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Ultrasound assisted dialysis of semi-permeable membrane devices for the simultaneous analysis of a wide number of persistent organic pollutants

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ABSTRACT

A new procedure based on ultrasound assisted dialysis (UAD) for the simultaneous and quantitative extraction of a wide number of persistent organic pollutants (POPs) such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) or some other organochlorinated pesticides (OCPs) contained in semi-permeable membrane devices (SPMDs) has been developed. This extraction technique combines the advantages of the organic solvent dialysis (OSD) and the speed of the ultrasound assisted extraction. The extraction was performed in an ultrasound bath for 32 min placing the SPMD in a glass flask covered with 80 mL of hexane. This set-up is able to extract simultaneously up to 8 samples. The proposed method entails good repeatabilities (RSD 2–13%) and recoveries (around 100% for almost every analyte). Limits of detection were at ng $SPMD^{-1}$ level and enough for the determination of the target analytes in a slightly polluted aquatic environment, as it was tested by successfully comparing the OSD to the proposed methodology. Therefore, the results obtained show that the UAD can be a good alternative for the extraction of POPs in SPMDs as it requires short extraction times and solvent volumes, and provides a cleaner extract for the subsequent clean-up step. Moreover, it fits better than the OSD to the general requirements of Green Chemistry.

1. Introduction

Even though the methodology suggested by Huckins et al. [1] for working with semi-permeable membrane devices (SPMDs) is still widely used in recent papers [2,3], this extraction technique is very time-consuming and the risk of analyte degradation and/or volatilisation during the process is a fact that may lead to some errors when it comes to the analysis of volatile or semi-volatile compounds [4]. Therefore there is a need to develop a more convenient extraction technique concerning these passive sampling devices.

A standard SPMD consists of a 91.4 cm long and 2.5 cm wide lay-flat low-density polyethylene (LDPE) filled with 1 mL pure triolein [5] and for the last few years it has been widely used as a reliable passive sampler in order to monitor non-polar persistent organic pollutants (POPs) in the environment [6]. However, even if SPMDs can be used with a very large group of POPs, they are not always applicable for all the contaminants in the environment such as metals, ionised compounds or very large or hydrophilic organic molecules [3,7,8]. Nevertheless, for those hydrophobic compounds with log $K_{ow} \geq 3$ such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) or some other organochlorinated pesticides (OCPs), SPMDs show up as appropriate passive sampler devices [9–12].

During the analysis of these hydrophobic compounds accumulated in the triolein within the SPMDs, the extraction and the clean-up steps are critical as the extractant requires being free from LDPE waxes and triolein or other lipidic substances [13]. Regarding the clean-up step, size exclusion chromatography (SEC) has been widely successfully used to remove the co-extracted residuals [9,14]. However, the extraction techniques employed in order to determine POPs in SPMDs are still far from being standardised. A widely used extraction technique consists of an organic solvent dialysis (OSD), hexane being the solvent used in most of the cases [1,9]. OSD is a suitable technique for the extraction of the analytes while triolein remains inside the SPMD, avoiding one of the major problems regarding SPMD analysis. Unfortunately, this methodology requires large extraction times (up to 72 h in some cases) and solvent volumes [15]. Therefore, other modern extraction techniques such as microwave-assisted

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extraction (MAE), accelerated solvent extraction (ASE) or ultrasound-assisted extraction (UAE) are being developed [16]. These new methodologies provide some advantages compared to the classic OSD, like lower extraction times and solvent volumes. However, in all of them the matrix concentration in the extract shows up as one of the major drawbacks. For this reason, a new procedure combining the advantages of the OSD and the speed of the new extraction techniques is required.

The use of ultrasonic energy in order to perform the extraction of the analytes from the SPMDs has already been developed and applied in previous works [17]. However, this methodology involves the lengthwise cutting of the membrane previous to the extraction, a procedure that entails the presence of a high amount of triolein in the extractant and, therefore, significant difficulties involving the clean-up step.

