



Reduction of congener specific PCBs in water and peanut oil by polyethylene packaging film

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The use of polyethylene film to reduce the concentration of selected polychlorinated biphenyl (PCB) congeners (one each from tri to deca chlorinated) from aqueous and oil systems, was investigated. The trend in removal of PCBs from the aqueous system showed an inverted mirror pattern of its uptake by the polyethylene film. There appeared to be little loss of PCBs from the oil during the duration of the study. The rate loss from each system was compared as a function of PCB congener types. The results show that the lower chlorinated congeners were removed at a faster rate from the water than their higher counterparts. In the oil, the congeners with higher chlorination generally were removed at a faster rate than those with lower chlorination. The mass balance ratio confirmed that the PCBs removed from the dissolved solutions were sorbed by the contacting polyethylene film. © 1997 Published by Elsevier Science Ltd

INTRODUCTION

Polychlorinated biphenyl (PCBs) congeners belong to a group of chlorinated hydrocarbons produced in the United States by Monsanto, Inc. and manufactured as complex mixtures called Aroclors. Belonging to a global group of highly lipophilic pollutants, 209 substituent arrangements (congeners) of PCBs are theoretically possible. Until cessation of their production in 1977, US industry used PCBs because of their high dielectric constant, chemical and thermal stability, non-flammability and their low cost. The industrial application of PCBs included insulating fluids in electrical transformers and capacitors, heat transfer substances, cutting oils, hydraulic fluids, lubricating oils, and as plasticizers in plastics manufacture. Application was also found for these compounds in paints, printing inks, carbonless copy paper, sealants, adhesives, and many other industrial applications (Swain, 1983).

Even though production has been discontinued and the importation and usage of PCBs highly restricted, PCBs remain in the environment at declining but still unacceptably high concentrations because of their past widespread application and chemical stability (Schmidt & Hesselberg, 1992). The identical characteristics which made them industrially desirable also make them persistent and allow accumulation in the environment. The

potential for toxicity has been shown in at least 18 and perhaps up to 36 of the 209 congeners because of their accumulation in fish/animal tissue and their demonstrated enzyme induction effects (McFarland & Clarke, 1989; Tanabe, 1988). This is understandable because their highly lipophilic nature results in adipose tissue accumulation when ingested, and thus reduces their potential for easy excretion.

Several researchers have reported on the toxic effects of PCBs in human, avian and aquatic organisms (Jacobson *et al.*, 1989; McFarland & Clarke, 1989; Schmidt & Hesselberg, 1992; and Gonzales, 1995). Thus, the reduction of PCBs from the environment, and from contaminated aquatic food sources in particular, is an issue of relevance to ensuring the safety of the food supply. In attempts to reduce PCBs in fish for example, preparation and cooking techniques by Zabik *et al.* (1995a,b, 1996), Armbruster *et al.* (1987) and Sanders and Haynes (1988) have resulted in varying loss of PCBs and other organic toxicants. This study seeks to create another model for PCBs reduction by focusing on the use of polyethylene film, normally used in food packaging, to reduce the concentration of PCBs in water and an edible oil.

Polyethylene is highly lipophilic because of its organic chemical structure—a phenomenon similar to PCBs. This principle formed the basis of this study, by assuming that PCBs would be expected to show greater partitioning for polyethylene if brought together in an aqueous system. The successful result of such exposure

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would certainly ensure a reduction in PCB concentration in the aqueous medium. Thus, the objectives of this study were: (1) to study the reduction of PCB concentrations in water exposed to low density polyethylene film; (2) to study the reduction of PCB concentrations in peanut oil exposed to low density polyethylene film and to compare this data with that from the aqueous study; (3) to create a model for the reduction of PCBs in food systems using food packaging plastic films.

MATERIALS AND METHODS

Materials

Polyethylene

Polyethylene-resin supplied as pellets by Dow Chemical (Midland, MI). The polyethylene pellets were extruded using a single screw 4 zone Killion model KLB-100 extruder, with a 76.2 cm diameter barrel attached. It was interfaced with a Killion chill roll attached to a model 8412-A sterico steritronic temperature control console. The temperature zones 1–4 were set to operate at 110, 121, 132, and 149°C, respectively, while the die temperature was set at 143°C. The screw speed was set to operate at 15 rev min⁻¹, the 20.32 cm wide and 15.24 cm diameter chill roll at the dial speed of 100 (30 rpm), with a cooling temperature of 120°C. The percent crystallinity of the polyethylene, after extrusion, was calculated to be 53.56%. It had a thickness of 2.286×10^{-3} cm.

