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Characteristics of sorption losses of polychlorinated biphenyl congeners onto glass surfaces

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Abstract

Sorption losses to glass surfaces of five polychlorinated biphenyl (PCB) congeners in aqueous solutions were investigated. Adsorption/desorption experiments were conducted under conditions that simulated actual sample handling procedures for environmental samples. It was found that the adsorption loss is related to the degree of chlorination. PCB congener 180 lost the most onto glass surfaces, followed by congeners 138, 101/28, and 52, in decreasing order. More PCB adsorption occurred onto glass under conditions of agitation and higher temperature (22°C) during the five-day experimental period. The salinity effect ("salting out effect") was also observed in this work. The efficiency of desorption (rinsing three times with solvent) was found to be ineffective in extracting adsorbed PCBs. It was necessary to use mechanical shaking for extraction. Storage of samples up to five days resulted in sorption losses as much as 30%, 17%, 30%, 40%, and 55% of PCB 28, 52, 101, 138, and 180, respectively. Sorption losses need to be considered when conducting water sampling or toxicological studies to avoid underestimation of the actual PCB concentrations and their toxic effects. © 2000 Elsevier Science Ltd. All rights reserved.

Keywords: Polychlorinated biphenyls; Sorption loss; Environmental sampling

1. Introduction

Polychlorinated biphenyls (PCBs) have been extensively studied due to their toxicity, omnipresence and persistence in the environment. PCBs in the environment tend to accumulate in sediments (Muir et al., 1996) and then diffuse from sediment pore water into the overlying water (Hunchak-Kariouk et al., 1997). Hydrophobic PCBs in water would either accumulate in biota (Jackson and Schindler, 1996; Beliaeff et al., 1997) or evaporate (Achman et al., 1993; Honrath et al., 1997). Assessment of PCB concentrations in water is important to understand the fate and transport of PCB congeners.

To avoid analytical interference from plastic products, glassware has to be used in virtually all sampling and extraction procedures and in laboratory chamber experiments involving PCB exposures. However, unaccountable sorption losses lead to underestimates of PCB concentrations and/or their toxic effects. Hattula and Karlog (1971) showed that the recovery of total PCBs in aqueous solution (0.1 ppm) was 11% after 52 h of storage in glass bottles. Pepe and Byrne (1980) found that two-thirds of 2,2′,4,4′,5,5′-hexachlorobiphenyl (IUPAC No. 153) (1.18 ppm) was lost to the glass surfaces after 144 h of storage. Muldrew et al. (1981) also reported a 45% loss of PCB 153 (5 ppm) after 72 h of storage. These losses during analytical

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manipulations were due to sorption rather than volatilization.

PCB congeners are expected to have different adsorption behaviors since they possess different physical and chemical properties. Earlier researchers focused on either total PCBs or single PCB congener adsorption in distilled water at room temperature; PCB adsorption behavior under different conditions was not examined. This paper describes adsorption/desorption characteristics of a five PCB congener mixture to glass under conditions designed to simulate actual sample handling procedures and different experimental conditions.

2. Methods and materials

Borosilicate glassware was chosen because it is used extensively in laboratories. It is comprised mostly of SiO_2 (81%) and B_2O_3 (13%), with lesser amounts of Al_2O_3, Na_2O, and K_2O (Corning Glass, 1994/1995). Forty-milliliter borosilicate glass vials with Teflon lined caps (Scientific Specialties Service, Randallstown, MD) were used as the experimental vessels. The total contact surface area between water and glass is approximately 68 cm^2 and the area to volume ratio is therefore 1:1.7.

Adsorption of PCBs to glass may be a function of the degree of chlorination (Mackay and Shih, 1992). One congener from each of the isomer groups Cl-3 to Cl-7 was selected, including congeners IUPAC No. 28 (2,4,4′-trichlorobiphenyl), 52 (2,2′,5,5′-tetrachlorobiphenyl), 101 (2,2′,4,5,5′-pentachlorobiphenyl), 138 (2,2′,3,4,4′,5-hexachlorobiphenyl), and 180 (2,2′,3,4,4′,5,5′-heptachlorobiphenyl). These five congeners were chosen based on their prevalence in the environment, their toxicity and their analytic advantages (do not coelute with other equally abundant congeners) (McFarland and Clarke, 1989; Schulz et al., 1989; Achman et al., 1993). Certified PCB congener solutions (99% pure in hexane) were purchased from Ultra Scientific (North Kingstown, RI); the PCB spiking solution was prepared by dissolving concentrated forms of these five congeners in acetone. All solvents were purchased from J.T. Baker, Phillipsburg, NJ (ultra resi-analyzed).

