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Removal of polycyclic aromatic hydrocarbons by low density polyethylene from liquid model and roasted meat

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Abstract

Low density polyethylene (LDPE) was used to remove polycyclic aromatic hydrocarbons (PAHs) from liquid media and roasted meat by sorption. Three liquid models and five carcinogenic PAHs were employed to monitor the sorption process, and amounts of chemicals were determined by GC-FID. More than 50% of the total adsorption occurred within 24 h for the selected PAHs in the three model systems. The water–oil system yielded the highest PAHs removal by LDPE; and the system containing phospholipid resisted the diffusion and resulted in the least adsorption among three models. Certain residual PAHs in the LDPE were significantly decreased to a range of 70.8–84.0% after 3 h of UV radiation, and benzo(a)pyrene was the most sensitive to UV among these PAHs. Removal of PAHs in roasted meat packaged under vacuum was achieved, and potent contamination by the PAHs in the LDPE may be avoided by subsequent UV irradiation.

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Keywords: PAHs; Sorption; LDPE; GC-FID; UV

1. Introduction

Polycyclic aromatic hydrocarbons (PAHs, also known as polycyclic organic matter or POM) are formed by the incomplete combustion of organic matter, in particular, from fossil fuels or when vegetation is burned. PAHs are ubiquitous environmental contaminants, and many have been found to be mutagenic and/ or carcinogenic, as well as to have cardiovascular, bone marrow or liver toxicity (Collins, Brown, Alexeeff, & Salmon, 1998; IARC, 1983, 1987; Phillips, 1999; USE-PA, 1987).

PAHs migrate through the food chain into hydrophobic compartments, and, thus, accumulate in lipid components due to their lipophilic nature (Chen & Chen, 2001; Madhaven & Naaidu, 1995; McLachlan, 1997; Roeder, Garber, & Schelling, 1998). The presence of PAHs in food is the predominant cause of human exposure to them. Food components, such as fats, cause

PAHs to be generated through thermal degradation or thermal polymerization, and different thermal processes affect their production quantitatively (Chen, 1997; Phillips, 1999). GC-FID has been widely used for the determination of PAHs in food analysis, because the GC-FID response is the same for all PAH compounds and linear over a large concentration range (ca. $1-10^6$) (Chen & Lin, 1997; Djozan & Assadi, 1999; Mottier, Parisod, & Turesky, 2000; Simko, 2002; Wu, Wong, Lee, Shi, & Ong, 1997).

PAHs are chemically inert and hydrophobic. Their carcinogenicity is initiated by their metabolic conversion in mammalian cells to diolepoxides that bind covalently to cellular macromolecules, including DNA, causing errors in DNA replication and mutation (Phillips & Grover, 1994). There is also evidence that other reactive intermediates are generated by a one-electron oxidation process that can result in the chemically unstable alkylation of DNA, leading to depurination (a potentially mutagenic event) (Rogan et al., 1993). Due to their carcinogenic activity, some European countries, such as Germany, have adopted a legal limit of 1 ppb for the benzo (a) pyrene (BaP) content in smoked foodstuff, 25

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ppb for total PAHs and 5 ppb for the heavy fraction (Moret & Conte, 2000). Currently, a reduction in PAH levels in food products can be achieved by alternative processes which suppress the formation of PAHs (Chen & Lin, 1997), as well as remove PAHs by sorption to the packaging film (Simko & Brunckova, 1993; Simko, Simon, Khunova, Brunckova, & Drdak, 1994; Simko, Khunova, Simon, & Hruba, 1995; Simko, Simon, & Khunova, 1999; Van Lune, Nijssen, & Linssen, 1997). Although PAHs are stable, degradations through photolysis and microorganisms are the major ways to decompose them (Manahan, 1991; Miller, Singer, Rosen, & Bartha, 1988; Sabate, Bayona, & Solanas, 2001; Saftic, Fedorak, & Anderson, 1992). Previous study (Bernstein et al., 1999) also showed a conversion of PAHs to oxidative derivatives on exposure to UV radiation in ice.