The aims of this study are (i) to reduce the volume of solvent and accelerate the dialytic process taking advantage of the ultrasound energy and (ii) to improve the performance of the cleaning step by limiting the undesirable presence of triolein and waxes in the extract. In this way, a greener chemistry would be achieved and a cleaner extract would be obtained in a much shorter time. To achieve these goals, a novel methodology for the simultaneous extraction of PAHs, PCBs and some OCPs in SPMDs using ultrasound-assisted dialysis (UAD) has been developed, optimised and successfully applied to field samples.

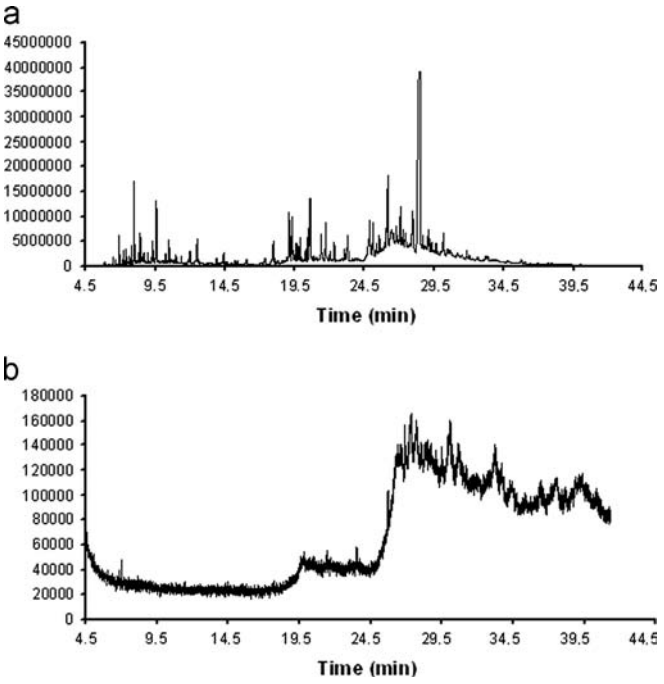

Fig. 1. Chromatograms of the extracts (SCAN mode) obtained after cleaning the LDPE tube in hexane for 45 min (a) and after a second cleaning for other 45 min (b).

2. Experimental

2.1. Materials and reagents

SPMDs were prepared in our laboratories using LDPE tubes purchased from Garciplast (Barcelona, Spain) and $>97\%$ purity triolein (Sigma-Aldrich, Steinheim, Germany). All solvents used were of HPLC grade and were obtained from Lab-Scan (Gliwice, Poland). NPD Control Standard S-4089 standard (including 16 EPA PAHs: naphtalene, *N*; Acenaphtylene, *Acy*; Acenaphtene, *Ace*; Fluorene, *Flu*; Phenantrene, *Phe*; Antracene, *Ant*; Fluoranthene, *Flt*; Pyrene, *Pyr*; Benz[a]anthracene, *B*[*a*]*A*; Chrysene, *Cry*; Benzo[k] fluoranthene, *B*[*k*]*F*; Benzo[b]fluoranthene, *B*[*b*]*F*; Benzo[a]pyrene; *B*[*a*]*P*; Indeno[1,2,3-cd]pyrene, *I*[*cd*]*P*; Dibenz[a,h]anthracene, *D*[*ah*]*A*; Benzo[g,h,i]perylene, *B*[*ghi*]*P*) and CEN PCB Congener Mix 1 (including *PCB*-18, *PCB*-28, *PCB*-31, *PCB*-52, *PCB*-44, *PCB*-101, *PCB*-153, *PCB*-118, *PCB*-138, *PCB*-149, *PCB*-180 and *PCB*-194) [18] were purchased from Supelco (Bellefonte, Pennsylvania, USA). In order to study the different OCPs, Pesticide Mix 11 and Pesticide Mix 164 standard solutions containing different isomers of hexachlorocyclohexane (HCH), dichlorodiphenyltrichloroethane (DDT), dichlorodiphenyldichlorethane (DDD) and dichlorodiphenyldichloroethylene (DDE) were obtained from Dr Ehrenstorfer (Augsburg, Germany). Perdeuterated Internal Standard S-4124 deuterated PAH mixture (Chiron AS. Trondheim, Norway) was used as surrogate.