To ensure that the solution was not saturated, 50% of the lowest value among commonly reported solubilities of these hydrophobic compounds (as single PCB in water (Mackay and Shih, 1992)) was chosen as the initial concentration in the distilled water experiments. It was approximately 6.1, 1.7, 2.2, 0.64, and 0.10 μg/l for congeners 28, 52, 101, 138, and 180, respectively. Since the addition of salts would enhance the polarity of aqueous solutions and reduce the solubilities of hydrophobic chemicals, the initial PCB concentrations in all saline water experiments were one-fifth of those in the distilled water experiments to account for the "salting out" effect (Schwarzenbach et al., 1992). PCB 180 was excluded from saline water experiments because the resulting concentration was close to the analytical detection limit.

PCB losses to glass surfaces may depend on the degree of agitation. To simulate agitating conditions, glass vials were filled with 40 ml water, spiked with PCBs, capped and sealed tightly with Teflon tape and placed on an orbital shaker (Lab-Line Instruments, Melrose Park, IL). Vials were then shaken at 300 rpm (room temperature, 22°C) for 5 min, 24, 48, 72, 96, and 120 h. PCB concentrations were determined for the aqueous solution (water sample), and adsorbed PCBs extracted from the glass surface by solvents (surface sample).

Adsorption under quiescent conditions was assessed following similar procedures from vials allowed to stand at room temperature (22°C) for 1, 24, 48, 72, 96, and 120 h. For these experiments, pre-spiked PCB solution was stored in a 500 ml borosilicate bottle at 4°C for at least five days to allow PCB partitioning to reach a quasi-equilibrium state before use. The resulting initial PCB concentrations in aqueous solutions were similar for both quiescent and agitating conditions.

To evaluate the effect of temperature, distilled and saline water experiments were conducted at room temperature (22°C) and 5°C. The temperature conditions at 5°C were maintained by conducting experiments inside an incubator. 1.4 g of artificial sea salt (Sigma, St. Louis, MO) was added to 40 ml distilled water to reach 3.5% salinity in saline water experiments.

The ratio of PCB amounts remaining in water was calculated to assess the decrease of PCB mass in the water solution (loss to glass surface). In addition, during the experimental process, PCBs may evaporate into the headspace of the glass vials. Mass balances were estimated to assess the significance of this route of PCB loss. PCB amounts obtained from water and surface samples were combined for comparison with PCB-spiked amounts to assess total recovery.

To extract adsorbed PCBs, solvent-rinsing of glass containers is a common practice (US EPA, 1984). In the desorption experiments, the efficiency of rinsing three times with solvent was evaluated. Glass vials were first conditioned by filling with 40 ml of PCB-spiked aqueous solution for 24 h under agitating/quiescent conditions at 22°C. The aqueous solution was then decanted. The vial was rinsed with 2:8 (v/v) acetone/hexane three times and shaken by hand vigorously for 20 s (first surface sample). The vial was then filled with 40 ml hexane and shaken at 300 rpm for different time intervals to obtain subsequent surface samples. Amounts extracted at each time interval were summed to provide the total extracted PCBs for this set of experiments.

Following adsorption or desorption experiments, the aqueous solution was decanted into a 50-ml extraction glass vial. Water samples were spiked with two surrogates (PCB 18 and 103) which assessed the efficiency of the extraction procedures. Samples were then extracted with 5 ml of hexane three times, each time hand-shaken vigorously for 30 s.

For surface samples, the experimental vial was filled with 2:8 (v/v) acetone/hexane and shaken for at least 2 h at 300 rpm. The desorption experiments (described previously) showed that shaking for 2 h was sufficient to extract PCBs adsorbed under various storage conditions up to 24 h. For samples with longer adsorption times, 24 h of shaking was needed to extract most PCBs out of glass surfaces. Samples were spiked with two surrogates after extraction to assess any loss in the following steps.