Edible oil

Peanut oil purchased from the Michigan State University food stores, East Lansing, MI. All the oil came from the same manufactured lot and was processed by Nabisco Foods, Planters Division, Winston-Salem, NC.

PCBs

Polychlorinated biphenyl congeners—Accustandard Inc. (New Haven, CT)—are shown in Table 1. These are based on the occurrence of the congeners in Aroclor[®] 1254 (Ballschmitter & Zell, 1980).

Method

Exposure of polyethylene film to PCB congeners

Deionized distilled water was spiked with a mixed PCB standard solution to produce a 10 ppb solution of each congener. This was done by initially dissolving equal concentrations of the eight PCB congeners using acetone as a solvent. After adding the desired quantity to give the 10 ppb solution, the system was heated at 40°C for 1 h with frequent shaking to evaporate any residual acetone. After cooling to 25°C, the solution was ready for contact with the polymeric materials. A 0.1 ppm stock solution of spiked peanut oil with PCBs was also prepared. This was made by dissolving 1 ml of a 50 ppm mixed PCB in hexane solution in approximately 5 ml of

peanut oil in a glass tube. The hexane in this solution was then evaporated for 30 min under nitrogen using an N-Vap Model III instrument (Organomation Assoc., Shrewsbury, MA). The resulting solution was then made up to 500 ml in a volumetric flask. This solution was then ready for contact with the polyethylene material.

Approximately 0.160 g of the polyethylene film was prepared by cutting into longitudinal strips approximately 4×1.5 cm. Each sample was then inserted in a 76 ml serum vial supplied by Wheaton Inc., Millville, NJ. Each vial was then brim-filled with the spiked aqueous PCB congener solution, sealed, and stored at 25°C in the absence of light. At given time intervals for a total of 72 h, samples were removed for PCB congener quantification. The polyethylene exposed to the peanut oil was prepared similarly to the polyethylene exposed to the water and also stored at 25°C for a maximum time of 72 h.

PCB congener analysis

The PCB analytical method, including the clean-up procedures used in this study, was adapted from electron capture gas chromatographic analyses for PCB as outlined by Price *et al.* (1986). The procedure is given in detail in Pascall *et al.* (1997a). All samples in this study were extracted in triplicates and subsequently quantified for PCBs.

After exposure of the oil to the polyethylene, the PCBs were extracted from the oil by first adding 25 ml acetonitrile (saturated with hexane) in a 250 ml separatory funnel. This was shaken for approximately 2 min, the lower acetonitrile layer was drained into a 500 ml separatory funnel. The remaining oil in the funnel was extracted twice more, each time with a fresh aliquot of 25 ml acetonitrile. In each case the acetonitrile layer was drained and combined in the 500 ml flask. To this combined solution was added 100 ml hexane and 100 ml of a 10% aqueous sodium chloride solution. After shaking for 2 min, 3 layers were observed. The bottom layer was discarded then the remaining solution was shaken again with fresh 100 ml aliquots of hexane and the 10% aqueous sodium chloride solution. If three layers appeared after this second extraction, a third 100 ml aliquot of each of hexane and sodium chloride was again added then extracted. However, if only two layers were observed, the lower layer was discarded and the upper layer collected into a 200 ml volumetric flask. In cases where three extractions were performed, this upper layer was also collected while the lower was discarded. Two layers were always present when the series of extractions ended. To the solution collected in the 200 ml volumetric flasks 8 g of anhydrous sodium sulfate was added. After allowing 1/2 h for the drying action of the Na₂SO₄, the extracts were reduced in the Turbo-Vap evaporator to approximately 1.0 ml volume at ambient temperature. Care was taken to avoid reducing the extracts to dryness. Each sample was then reduced to approximately 0.1 ml by nitrogen evaporation using the N-Vap Model III instrument then made up with iso-octane to exactly 0.5 ml in a volumetric flask.

Table 1. Systematic names of the PCB congeners used in this study

IUPAC No.	Chlorobiphenyl systematic names
Internal Standard No.30	2,4,6-Trichlorobiphenyl
Isomer No.	Biphenyl Compound
31	2,4',5-Trichlorobiphenyl
47	2,2',4,4'-Tetrachlorobiphenyl
103	2,2',4,5',6-Pentachlorobiphenyl
128	2,2',3,3',4,4'-Hexachlorobiphenyl
171	2,2',3,3',4,4',6-Heptachlorobiphenyl
200	2,2',3,3',4,5',6,6'-Octachlorobiphenyl
206	2,3,4,5,6,2',3',4',5'-Nonachlorobiphenyl
209	2,3,4,5,6,2',3',4',5',6'-Decachlorobiphenyl

(Ballschmiter & Zell, 1980).

