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Ionic liquid based hollow fiber supported liquid phase microextraction of ultraviolet filters

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ABSTRACT

Hollow fiber protected liquid phase microextraction using an ionic liquid as supported phase and acceptor phase (IL-HF-LPME) is proposed for the determination of four ultraviolet (UV) filters (benzophenone, 3-(4-methylbenzylidene)-camphor, 2-hydroxy-4-methoxybenzophenone and 2,4-dihydroxybenzophenone) in water samples for the first time. In the present study, four different ILs 1-hexyl-3-methylimidazolium tris(pentafluoroethyl)trifluorophosphate) [HMIM][FAP], 1-butyl-1-methylpyrrolidinium tris(pentafluoroethyl)trifluorophosphate [BMPL][FAP], 1-butyl-3-methylimidazolium phosphate ([BMIM][PO_4]) and 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF_6]) were evaluated as extraction solvent. Only [HMIM][FAP] showed high chemical affinity to the analytes which permits a selective isolation of the UV filters from the sample matrix, allowing also their preconcentration. IL-HF-LPME and high performance liquid chromatography provides repeatability from 1.1% to 8.2% and limits of detection between 0.3 and 0.5 ng/ml. Real water samples spiked with the analytes extracted were analyzed, and yielded relative recoveries ranging from 82.6% to 105.9%.

1. Introduction

Currently, ultraviolet (UV) filters are applied to sunscreen, cosmetics and other personal care products in order to filter UV-A and UV-B radiation from sunlight. There are two types of UV filters, organic UV filters, which work by absorbing UV filters, and inorganic UV filters (TiO_2, ZnO), which work by reflecting and scattering UV light. In the European Union, 28 UV filters [1] and in Switzerland 30 UV filters (29 organic, 1 inorganic) [2] are allowed in cosmetics. The amounts of UV filters integrated in many cosmetic formulations are between 0.1% and 10%.

UV filters enter the environment by different ways due to their various applications. It has been found that some of the UV filters are introduced to surface waters (rivers, lakes, and coastal seawaters) via release from the skin during swimming and bathing [3–5]. Besides this, indirect input (e.g. rubbed off with towels, washed off during showering, etc.) via wastewater treatment plants (WWTPs) is possible. Finally, the lipophilic character of the UV filters has also led to bioaccumulation in fish [6–8], marine sediments [9], and soils [10].

A few recent studies have shown that some UV filters are hormonally active (estrogenic, antiestrogenic, androgenic and antiandrogenic) in vitro and in vivo. Estrogenic effects were demonstrated with the yeast estrogen screen (YES) assay for up to 10 UV filters [11–13]. However, their possible effect on the environment is still quite unknown. The development of sensitive analytical methods to assess the pollution of water by UV filters is therefore required [14].

UV filters in the aqueous environment have been mainly determined by gas chromatography–mass spectrometry (GC–MS) [15], liquid chromatography–UV (LC–UV) [16,17] and LC–MS–MS [18] after proper sample pretreatment. Sample preparation methods such as liquid–liquid extraction (LLE) [19] and solid phase extraction (SPE) [20–22], have been developed for the preconcentration of UV filters. However, traditional sample preparation methods are time-consuming, solvent-intensive and laborious. Alternatively, single-drop microextraction (SDME), solid-phase microextraction (SPME) [23] and stir-bar sorptive extraction (SBSE) [15] have been proposed for the extraction of UV filters. Another promising method to reduce solvent, time and labour is hollow fiber based liquid phase microextraction (HF-LPME).

HF-LPME is a simple and solvent-minimized technique in which a hollow fiber, containing extraction solvent, is affixed to the tip of the syringe needle for the extraction of analytes from an aqueous sample. Upon completion of extraction, the extraction solvent is withdrawn into the syringe and injected into a GC or a GC–MS system for analysis. Organic solvents are widely used as extraction solvent for HF-LPME [24]. These may add to the environmental pollution burden. Therefore, ionic liquids (ILs), generally considered environmentally sound materials, have been used as green solvents for extraction. Special features like low-vapor pressure, high viscosity, dual natural polarity, good thermal stability and a

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wide range of miscibility with water and other organic solvents are generally attributed to ILs. ILs containing the hexafluorophosphate anion (PF_6^-) have been applied as extraction solvents in SDME [25], three-phase HF-LPME [26,27] and dynamic LPME (dLPME) [28] for a variety of analytes. Recently, Yao et al. employed a new class of ILs containing the tris(pentafluoroethyl)trifluorophosphate anion (FAP^-) for SDME [29]. The new type of ILs show excellent hydrolytic, thermal and electrochemical stability. One of the most peculiar properties is their ultra-hydrophobic nature. It has been shown that the water uptake of these ILs is more than 10 times less than that of ILs with the PF_6^- [30].