2.2. SPMD preparation

In order to maintain the characteristics of a commercial SPMD such as the surface area and lipid-to-membrane ratio [6], the SPMDs prepared in our laboratory were made using 3 cm wide and 76.6 cm long LDPE tubes containing 1 mL of high purity triolein. Before preparing the SPMDs, an 80 μm wall thickness and 10 Å pore size LDPE tube was cut into approximately 84 cm long pieces, placed into glass flask together with 200 mL of hexane and cleaned using sonication energy (400 W, ultrasonic bath, JP Selecta. Barcelona, Spain) for 45 min. The extraction time and volume were settled after studying the chromatograms obtained from the concentrated extracts (ca. 1 mL) and guaranteeing that the LDPE tube was clean enough and analyte free for the making of the SPMDs (Fig. 1). After the cleaning step, tubes were kept at 40 °C till dryness. Afterwards one of the ends of the pre-cleaned LDPE tube was heat-sealed, adding a second "safety" seal to obtain a loop for proper deployment when sampling. Then, the tube was filled with 1 mL of triolein and once again heat-sealed on its other end. SPMDs were stored in the dark at −20 °C until future sampling campaigns.

2.3. Ultrasound-assisted dialysis

In order to optimise the UAD, spiked SPMDs were prepared using 75% purity triolein (Fluka, Gallen, Switzerland) and standard solutions of target analytes (ca. 500 ng). Analytes were first dissolved in the triolein and, afterwards, the spiked triolein was introduced inside the LDPE tube before heat-sealing. The spiked SPMDs were placed inside glass flasks and covered with 80 mL of hexane. Membranes were placed in an ultrasound bath and sonicated for different times at a constant temperature of 20 °C. The bulk solvent was first vacuum evaporated in a Laborota 4000 rotary evaporator (Heidolph. Schwabach, Germany) and finally reduced to approximately 0.5 mL using a gentle nitrogen stream in a Turbo Vap LV evaporator (Zymark, Allschwil, Switzerland).

2.4. Organic solvent dialysis

A comparison between the UAD and the OSD was performed using field samples in order to prove the effectiveness of the UAD. Briefly, SPMDs were placed inside glass flasks together with 250 mL of hexane and were left to dialyse for 48 h [19,20]. The bulk solvent was also evaporated to approximately 0.5 mL using a rotary evaporator and a gentle nitrogen stream.

2.5. Size exclusion chromatography (SEC)

Size exclusion chromatography was the chosen technique in order to remove any residual triolein and impurities from the extract. 300 μL of the extract was filtered through 0.2 μm Syringe Filter (GHP Acrodisc, PALL) and then fractionated using high

performance liquid chromatography (1100 Series, Hewlett Packard) with a SEC size exclusion column (350 × 21.2 mm^2, Envirosep ABC, Phenomenex) using dichloromethane as mobile phase with a constant flow of 5 mL min^{-1} [21].

Two detectors were used in a qualitative way in order to select the fraction to be collected from the size exclusion process. The diode array detector (DAD) used was obtained from Hewlett Packard and signals were recorded at 286 nm for all the chromatograms. A fluorescence detector (FLD) purchased from the same company was used as a second detector collecting 250 nm excitation wavelength while working at 410 nm emission wavelength.

Besides, in order to further specify the elution time, fractions of 2.5 mL were collected, concentrated to dryness, re-dissolved in 200 μL of hexane and kept in the dark at −20 °C until analysis by gas chromatography–mass spectrometry (GC–MS).

2.6. Chemical analysis

Dilutions in hexane of the NPD Control Standard S-4089 standard, CEN PCB Congener Mix 1 and Pesticide Mix 11 and Pesticide Mix 164 were used to prepare calibration standards for all the analytes. Extracts were analysed in a 6890N Agilent gas chromatograph (GC) coupled to a 5973N Agilent mass spectrometer (MS) with a 7683 Agilent autosampler (Agilent Technologies) using hydrogen as carrier gas (AD-1020, Hydrogen Generator, H_2). The experimental conditions of the analysis are summarised in Table 1.