The polyethylene and oil extracts were cleaned up using florisil followed by silica gel low pressure chromatography as described previously (Pascall *et al.*, 1997a).

PCB quantification

The concentration of individual PCB congeners in each extract was determined by gas chromatography (GC) with a ^{63}Ni electron capture detector (Hewlett Packard 5890 series II, Avondale, PA) and equipped with a DB-5 capillary column (60 m \times 0.25 mm i.d.; 0.25 μ film thickness). PCBs would generally elute in order of chlorination: $\text{C}_{12}\text{H}_9\text{Cl}$ first, $\text{C}_{12}\text{Cl}_{10}$ last.

The separated peaks from the GC column were detected at different times (retention times). The retention time of each PCB congener was confirmed by comparing the individual peaks with the retention time of the 12 PCB congeners mixed standard. These 12 congeners are found in Aroclor 1254[®] and run under the same conditions used for Aroclor 1254[®] quantitation. The standard PCB congeners used were all the same concentration and are listed in Table 1.

All quantification was based on peak areas relative to the area of individual congener standards and congener No. 30. A standard curve was constructed for each PCB congener from different concentrations of the standard mixture. The GC conditions for peak separation and quantification were given previously (Pascall *et al.*, 1997a). The polyethylene, oil and water samples were quantified by comparison of peak area using the following equations:

$$\text{Conc. (ppm) inj. vol} = \frac{LS \text{ of congener} \times RA \text{ of congener}}{\text{Recovery \%}} \quad (1)$$

$$\text{Wt. PCB (mg) in sample} = \frac{\text{Conc. inj. vol} \times 0.5 \text{ ml} \times 0.076 \text{ liter}}{0.003 \text{ ml}} \quad (2)$$

In eqn 1, *LS* is line slope and *RA* is retention area.

Data analysis

Mass balance determination A mass balance of the weights of PCBs in the water and the polyethylene were performed. The results of this determination can be used to show that the PCB loss from the aqueous phase diffused into the polyethylene material. These ratios were obtained by taking the sum of the PCB concentrations in the water and in the polyethylene and dividing by the initial PCB concentration in the water. This was expressed in the following equation:

$$\text{Mass bal. ratio} = \frac{(\text{wt. PCB in H}_2\text{O at time}_t) + (\text{wt. PCB in plastic at time}_t)}{\text{wt. PCB in H}_2\text{O at time}_o} \quad (3)$$

In an ideal system, the mass balance ratio should be equal to one (1). In such cases, the concentration of the penetrant removed by the polymer would be equal to the concentration lost from the surrounding medium. However, due to experimental errors one might get small fluctuations above and below this ideal.

A mass balance determination was not performed on the oil/polyethylene system because the polyethylene in this case was not analyzed for its PCB content. This was not done because of difficulty with removal of residual oil from the material without removing adsorbed PCB on the material's surface.

RESULTS AND DISCUSSION

Weights of the PCB congeners (g) in the water exposed to the polyethylene as a factor of time (h) are presented graphically in Figs 1 and 2. Figure 1 shows the tri- to penta-chlorinated PCBs while hexa-chloro to deca-chlorinated PCBs are depicted in Fig. 2. The weights of these congeners in the polyethylene material are similarly shown in Figs 3 and 4. The graphs showing the uptake of PCBs by the polymer seem to be an inverted mirror image of the graphs showing the weight loss of PCBs from the water. This is confirmed by the mass balance ratios shown in Table 2. Therefore, mass balance demonstrates good recovery of the original