PCB analysis was performed on a Hewlett Packard 5890A Gas Chromatography, equipped with a 30 m × 0.25 mm ID × 0.25 μm Alltech DB-5 capillary column. The temperatures of the injection port and the electron capture detector were 280°C and 300°C, respectively. Quantification of GC results was conducted by the internal standard method.

3. Results

3.1. QA/QC

The results of 13 procedural and laboratory blanks showed minimal contamination, mostly less than 0.3 ng for the congener specific analysis. Samples were not corrected for blank values. Method detection limits (MDLs) were 0.32, 0.46, 0.77, 0.45, and 0.27 μg/l for PCB congeners 28, 52, 101, 138, and 180, respectively. All data used in this paper were above the MDLs.

Ten triplicates were examined in the adsorption experiments (≈10% of total samples) to assess precision. US EPA (1994a) method 525.2 accepts percent differences between replicates as high as 30%. All triplicate data for five congeners (total $N = 50$) had %RSD (standard deviation over mean) less than 16% except for two, which were 25% and 34%. The acceptable range in US EPA (1989b) method 508 for percent recovery is from 70% to 130%. The percent recoveries of spiked surrogates were all within 69–109%. The majority (96.6%) of the recoveries was above 80%, indicating good extraction efficiency. Eighty-seven percent of total recoveries (for mass balance calculation) of five congeners in adsorption experiments was between 80% and 109%.

3.2. Adsorption experiments

The results of the adsorption experiments are presented in Figs. 1(a)–(d) and 2(a)–(d). Figs. 1(a)–(d) show a clear trend of PCB mass decrease in distilled water solutions. At 5°C under quiescent conditions, the percent losses were not substantially different among congeners (Fig. 1(a)), ranging from 10% to 20% after 120 h. However, under agitating condition, there were large differences among congeners (Fig. 1(b)). Approximately 60% of PCB 180 was lost to adsorption after 120 h, while PCB 52 only lost around 11% of its original amount.

After 120 h at 22°C, PCB 52 lost about 14% and PCB 180 lost around 40% of their mass from distilled water under quiescent conditions (Fig. 1(c)), with the other congener losses in between. Fig. 1(d) shows that all five congeners suffered the greatest losses under agitating conditions at 22°C, compared with the other distilled water experiments (Figs. 1(a)–(c)). At 120 h, PCB 28, 52, 101, 138, and 180 lost about 45%, 22%, 45%, 67%, and 87% of their amounts, respectively.

Figs. 2(a)–(d) show PCB mass decrease in saline water. Similar to distilled water, at 5°C, under quiescent conditions, approximately 10–20% PCBs were lost at the end of the experimental period, with little difference among congeners (Fig. 2(a)). Under agitating condition at 5°C, PCB 28, 52, 101, and 138 lost approximately 33%, 18%, 43%, and 59%, respectively, after 120 h (Fig. 2(b)).

Fig. 2(c) shows PCB losses in saline water under quiescent conditions at 22°C. PCB 138 was omitted from the dataset due to suspected contamination of this particular set of samples. At 120 h, PCB losses were somewhat greater than those in distilled/saline water at 5°C (Figs. 1(a) and 2(a)), and those in distilled water at 22°C under quiescent conditions (Fig. 1(c)). Under agitating conditions at 22°C (Fig. 2(d)), PCB losses in saline water were quite different among congeners, with PCB 28, 52, 101, and 138 losing about 46%, 22%, 48%, and 60% of their mass, respectively. The loss of PCB 138 was slightly less than that under agitating conditions at 22°C in distilled water (Fig. 1(d)), while the losses of other congeners were quite similar.

3.3. Desorption experiments

Desorption experiments were conducted only on vials adsorbing PCBs for 24 h under agitating and quiescent conditions at 22°C. Figs. 3(a)–(c) show the time course of solvent-extraction of PCB congeners from glass surfaces. The additional amounts extracted after 120 min were negligible; thus, only the PCB amount extracted in the first 2 h were summed as 100% adsorbed PCBs. The percentages of PCBs extracted by initially rinsing three times with solvent are indicated at time zero, representing the efficiency of common laboratory desorption practices.

