



Polychlorinated biphenyl (PCB) recovery from spiked organic matrix using accelerated solvent extraction (ASE) and Soxhlet extraction

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Abstract

The recovery of five PCB congeners from PCB spiked organic matrices was studied using Accelerated solvent extraction (ASE) and Soxhlet extraction (SE). The chromatogram of ASE extract was found to be relatively clean and similar to that of SE extract. ASE extraction efficiency was dependent on the operation temperature and sample size loading. ASE showed extraction efficiency comparable or slightly higher to that of SE for the PCB spiked organic matrix. PCB recovery from spiked matrix was dependent on the type and molecular weight of congener, and nature of matrix. For some selected PCB congeners, *ortho*-substitution did influence the PCB recovery from graphite matrix. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Organic matrix; Graphite; Coal; PCB congeners; Accelerated solvent extraction

1. Introduction

From 1929 to 1979, approximately 1.4 million pounds of polychlorinated biphenyls (PCBs) have been produced worldwide for usage as dielectric fluids in capacitors and transformers, hydraulic fluids, fire retardants, and plasticizers [1]. For several of these applications, PCBs were chosen because of their physical and chemical stability and their electrical insulating properties. The chemical and biological stability of PCBs has been primarily responsible for PCB accumulation in the environment.

During the period of widespread use of PCBs, the lack of proper disposal methods at industrial discharge points and the accidental release of PCBs have contributed to the

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contamination of soil and sediment near the point of release [2]. PCB contaminated environmental samples have received major attention due to the mutagenic characteristic of PCBs and their known ability to evoke toxic responses in a number of animal species and human beings at high concentrations [3,4]. Because of the mutagenic characteristic of PCB, reliable assessment of contaminant concentration in the environmental samples is important. Measurement of contaminant concentration at the parts-per-million or lower level in complex matrices such as soil with wide range of organic carbon is often challenging. However, accurate quantification of PCBs at these trace levels requires efficient extraction and detection methods to minimize the interferences contributed by the matrix. Several extraction and detection procedures for recovery of polyaromatic hydrocarbons (PAHs), PCBs, and alkanes from soil, sediment, marine tissue, and air particulates have been described in the literature [5–9]. This article uses a traditional (Soxhlet extraction) and new (accelerated solvent extraction) recovery method for the extraction of PCB congeners from spiked organic matrix containing mixed PCB congeners.

Traditionally, PCB recovery has been performed by Soxhlet or sonication techniques [6]. These techniques are often time-consuming and require large volumes of organic solvent. The use of large volumes of extracting solvent adds additional cost because of the fee associated with purchasing and disposal of toxic solvents. In the last decade, alternative extraction techniques that reduce the volume of solvent usage for extraction, and the time of extraction have been considered [7,10]. For example, some of the newer techniques are microwave extraction, supercritical fluid extraction, and accelerated solvent extraction (ASE) [6,7,11].

ASE is a new technique that uses limited amounts of organic solvent in recovering semivolatile organics [12–14]. ASE operates at high pressures and temperatures above the boiling point of the organic solvent. The use of higher temperature increases the ability of solvent to solubilize the analyte, decreases the viscosity of liquid solvents, thus allowing better penetration of the solvent into the matrix and promotes ‘wetting’ of the matrix particles so as to achieve contaminant recovery [15]. The use of higher pressure facilitates the extraction of analyte from samples by improving the solvent accessibility to the analytes that are trapped in the matrix pores. Table 1 is a comparison of the ASE and Soxhlet extraction techniques.

Ritcher et al. [15] and Schantz et al. [16] have analyzed reference samples and found that the extraction temperature and volume of solvent, played an important role in influencing the extraction efficiency in ASE. EPA recently proposed a revised ASE method for assay of environmental sample [17]. Although, the literature provides a wide variety of SE and/or ASE recovery data, the dependence of PCB extractability upon type of matrix has not been investigated.

