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Pressurized fluid extraction of polychlorinated biphenyls in solid environmental samples

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Abstract

The effectiveness of extracting native (not spiked) polychlorinated biphenyls (PCBs) from solid environmental samples by means of pressurized fluid extraction (PFE, Dionex trade name Accelerated Solvent Extraction) according to US Environmental Protection Agency (EPA) Method 3545, was determined. Three different certified reference materials, two sediments and one sewage sludge, were utilized. As opposed to most of the previous investigations, a thorough quantitative determination of the extracts obtained by PFE was performed and compared to certified values. Obtained data were in good agreement with certified values for all materials. However, materials with different particle sizes seemed to have influence on the extraction efficiency, with enhanced extraction for smaller particle size samples. PFE is concluded to be at least as effective as previously used methods in terms of quantitative extraction. When compared to data obtained with supercritical fluid extraction (SFE) using EPA Method 3562, the recoveries where slightly higher. This was explained by the less clean extracts obtained in PFE despite clean-up of the extracts. This is contrary to the clean extracts obtained by SFE which are ready for analysis. It can, however, not be excluded that PFE is really more efficient for extraction of very strongly bound analytes.

Keywords: Pressurized fluid extraction; Extraction methods; Environmental analysis; Polychlorinated biphenyls

1. Introduction

Pressurized fluid extraction (PFE, Dionex trade name Accelerated Solvent Extraction) is a relatively new sample preparation technique for automated extraction of analytes in solid materials. At present it is competing with other techniques like microwave-assisted extraction (MAE) and supercritical fluid extraction (SFE) for the extraction of organic contaminants from various solid matrices [1,2]. There are several reasons why these methodologies have evolved, and according to Wan and Wong [3] one of the major driving forces is the increasing demands from authorities to reduce the large volumes of organic solvents consumed by classic extraction methods like Soxhlet. The first reports on PFE appeared in 1995, presenting the basic experimental setup as well as extraction results for spiked pesticides and herbicides in soils and polynuclear aromatic hydrocarbons (PAHs) in urban dust [4,5]. The recoveries obtained were in good agreement with Soxhlet data, demonstrating the great potential of PFE in terms of speed and reduced organic solvent consumption. The success of PFE, with a matrix

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independent quantitative recovery of a number of compounds after only a few minutes of static extraction, have been explained by the enhanced solubilization and desorption of analytes from the matrix occurring at elevated temperatures (50–200°C) and pressures (7–20 MPa) [4–6]. The effects and relative importance of different extraction parameters have been evaluated and discussed in more detail by several independent researchers, using a number of real world and model matrices [7–9].

Since PFE was demonstrated to be very efficient, the technique was rapidly accepted by the US Environmental Protection Agency (EPA) as a method for evaluation of solid wastes [10]. Another contributing factor for the rapid acceptance of PFE is that method development is rather straight-forward. Often the organic solvent or combination of solvents utilized in existing Soxhlet methods can simply be adopted by the PFE method [5,6]. Consequently, the year after the first publications on PFE appeared, several publications dealing with persistent organic pollutants (POPs) in soils and sediments were presented [2,6,11]. Ever since, the number of publications dealing with PFE of POPs have increased, where the main focus has been on PAHs [12–18]. Surprisingly, one of the most well-known POPs world-wide, namely polychlorinated biphenyls (PCBs) [19,20], have been paid relatively little attention and the number of papers presented until today is limited. PCB extractions are often discussed very briefly within some of the existing publications [5,7,16], and to our knowledge only two articles are mainly devoted to PCBs. [11,21]. Schantz et al. [21] thoroughly evaluated PFE for the extraction of several PAHs, PCBs and chlorinated pesticides from eight different reference materials. They concluded that PFE is a suitable alternative to Soxhlet extraction, and in some cases it is even more efficient than Soxhlet. In the investigation performed by Donnelly et al. [11] no quantitative data were presented, and consequently no information regarding the effectiveness of PFE could be found in this work.

In this paper the effectiveness of PFE EPA method 3545 [10] has been thoroughly investigated for native (not spiked) PCBs by extracting three certified reference materials; two sediments and one sewage sludge. The conditions proposed in method 3545 were used throughout, and special attention was paid to the extraction time. The reason for this is that current literature has differing reports on the time needed to give a complete recovery [7,13]. The proposed length of the static step in PFE EPA method 3545 is 5 min, and for all matrices investigated two subsequent extractions (2×5 min) were performed to investigate the exhaustiveness of the method. For one of the sediments a comparison with SFE (EPA method 3562) [22] was also performed.