Figs. 3(a) and (b) show the time course of PCB desorption for vials previously containing distilled water under agitating and quiescent conditions, respectively. The percentages of PCBs extracted by three solvent rinses were less than 40% for all congeners; the

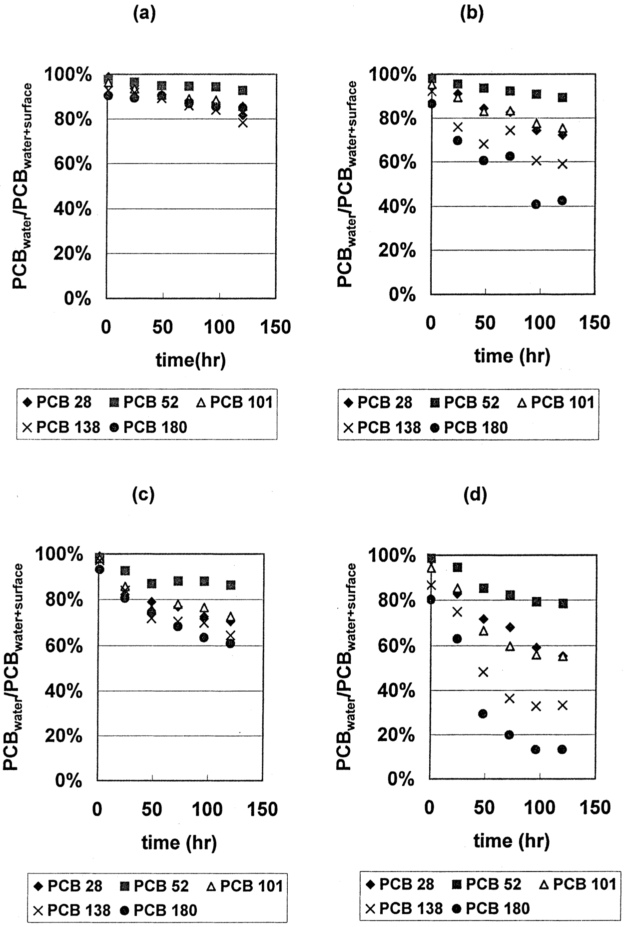

Fig. 1. Time courses of PCB congener losses in distilled water due to glass adsorption under various conditions. PCB_{water}: PCB amounts remaining in water, $PCB_{water+surface}$: PCB amounts remaining in water plus PCBs adsorbed on glass surfaces; (a) at 5°C, quiescence; (b) at 5°C, agitation; (c) at 22°C, quiescence; (d) at 22°C, agitation.

percentages varied about 15% among congeners in any given time.

In contrast, the percentages of PCBs extracted by three solvent rinses were higher for vials previously containing saline water under quiescent condition (Fig. 3(c)). There were substantial differences between congeners, with higher chlorinated congeners having greater percentage recoveries at any given time. The percentages were somewhat higher than those in the distilled water experiments although lower PCB concentrations were initially added to saline water.

4. Discussion

4.1. QA/QC

The total recoveries of our samples ranged from 58% to 121%, with the majority (87%) between 80% and 109%. Higher chlorinated congeners (less volatile) had slightly higher total recoveries. Most of the variability could be attributed to percent recoveries of the extraction procedures, which were within approximately the same range (69–109%). However, three sets of total recoveries (below 69%) could not be explained by the losses during the extraction process. Those were data of PCB 28 in saline water of vials shaken at 22°C for more than 48 h. The most likely route for additional PCB loss was evaporation. The vials were capped tightly and sealed with Teflon tape during experiments, resulting in less than 3.5 cm^3 headspace. Under lower temperatures and/or quiescent conditions, PCB loss due to evaporation was negligible. However, when vials were shaken at higher temperature, the partitioning of PCBs into the air may have become more and more significant. PCB 28 is the most volatile congener of these five PCBs; thus, substantial evaporation may occur under agitation longer than 48 h.