Deuterated analogues (naphthalene-d_8, biphenyl-d_{10}, phenanthrene-d_{10}, pyrene-d_{10}, benzo[a]anthracene-d_{12}, benzo[a]pyrene-d_{12} and benzo[g,h,i]-perylene-d_{10}) were used for both recovery and quantification corrections.

Table 1
GC experimental conditions for the analysis of the studied organic compounds.

HP 6890N Agilent GC					
Injection mode	Splitless				
Injection volume	2 μL				
Injection temp.	270 °C				
Column	HP-5MS (30 m length × 0.25 mm i.d., 0.25 μm film)				
	Ramp	**Initial (°C/min)**	**Next (°C)**	**Hold (min)**	**Total (min)**
Temp. programme	Initial		50	2.00	2.00
	1. Ramp	40.00	100	0.00	3.25
	2. Ramp	25.00	200	10.00	17.25
	3. Ramp	20.00	250	5.00	24.75
	4. Ramp	25.00	290	15.00	41.35

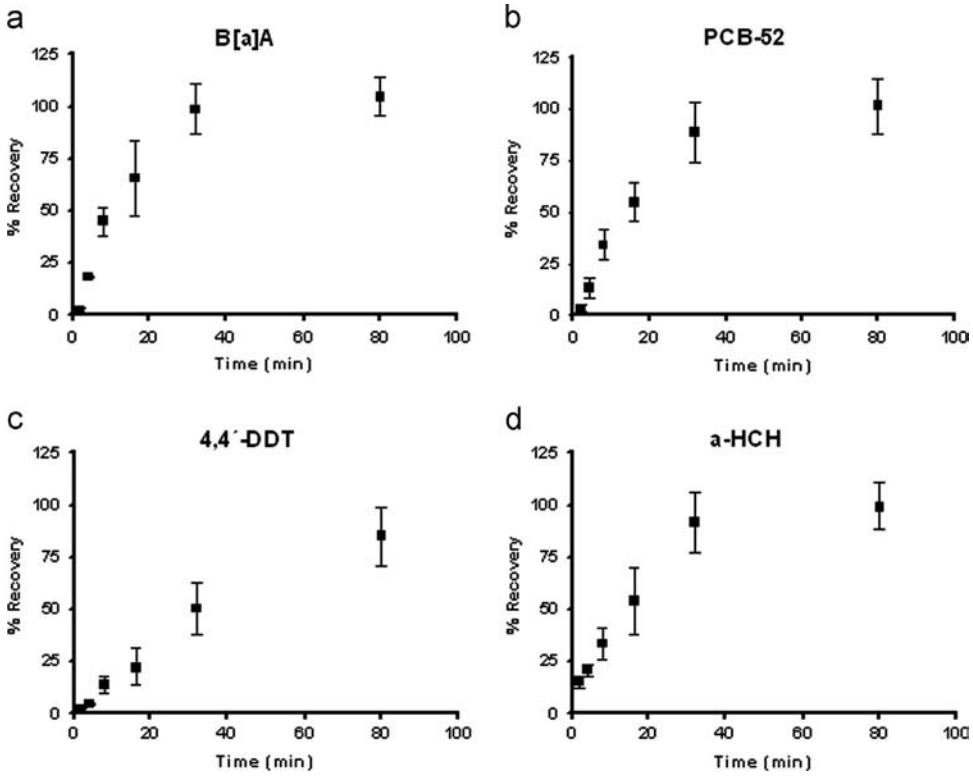

Fig. 2. Comparison of the average ($n=3$) recovery percentages obtained at different extraction time for (a) B[a]A, (b) PCB-52, (c) 4,4′-DDT and d) α-HCH.

