Porapak and then analyzed by TD-GC-MS were determined. Linear range studied was from 1.31×10^{-4} to 20×10^{-1} mg/kg of simulant in all cases. No higher or lower values were analyzed as they were not present in the real samples under study.

Before the analysis of the samples, a recovery study was carried out in order to be sure that the applied analytical procedure was quantitative. The calibration plot was obtained following the procedure described under the Materials and Methods. The results of the recovery studies are shown in **Table 1**. As can be seen, in both simulants the values were higher than 80%. No difference was observed either within the simulants or with the compounds.

Migration Tests. Microwave ovens were created to accelerate the preparation of the foodstuffs either for heating or cooking. Usually the food is heated in contact with the container in an interval of time that ranges between 5 and 30 min as maximum, depending on if the food is precooked or not. For this reason, 30 min was selected as the heating time. Another parameter to select was the temperature, which depends on the described indications from the manufacturers of each plastic container. According to this, polycarbonate packages can support a maximum temperature of 140 °C without undergoing any physical deformation or considerable damage of the material. Although previous studies showed that PC can reach higher temperature values (*18*), 150 °C was chosen as the worst case for PC containers since higher values damaged the material. For the three types of polypropylene and SAN packages the maximum temperature used was 120 °C, also selected as the worst case (*19*).
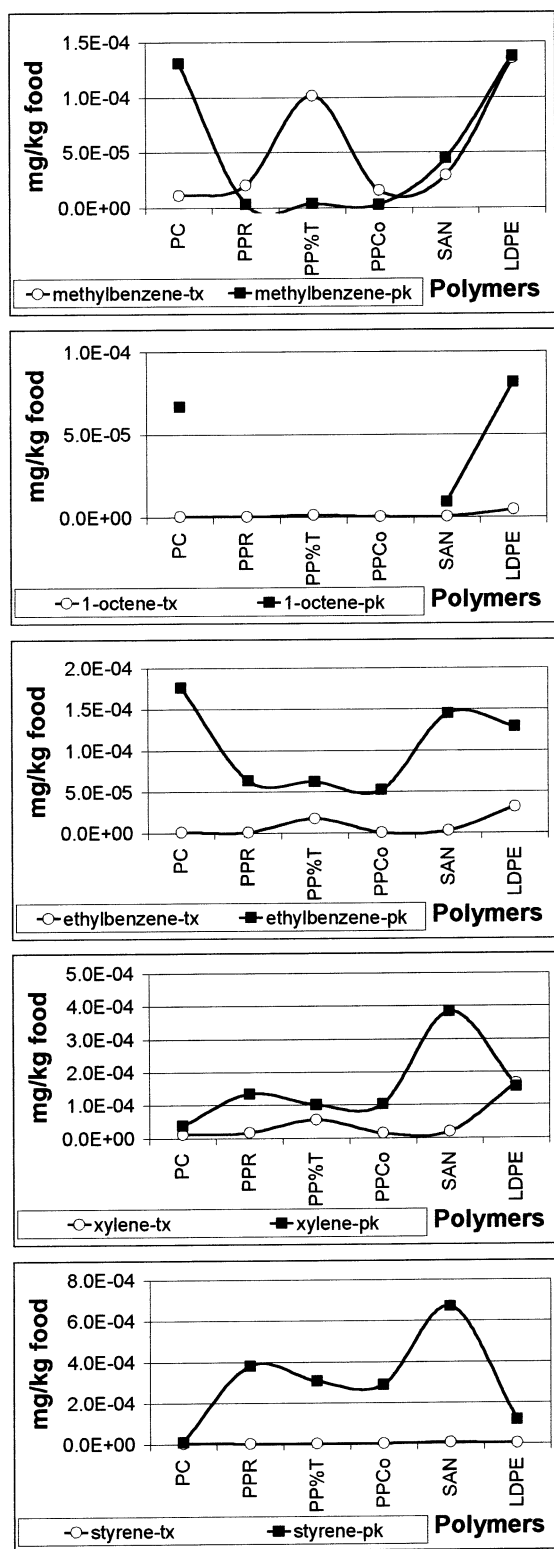
Figure 2. Comparison between Tenax and Porapak as food simulants in contact with several plastic materials versus several volatile compounds.

The study was carried out in a conventional oven instead of using a microwave oven so that the accurated control of temperature was possible. The adsorbent amount used was limited by the maximum capacity of the stainless steel tube of the TD-GC-MS system used, which means 0.3 g of Tenax and 0.8 g of Porapak.