2. Experimental

2.1. Chemicals

Three certified reference materials were used in this study: marine sediment SRM 1944 [National Institute of Standards and Technology (NIST), Gaithersburg, MD, USA], harbor sediment CRM 536 [Community Bureau of References (BCR) Brussels, Belgium] and sewage sludge BCR 392 (Community Bureau of References). Harbor sediment CRM 536 was available in two particle sizes, a starting material with sizes ranging from 75 to 1000 μm, and a sample with particles <15 μm (IRMM, Geel, Belgium).

Eleven PCBs, IUPAC Nos. 28, 52, 101, 105, 118, 128, 138, 149, 153, 156 and 180, were used (BCR and Ultra Scientific). All solvents used (acetone, *n*-hexane, *n*-heptane and methylene chloride) were of pesticide grade (Merck, Darmstadt, Germany).

2.2. Pressurized fluid extraction

Extractions were performed according to PFE EPA method 3545 [10], using an ASE 200 accelerated solvent extraction system (Dionex, Sunnyvale, CA, USA). For SRM 1944, CRM 536 and BCR 392, about 1.5 g, 1.5 g and 0.5 g of sediment was mixed with an equal volume of anhydrous sodium sulfate and transferred to Dionex standard stainless steel cells (11 ml). To prevent clogging of the metal frit, a filter paper (diameter=20 mm, Waters) was placed at the exit of the cell. After loading the cell, it is placed in the extractor and the extraction starts. First the mixed organic solvent, consisting of *n*-hexane–acetone (1:1, v/v), is pumped into the cell. This solvent

mixture has previously been demonstrated to give good recoveries for PCBs [7,11,21] and organochlorine pesticides (OCPs) [13,21] in environmental matrices. The cell is then preheated for 5 min to reach the set temperature (100°C), followed by a static extraction step at this temperature. It has earlier been demonstrated that 100°C is a suitable temperature for extracting PCBs and OCPs [5,13,21]. During the following static extraction (5 min), the cell is held at a constant pressure and temperature. After its conclusion, the pressure is released and the extract is collected in 25-ml glass vials. To ensure that all extracted analytes reach the collection vial, the cell is rinsed with fresh solvent. The rinsing volume is normally 60% of the extraction cell volume, as set by the software. Finally, pure nitrogen (''Plus'' quality, AGA Gas, Sundbyberg, Sweden) is purged through the extraction cell for 1 min to assure that the solvent is completely transferred to the collection vial.

2.3. PFE extract clean-up

One ml of *n*-heptane was added to the 15-ml PFE extracts, after which they where evaporated under a gentle stream of nitrogen down to ca. 1 ml. Internal standards (PCB 35 and PCB 169) were added to the residue. Each extract was then loaded onto a 15 mm I.D. glass column filled with 5 cm of activated silica, impregnated with 40% (w/w) sulfuric acid. The PCBs where eluted from the column with 50 ml *n*-hexane, and after its completion another milliliter of *n*-heptane was added prior to evaporation down to 1 ml on a rotary evaporator. The final volume was adjusted to 1.8 ml with *n*-heptane. A small amount of copper powder was added to the vials prior to gas chromatography (GC). The vials were then left overnight in order to eliminate possible sulfur interferences contained in the extracts.

2.4. Supercritical fluid extraction

The extractions were done on a HP 7680T supercritical fluid extraction unit (Hewlett-Packard, Wilmington, DE, USA), according to the conditions in SFE EPA method 3562 [22]. About 1 g of sediment CRM 536 <15 μm was used. The samples were mixed with 7 g of anhydrous sodium sulfate and 2 g of pre-rinsed electrolytic grade copper powder and transferred to Hewlett-Packard 7-ml standard stainless steel extraction cells. It has previously been demonstrated that adding copper like this is an efficient way of eliminating interfering sulfur [23]. A glass filter paper (Whatman, GF/B) was placed at both ends of the cell to avoid metal frit clogging. The extraction fluid in all experiments was SFC grade carbon dioxide (Scott Specialty Gases), while food-grade carbon dioxide was used as the cryo gas required for cooling different zones in the SFE apparatus. The samples were extracted at 80°C and a density of 0.75 g/ml (305 bar), 10 min in the static mode, followed by 40 min of dynamic extraction. The flow-rate was set to 1.0 ml/min, corresponding to seven sweeps of the extraction cell. The extracted PCBs were collected on a solid phase trap containing approximately 1 ml of Florisil (0.16–0.25 mm particles), which has been shown to give clean extracts [23,24]. The temperature of the trap and the nozzle during the extraction was 20 and 45°C, respectively. Elution of trapped analytes were done with 2×1.5 ml *n*-heptane, followed by 4 ml of methylene chloride–acetone (1:1, v/v) and another 1.5 ml portion of *n*-heptane (for reconditioning of the trap). The only fraction analyzed was the first 1.5 ml of *n*-heptane. After addition of internal standards (PCB 35 and PCB 169), the samples were ready for analysis without further sample clean-up.