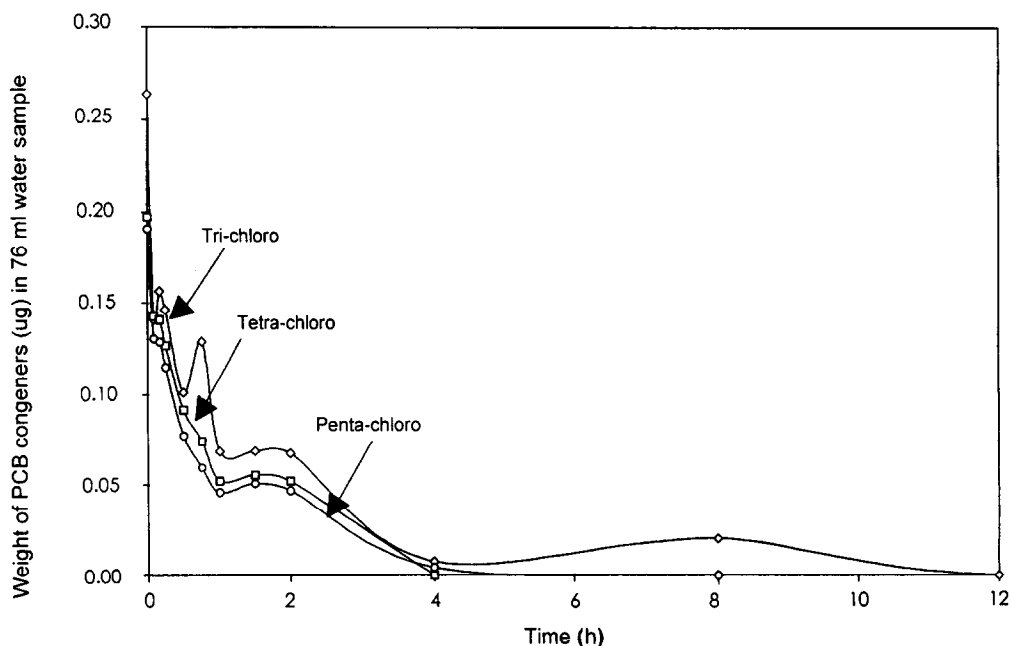


Fig. 1. Weight of tri- to penta-chloro PCB in water exposed to polyethylene in an aqueous system.

PCB and that the analyte in the water moved into the plastic. Table 3 gives the mean concentrations and standard deviations of each PCB congener in water and oil. The PCB congener concentrations were significantly higher ($p < 0.01$) in the oil than in the water. Variation was greater for the mean concentrations for the low levels in the water than for the higher levels in the oil.

The percent PCB congeners retained in the peanut oil are graphically depicted in Figs 5 and 6. These figures show relatively little loss of PCB from the oil. The

rate loss of PCBs from the water and from the oil is presented in Fig. 7. Comparison of each curve shows that the rate loss of PCBs from water is greater than that from the oil. The lower chlorinated congeners showed a larger rate loss than the congeners with higher chlorination in the aqueous medium. In the oil a reverse trend is observed, where the higher chlorinated congeners generally show a larger rate loss.

Water differs from PCBs and other similar organic compounds by its smaller molecular size and polarity. It

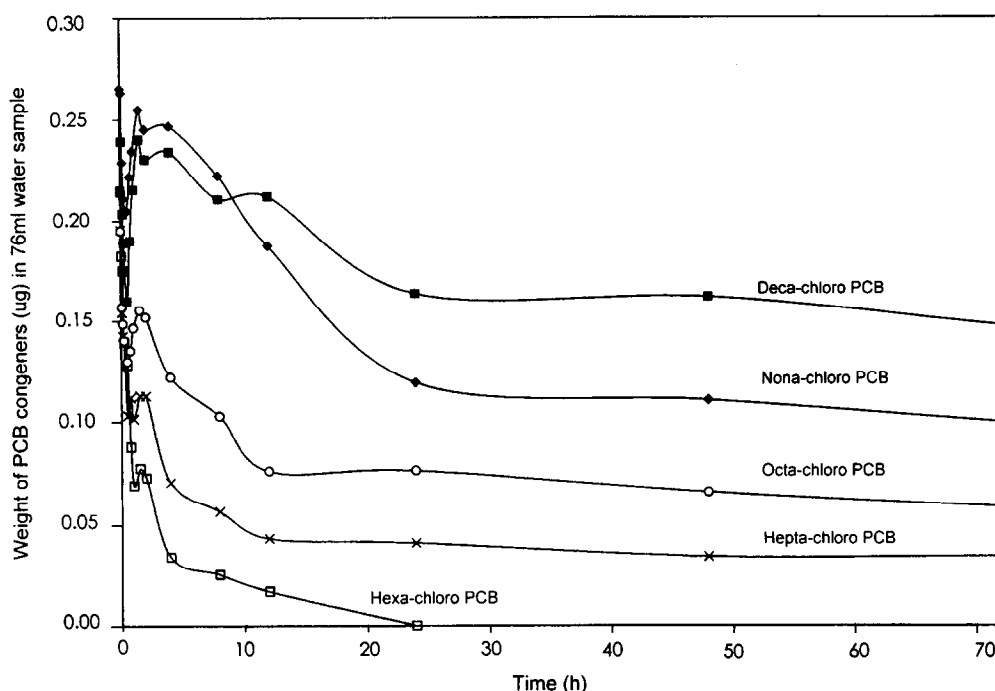


Fig. 2. Weight of hexa- to deca-chloro PCB in water exposed to polyethylene in an aqueous system.