After the migration tests, the simulants were analyzed by thermal desorption coupled in line to a GC-MS. **Tables 2** and

3 show the results of the migration tests expressed on the migration units (milligrams of compound per square decimeter of plastic surface) as well as per mass unit of simulant. To compare the values with the specific migration units (SML), which means the milligrams of compound per kilogram of food, it was taken into account that 6 dm^2 of plastic surface were in contact with 1 kg of food (or food simulant).

Figure 2 shows the comparison of both simulants in the migration tests. As can be seen, Tenax is able to trap the less volatile compounds better than Porapak, while the most volatiles are better trapped into Porapak. This behavior is surprising since Tenax is always recommended as the most appropriate solid food simulant, mainly for volatile compounds. However, in high-temperature applications in which the simulant is also heated in contact with the packaging material, Tenax does the thermal desorption. The compounds released from the plastic are trapped onto Tenax and then distributed into the vapor phase, so that the equilibrium between the three phases, the vapor phase, the simulant, and the plastic, is reached. When the migration test is carried out in an open cell, such as the Petri dish used in this case, the equilibrium in the vapor phase is not reached but Tenax releases the compounds at high temperature (thermal desorption). For this reason, Tenax cannot be recommended as a food simulant for high temperature applications, as it does not represent properly the behavior of foodstuffs under these conditions. This behavior has also been shown in other papers, in which Tenax and milk powder have been studied as food simulants for paper and board (*20, 21*).

In contrast, the behavior of Porapak is worst for thermal desorption process, as it requires higher temperature to release the trapped volatile compounds and, consequently, higher values that with Tenax were obtained in the migration test at high temperature.

Looking at **Figure 2,** some differences between the plastics can be pointed out. SAN in contact with Porapak released higher amount of xylene, styrene, and ethylbenzene, whereas PC released methylbenzene and ethylbenzene. Very similar behavior was obtained within the three types of PP. Theoretically, the same behavior could be expected for each compound on each adsorbent, but this is not the case for methylbenzene. In fact, the results obtained showed that Porapak traps more methylbenzene than Tenax from PC but the opposite happens working with PP%T. To explain these results, the temperature of the test has to be checked. The temperature applied to PC in contact with the simulants was 150 °C, while that applied to PP%T was 120 °C. Tenax probably traps more methylbenzene during the heating but it loses it through a thermal desorption process, and consequently, the final amount of methylbenzene adsorbed on Tenax is lower than on Porapak at thigh temperatures. However, when PP%T is studied at 120 °C, the lose of Tenax is lower than at 150 °C. This could be the reason the behavior Tenax and Porapak is opposite that of methylbenzene.

LDPE is not recommended for high-temperature applications but it is often used by consumers to cover the food containers in the microwave ovens. According to the results obtained, this situation does not involve a health risk, either for LDPE or for the other plastics under study, since all the migration values obtained are well below the SML values established in the European Directive for the mentioned compounds, which are the following: methylbenzene, 1 mg/kg of food; 1-octene, 15 mg/kg of food; ethylbenzene, 0.6 mg/kg of food; xylene, 1.2 mg/kg of food; styrene, 0.6 mg/kg of food.

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CONCLUSIONS

Several conclusions can be highlighted from the study carried out:

1. Both Tenax and Porapak can be used as solid food simulants in high-temperature applications, but the migration results will depend on the volatility of the compounds. The most volatile compounds can be lost from Tenax in a thermal desorption process carried out during the migration test. Consequently, for the most volatile compounds, Porapak is a better simulant at high temperature.

2. All the food containers under study (PC, PPrandom, PPco, PP%T, SAN, and LDPE) gave very low migration values and in all cases these values were below the recommended SML.

3. Volatile compounds released from the plastics can be easily trapped onto the solid simulants and then analyzed by TD-GC-MS.

4. Thermal desorption (TD) coupled in line to GC-MS is a good and sensitive technique for specific migration analysis of volatile compounds.

SAFETY

1-Octene, methylbenzene, ethylbenzene, xylene, and styrene are flammable as liquid and vapor, cause respiratory and digestive tract irritation, and may cause central nervous system depression, as well as eye and skin irritation. The target organ is the central nervous system. They require the use of proper personal protective equipment and use with adequate ventilation. Keep away from heat, sparks, and flames. Store in a cool, dry, well-ventilated area away from incompatible substances.

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