2.5. Gas chromatographic analysis

Analysis was done according to a previously described dual column high-resolution GC method [25,26]. An electronic pressure controlled HP Model 5890 Series II GC system equipped with on-column injector and two ^{63}Ni electron capture detectors (300°C, purged with N_2, 60 ml/min) was used. Hydrogen was used as carrier gas, with a linear velocity of ca. 43 cm/s, held constant throughout the whole temperature program. Aliquots of 1 μl were injected on-column on two parallel coupled columns, a 60 m×0.25 mm, 0.25 μm 50% diphenyldimethylsiloxane HP 50+ column (Hewlett-Packard) and a 25 m×0.25 mm, 0.25 μm 5% diphenyldimethylsiloxane HP-5ms (Hewlett-Packard) in series with 25m×0.22 mm, 0.10 μm 1,7-dicarba-*closo*-dodecarborane-dimethyl-siloxane HT-

5 column (Scientific Glass Engineering). A quick-seal glass ''T'' connected to a deactivated retention gap (2 m×0.53 mm fused silica) was used to connect the two columns in parallel. The GC system was programmed as follows: initial temperature at 90°C for 2 min, followed by an increase to 170°C at a rate of 20°C/min, retained for 7.5 min, then increased at a rate of 3°C/min to 275°C and held for 10 min (total time 58.5 min). Quantitation was based on an eight-point multi-level calibration curve in the concentration interval 0.5–441 pg/l for the individual PCB congeners and PCB 35 and PCB 169 as internal standards.

3. Results and discussion

3.1. Initial PFE experiments on marine sediment SRM 1944

Marine sediment SRM 1944 has previously been demonstrated to be relatively easy to extract, e.g., SFE with pure carbon dioxide at 100°C, extracted about 95% of the PCBs present in the matrix [27,28]. Therefore this matrix was initially extracted to verify that PFE was capable of extracting PCBs from a sediment previously known not to cause any severe problems. The results from the PFE extractions are presented in Table 1.

For this sediment a single 5 min static extraction step seems sufficient to achieve a quantitative extraction, since the mean recovery for all investigated congeners was 99% compared to certified values. The recoveries for individual congeners were normally also very close to certified and typically ranged from 80 to 120%. For a few of the reported congeners the recoveries may seem low. This is explained by differences in the final analysis. For example, the concentration reported by NIST for PCB 138 also contains PCB 163 and 164. The concentrations presented here are, however, free from PCB 163 [29]. The exhaustiveness is further verified by the fact that the average concentration of PCBs found in the second static step was 0.8%, which is negligible compared to the first static step. Additionally, the standard deviations from the five independent experiments are low, normally below or close to 5%. The results presented in Table 1 demonstrates that PFE is capable of extracting PCBs from sediments with the conditions proposed in PFE EPA method 3545. However, one material is too little to draw any certain conclusions and therefore a different type of matrix (sewage sludge BCR 392), as well as a sediment with a greater variation in

Table 1
Pressurized fluid extraction of SRM 1944 (n=5)

PCB	NIST values[c] (ng/g)	Amount (ng/g) 5 min static 1st step	RSD (%)	Recovery vs. certified values (%)	Found in 2nd step (%)
28	75.8±2.2	85.2[a]	3.8	112	1.0
52	78.9±1.7	79.5[a]	2.5	101	0.7
101	73.3±1.5	64.6[a]	2.9	88	0.6
149	49.1±1.7	47.9[a]	2.4	98	0.9
118	57.6±1.3	51.7[b]	4.9	90	0.8
153	73.5±1.5	60.9[a]	4.5	83	0.5
105	22.4±0.8	23.7[a]	2.6	106	0.4
138	59.7±1.5	49.8[a]	4.6	83	0.6
128	8.21±0.75	10.25[a]	2.8	125	0.9
156	6.34±0.23	6.24[b]	3.3	98	1.4
180	41.7±1.0	44.0[b]	10.1	106	0.7

[a] Determined using the HP-50+ column.
[b] Determined using the HP5-HT5 column combination.
[c] These values are not the final certified, these will be available from NIST shortly.

particle size distribution (CRM 536 starting material) was investigated.