In an attempt to elucidate the relationship of PCB recovery to the nature of matrix, a series of recovery experiments were conducted following the spiking of a known amount of contaminant (i.e. mixed congener) on the matrix. It should be mentioned that contaminant recovery from spiked sample matrix is not comparable to native sample and only provides reliable bench marks for further investigation.

The selection of a complex organic matrix, to simulate the recovery of contaminant from a native soil sample is not straightforward. Recently Kenney and Oleslik recovered PAHs from complex matrices such as lignite and bituminous coal fly ash using ASE and SE

Table 1
Comparison of ASE and Soxhlet extraction technique

| Parameters | ASE | Soxhlet |
|------------------------------------|---|--|
| Extraction time | 15 min | 20–24 h |
| Solvent consumption | 20–30 ml | 200–500 ml |
| Capital investment | High | Low |
| Automatic/manual | Automatic | Manual |
| Detection limit | ppm–ppb | ppm–ppb |
| Conditions employed for Extraction | Harsh | Harsh |
| Concentrating the extract | Small solvent volume requires minimal concentration of analyte | Large solvent volume requires considerable concentration of the analyte |
| Labor intensive | Concern | Major concern |
| Analyte loss | Since concentration step is shorter or not required, the analyte loss is lower due to sample handling | Since concentration step is required and longer, the analyte loss is higher due to sample handling |

procedures [18,19]. In these studies, it was shown that the composition of fly ash and type of PAH were responsible for the slow recovery of PAH. Similarly, in this study, the effect of matrix composition on PCB extractability, have been studied using the ASE and SE techniques. For this investigation, matrices with different amount of organic carbon such as humic acid, low and high rank coal, and graphite, were spiked with PCB congeners with different molecular mass.

2. Materials and method

HPLC grade solvents purchased from Fisher Scientific Company were used in this study. PCB contaminants were obtained from Ultra Scientific Company.

To study the matrix effect, four types of organic matrix: (i) low rank coal (Beulah-Zap North Dakota lignite); (ii) high rank coal (Pocahontas low volatile bituminous); (iii) graphite and (iv) humic acid were chosen. Coal samples of –20 mesh were purchased from Argonne National Laboratory, IL; graphite was obtained from Alfa-Aesar; and sodium salt of humic acid was purchased from Acros Chemical.

2.1. Spiking procedure

The organic matrix was spiked with mixed PCB congeners [2,3-dichlorobiphenyl (PCB-5), 2,4',5-trichlorobiphenyl (PCB-31), 2,2',5,5'-tetrachlorobiphenyl (PCB-52), 2,2',4,5,5'-pentachlorobiphenyl (PCB-101) and 2,2',4,4',5,5'-hexachlorobiphenyl (PCB-153)]. The PCB mixture was prepared in hexane by mixing equal volumes of 50 ppm of PCB-5, PCB-31, PCB-52, PCB-101, and PCB-153 and the solution was spiked on 5 g of organic matrix. Following the completion of spiking, 50 ml of hexane was added for complete wetting of the sample. The solution was hand stirred for 1 h to obtain homogeneous solution. After 48 h contact time, hexane was allowed to evaporate at $23 \pm 2^\circ\text{C}$ and the PCB spiked organic matrix was brought to dryness under a stream of nitrogen. In the past, the spiked

analyte have been allowed to equilibrate anywhere from 2 days to 2 weeks [18–20]. In this study, the analyte was allowed to equilibrate with the matrix for a minimum of 48 h. The resulting spiked sample was stored in amber colored vial so as to prevent any photodegradation. The bottles were periodically shaken during the initial period of equilibration. The control sample was prepared in an identical fashion except no PCB congener was added during the spiking procedure to the matrix. Then, ASE and SE studies were conducted on spiked matrix system.