The ability of plastic to adsorb some compounds in food represents an effective route to eliminate some hazardous components from foods. The aims of this study were to evaluate the use of LDPE in removing PAHs in liquid medium models, including aqueous, water–oil and water–oil containing phospholipid systems. Additionally, the procedure was applied to remove PAHs from roasted duck skin packaged in a LDPE pouch at ambient temperature. Subsequently, reduction of PAHs in PE materials by exposure to UV radiation was also investigated.

2. Materials and methods

2.1. Chemicals

Sixteen PAH standards, including naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, dibenzo[a,h]anthracene, benzo[g,h,i] perylene, and indeno[1,2,3-c,d]pyrene were purchased from Accustandard Co. (New Haven, CT), and used to determine the retention time and peak area response by GC analysis. Five carcinogenic PAHs, including benzo[a]anthracene (BaA), benzo[b]fluoranthene (BbF), benzo[a]pyrene (BaP), dibenzo[a,h]anthracene (DBahA), and indeno[1,2,3-c,d]pyrene (IcdPy) (IARC, 1983, 1987) were selected as markers to determine the sorption of PAHs to LDPE film. Phosphatidylcholine was purchased from Sigma Co. Propylene alcohol (polarity index 3.9) used to stimulate lipid phase in meat product, ethanol, and $CH₂Cl₂$ were obtained from Merck Co.

2.2. Sorption of PAHs by LDPE in liquid media models

Three liquid models were employed to study the removal of PAHs by LDPE via adsorption mechanism, and the adsorption process was then applied to remove PAHs in roasted duck skin. To simulate the migration of PAHs from meat product to package material, a water–oil model, composed of distilled water and propylene alcohol, was used, instead of the aqueous phase alone. Moreover, meat products contain 1% phospholipids which act as an emulsifier; an emulsion model was also accomplished by adding 1% phosphatidylcholine to the water–oil system.

Five carcinogenic PAHs (each 50μ g) were dissolved in 0.5 ml methanol, and then spiked to 49.5 ml of distilled water (the final PAH concentration was 100 ppm of each). In the water–oil model, the moisture and lipid contents were pre-determined by the oven-drying method at 103 °C and the modified Soxhlet method (AOAC, 1995), respectively. The moisture and crude fat contents in roasted duck skin were $45.1 \pm 6.9\%$ and 50.7 ± 11.6 %, respectively. The ratio of moisture to fat content in duck skin was calculated as 1:1; therefore, the composition of medium used in the water–oil model was set for water: propylene alcohol $= 1:1$. In the emulsion model, a natural emulsifier, such as phospholipid, was applied to the water–oil model. Phosphatidylcholine (0.5 g), a phospholipid mainly presents in membrane, was dissolved in 1 ml methanol, and then added to 49 ml of water–propylene alcohol mixture (1:1) for the water–oil model containing 1% phospholipid (water–oil + PL) model.

Commercial LDPE films, used in liquid models to absorb PAHs, were cut to a round portion (14.0 cm diameter \times 0.08 mm thickness, average weight 1.2 ± 0.08 g), and washed by immersing into a 95% ethanol solution for 10 min (with metal clips to ensure that the films submerged), then rinsed by distilled water. The density of the LDPE was ca. 0.91 g/cm3, and it therefore floated on the liquid surface that was employed to simulate the single side absorption. In general, ready-to-eat roasted duck product packages are shelfed in air-conditioned retail stores in Taiwan, the storage temperature conditions ranging from 23 to 28 $^{\circ}$ C (averaged ca. 25 $^{\circ}$ C). Therefore, LDPE film and liquid medium were placed in an oven at 25 $\mathrm{^{\circ}C}$ for 7 days, and the amounts of PAHs in the LDPE and liquid medium were determined at days 0, 1, 3 and 7.

2.3. Extraction of PAHs

PE film was cut into $2 \text{ cm} \times 2 \text{ cm}$ rectangles and placed in a 250 ml flask with 50 ml CH_2Cl_2 , the flask was shaken for 30 min and then sonicated for 5 min. Each extraction was repeated three times. After filtering, the CH_2Cl_2 layer was evaporated to near dryness, and the residue was rewetted with 4 ml of CH_2Cl_2 . The extract was then placed in a brown vial crimped by an aluminium-cap and stored in a freezer $(-20 \degree C)$ prior to GC analysis.