3.2. Evaluation of the 5 min static extraction step using different matrices

3.2.1. Sewage sludge BCR 392

Additional experiments to verify the effectiveness of the proposed static step of 5 min were done by extracting sewage sludge BCR 392. These results are presented in Table 2.

Also for this matrix a 5 min static step gives a quantitative extraction, with an average recovery for six investigated congeners of 101%. Individual congener concentrations also matched certified values very well, and the recoveries ranged from 90 to 110%. Once again the second static step contained very small amounts of PCBs; for this material only 1.4% of the concentrations detected during the first step was found in a second fraction. The standard deviations were also very satisfying, and never exceeded 5%.

3.2.2. Harbor sediment CRM 536, starting material

The second verification experiment was performed on CRM 536 (starting material), and the results are presented in Table 3.

For this sediment the concentrations were once again close to certified values, with individual congener recoveries normally in the range of 90 to 120%, and an average recovery of 107% (excluding PCB 105). However, the concentrations determined in the second static step implies that the first static step is incomplete. The average concentration found in step two is about 7% of the PCBs present in the first extract. In no case was the concentration in the second extract below 4% of that in the first step (except for PCB 105 which will be discussed later in Section 3.4). For PCB 52 as much as 14% was found in the second extract. The relative standard deviations (RSDs) were also rather high, normally larger than 7%. Consequently, for this material, the concentration determined in the second extraction step was added to the concentration in the first step, and the sum was used as the total concentration. By doing this, the average recovery increased to 114% and the RSD values decreased to about 5%, which are similar to the RSD values obtained for sediment SRM 1944 (Table 1).

One reason for the low recoveries and the relatively larger standard deviations of the individual congeners after the first extraction step for CRM 536 starting material might be caused by the large differences in particles size, leading to a more inhomogeneous diffusion path distribution and con-

Table 2
Pressurized fluid extraction of BCR 392 ($n=5$)

PCB	Certified values (ng/g)	Amount (ng/g) 5 min static 1st step	RSD (%)	Recovery vs. certified values (%)	Found in 2nd step (%)
28	100±10	99.5[a]	1.1	99	0.9
52	78±8	80.2[a]	4.7	103	1.0
101	134±10	137.3[a]	2.2	102	1.1
149	–	239.2[a]	3.2	–	1.0
118	97±10	87.9[b]	1.2	91	1.6
153	288±18	295.7[a]	2.0	103	0.9
105	–	37.5[b]	1.5	–	2.5
138	–	209.4[a]	1.9	–	1.0
128	–	28.0[b]	1.9	–	2.1
156	–	22.3[b]	1.4	–	1.8
180	311±24	339.9[b]	0.9	109	1.1

[a] Determined using the HP-50+ column.
[b] Determined using the HP5-HT5 column combination.

Table 3
Pressurized fluid extraction of CRM 536, starting material ($n=5$)

PCB	Certified values (ng/g)	Amount (ng/g) 5 min static 1st step	RSD (%)	Recovery vs. certified values (%)	Found in 2nd step (%)	Amount (ng/g) 5+5 min static	RSD (%)	Recovery vs. certified values (%)
28	44.40±6.26	54.55[b]	6.2	123	6.6	58.13[b]	4.8	131
52	38.42±7.09	46.08[b]	7.2	120	14.5	51.79[a]	2.2	135
101	43.66±6.17	47.85[a]	8.5	110	5.5	50.51[a]	3.7	116
149	48.77±5.87	45.55[b]	6.8	93	6.2	48.38[b]	4.9	99
118	27.55±4.02	28.42[b]	5.1	103	4.5	29.69[b]	6.8	108
153	50.30±5.70	53.80[b]	7.5	107	5.8	56.92[a]	5.0	113
105	3.50±0.65	6.74[a]	7.5	193	3.3	6.96[a]	4.9	199
138	26.84±3.89	28.53[a]	9.1	106	5.2	30.02[a]	4.8	112
128	5.39±1.43	5.48[a]	3.2	102	4.0	5.70[a]	6.5	106
156	3.04±0.44	3.25[b]	8.0	107	6.3	3.46[b]	6.9	114
180	22.44±3.59	22.24[b]	8.3	99	6.6	23.72[b]	5.1	106