2.2. ASE extraction

ASE was carried out in 11 ml extraction cell. A weighed amount (0.5 g) of the mixed congener spiked matrix was placed into the sample cell (11 ml), and the remaining volume of the cell was completely filled with anhydrous sodium sulfate. When the sample cells were loaded into the carousel of the ASE 200 system [Dionex (UK)], extractions were performed by filling the cell with solvent before heating (pre-fill method). For performing extraction, approximately 15–30 ml of solvent was used. The system was filled with a 1:1 mixture of hexane and acetone (HPLC grade) [21]. The operating conditions were as follows: heating for 5 min, equilibrating for 5 min and performing static extraction for 5 min at a pressure of 2000 psi (14 MPa). The conditions were chosen based on prior work of Schantez et al. [16], Ritcher et al. [15], and EPA method 3545 [17] on a number of systems such as contaminated soil, sediments, air particulates, tissues, and standard reference materials. To assess the effect of extraction temperature on the recovery of analytes adsorbed to the organic matrices, the oven temperatures was maintained at 50, 75 and 125°C. The extracted analytes were then purged from the sample cell using nitrogen gas for 90 s. The extracted analytes were collected in suitable vials, and the total volume of analyte collected along with solvent was 20–30 ml. The internal standard, lindane (1 α , 2 α , 3 β , 4 α , 5 α , 6 β hexachloro cyclohexane) was added to the extracted volume so as to account for any loss during handling and analysis. Similarly, extractions were performed on clean organic matrix.

2.3. Soxhlet extraction

Soxhlet extraction was performed using 0.5 g portion of the mixed congener spiked matrix. The sample was transferred into a cellulose extraction thimble and inserted into a Soxhlet assembly for extraction. In the past, hexane and acetone mixture as well as methylene chloride–acetone mixture have been used as solvent mixtures for the recovery of contaminants from soil [9]. However, the presence of methylene chloride in the latter solvent mixture requires solvent exchange prior to gas chromatographic analysis if electron capture detection method is used [22]. Because the selection of methylene chloride/acetone mixture adds additional step to PCB recovery, in our study, a 300 ml portion of 1:1 ratio of hexane and acetone was placed in the 500 ml round bottom flask and the Soxhlet assembly was refluxed for 24 h using heating mantle. Based on our prior work and EPA method 3540 [21], the optimum time period for extraction was chosen to be 24 h. Upon completion of 24 h extraction, the extracts were allowed to attain room temperature, and concentrated to 20 ml by fractional distillation using a three-ball macro snyder column as a fractionating column. Finally, the extracts were concentrated in the condenser to 5 ml. To the concentrated extract,

5 ml of 1 ppm internal standard (lindane) were added for PCB quantification. The control samples were Soxhlet extracted in an identical manner as that of PCB spiked matrix.

2.4. Ultrasonic and thermal extraction of PCB sorbed graphitic sheet

Additional experiments were conducted to study the recovery of PCB from graphite using ultrasonic and thermal extraction techniques. 60 mg of graphitic sheet of dimension (1.5 cm × 1.5 cm × 0.0254 cm) was immersed in 5 ml of 10 ppm solution of PCB-5 at room temperature (23 ± 2°C) for 24 h. Similarly, graphite sheets were immersed in individual solutions of PCB-153 and 2,6 dichlorobiphenyl (PCB-10) congener. At the end of the period, the sheets were removed from each solution and allowed to dry for 24 h. Some of the dried PCB sorbed graphite was sonicated followed by thermal extraction.

For performing ultrasonication of PCB sorbed graphite, Branson 200 ultrasonicator was used. Approximately 60 mg of the PCB sorbed graphite was sonicated along with 5 ml HPLC grade hexane for 3 min. To the sonication extract, 5 ml of 1 ppm internal standard was added for PCB quantification.