Residual PAHs in the liquid medium was determined by extraction with two 50 ml portions of CH_2Cl_2 in a separatory funnel. After filtering, the $CH₂Cl₂$ layer was evaporated to near dryness, and rewetted with 4 ml of CH_2Cl_2 . The extract was stored in a brown vial at -20 -C prior to GC analysis.

Recovery of PAHs used in the model study was calculated by

$$
Recovery = \frac{amount\ of\ PAH\ on\ PE + residue\ in\ medium}{amount\ of\ PAH\ spiked\ in\ medium}
$$

$$
\times 100\%.
$$

2.4. Identification of PAHs by GC

GC-FID is widely used for the determination of PAHs in food, due to the fact that the GC-FID response is the same for all PAH compounds and linear over a large concentration range (ca. $1-10^6$) (Chen & Lin, 1997; Djozan & Assadi, 1999; Mottier et al., 2000; Simko, 2002; Wu et al., 1997). A Perkin–Elmer model Autosystem XL equipped with an FID was employed for the PAH analysis. A 60 m \times 0.32 mm i.d. (0.25 µm thickness) DB-1 fused silica capillary column (J&W, Folsom, CA) was used with N_2 carrier gas at a flow rate of 2 ml/ min. The temperatures of the injector and detector were 280 and 280 °C, respectively. The oven was programmed from 35 °C for 2 min, then heated to 130 °C at 10 °C/ min and held for 2 min, followed by an increase at $5 \text{ }^{\circ}\text{C}/$ min to 210 \degree C and then held for 2 min. Subsequently, the oven was heated to a final temperature of 280 \degree C at 2.5 \degree C/min, and maintained for 40 min. Peak areas were integrated using Turbochrom software (ver. 4.1, Perkin– Elmer).

Standard curves for PAH concentrations (0.10–200 ppm) vs. response peak area were established and used to determine the PAHs contents. An internal standard of triphenylene (10 ppm) was added to calibrate the GC-FID.

2.5. Preparation and analysis of roasted meat

Whole duck samples were placed in an oven, roasted for 60 min at 225 \degree C, and then vacuumpacked in LDPE pouches after cooling. Carcinogenic PAHs in the roasted meat were determined after storage at 25 °C for 24 h.

Each roasted duck skin sample (50 g) was mixed with 60 ml of 50% KOH in a flask, and then shaken at 100 rpm for 30 min in a 70 \degree C water bath. The mixture was extracted with 50 ml of $CH₂Cl₂$ twice and the extract evaporated to 5 ml. The concentrate was poured into a Sep-Pak Florisil cartridge which was activated with methanol. A 10 ml aliquot of methanol was used to elute the chemicals, and the eluate was collected, evaporated to dryness and rewetted with $CH₂Cl₂$.

2.6. UV irradiation of PAH in LDPE

LDPE was immersed in 50 ml of the water–oil model medium spiked with the five selected PAHs (1 ppm each) at 25 °C for 24 h. The test LDPE was then transferred to a UV chamber and irradiated at 254 nm (10 cm distance from a UV source) for 0–3 h (Bernstein et al., 1999).

2.7. Statistical analysis

All experiments were accomplished in triplicate, and the data were subject to an analysis of variance and Duncan's multiple range tests using the SAS software package (SAS Institute, Inc., Cary, NC, USA). A nonlinear least error model for adsorption over time was achieved using the Quasi-Newton analysis.

3. Results and discussion

3.1. Identification and quantification of PAHs by GC

Identification and quantification of the PAHs was achieved by gas chromatography modified from Wu et al. (1997). The 16 PAH standards were separated and identified under the GC conditions employed (Fig. 1(a)), and the selected PAHs adsorbed to the LDPE were also identified under the three liquid models (Fig. $1(b)$ – (d)).The limits of detection for the selected carcinogenic PAHs, including BaA, BbF, BaP, DBahA and IcdPy, were 0.10 ppm, and the R^2 values of each standard PAH curve (concentration of 0.10–200 ppm vs. peak area) were greater than 0.99. Clearly, this method showed a high specificity and sensitivity, and thus should be adequate for the identification and quantification of the selected PAHs.