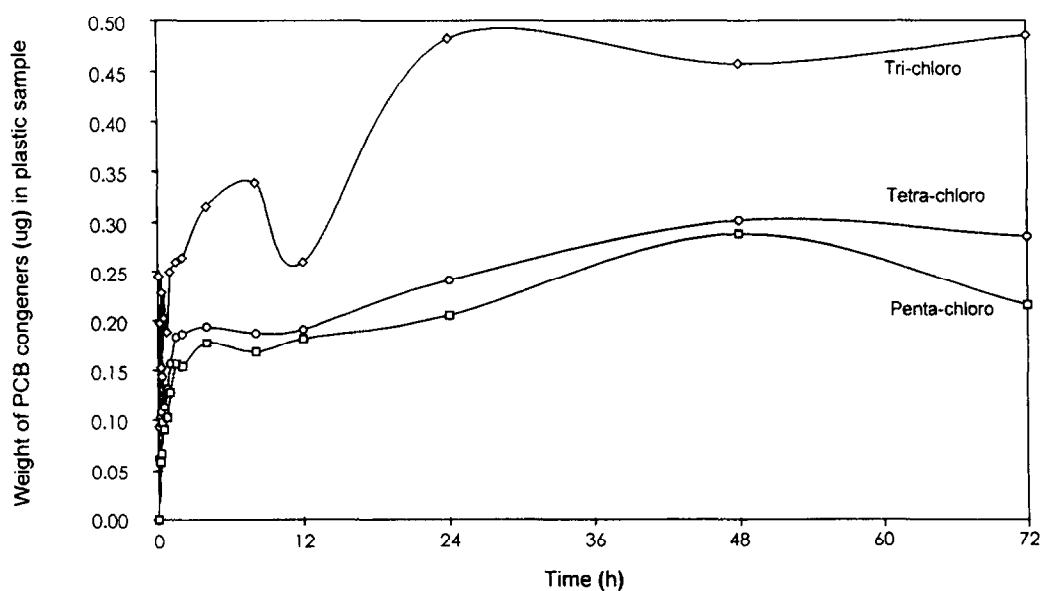
Table 2. Mass balance ratios for PCB congeners in polyethylene/water. Quantitation corrected for recovery of the internal standard

Time (h)	Tri	Tetra	Penta	Hexa	Hepta	Octa	Nona	Deca
0.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
0.08	1.46	1.20	1.00	1.21	1.20	0.94	0.99	1.12
0.17	1.34	1.23	0.98	1.23	1.21	0.97	0.86	0.95
0.25	1.13	1.19	0.95	1.08	1.21	0.94	0.80	0.88
0.50	1.15	1.04	0.88	1.09	1.08	0.94	0.77	0.75
0.75	1.21	1.04	0.85	0.96	1.12	0.98	0.84	0.89
1.00	1.21	1.06	0.91	0.99	1.12	1.10	0.88	1.01
1.50	1.25	1.21	1.09	1.14	1.24	1.23	0.96	1.12
2.00	1.26	1.21	1.05	1.06	1.40	1.16	0.92	1.08
4.00	1.23	0.98	0.95	0.97	1.09	1.12	1.16	1.09
8.00	1.36	0.95	0.88	0.88	1.10	1.07	1.06	1.13
12.00	0.98	0.97	0.95	0.91	1.05	1.05	1.06	1.32
24.00	1.83	1.22	1.08	1.17	1.17	1.16	0.84	1.20
48.00	1.74	1.53	1.51	1.30	1.39	1.40	1.00	1.39
72.00	1.84	1.45	1.13	1.19	1.66	1.23	0.90	1.26

can also be described as being in a condensed state with the formation of hydrogen bonds (Chainey, 1989). Thus, in contact with the lipophilic polyethylene, one should expect clustering of the water molecules to occur. This has been shown in experiments performed by Thornton *et al.* (1958); Puffr & Sabenda (1967); Vergnaud (1986); and Yang *et al.* (1985). When in contact with polyethylene, this clustering will be expected to hinder the transport of dissolved PCBs through the polymer, since only the monomeric species are small enough to diffuse. Hernandez *et al.* (1992) reported that clustering could reduce the effective mobility of water either by increasing the size of the diffusing group or by increasing the tortuosity of the diffusion pathway. These processes are by no means simple and clear cut. Chainey (1989) reported that complications can arise depending on the polymer type exposed to the water. He further

stated that heat of sorption can be large, producing temperature rises of several degrees which can lead to errors in interpretation of the data, and indeed this may have been a factor in this study.