For performing thermal extraction of sonicated and non-sonicated PCB sorbed graphite, a Ruska Laboratories thermal extraction inlet system that was interfaced to HP5890 SII GC was used. The GC system was equipped with a (60 m × 0.25 mm × 0.25 μm) capillary column with DB-5 stationary phase, and an electron capture detector (ECD). Approximately 60 mg of the PCB sorbed graphite was placed in a quartz crucible inside the THERMEX oven. The crucible was heated up to about 350°C under a stream of helium gas. The organic vapor was swept through a heated capillary transfer line into the inlet of the GC port. The injector and detector were maintained at 320°C. The system was maintained at 150°C for 10 min and then ramped from 150 to 280°C at 5°C/min. Quantification was performed by internal standard calibration.

2.5. Analysis of the liquid extract

All of the extracts (ASE, SE, and Ultrasonication) were analyzed using a gas chromatography (GC) with an ECD using the Hewlett-Packard 5890 series II GC equipped with a split-splitless injector. All of the extracts were treated identically and analyzed using similar conditions. By injecting 1 μl of the extracted solution directly into a GC-ECD, the analysis of the extract was performed. The column used for the current study was a 0.53 mm × 30 m fused silica capillary column containing a 5% phenyl-substituted methylpolysiloxane phase, 0.88 μm film thickness. The injector and detector were maintained at 320°C. The system was ramped from 180 to 245°C at 5°C/min. Quantification was performed by internal standard calibration.

3. Results and discussion

As mentioned in the introduction, we spiked organic matrices (humic acid, high rank coal, low rank coal, and graphite) with mixed PCB congeners. A 1:1 mixture of hexane and acetone was used as PCB extracting solvent for both ASE and Soxhlet extraction. Since,

Table 2
ASE recovery data for PCB-101 from coal matrix

| Test System | Extraction conditions | Sample size (g) | Percent extraction | Relative standard deviation (%) |
|-------------------------------|-----------------------|-----------------|--------------------|---------------------------------|
| High ranked coal with PCB-101 | At 125°C and 2000 psi | 0.5 | 86.95 | 5.8 |
| | | 1.0 | 73.71 | 1.58 |
| | | 1.5 | 72.74 | 1.43 |
| | | 2.0 | 73.03 | – |

the role of pressure for ASE extraction may be important in that a high pressure keeps the solvent in the liquid phase at elevated temperatures. Therefore, for the current investigation a high operating pressure was chosen. ASE was initially operated at 125°C and 2000 psi. The literature also provides evidence to support the use of an extraction pressure of 2000 psi to achieve optimum recovery from matrix [15,16]. We studied the effect of sample size and extraction temperature as variables for PCB recovery from the above mentioned matrices.

3.1. Effect of sample size on PCB recovery

To study the sample size effect, extractions were performed on 0.5, 1.0, 1.5, and 2.0 g of spiked high rank coal. Table 2 shows the PCB-101 recovery data for ASE conditions from spiked organic matrix. It should be mentioned that the results presented are averages of triplicate sample measurements. As shown in the table, the standard deviation (S.D.) of the ASE method varied from 1.4 to 5.8%. We noticed slightly higher variation in PCB recovery data when the sample size was small and lesser variation in PCB recovery data when the sample size was 1.0 g or greater. These variations may be a reflection of the heterogeneous nature of the spiked matrix.

3.2. Effect of extraction temperature on PCB recovery

Fig. 1 shows the effect of ASE oven temperature on the recovery of PCB 101 from coal matrix at constant pressure (2000 psi). Comparing results for different temperatures, it

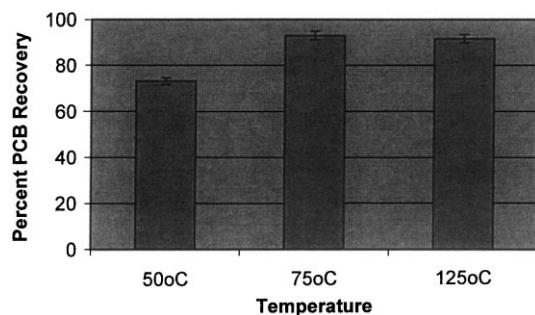


Fig. 1. Effect of ASE extraction temperature on PCB-101 recovery from coal matrix.