In the present study, a new IL, 1-hexyl-3-methylimidazolium tris(pentafluoroethyl)trifluorophosphate ([HMIM][FAP]) was applied to the HF-LPME (IL-HF-LPME) of several UV filters from aqueous samples for the first time. Extraction parameters including stirring rate, pH of aqueous samples, salt concentration and extraction time were optimized. The proposed procedure was applied to determine UV filters in environmental water samples.

2. Experimental

2.1. Materials and chemicals

Benzophenone (99%) (BP), 3-(4-methylbenzylidene)-camphor (99+%) (4-MBC), 2-hydroxy-4-methoxybenzophenone (98+%) (BP-3) and 2,4-dihydroxybenzophenone (99%) (DHB) were obtained from Alfa Aesar (Heysham, England). The ILs used in this study, [HMIM][FAP] and 1-butyl-1-methylpyrrolidinium tris(pentafluoroethyl)trifluorophosphate ([BMPL][FAP]) were provided by Merck (Darmstadt, Germany), 1-butyl-3-methylimidazolium phosphate ([BMIM][PO_4]) and 1-butyl-3-methylimidazolium hexafluorophosphate ([BMIM][PF_6]) were purchased from Strem Chemicals (Newburyport, MA, USA). All solvents used were of HPLC grade. Methanol and acetone were obtained from Merck and ethanol was purchased from Fisher (Loughborough, UK). Sodium chloride (NaCl) was obtained from GCE (ChulaVista, CA, USA). Ultrapure water was obtained from a Milli-Q water purification system (Millipore, Billerica, MA, USA).

Individual stock solutions of 1000 μg/ml of each pure UV filters were prepared in methanol and stored at 4 °C. Working solutions containing all of the UV filters at different concentrations were prepared by spiking them into ultrapure water. Working solutions at the concentration of 1 μg/ml were used for the optimization experiments. Environmental water samples were collected from the Singapore River.

Accurel Q3/2 polypropylene hollow fiber membrane with an inner diameter of 600 μm, wall thickness of 200 μm and wall pore size of 0.2 μm was purchased from Membrana (Wuppertal, Germany).

2.2. Blank contamination

Contamination is a common problem in UV filter analysis, since these substances are widely used in many cosmetics and personal care product such as sunscreens, soaps and shampoos. These compounds could contaminate glassware during sample preparation processing. In order to minimize these contaminations, some precautions needed to be taken. Thus, surgical gloves were worn during sample preparation and scrupulously clean glassware was used. All glassware was rinsed three times each with acetone, methanol and ultrapure water after use and once again immediately before use to eliminate sample contamination.

2.3. Instrumentation

Chromatographic analyses were performed on a Waters (Milford, MA, USA) high performance LC (HPLC) system with a Rheodyne (Cotati, CA, USA) 77251 injector equipped with a 20 μl sample loop, a Degasys DG-2410 vacuum degasser, a waters 1525μ binary HPLC pump and a Waters 2487 dual λ absorbance detector. Data acquisition and processing were accomplished by Empower version 5.0 (Waters) data analysis software.

Chromatographic separation was based on a Metaphase KR100-5-C18 (Bioscience, Kuala Lumpur, Malaysia) column (25 cm × 4.6 mm i.d., packed with 5 μm particle size C_{18} stationary phase). The mobile phase used consisted of ethanol: 1% acetic acid 60:40 (v/v) at 1 ml/min flow rate. The analytes were monitored at 289 nm.

2.4. IL-HF-LPME procedure

Briefly, IL-HF-LPME was performed as follows: prior to extraction, the hollow fiber was cut manually into 2.8 cm lengths and one end of it was heat sealed. In order to eliminate blank contamination, these segments were cleaned in acetone and methanol separately by sonication for 10 min and dried in air before use. A 10 ml volume of sample solution was added to a 15 ml sample vial with a 15 mm × 6 mm magnetic stirring bar. The sample vial was placed on a MR3001K magnetic stirrer plate (Heidolph, Kelheim, Germany). A 7.0 μl aliquot of IL was withdrawn into a 25-μl microsyringe with a flat needle tip. A 15 ml sample vial septum cap was pierced by the microsyringe. The needle tip was inserted into a hollow fiber and then the fiber was immersed in IL for 5 s for impregnation of the porous wall. Subsequently the IL contained in the microsyringe was injected into the hollow fiber, after which the latter was immersed in the sample immediately for extraction. The sample vial was capped during extraction. After extraction, the hollow fiber with microsyringe was removed from the sample solution. The extracted solution was withdrawn into the microsyringe and injected directly into HPLC-UV system for analysis. The used hollow fiber was discarded and a fresh one was used for the next extraction.

