Comparison of Pressurized Fluid (PFE) and Soxhlet Extraction of PCDD/F from Soil Samples

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Introduction

Pressurized fluid extraction was evaluated to extract polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/F) (1, 2) from soil. One lower contaminated soil sample (Para 1, I-TEQ about 5 pg/g d.m.) and one higher contaminated soil sample (Marsch 2, I-TEQ about 100 pg/g d.m.) were extracted as duplicates by pressurized fluid and soxhlet extraction (Tab. 2).

Keywords

Pressurized fluid extraction (PFE), accelerated solvent extraction (ASE®), soxhlet extraction, polychlorinated dibenzo-p-dioxins and dibenzofurans (PCDD/F), soil.

Material and Methods

The pressurized fluid extractor used was a Dionex accelerated solvent extractor (ASE® 200), the soxhlet extractor was a Knöfler-Böhm type. The two soil samples were prepared for use in a round robin study (3) and their elemental homogeneity was verified by analysing 9-10 metals with ICP-OES. For the PCDD/F peparation conditions see Tab. 1.

Tab. 1. PCDD/F Preparation Conditions of the Soil Samples Para 1 and Marsch 2

	Pressurized Fluid Extraction	**Soxhlet Extraction**
Sample Size		
Para 1	30 g	30 g
Marsch 2	10 g	10 g
Internal Standard	1 ng $^{13}C_{12}$-Te-HpCDD/F, 2 ng $^{13}C_{12}$-OCDD/F	
Solvent	Toluene, 60 ml	Toluene, 300 ml
Temperature	180 °C	111 °C
Pressure	15 mpa	Atmospheric
Time	Heat up 9 min, Static 3 x 15 min, Purge 5 min, Total 59 min	18 h
Clean Up	Al_2O_3 B Super I; SiO_2-$AgNO_3$, H_2SO_4, NaOH; Bio-Beads S-X3; Al_2O_3 B Super I	
Analytical	HRGC-LRMS	HRGC-LRMS

Tab. 2: Comparison of Pressurized Fluid (PFE) and Soxhlet Extraction of PCDD/F from the Soil Samples Para 1 and Marsch 2. PCDD/F in pg/g d.m.

PCDD/F	Para 1				Marsch 2			
	PFE Mean n = 2	RSD %	Soxhlet Mean n = 2	RSD %	PFE Mean n = 2	RSD %	Soxhlet Mean n = 2	RSD %
2,3,7,8-TCDD	<0.08	-	<0.08	-	2.40	1.8	2.51	0.7
Total TCDD	2.57	0.2	2.70	30	100	1.4	82.5	32
1,2,3,7,8-PeCDD	0.64	10	0.53	5.2	<0.5	-	7.89	-
Total PeCDD	8.35	1.0	8.48	18	297	1.3	229	12
1,2,3,4,7,8-HxCDD	0.80	3.0	0.61	14	<1.5	-	<1.5	-
1,2,3,6,7,8-HxCDD	1.26	1.6	0.98	7.0	15.1	5.7	14.6	3.6
1,2,3,7,8,9-HxCDD	1.04	5.7	1.12	12	34.6	2.5	27.9	0.7
Total HxCDD	16.6	3.3	14.0	1.5	152	5.1	131	10
1,2,3,4,6,7,8-HpCDD	11.7	1.1	9.30	0.7	123	6.9	80.5	14
Total HpCDD	25.0	0.6	18.8	3.9	222	6.2	143	12
OCDD	62.1	0.9	42.1	2.4	412	8.0	262	5.9
PCDD	115	1.1	86.1	1.0	1183	2.8	848	5.1
2,3,7,8-TCDF	1.30	4.0	1.11	2.5	55.8	0.2	48.8	1.1
Total TCDF	17.9	36	18.2	0.03	1184	1.1	976	16
1,2,3,7,8+(4,8)-PeCDF	2.19	14	1.97	3.7	187	3.7	143	12
2,3,4,7,8-PeCDF	1.68	3.1	1.48	0.7	45.5	0.5	39.1	6.4
Total PeCDF	19.9	11	17.0	0.3	1313	2.2	1027	8.8
1,2,3,4,7,8+(7,9)-HxCDF	3.90	3.3	3.01	2.5	398	2.2	304	8.6
1,2,3,6,7,8-HxCDF	2.34	3.3	1.83	3.2	248	7.2	187	12
1,2,3,7,8,9-HxCDF	0.88	0.5	0.77	22	65.8	4.0	52.5	15
2,3,4,6,7,8-HxCDF	2.00	3.9	1.76	2.2	55.4	7.3	44.0	12
Total HxCDF	24.2	3.5	19.7	3.6	1824	3.2	1403	11
1,2,3,4,6,7,8-HpCDF	24.2	3.8	18.3	1.4	1200	8.2	723	15
1,2,3,4,7,8,9-HpCDF	2.28	6.2	1.83	0.2	377	2.2	283	8.8
Total HpCDF	31.5	5.9	23.6	0.4	2045	8.3	1217	12
OCDF	66.6	8.9	50.6	0.7	3322	7.1	1981	14
PCDF	160	11	129	1.0	9688	5.2	6604	12
PCDD/F	275	6.8	215	1.0	10871	5.0	7453	11
I-TEQ	3.13	2.3	2.61	2.6	142	3.0	112	6.4