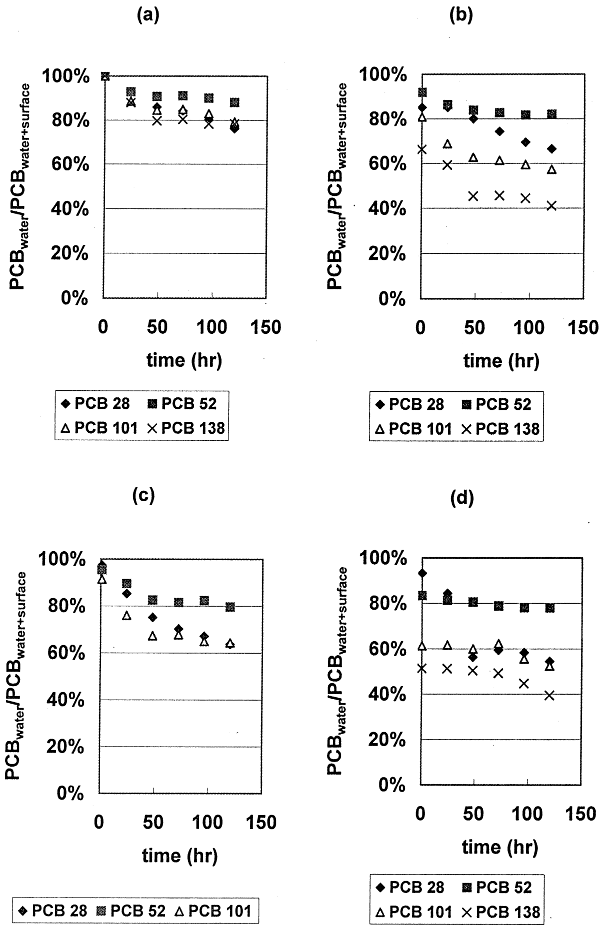

Fig. 2. Time courses of PCB congener losses in saline water due to glass adsorption under various conditions. PCB_{water}: PCB amounts remaining in water, $PCB_{water+surface}$: PCB amounts remaining in water plus PCBs adsorbed on glass surfaces; (a) at 5°C, quiescence; (b) at 5°C, agitation; (c) at 22°C, quiescence; (d) at 22°C, agitation.

4.2. Experiments

The percent losses of congener 180 were the greatest among congeners in any period of time, regardless of agitating or quiescent conditions. The losses of congeners 138, 101/28, and 52 followed in decreasing order. The more highly chlorinated PCBs are more hydrophobic and hence adsorbing more to relatively non-polar glass surfaces. However, congener 28 was an exception. The percent losses of PCB 28 were greater than those of PCB 52, which has one more chlorine atom and was thought to be more hydrophobic (the reported solubility of PCB 28 is greater than that of PCB 52 (Mackay and Shih, 1992)). One possible explanation is that the orientation of the molecules somehow affects the adsorption of PCB 28. The other congeners all have chlorine atoms on positions 2 and 2′ so they cannot assume the coplanar configuration; PCB 28 is the only one that can rotate freely and assume any configuration. As PCB 28 is in the coplanar configuration, its own polarity is reduced and may become more easily associated with glass surfaces than PCB 52. PCB adsorption behaviors may, therefore, not only relate to degree of chlorination but also the configuration of the congener. The adsorption losses of these five congeners cannot therefore be taken as entirely representative of their isomer groups. However, they can serve as a rough estimation of the losses of other similarly structured congeners within the groups.

All congeners adsorbed more onto glass surfaces under conditions of agitation, which was expected since kinetic processes would be accelerated. The PCB amounts adsorbed under agitation were as much as two to four times of those under quiescent conditions. The extent of PCB adsorption during actual sample handling and experimental conditions may fall between the two extreme conditions, quiescent and agitating. Samples in rough shipping conditions and chamber experiments