2.7. SPMD deployments and retrievals

In order to prove the effectiveness of the new methodology in field samples, a single sampling campaign was accomplished at a harbour in Mundaka within the Reserve of the Biosphere of Urdaibai (Biscay, Basque Country) in June 2011. This sampling point was chosen due to the experience acquired by the group when working with SPMDs in previous sampling campaigns in the same area [22]. The deployment of SPMDs into the water was carried out using a 14 m steel chain with a block attached to one of its ends and a buoy on the other to avoid the loss of the whole structure. The items were placed inside stainless steel canisters and moored to the chain with plastic bridles and carabiners. Eight SPMDs were deployed at time 0. Half of them were retrieved 2 weeks after the deployment and the rest of them 2 weeks later.

SPMDs were carried to the sampling points stored in plastic zipper bags inside portable coolers. Field blank SPMDs accompanied the deployment SPMDs during transportation to the field, deployment, retrieval, and transportation back to the laboratory. During deployment and retrieval, care was taken to minimise contamination through handling or atmospheric inputs.

3. Results and discussion

3.1. UAD optimisation

Hexane was the chosen extractant for the UAD optimisation as it is the most used solvent in different extraction techniques for different POPs [10,15,23–25]. The extraction volume was fixed at 80 mL, as this is the minimum volume to cover all the SPMDs in the extraction flask. To optimise the extraction time, sonication of triplicate spiked samples was carried out at 2, 4, 8, 16, 32 and 80 min at a constant temperature of 20 °C. 20 μL of the surrogate solution was added to the bulk solvent in order to maintain under control the possible losses of the analytes during evaporation and SEC processes.

Time responses for B[a]A, PCB-52, 4,4′-DDT and α-HCH have been selected as examples to show the effectiveness of the extraction process for, respectively, PAHs, PCBs and OCPs (Fig. 2). After studying the obtained curves, it was observed that 32 min time is enough to perform the total extraction of almost every analyte (recoveries: PAHs, 82–109%; PCBs, 73–108%).

However, for compounds like 2,4′-DDT and 4,4′-DDT (Fig. 2c), and β-HCH and δ-HCH the recoveries obtained were inferior (42–79%). No clear explanations could be found for these low recoveries for the mentioned analytes. Thus, in order to improve the extraction, experiments using different mixtures of acetone: hexane (1:1 and 1:4) as extractant were performed. Nevertheless, no improvement was observed in the extraction of these compounds even in the time required for the total extraction for the rest of the analytes. Hence, as a last option to increase the recoveries, after a first extraction using hexane as extractant (32 min), a second extraction was carried out also with hexane

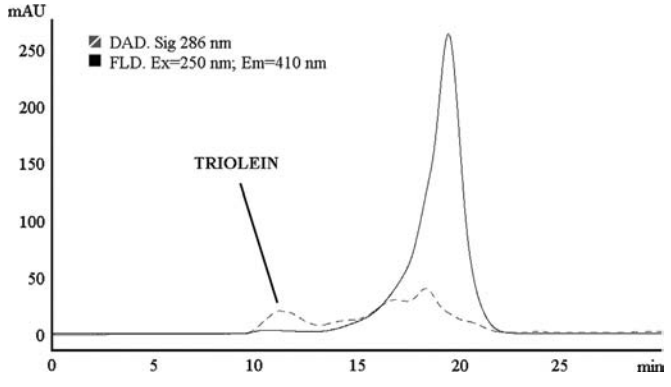

Fig. 3. Obtained signals by both DAD (wavelength, 286 nm) and FLD (excitation wavelength, 250 nm; emission wavelength, 410 nm) during size exclusion process.

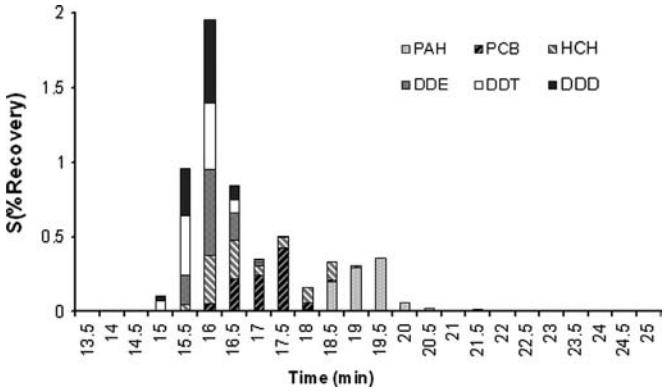

Fig. 4. The sum of the different recoveries for each family of analytes in the collected fractions between 13 and 25 min.