3.2. Adsorption of PAHs by LDPE film in model studies

Three liquid models were used to investigate the effect of medium on the adsorption of PAHs on LDPE in this study. The efficiency of the extraction was tested by recovery studies, and the average recovery of PAHs in each experiment was 97.3% (in the range of 86.3– 107.1%). The adsorption of PAHs on the LDPE film in the model studies was determined. Most of PAHs were adsorbed within 12 h, and reached a plateau at day 3 (Figs. 2–6). The adsorption of PAHs (including BbF, BaP, IcdPy and DBahA) was found to be greater in the water–oil model among the three systems (Figs. 3–6), except that BaA was adsorbed to a greater extent in the aqueous model than in the others (Fig. 2). The adsorption rate of each PAH was determined for each of the liquid models; the PAHs adsorbed by the LDPE was found to be greatest in the water–oil system and least in the water–oil containing PL system (Table 1). The

Fig. 1. Gas chromatogram of PAHs. (a) standard solution containing 16 PAHs (peaks: 1, N; 2, APL; 3, AP; 4, FL; 5, Phen; 6, A; 7, F; 8, Py; 9, BaA; 10, Ch; 11, BbF; 12, BkF; 13, BaP; 14, DBahA; 15, BghiP; 16, IcdPy); PC, phosphatidylcholine; IS indicates the internal standard (triphenylene). (b) Selected PAHs adsorbed to LDPE in the aqueous model system. (c) Selected PAHs adsorbed to LDPE in the water–oil model system. (d) Selected PAHs adsorbed to LDPE in the emulsion model system.

average adsorption rate for each PAH in the system containing PL was about half that of the other two models. In the aqueous model, the order of adsorption rate in 24 h was $BaA > BaP > DBahF > BbF > IcdPy$; however, the adsorption of Icd Py was found to be greater in the other liquid models (Table 1).

The ability of plastics to sorb certain types of compounds is based on a complex process which involves the diffusion of PAHs into a solution, followed by the affinity to the surface due to similar polarity, and eventually subsequent diffusion to the interior of the packaging film (Van Lune et al., 1997). Reports have also indicated that the sorption of PAHs from an aqueous phase by a PE film is limited by diffusion in the medium and the nature of the PAHs (Simko et al., 1994; Simko et al., 1999). PAHs migrate from a strongly polar

Fig. 2. Adsorption of BaA to LDPE film in model systems. The regression equations and correlation coefficients are as follows – aqueous: $Y = 30.84 + 4.38 \text{Ln}(X) R^2 = 0.98$; water–oil: $Y = 29.10 +$ 4.16Ln(X) $R^2 = 0.98$; water-oil + PL: $Y = 14.93 + 2.08$ Ln(X) $R^2 =$ 0.97 (Y: adsorption, µg PAH/g film; X : time, day).

Fig. 4. Adsorption of BaP to LDPE film in model systems. The regression equations and correlation coefficients are as follows – aqueous: $Y = 27.03 + 3.92 \text{Ln}(X) R^2 = 0.95$; water–oil: $Y = 32.17 +$ 4.50 Ln(X) $R^2 = 0.99$; water-oil + PL: $Y = 15.37 + 2.13$ Ln(X) $R^2 =$ 0.97 (Y: adsorption, μ g PAH/g film; X: time, day).