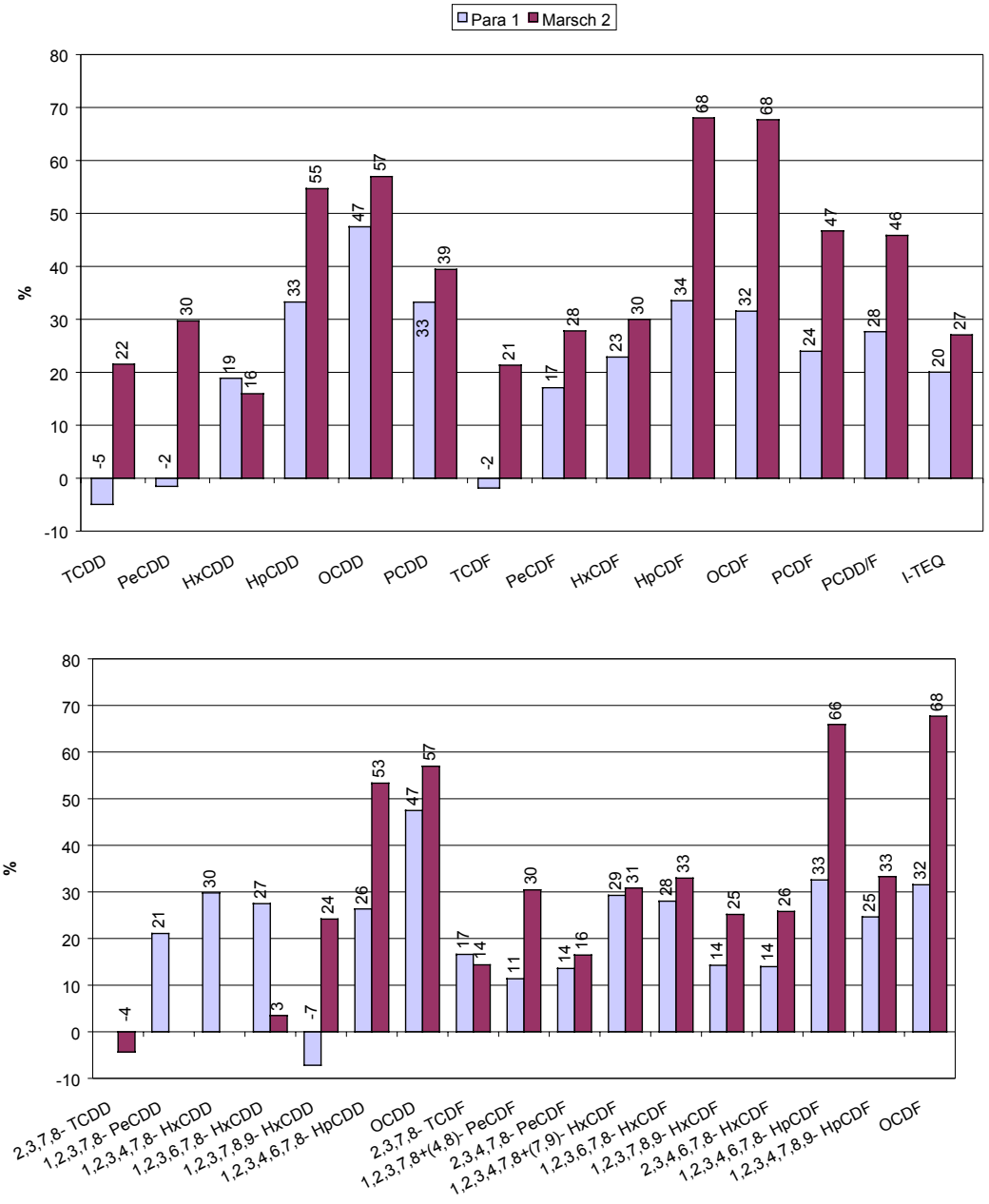

Fig. 1 and 2: PCDD/F more or less extracted by Pressurized Fluid (PFE) or Soxhlet Extraction (= 100 %) of the Soil Samples Para 1 and Marsch 2 in %:
(Mean $_{PFE}$ – Mean $_{Soxhlet}$) 100 / Mean $_{Soxhlet}$

Results and Discussion

With pressurized fluid extraction significantly more PCDD/F are extracted than with soxhlet extraction because of the much higher extraction temperature. The I-TEQ value of the lower contaminated soil sample (Para 1, I-TEQ about 5 pg/g d.m.) was found to be 20 % and of the higher contaminated soil sample (Marsch 2, I-TEQ about 100 pg/g d.m.) to be 27 % higher (Soxhlet = 100 %) (Fig. 1 and 2).

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References

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