Table 2
The performance characteristics of the current method including recoveries, relative standard deviation (RSD) and limit of detection (LOD) for each analyte.

Compound	Recovery ($n=3$; 500 ng SPMD^{-1}) (%)	RSD (%)	LOD (ng SPMD^{-1})
PCB-18	101	6	12
PCB-31+PCB-28	98	2	11
PCB-52	95	4	19
PCB-44	96	6	10
PCB-101	97	5	17
PCB-153	103	3	3
PCB-118	98	5	11
PCB-138	96	9	5
PCB-149	91	7	5
PCB-180	86	11	1
PCB-194	87	9	29
Nap	93	5	8
Acy	103	2	9
Ace	103	6	24
Flu	102	3	11
Phe	97	4	10
Ant	97	8	9
Flr	94	5	6
Pyr	99	9	32
B(a)A	95	9	7
Cry	102	7	9
B[b]F	101	8	10
B[k]F	100	3	11
B[a]P	95	6	11
Ind	95	6	13
D[ab]A	92	12	13
B[ghi]P	81	13	13
α-HCH	102	3	39
β-HCH	52	9	25
γ-HCH	102	4	28
δ-HCH	56	9	15
2,4′-DDE	102	5	7
4,4′-DDE	95	6	7
2,4′-DDT	77	12	7
4,4′-DDT	75	8	4
2,4′-DDD	101	6	10
4,4′-DDD	98	2	8

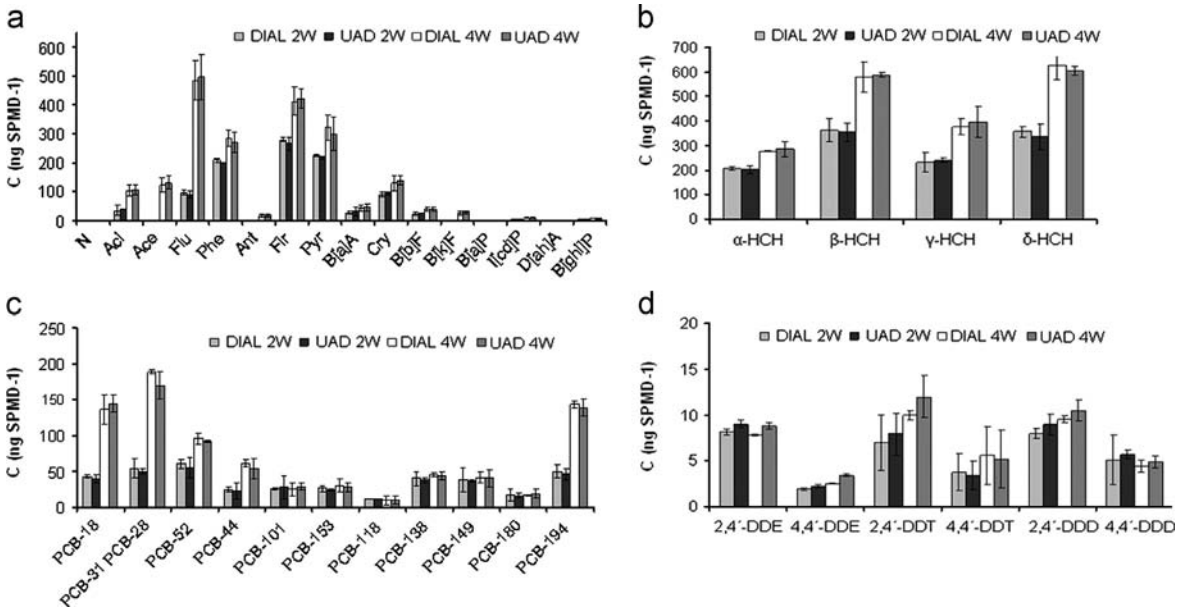

Fig. 5. Concentration ($n=2$, ng SPMD^{-1}) of PAHs (a), PCBs (c) and OCPs (b and d) obtained in SPMDs deployed in Mundaka harbour and retrieved 2 weeks (2W) and 4 weeks (4W) after using Dialysis (DIAL) and UAD. Concentrations were corrected using blank samples.