Another factor influencing the nature of the data obtained for the loss of PCBs from the aqueous system is the difference in molecular size of the congener species tri- to deca- chlorinated. This has been dealt with in another paper by Pascall *et al.* (1997b). The molecular size of each congener was correlated to its diffusion potential. From this deduction, the conclusion was made that the larger molecular size congeners showed more difficulty in diffusing through the polymeric matrix in an aqueous solution. The result shown in Fig. 7 in this study confirms this notion, showing that the smaller congeners generally displayed a greater rate loss from the water than their larger counterparts. The

**Fig. 3.** Weight of tri- to penta-chloro PCB uptake by polyethylene in an aqueous system.

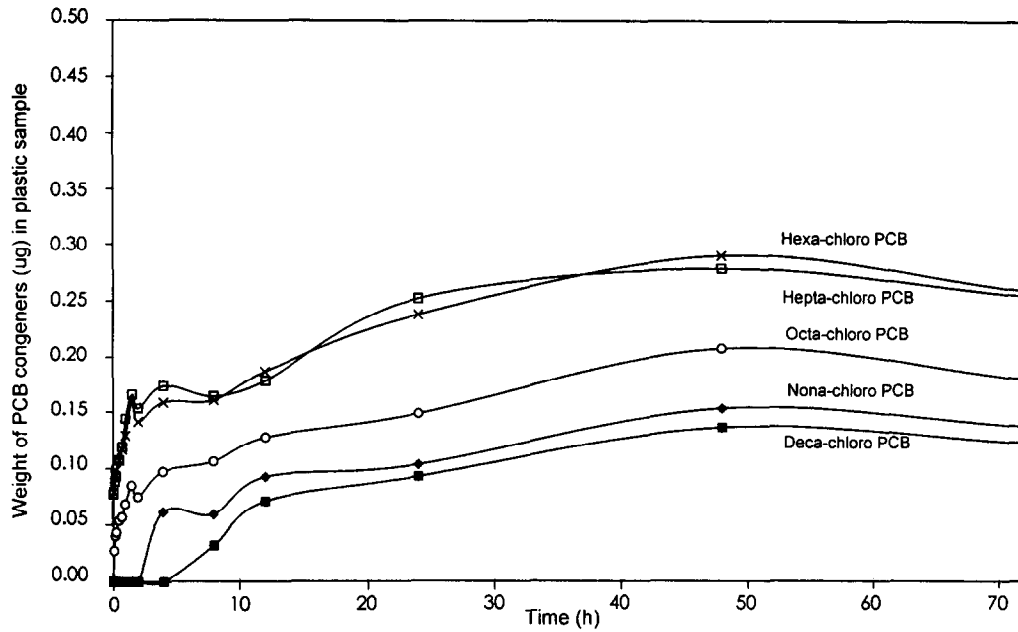


Fig. 4. Weight of hexa- to deca-chloro PCB uptake by polyethylene in an aqueous system.

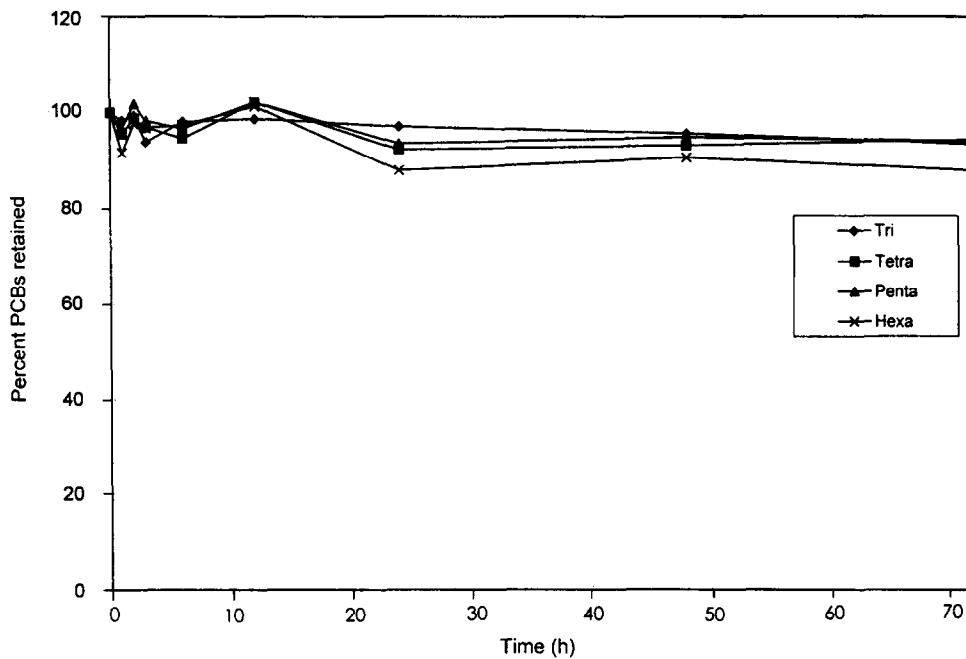


Fig. 5. Percent tri- to hexa-chloro PCB retained in oil exposed to polyethylene.