[a] Determined using the HP-50+ column.
[b] Determined using the HP5-HT5 column combination.

sequently different degree of entrapment of the analytes. The particle size distribution for CRM 536 starting material is between 75 and 1000 μm for 80% of the material, while 10% is over 1000 μm, and 10% is below 75 μm. In the case of SRM 1944 however, 90% is in the range of 75 and 250 μm, and the additional 10% is below 75 μm. Corresponding size fraction values for BCR 392 are not known, but are probably close to those reported for SRM 1944. The reason for this is that both materials were homogenized with a ball mill, contrary to CRM 536 <15 μm where a jet milling process was used. The results suggests that 5 min might not always be enough to assure a completely exhaustive extraction for very inhomogenous samples, and if 100°C is to be used it might be advantageous to perform a 2×5 min extraction. This has previously been demonstrated to give the highest recovery for OCPs, where a 2×5 min extraction was superior to both a 5 min, a 10 min and a 15 min static step [13]. To further study the effects of particle size, a jet milled batch of sediment CRM 536 with particle sizes below 15 μm was investigated.

3.3. Influence of particle size

3.3.1. Harbor sediment CRM 536, <15 μm

The results for the extraction of harbor sediment CRM 536 with a smaller particle size (<15 μm) are presented in Table 4.

For this sediment, the average recovery for the investigated congeners is 122%, with individual congeners ranging from about 110–140%. The recovery of 122% is somewhat higher than the 2×5 min value presented for the starting material of the same sediment above (114%). This is probably an indication that the PCBs in the small particle material presented in Table 4 are made more accessible during the jet milling processes (possibly yielding shorter diffusion path lengths) compared to the PCBs in the starting material. This is further supported by the fact that summation of the concentrations obtained in step 1 and step 2 (as was done in Table 3) does not cause a large increase in the average recovery (127%). Additionally, the RSDs of the results obtained from the small particle size fraction are very good. Generally they are close to 2%, both for the first static extraction and for the combined 2×5 min static extraction. These RSDs seem to be better than those obtained for CRM 536 starting material above, but a larger number of experiments would have to be performed in order to prove the difference to be statistically significant. It must be pointed out though, that the above results could be explained also by the smaller particle size fraction containing slightly higher amounts of PCBs. The reason for this is that the jet milling process involves

Table 4
Pressurized fluid extraction of CRM 536, particle size <15 μm (n=5)

PCB	Certified values (ng/g)	Amount (ng/g) 5 min static 1st step	RSD (%)	Recovery vs. certified values (%)	Found in 2nd step (%)	Amount (ng/g) 5+5 min static	RSD (%)	Recovery vs. certified values (%)
28	44.40±6.26	64.93[b]	0.8	146	3.1	66.11[a]	2.4	149
52	38.42±7.09	54.58[b]	2.0	142	9.4	55.68[a]	5.4	145
101	43.66±6.17	55.64[b]	0.5	127	3.1	57.36[b]	1.1	131
149	48.77±5.87	52.00[b]	1.0	107	3.5	53.06[a]	3.2	109
118	27.55±4.02	32.17[b]	2.8	117	2.9	33.09[b]	2.0	120
153	50.30±5.70	61.22[b]	0.8	122	3.3	63.21[b]	1.3	126
105	3.50±0.65	7.27[a]	1.8	208	1.5	7.37[a]	1.8	211
138	26.84±3.89	32.31[a]	2.5	120	2.9	33.24[a]	2.3	124
128	5.39±1.43	5.86[a]	1.3	109	2.4	6.00[a]	0.7	111
156	3.04±0.44	3.59[b]	3.6	118	4.1	3.73[b]	4.1	123
180	22.44±3.59	25.57[b]	1.6	114	3.7	26.52[b]	1.3	118

[a] Determined using the HP-50+ column.
[b] Determined using the HP5-HT5 column combination.

a particle size fractionation, and selecting only the smaller particles could give rise to higher concentrations assuming higher PCB amounts per gram of sediment for the smaller particle sizes.