Fig. 3. Adsorption of BbF to LDPE film in model systems. The regression equations and correlation coefficients are as follows – aqueous: $Y = 23.94 + 3.42 \text{Ln}(X) R^2 = 0.94$; water–oil: $Y = 30.77 +$ 4.29 Ln(X) $R^2 = 0.99$; water–oil + PL: $Y = 12.24 + 1.59$ Ln(X) $R^2 =$ 0.99 (Y: adsorption, μ g PAH/g film; X: time, day).

medium into a non-polar medium, where van der Waals forces have a decisive influence on the sorption of nonpolar PAHs into the packaging material (Simko & Brunckova, 1993). Since PAHs and LDPE are non-polar compounds, more PAHs become distributed in the

Fig. 5. Adsorption of IcdPy to LDPE film in model systems. The regression equations and correlation coefficients are as follows – aqueous: $Y = 22.92 + 3.30 \text{Ln}(X) R^2 = 0.92$; water-oil: $Y = 35.39 +$ 4.88 Ln(X) $R^2 = 0.98$; water-oil + PL: $Y = 17.21 + 2.37$ Ln(X) $R^2 =$ 0.99 (Y: adsorption, μ g PAH/g film; X: time, day).

less polar layer of the propylene alcohol and are then readily adsorbed by the LDPE film. The viscosities of the liquid media also play an important role in overall sorption. The viscosity coefficient (dyne $s/cm²$) of each liquid model was determined to be 0.17 ± 0.003 ,

Fig. 6. Adsorption of DBahA to LDPE film in model systems. The regression equations and correlation coefficients are as follows – aqueous: $Y = 19.57 + 2.63 \text{Ln}(X) R^2 = 0.96$; water–oil: $Y = 33.38 +$ 4.62Ln(X) $R^2 = 0.98$; water–oil + PL: $Y = 14.05 + 1.96$ Ln(X) $R^2 =$ 0.97 (Y: adsorption, μ g PAH/g film; X: time, day).

 0.31 ± 0.006 and 0.34 ± 0.006 for aqueous, water–oil and water–oil + PL models, respectively. The water–oil system containing PL was found to be the most viscous among the three models; thus the sorption of PAHs from this liquid medium was the least. Additionally, increasing the molar mass and size of the compounds causes a decrease in solubility and diffusion coefficients of PAHs to LDPE materials (Charara, Williams, Schmidt, & Marshall, 1992; Fayouz, Seuvre, & Voilley, 1997; Simko et al., 1995, 1999).

Most adsorption PAHs to plastic films occurs within 24 h. In fact, the sorption process starts immediately after immersing the film in the liquid medium. It was possible to determine the adsorption of PAHs on the contact layer of a PE sheet as early as after 0.75 h, and in a further layer after 69 h (Simko et al., 1999). Therefore, the use of non-polar packaging material to sorb PAHs in roasted meat products should be beneficial in terms of eliminating hazardous compounds.

3.3. Removal of PAHs in roasted meat by LDPE

In the model studies, LDPE functioned to remove PAHs, thus permitting the removal of PAHs. Whole duck samples were roasted at 225 \degree C for 60 min, and skin samples were peeled off for analysis. Roasted skin samples were packed in a LDPE pouch under vacuum to ensure that the meat was in contact with the film, and then held at room temperature for 24 h prior to PAH determination.

Three carcinogenic PAH compounds were detected in the freshly roasted duck skin, and the amounts of BaA, BbF and BaP were 143, 3.7 and 3.5 ng/g, respectively (Table 2). However, no IcdPy or DBahA were found. A reduction of PAH contents in the roasted samples was observed after packaging in the LDPE pouch for 24 h. The residual PAHs were BaA, BbF and BaP as 130, 1.7 and 0.9 ng/g, respectively, and a significant decrease in BbF and BaP contents was achieved (Table 2).

Many reports have demonstrated that carcinogenic PAHs are formed through the grilling and smoking of foods (Chen & Lin, 1997; Mottier et al., 2000; Ova & Onaran, 1998; Phillips, 1999; Swallow, 1976; Wu et al., 1997). To remedy the production of the hazardous compounds in food products, the use of packaging

Table 1

 a 50 µg of each PAH was spiked into 50 ml of liquid model system.