Table 3. Mean concentrations and standard deviations of PCB congeners in water and oil at 25°C

Congener	Mean ^a + S.D. for Water	Mean ^b + S.D. for Oil	F-Value
Trichlorinated	0.081 ± 0.075	2.575 ± 0.097	15351
Tetrachlorinated	0.069 ± 0.065	1.680 ± 0.071	9952
Pentachlorinated	0.063 ± 0.061	1.716 ± 0.075	10705
Hexachlorinated	0.081 ± 0.074	1.819 ± 0.123	5820
Heptachlorinated	0.096 ± 0.051	1.050 ± 0.071	4545
Octachlorinated	0.121 ± 0.042	0.790 ± 0.052	3769
Nonachlorinated	0.202 ± 0.059	0.478 ± 0.039	474
Decachlorinated	0.190 ± 0.049	0.430 ± 0.031	524

^an = 48 ^bn = 27.

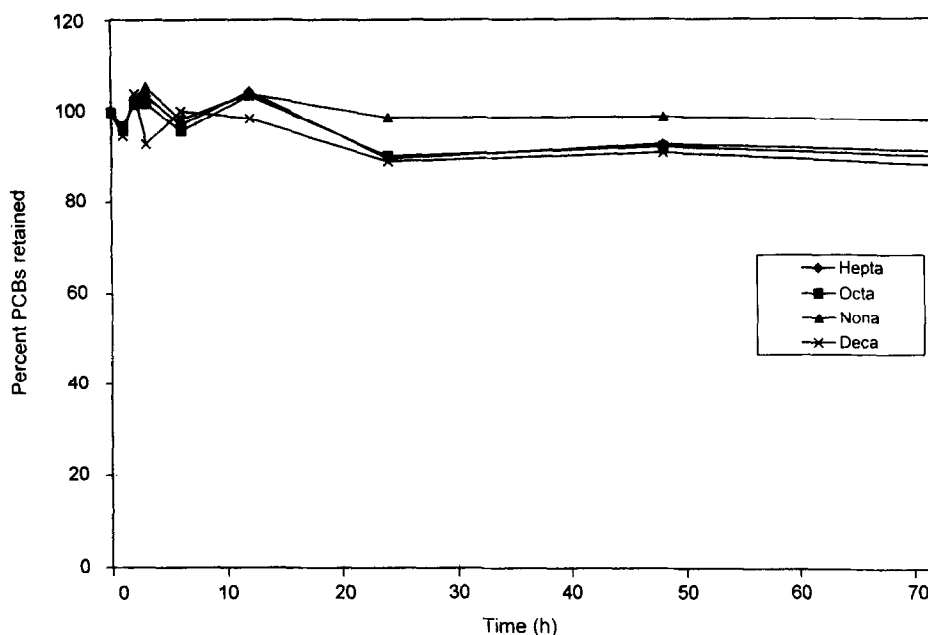


Fig. 6. Percent hepta- to deca-chloro PCB retained in oil exposed to polyethylene.

lower rate loss obtained for the tri-chlorinated congener when compared to that of the tetra and penta congeners (Fig. 7) may be due to the lower sensitivity of the electron capture (EC) detector on the gas chromatograph instrument for compounds with lower numbers of chlorine atoms as compared to others with larger numbers of chlorines.

In the case of polyethylene exposed to the oil, it can be reasonably assumed that both PCB and the oil would have a higher partition coefficient for the non-polar polymer when compared to water molecules. Thus, if both the PCB and oil exerted a plasticizing effect on the plastic material, swelling of the film would have been

expected. Indeed, this may explain why the higher chlorinated congeners appeared to diffuse with greater concentration into the plastic when compared to the trend in the aqueous system. A deviation from this trend was observed for the nona-chlorinated congener when compared to its neighboring counterparts in Fig. 7. This may be due to the arrangement of the chlorine atoms on its biphenyl rings. Since several congeners of nona-chlorinated PCBs are known to exist, their differing molecular arrangements may influence relative differences in their uptake rates by a given polymer to which they may be exposed. Thus, the selection of another nona-chlorinated congener might