3. Results and discussion

In order to quantitatively assess and compare the performance of individual extractions, the enrichment factor (EF) was used. The EF is defined as the ratio of the analyte concentration in the extraction solvent after extraction and the initial analyte concentration in the aqueous sample solution. Calibration plots were also prepared using IL ([HMIM][FAP], see below) to show that they were satisfactorily linear; the data are shown in Table S1, Supplementary material. Due to the high viscosity of IL, only around 4–5 μl of IL could be withdrawn into the microsyringe after extraction when 7 μl IL used for extraction. A volume of 4 μl was used for HPLC analysis directly since it was completely soluble in the mobile phase.

3.1. Effect of IL solvents

The selection of an appropriate extraction solvent is very important for IL-HF-LPME. Organic solvents such as toluene, *n*-nonane, and isooctane are commonly used in HF-LPME. Here, both the wall pores of the hollow fiber membrane and the channel were filled with the organic solvent. In this work, ILs were used instead. The involved ILs are water immiscible and have good solubility for many organic solvent. Fig. 1 clearly shows that higher EFs were achieved using [HMIM][FAP] when compared with the other ILs, making it the IL of choice for subsequent experiments. The possible reason is its better affinity for the UV filters resulting from its relatively higher polarity ("like dissolves like" principle) and ultra-hydrophobic ability.

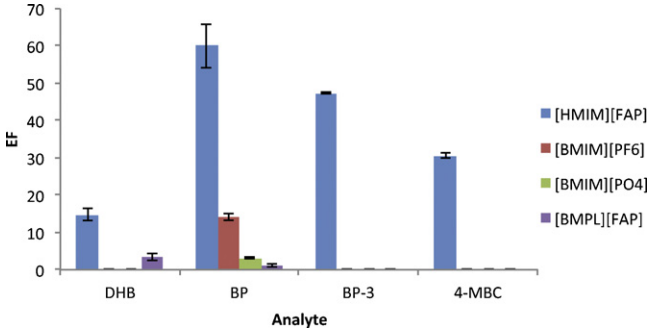

Fig. 1. Effect of different ionic liquid on HF-LPME. Spiked concentration: 1 μg/ml for individual analyte; pH of sample solution: 2.5; stirring rate: 400 rpm, extraction time: 20 min. Three replicate experiments were conducted.

3.2. Effect of pH of the aqueous phase

The effect of pH of the sample solution on IL-HF-LPME was studied. As the results show in Fig. 2, BP and 4-MBC showed almost no significant difference in terms of EF when the pH was varied between 2 and 12 (pH 1, 3, 6, 8, and 11), not surprisingly since these compounds are relatively neutral and hydrophobic. Better EFs of BP-3 and DHB were observed at pH 3. The pK_a value of BP-3 and DHB are 7.56 and 7.53. Thus, acidic pH values favor the extraction due to the reduction of their ionic states. At higher pH values, hydrolysis conceivably caused a deterioration in extraction. A pH of 3 was enough to maintain all analytes in their unionized forms. Therefore, a pH value of 3 was adopted for further experiments.

3.3. Effect of stirring rate

The effect of stirring rate on EFs was obtained over a range of 200 rpm to the maximum of 1000 rpm (200, 400, 700, 1000 rpm). Fig. 3 shows that for the four UV filters, 400 rpm was appropriate to obtain the highest EF. Higher stirring rate than 400 rpm resulted in air entering the solution and the formation of air bubbles that were attached to the surface of the hollow fiber that might occupy contact sites on the surface, thus impeding mass transfer of the analytes. Therefore, 400 rpm was considered the optimum stirring rate.

3.4. Effect of extraction time

IL-HF-LPME is an equilibrium extraction process. Fig. 4 shows the time profiles from 20 to 60 min at 10 min intervals. All of the EFs increased with increase in extraction time up to 50 min, but a decrease thereafter. The deterioration in extraction after 50 min

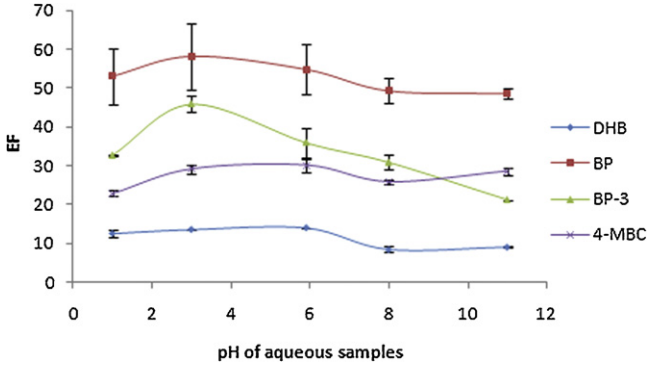

Fig. 2. Effect of pH of aqueous phase on IL-HF-LPME. Spiked concentration: 1 μg/ml for individual analyte; extraction solvent: [HMIM][FAP]; extraction time: 20 min; stirring rate: 400 rpm. Three replicate experiments were conducted.