	Content (ng/g)		Reduction [*] $(\%)$	
	0 _h	24 h		
BaA	143 ^a	130 ^a	8.01 ± 4.09	
	$(77.7-208)$	$(73.7 - 185)$		
BbF	3.69a	1.69 ^b	54.3 ± 0.43	
	$(2.78-4.6)$	$(1.28 - 2.09)$		
BaP	3.50 ^a	$0.94^{\rm b}$	73.0 ± 0.58	
	$(1.57 - 5.42)$	$(0.43 - 1.44)$		
IcdPy	ND ^c	ND	$\overline{}$	
DBahA	ND	ND		

Table 2 PAH contents in roasted duck skin packed in LDPE pouches

^{a,b} Values with different superscripts in the same PAH indicate a significant difference ($p < 0.05$).

^c ND: not detected.

* Reduction is calculated by [initial amount (at $(0 h)$ – final amount (at $(24 h)$]/initial amount.

Table 3 Effect of UV irradiation on PAHs adsorbed to LDPE film

Duration (h)	BaA	BbF	BaP	IcdPy	DBahA
	$100^{*,a}$	$100^{\rm a}$	100 ^a	100 ^a	$100^{\rm a}$
	$102^{\rm a}$	103 ^a	102 ^a	101 ^a	102 ^a
_	85.9 ^b	89.1 ^b	86.5^{b}	$92.4^{\rm a}$	92.0^{b}
	74.6 ^c	84.0°	70.8 ^c	89.1ª	82.1°

^{a-c} Values with different superscripts in each column indicate significant difference ($p < 0.05$).
*Values used are relative amounts of PAHs in LDPE, a value of 100% is assigned for the initial PAHs prior to UV irradiat

material to remove PAHs represents is a potential solution. Since the surface of the roasted/smoked meat contains the most PAHs, the sorption of PAHs by contacting packaging material is feasible. The results in this study demonstrate the sorptive effect on PAHs, and the process can be completed within 24 h.

3.4. Effect of UV irradiation on PAHs in LDPE film

To prevent the potent contamination by PAHs of the environment, the elimination of PAHs in LDPE by UV irradiation was also investigated. LDPE film was placed on a water–oil medium containing selective carcinogenic PAHs for 24 h, and then exposed to UV irradiation. PAH contents in the LDPE were measured every hour for up to a 3 h duration. The decrease in PAH content in the LDPE was clearly associated with UV irradiation. No significant reduction was found within a 2 h irradiation, the residual PAHs in the LDPE were in the range 85–92% after a 2 h treatment (Table 3). A significant decrease in carcinogenic PAH content in the LDPE was found after a 3 h exposure, except for IcdPy (Table 3). Among the five selected PAHs, BaP was found to be the most sensitive to UV radiation, while IcdPy was the least (Table 3).

Photolysis and microbial degradation are the main processes for eliminating PAHs (Manahan, 1991; Miller et al., 1988; Sabate et al., 2001; Saftic et al., 1992). According to their chemical structures, PAHs readily absorb sunlight and ultraviolet radiation, and are particularly sensitive to the photochemical effects of UV irradiation (Chen, Quan, Yan, Yang, & Peijnenburg, 2001; Nico, Schouten, Gerrit, & Van Der Stegen, 1987; Manahan, 1991; Miller et al., 1988; Sabate et al., 2001). PAHs with a high molecular weight (bulkiness) tend to photolyze faster, but chemically derivatized samples are more resistant to photolysis (Chen et al., 2001; Sabate et al., 2001). Bernstein et al. (1999) demonstrated that PAHs are converted to aromatic alcohols, ketones, and ethers by oxidation of peripheral carbon atoms when they are exposed to UV radiation in ice. Those results indicate a possible method for eliminating adsorbed PAHs in packaging materials. A similar process was employed in this study to reduce the carcinogenic PAHs content in LDPE. A decrease in residual PAHs in LDPE was observed after the UV radiation, and the reducing effect was related to the duration of exposure. However, the specific mechanism that controls this process will require further investigation.

4. Conclusion

The results obtained in this study lead to the following conclusions. The sorption of PAHs to LDPE in liquid models was related to the mobility of chemical in the liquid, and most of the total adsorption occurred within 24 h. PAHs in roasted meat products may be removed by being in contact with the packaging film, and then eliminated by UV radiation. This approach of removal of PAH compounds from meat products is beneficial for eliminating these hazardous chemicals.

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