appears that the ASE operating temperature may have a role to play in the PCB recovery from matrix. A paired *t*-test was used to examine whether there was statistically significant difference in the average recovery of PCB congeners at different temperatures. The *t*-value was calculated to determine the level of confidence at which the averages are statistically different. Any confidence level close to 95% was treated as significant difference. A significant difference in the PCB recovery was found when the oven temperature was increased from 50 to 75°C, while for temperatures higher than 75°C, the difference was not significant. In Fig. 1, we see a plateauing in the PCB recovery beyond 75°C. A similar trend was noticed for other congeners. Similar temperature effect have been previously observed in ASE extraction of PAH and PCB from complex matrix [16]. An increase in temperature is expected to induce temperature-dependent solubility of contaminant in solvent, weaken the analyte–matrix interaction, and increase the vapor pressure of the analyte. ASE extraction results to be discussed, hereafter, were conducted at optimum temperature, unless otherwise stated.

By performing ASE extraction at elevated temperatures, we have shown that an improvement in the recovery of PCB congener from organic matrix can be obtained. To study the effect of high extraction temperature on the co-extraction of other organic substances from matrix and/or the formation of degraded products of the parent compound, the GC chromatograms of ASE extract and SE extract were compared. It should be mentioned that Soxhlet extraction was performed at $75 \pm 5^\circ\text{C}$. The chromatogram of the ASE extract (data not shown) has congener peaks similar to that of SE extract (data not shown). This suggests that ASE extract is as clean as Soxhlet extract. A close observation of SE extract shows a small peak besides the major congener peaks, this may be a co-eluting peak from the matrix, under the conditions studied. However, this observation needs to be confirmed by performing additional experiments at different temperatures.

Table 3 shows the congener recovery from spiked low rank coal matrix for ASE and SE conditions, respectively. The reported congener concentration is a mean of three or more analyses. A paired *t*-test was used to compare the mean PCB recovery from coal using ASE and SE techniques. Any confidence level close to 95% was treated as significant. For example, for low rank coal, the difference in the recovery of the congeners by ASE and SE technique was either comparable or slightly higher. The slightly higher PCB recovery by ASE technique may be a result of the use of high pressure and elevated temperature conditions in ASE technique compared to milder conditions in SE technique.

Table 3
PCB recovery data from low rank coal using ASE and SE method^a

| Type of congeners | ASE | SE |
|-------------------|---------------|---------------|
| PCB-5 | 126.60 (7.44) | 115.72 (6.84) |
| PCB-31 | 100.27 (3.88) | 90.25 (2.96) |
| PCB-52 | 92.12 (5.07) | 86.53 (14.41) |
| PCB-101 | 104.02 (7.89) | 86.57 (13.93) |
| PCB-153 | 75.48 (12.36) | 72.95 (0.58) |

^a Numbers in parentheses represent standard deviation.

Table 4
Comparison of PCB recovery from spiked organic matrix using ASE method^a

| Type of congeners | Humic acid | Low rank coal | High rank coal | Graphite |
|-------------------|--------------|---------------|----------------|----------------|
| PCB-5 | – | 126.60 (7.44) | 131.82 (4.57) | 43.91 |
| PCB-31 | 11.05 (2.19) | 100.27 (3.88) | 101.57 (0.46) | 68.59 (13.3) |
| PCB-52 | 23.80 (0.26) | 92.12 (5.07) | 93.30 (0.46) | 74.33 (9.39) |
| PCB-101 | 82.70 (2.12) | 104.02 (7.89) | 109.68 (0.77) | 103.74 (11.42) |
| PCB-153 | 86.65 (0.21) | 75.48 (12.36) | 89.23 (1.17) | 84.64 (13.68) |

^a Numbers in parentheses represent standard deviation.