The results presented in Tables 1–4 demonstrates that the conditions proposed in PFE EPA method 3545 (with a static step of 5 min) is capable of extracting the majority of PCBs from a number of matrices. In some cases, however, a 5 min step is somewhat short, and up to 7% of the PCBs might be left unextracted for heterogeneous samples with a large span of particle sizes (Table 3). For these types of materials it is advantageous to either grind the sample to smaller sizes (Table 4) or perform a 2×5 min static extraction (Table 3) in order to quantitatively extract all PCBs.

3.4. Comparing PFE EPA method 3545 and SFE EPA method 3562

PFE has evolved not only as a replacement to Soxhlet, but also as an alternative to supercritical fluid extraction. Therefore it is interesting to compare the results obtained for the two techniques. A comparison between PFE and SFE was performed by extracting sediment CRM 536 <15 μm using SFE EPA method 3562. These results are presented in Table 5.

The concentrations obtained for the investigated congeners are closer to certified data using SFE (97%) than using PFE (122%). The most striking difference is that while the recoveries for PCB 101, 149, 153 and 138 using SFE is 90% or below, corresponding values for PFE are over 100%, and in some cases as high as 127%. A number of explanations to this clear difference between the two techniques can be brought forward. First of all the SFE conditions at 80°C might be somewhat low. It is likely that a more exhaustive extraction could be

Table 5
Supercritical fluid extraction of CRM 536, <15 μm (n=3)

PCB	Certified values (ng/g)	Amount (ng/g)	RSD (%)	Recovery vs. certified values (%)
28	44.40±6.26	50.98[a]	2.2	115
52	38.42±7.09	38.37[a]	1.2	100
101	43.66±6.17	39.20[a]	2.1	90
149	48.77±5.87	38.51[a]	1.9	79
118	27.55±4.02	25.76[a]	2.7	94
153	50.30±5.70	44.56[a]	3.4	89
105	3.50±0.65	4.93[a]	2.3	141
138	26.84±3.89	23.81[a]	2.9	89
128	5.39±1.43	5.02[b]	3.0	93
156	3.04±0.44	3.96[a]	1.6	130[c]
180	22.44±3.59	21.35[a]	2.4	95

[a] Determined using the HP-50+ column.
[b] Determined using the HP5-HT5 column combination.
[c] Interference from PCB 171 in the determination.

obtained using higher temperatures [27]. Additionally the SFE method which traps the analytes on a solid phase trap packed with Florisil generates very clean extracts, both compared to Soxhlet extraction (which many of the certifying laboratories use) and PFE. Therefore the number of interfering compounds is expected to be higher in PFE, despite the clean-up step. One example is PCB 105, which in the PFE experiments on CRM 536 (table 3 and 4) gave recoveries close to 200% and therefore clearly contained interfering compounds, while corresponding recoveries using SFE, despite obvious interference, only reached 140%.

Furthermore, when using pure supercritical carbon dioxide very clean extracts are generated, since most organic material in the sample is left intact [30]. Thus the lower recoveries might simply be a product of cleaner extracts. This has been demonstrated for Soxhlet extracts of sediments, soils and lyophilized muscle tissue, which had to be cleaned twice before acceptably low levels of contaminants were reached in the extracts [28].

However it must be stressed that even though the higher recoveries obtained in PFE to some extent might be caused by a larger number of interferences, it is not unlikely that PFE in some cases has a higher extraction efficiency than other techniques, and the values reported by the certifying agencies, therefore, might be somewhat underestimated. The results presented in Tables 4 and 5 are good examples of the problems involved in defining a 100% recovery. It is quite clear though, that the statements made regarding PFE as a matrix-independent solution to most environmental applications [5,6] should be taken with a grain of salt. Certainly the problems involved in the field of sample preparation of environmental samples are more complex than that.

4. Conclusions

Sample preparation with PFE is a very fast alternative in terms of extraction time compared to conventional methods like Soxhlet, and also in comparison to more recent techniques like SFE. However, compared to the latter, extensive sample clean-up is needed, which drastically decreases the competitiveness of PFE. In PFE, the static step must be long enough to assure a complete transfer of analytes out of the sample matrix, or alternatively that the sample is well homogenized. This is especially important for samples with a large range of particle sizes. However, if the above criteria are considered, PFE might be a good tool for completely extracting all PCB molecules present in a sample. For routine measurements, cutting overall costs, SFE is probably the better choice though, due to its inherent selectivity towards analytes of interest.

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