(32 min). Once again, no significant improvement was observed as recoveries were increased only up to 85%.

Therefore, the extraction time was finally fixed at 32 min using hexane as extractant.

In order to study the robustness of the UAD, spiked samples were placed in 8 different positions inside the ultrasound bath. After studying the results for every position ($n=3$) and performing an analysis of variance (ANOVA, $\alpha=0.05$), no influence in the positioning of the sample inside the ultrasound bath or in the number of ultrasonicated samples was observed.

3.2. Optimisation of the clean-up step

In order to determine the elution time of the target analytes in the cleaning step, sample extracts were subjected to SEC. After studying the obtained chromatograms (Fig. 3), and taking into account that the peak observed between 9.5 and 13 min in the DAD chromatogram corresponds to traces of triolein contained in the extract, fractions of 2.5 mL between 13 and 25 min were collected and analysed by GC–MS to further investigate the elution time of the different analytes.

The sum of the different recoveries for each analyte family in each collected fraction is shown in Fig. 4. The obtained signals verified that the desired fraction including all the analytes was the one between 14.5 and 22 min.

3.3. Performance of the analytical procedure

To study the performance of the analytical procedure, spiked SPMDs were prepared in a way similar to those for the optimisation, using in this case a 97% purity triolein purchased from Sigma-Aldrich instead of the one used during the optimisation (75% purity). The performance characteristics of the analytical procedure employed in this study are summarised in Table 2. The proposed method entails good repeatability (RSD 2–13%) and recoveries (around 100% for almost every analyte except for β-HCH, δ-HCH, 2,4′-DDT and 4,4′-DDT). Limits of detection were calculated using the average signal ($n=3$) plus three times the standard deviation of laboratory blank samples.

Comparing these results to other alternative extraction methods [16,26], it is observed that recoveries are, in general, acceptable and around 100%. The recoveries for β-HCH, δ-HCH, 2,4′-DDT and 4,4′-DDT are lower but still acceptable, taking on account the large amount of analytes involved in this study and the good repeatability obtained for all the compounds. LODs were of ppb level and low enough to detect the concentrations of analytes usually found in slightly-polluted aquatic environments.

3.4. Application to field samples

The optimised method was applied to field samples for the determination of PAHs, PCBs, HCHs, DDDs, DDTs and DDEs. The SPMDs deployed in the Mundaka harbour and retrieved after 2 and 4 weeks, as described in Section 2.6, were submitted to OSD and to the extraction technique proposed in this work (UAD) in order to compare both methods and study the applicability of the UAD. No significant difference was observed between both methods when working with field samples (Fig. 5).

Moreover, concentrations found in the exposed SPMDs are in the same level of the ones obtained in other studies with different environmental conditions [27], suggesting that this new methodology of extraction of SPMDs might be applicable to other sampling sites with different characteristics. However, in order to assure this statement further sampling campaigns should be carried out in different locations including different concentration levels of analytes.

4. Conclusions

In this study an ultrasound assisted dialysis (UAD) based extraction method has been developed and optimised to simultaneously determine PAHs, PCBs and OCPs contained in SPMDs. This novel methodology also contributes to obtain a greener chemistry as it requires shorter extraction time (32 min) and lower extractant volume (80 mL hexane) than those of other extraction techniques, including the OSD. Moreover, in a standard ultrasonic bath, up to 8 samples can be simultaneously treated using this technique. LODs calculated with laboratory blank samples were low enough to apply the proposed extraction technique to field samples, even in slightly polluted environments. In fact, experiments performed with SPMDs previously deployed in and

retrieved from a slightly polluted environment showed that the results obtained with the proposed methodology are comparable to those obtained with the OSD.

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