3.3. Dependence of PCB recovery on the type of matrix

Low rank coal, high rank coal, graphite, and humic acid were chosen for investigating the dependence of PCB recovery on the type of matrix. Table 4 compares the PCB recovery from spiked matrix using ASE extraction method. A similar PCB recovery was obtained by SE technique. The overall PCB congener recovery varied from 11 to 126%. In general, for the lower chlorinated PCB congeners (PCB-31, PCB-52), the PCB recovery was poor from humic acid than that from coal matrix. It should be mentioned that the humic acid content in the acid salt was only 60%. Furthermore, the carbon content of most humic substance can vary from 45 to 55% depending on the source of humic material [23]. Lower the carbon content of the matrix, poor is the adsorption of the analyte to the matrix, and poor is the recovery of lower chlorinated congener from humic acid. Similar recovery results have been reported for low molecular weight PAHs sorbed on fly ash [18].

Unlike the poor recovery of lower chlorinated PCB congener from humic acid, a high recovery from spiked low and high rank coal was observed. This may be attributed primarily to the relatively high carbon content of low and high rank coal, i.e. 73 and 91%, respectively and not to the porosity. Because, the porosity of the low rank coal and high rank coal (>20 nm) is similar. When students *t*-test at 95% confidence limit was applied to the recovery data of lower chlorinated congener from humic acid, and high and low rank coal, we noticed statistically significant difference in the mean recovery. However, the difference in the recovery of higher chlorinated congeners (PCB-101 and PCB-153), for humic acid, high and low rank coal was not significantly different. This may be attributed to the role of bulkiness of congener on the analyte-matrix interaction. Based on the above evidence, it can be mentioned that percent PCB recovery is sensitive to carbon content of the matrix and the type of congener.

From the above observation, it is expected that for graphitic system (high carbon containing matrix) the PCB adsorption and recovery should be the highest. On the contrary, poor recovery of lower chlorinated PCBs and improved recovery of high chlorinated PCBs from PCB sorbed graphite was observed. These results suggest that in addition to carbon content also the structure of matrix influence PCB recovery. A probable explanation for poor lower chlorinated PCB recovery from graphite matrix is that the layered structure of graphite would allow preferential diffusion of smaller molecules into the structure. Upon the diffusion of the molecule in to the structure, the trapped congeners are difficult to elute. It has been shown that an efficient way of separating different congeners is by adsorbing PCB

mixture on to graphite followed by elution. The elution of PCB congeners from graphitic system has been shown to closely follow the size and structure of congener molecule [9]. It should also be mentioned that the structure of PCB molecule is influenced by the degree of substitution of chlorine atom at the *ortho*-position of PCB congener [9]. It is suspected that the combination of structural configuration and size of PCB molecule would dictate the movement of PCB congeners into the interstices of graphitic layered structure. For PCB-153, it is believed that the congener being bulky and having non-planar configuration would be present predominantly on the surface and therefore should be more readily accessible by solvent. Although our observations are based on limited trials, they support the higher recovery of PCB-153 from the spiked graphitic matrix.

3.4. Dependence of PCB recovery from adsorbed graphitic sheets on the type of congener

A more systematic study was conducted to provide further evidence to support our earlier suspicion, i.e. the more the chlorine at *ortho*-position, the more easily the PCB molecule is accessible by solvent from graphitic system. The influence of chlorine substitution on PCB extractability was examined by using PCB with six chlorine substituents and two PCB congeners with two chlorine substituents. The rationale for using PCB-5 and PCB-10 is that they vary only in the *ortho*-substitution while that of PCB-153 and PCB-10 have similar number of *ortho*-substitution, but they vary in the number of chlorine substitution. For convenience, in this exploratory study, the work was limited only to graphite in the belief that similar effects would be noticed for matrices with layered structure.

We performed sonication experiments on PCB-5 (with 1-*ortho*-substitution), PCB-10 (with 2-*ortho*-substitution), and PCB-153 (with 2-*ortho*-substitution) sorbed on graphitic sheets, and this was followed by thermal extraction of PCB sorbed graphitic sheet. The underlying assumption is that sonication of spiked matrix when performed for short time interval would extract only PCB present on the surface of graphite, while thermal extraction of sonicated graphite should recover the PCB trapped in the matrix.