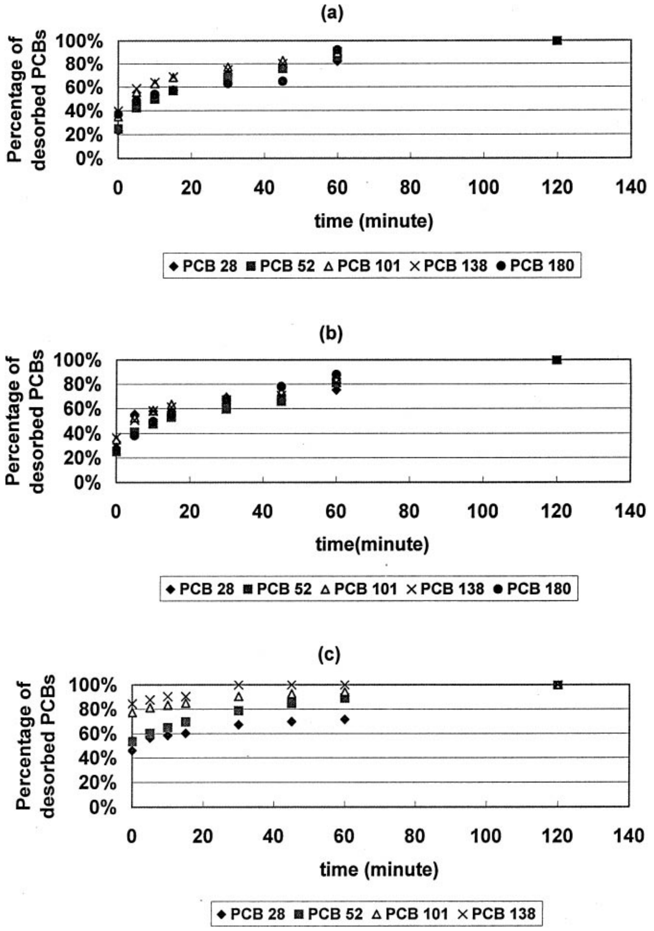

Fig. 3. (a) Desorption of PCB congeners in distilled water under agitating conditions; (b) desorption of PCB congeners in distilled water under quiescent conditions; (c) desorption of PCB congeners in saline water under quiescent conditions.

requiring agitation would suffer higher losses of PCBs onto glass containers.

It was speculated that PCB congeners might adsorb more onto glass surfaces in saline water due to salting out effects, i.e., hydrophobic PCBs are less soluble in the more polar aqueous environment. More congener loss was noted in saline water than distilled water under quiescent conditions and at 5°C under agitation; most of the losses were no more than twice of those losses in distilled water. However, PCB losses were almost the same in distilled and saline water at 22°C under agitation, except for PCB 138. Lower total recoveries discussed earlier under this experimental condition may result in this discrepancy. As a result of their lower solubility, PCBs might be more susceptible to evaporation in saline water. Thus, the salting out effect was partially observed in our work; it could be masked by other factors, such as evaporation. The effect of salinity is therefore difficult to predict and warrants further research.

Congener losses were greater at 22°C in the five-day experimental period, again reflecting faster kinetic processes at higher temperatures. The losses at 22°C can be as much as twice the losses at 5°C. Therefore, PCB adsorption can clearly be reduced by keeping samples at lower temperatures. For chamber experiments designed to run at higher temperatures, PCB adsorption loss should be considered in interpreting results. Higher temperatures may also increase evaporation and, therefore, more PCBs could be lost to the headspace. However, the area for evaporation is limited to the top layer of the aqueous solution. In general, losses to adsorption were much greater than losses to evaporation, as determined by the total recovery calculation. This is consistent with the findings of an earlier study (Muldrew et al., 1981).

From the results, it was not obvious that the adsorption processes reached equilibrium. The percent losses of mass at the end of the experimental period (120 h) may be an underestimate for samples with long storage or experiment duration. There are several official EPA methods relevant to the sampling and analysis of PCBs; samples are allowed to be stored from seven to 14 days in those methods (US EPA 1989a,b, 1994a,b). There are no indications of a preservation method to minimize PCB adsorption losses. The results of our experiments can be used as a basis to estimate PCB adsorption losses during storage and experiment duration. In addition, water chambers have been used in toxicological studies to investigate the accumulation and the toxic effects of PCBs on the food web (Safe, 1988). Adsorption losses could potentially result in significant errors in data interpretation in conducting those studies.