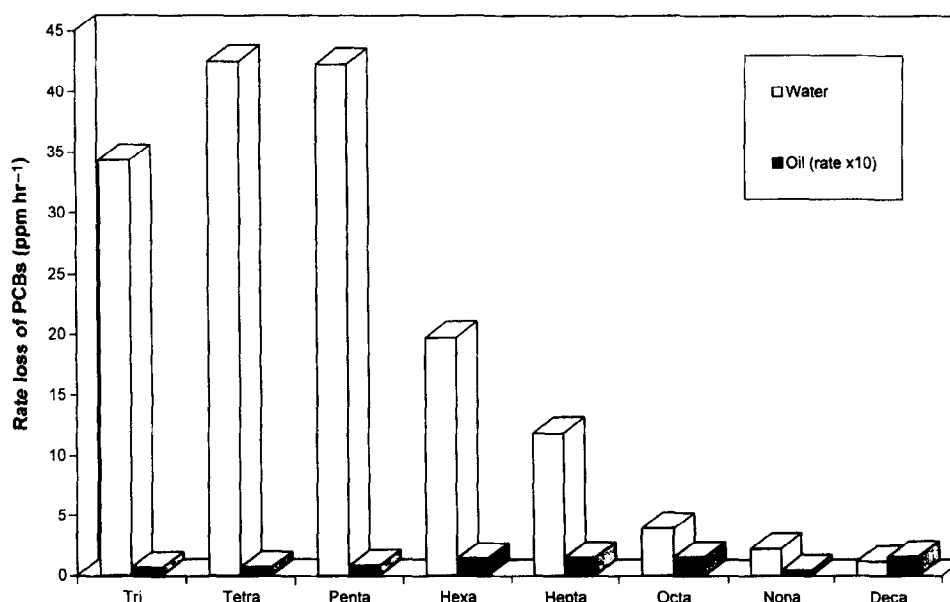


Fig. 7. Comparison of rate loss for all congeners in water and oil exposed to polyethylene.

Table 4. Food and food simulants (Vergnaud, 1986)

Group	Food	Food simulant
I	Watery, nonacidic food (pH > 5)	Distilled water
II	Watery, acidic food (pH < 5)	3 wt % acetic acid
III	Watery, acidic or nonacidic fatty food	Same as those for Groups I, II, & IV
IV	Fatty foods	Heptane, diethyl ether, edible oil
V	Liquids with 5% ethanol	20–50 wt % ethanol

have given a more uniform trend. Since the partitioning of PCBs for the oil is also expected to be relatively higher, it is not surprising that the rate loss of PCBs from the oil (Fig. 7) was quite low, when compared to the rate loss of PCBs in the aqueous phase.

The second major factor influencing diffusion of the PCB congeners in the oil is the change in polarity of the biphenyl rings as we go from tri- to deca-chlorination. Hutzinger *et al.* (1974) reported that the solubility of PCB decreases in water with increasing chlorination. This is indeed so because the influence of chlorination disrupts the electron cloud within the biphenyl rings and thus introduces greater non-polar influences. This may explain why a small, but increasing rate loss of the congeners from tri- to deca-chlorination in the oil was obtained when exposed to polyethylene (Fig. 7). In addition, the synergistic plasticizing effect of both oil and PCB on the material would be expected to increase the ease to which the higher chlorinated congeners would diffuse into the polymer. This trend in diffusion pattern of the congeners differs from that obtained for the polymer/water exposures (Fig. 7), where the effect of increasing molecular size (from tri- to deca-chlorinated) had a limited effect on the diffusion of the larger size congeners.

Similar results were obtained by Grigorakakis *et al.* (1985) when they studied the diffusion of plasticizers in PVC exposed to vinegar and acetic acid solutions with an increasing water percent mixture. Obtaining similar trends in a different study, Messadi and Vergnaud (1981) reported that the effect of water is an important factor in the transport of diffusants in packages made of PVC.

Due to the complex nature of many commonly consumed food systems, problems arise in identifying many species migrating from foods to packaging systems. Thus having a model system with a resemblance to the physical/chemical characteristics of the real food in its behavior and interaction with the plastic packaging allows for the determination of the translocation of a chemical component from the food to the package system. Vergnaud (1986) also reported on the use of simulants for various types of foods; these are shown in Table 4.

The use of peanut oil and water in this study can thus be used to model the diffusion characteristics of many polar and non-polar compounds naturally found in foods or added either intentionally or unintentionally. Many researchers have shown the migration of limo-

nene for example, from orange juice to low density polyethylene (Sadler & Braddock, 1991; Habek & Luttman, 1991). On the other hand, the migration of polymeric adjuvants to various food types has been documented by Limm and Hollifield (1995) and Little (1990). Thus, if a given food contaminant is exposed to judiciously selected polymers, a reduction of this compound in the food system can be achieved. This is significant since there is little published research on the use of food grade packaging polymeric films as vehicles for contaminant reduction in food systems.

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