Table 5 shows the extraction results from sonication, and thermal extraction after sonication. The reported results are means of triplicate or more data. It must be mentioned that since these results were based on a small number of PCB congeners with different substitutions and single matrices, the conclusion should be viewed with caution. For identical mass of PCB sorbed, the recovery data suggests that sonication may recover to a larger extent the PCB-10 from graphite than the dichlorobiphenyl PCB-5. On the other hand, thermal extraction recovers a larger fraction of residual PCB-5 from graphite. This suggests that improved recovery of PCB-5 from graphite is facilitated at elevated temperature. This

Table 5
PCB recovery from spiked graphitic sheet using sonication and thermal extraction technique^a

| Type of congeners | <i>Ortho</i> -position of chlorine atom | % Recovery by sonication | % Recovery by thermax |
|-------------------|---|--------------------------|-----------------------|
| PCB-5 | 1 | 37 (15) | 62 (13) |
| PCB-10 | 2 | 66 (9) | 5 (1) |
| PCB-153 | 2 | 64 (16) | 37 (14) |

^a Numbers in parentheses represent standard deviation.

observation is in agreement with the commonly accepted view that a congener with less *ortho*-substitution can diffuse into the graphitic layered structure and be trapped. In other words, once a molecule is in the entrapment site of the matrix, it has to travel the tortuous path of the matrix, before it is released from the matrix. It is believed that temperature facilitates desorption of trapped congener from high carbon containing matrix. Consequently during thermal extraction, we notice improved recovery of PCB-5 from sorbed graphite.

A comparison of PCB-10 and PCB-153 gave similar sonication recovery. This suggests that congeners with two *ortho*-substitution exhibit similar sorption and desorption behavior on graphite matrix. Further, these results indicate that the substitutional pattern of non-*ortho*-substituents did not noticeably affect the PCB recovery from graphite matrix. This finding is in agreement with the Jensen and Sundstorm reported data for PCB congeners [24].

We also extended the study to a planar PCB molecule, (i.e. 4,4'-dichlorobiphenyl (PCB-15)) and noticed a high amount of PCB sorption and poor PCB recovery. The high sorption results might be explained based on the strong interaction of graphitic layer with the aromatic character of PCBs [9]. This observation is in agreement with well known fact that planar PCBs are much more sorbed on to graphite than their non-coplanar isomers. Because the mass of PCB-15 sorbed was considerably higher, no attempt was made to correlate the recovery data of planar and non-planar congeners by SE and TE techniques.

It must be mentioned that additional adsorption–desorption studies are needed to confirm the above stated claim by selecting several planar and non-planar congeners and by considering other matrix.

4. Conclusions and recommendations

Based on our preliminary study, the following conclusions were drawn:

1. Temperature in ASE extraction has been shown to play a small but important role in the recovery of analyte from complex matrix in a narrow range (50–75°C) and plateauing beyond 75°C.
2. The chromatogram of ASE extract is relatively clean similar to that of SE extracts. ASE recovery results are comparable or slightly higher to that of SE recovery results.
3. Within the four matrix studied, two subgroups showing similar extractability emerged. Humic acid and graphite showed poor extractability for lower congeners while high and low rank coal showed higher extractability for lower congeners. Possibly the PCB extractability from a matrix is highly sensitive to the carbon content and the layered structure of matrix.
4. An apparent difference in the recovery of planar and non-planar PCBs from graphite was noticed. Possibly the ease of extraction depends on the configuration of congener, and the structure of the matrix.

It is recommended that further tests be conducted to provide more information on the effects of congener configuration, structure of matrix on the extractability of PCBs from spiked organic matrix. The study should be extended to include several other spiked and native organic systems, so as to verify the observations before it can be applied to native samples.

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