Brownawell (1986) observed that 7.3%, 12%, 21%, 29%, and 58% of the congeners 8, 28, 70, 87, and 138 were lost to the glass walls, respectively, in a 1-l Pyrex glass bottle after 24 h on an orbital shaker at room temperature. Under similar conditions, 17%, 5%, 15%, 25%, and 37% of congeners 28, 52, 101, 138, and 180 (isomer groups 3–7), respectively, were adsorbed onto glass surfaces in our experiments. PCB 153, from the same isomer group as PCB 138, was lost 45% and 66% after three and six days of storage in distilled water at room temperature, respectively (Pepe and Byrne, 1980; Muldrew et al., 1981). From our experiments, about 29% and 35% of PCB 138 were lost to glass surfaces after three and five days of quiescent storage at 22°C, respectively. Differences in glass containers (with different area to volume ratios), chosen congeners, and experimental conditions may account for part of the discrepancies among studies. Nevertheless, previous results all indicated significant loss of PCBs to glass surfaces when sampling for PCBs or conducting experiments.

Our desorption experiments showed that the common practice of rinsing glass containers with solvents to extract adsorbed-PCBs was inefficient. Solvent-rinsing three times extracted less than 40% of the adsorbed congeners in vials containing distilled water after 24 h of storage. Two hours of mechanical shaking was needed to extract almost all the adsorbed PCBs from the 24-h experiments. For experiments longer than 24 h, shaking for 24 h was necessary to extract most PCBs from the glass surfaces.

The sorption loss to glass surfaces is the product of the percent adsorbed PCBs and the percentage which cannot be extracted by three solvent rinses. Thus, the sorption loss can be expressed as follows:

$$\begin{aligned}&\text{percentage of sorption loss}\\&\quad=\text{percentage of adsorbed PCBs}\\&\qquad\times(100\%-\text{percentage of desorbed PCBs}).\end{aligned}\tag{1}$$

The results of Eq. (1) based on two different scenarios are shown in Table 1. In the most conservative estimation, samples in distilled water solutions were kept at 5°C under quiescent conditions for 24 h before extraction. The losses are not substantial (Table 1(a)). However, in some field surveys, it is not feasible to analyze PCBs in one day. In the worst-case scenario in our study, samples in distilled water were kept at room temperature (22°C) under agitation for five days (Table 1(b)). The unrecoverable losses of all five congeners are more than 10% of their initial values and are also different among congeners. Thus, this sample handling procedure would underestimate actual concentrations in the environment and would also give inaccurate information about the congener profile. The extent of PCB sorption losses during actual sample handling and experimental conditions should fall between the previous two extremes, given less than five days of storage. However, if the storage period was seven to 14 days, as allowed in EPA methods, the losses would be much higher.

Table 1
Sorption losses of PCB congener mass: (a) stored for 24 h in distilled water under quiescent conditions at 5°C, (b) stored for five days in distilled water under agitating condition at 22°C

PCB congener	Adsorption (%)	Efficiency of desorption (%)	100% – efficiency of desorption (%)	Sorption loss (%)
(a)				
PCB 28	5	33	67	3
PCB 52	3	25	75	2
PCB 101	6	35	65	4
PCB 138	8	37	63	5
PCB 180	11	27	73	8
(b)				
PCB 28	45	23	67	30
PCB 52	22	25	75	17
PCB 101	45	35	65	30
PCB 138	67	40	60	40
PCB 180	87	37	63	55

5. Conclusion

The significance of sorption losses of PCB congeners to glass surfaces in aqueous solution was demonstrated in this work. Important factors affecting the kinetic process of adsorption included higher degree of chlorination, higher temperature (22°C) and conditions of agitation. The efficiency of desorption (three solvent rinses) was evaluated and shown to be ineffective in extracting adsorbed PCBs. In five days, the sorption losses were as much as 30%, 17%, 30%, 40%, and 55% of PCB 28, 52, 101, 138, and 180, respectively. Mechanical shaking is necessary to minimize these losses when conducting water sampling for PCBs, in order to estimate actual PCB concentrations and provide accurate congener profiles. Along with the advancement of analytical technology, scientists can detect concentrations of these pollutants in ppt levels. With the current sensitivity of analytical techniques, the percent losses of PCB congeners to glass surfaces would result in serious errors in estimating PCB concentrations and subsequent ecological and human risk. Furthermore, in toxicological chamber studies, sorption loss may be difficult to estimate, resulting in underestimation of PCB toxicity.

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