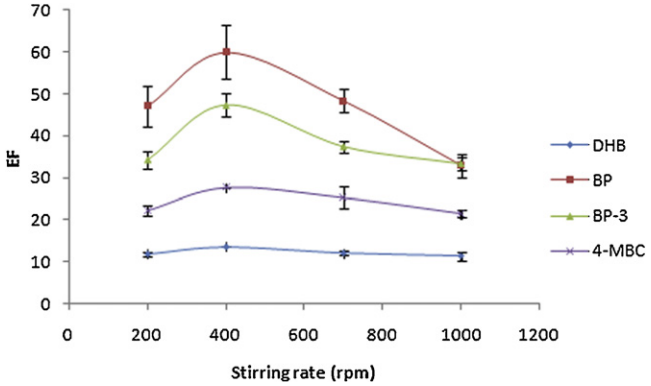

Fig. 3. Effect of stirring rate on IL-HF-LPME. Spiked concentration: 1 μg/ml for individual analyte; extraction solvent: [HMIM][FAP]; extraction time: 20 min; pH of aqueous samples: 3. Three replicate experiments were conducted.

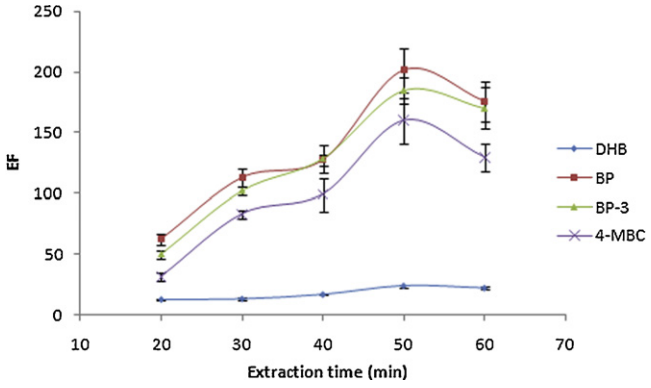

Fig. 4. Effect of extraction time on IL-HF-LPME. Spiked concentration: 1 μg/ml for individual analyte; extraction solvent: [HMIM][FAP]; stirring rate: 400 rpm; pH of aqueous samples: 3. Three replicate experiments were conducted.

may be attributed to the partitioning of water into the IL, which reduced the mass transfer of the analytes into the IL. Based on this observation, the optimum extraction time was set at 50 min.

3.5. Effect of salt concentration

The addition of salt (NaCl) to the sample solution leads to a decrease of solubility of the analytes in solution, and improves the extraction efficiency. The influence of salt on the extraction of IL-HF-LPME was adjusted by addition of NaCl from 0 (w/v) to 40% at intervals of 10% to exploit the salting-out effect. The experiment results (Fig. 5) indicated that the optimum salt concentration in

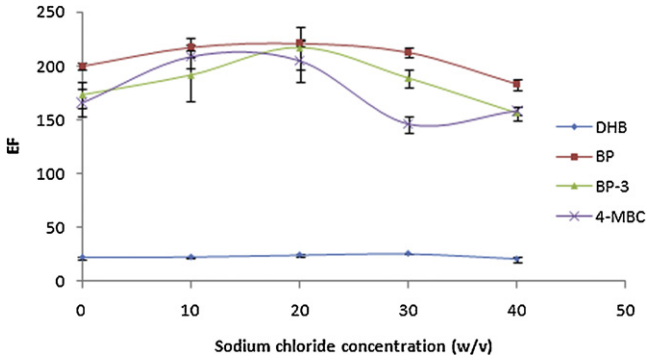

Fig. 5. Effect of salt concentration on IL-HF-LPME. Spiked concentration: 1 μg/ml for individual analyte; extraction solvent: [HMIM][FAP]; stirring rate: 400 rpm; pH of aqueous samples: 3. Three replicate experiments were conducted.

Table 1
Quantitative results of IL-HF-LPME ($n = 3$).

Analytes	Linearity range (μg/ml)	Correlation coefficient (r)	LOD (ng/ml)	RSD (%, $n = 5$)	EF	ER%	Ref. [33] EF	Ref. [32] EF
DHB	0.01–1	0.995	0.5 ± 0.04	8.2	25 ± 1	1.0	101	
BP	0.005–1	0.997	0.2 ± 0.006	3.4	221 ± 4	8.9	85	
BP-3	0.005–1	0.996	0.2 ± 0.002	1.1	216 ± 10	8.6	107	98
4-MBC	0.005–1	0.993	0.3 ± 0.012	3.5	205 ± 11	8.2		48

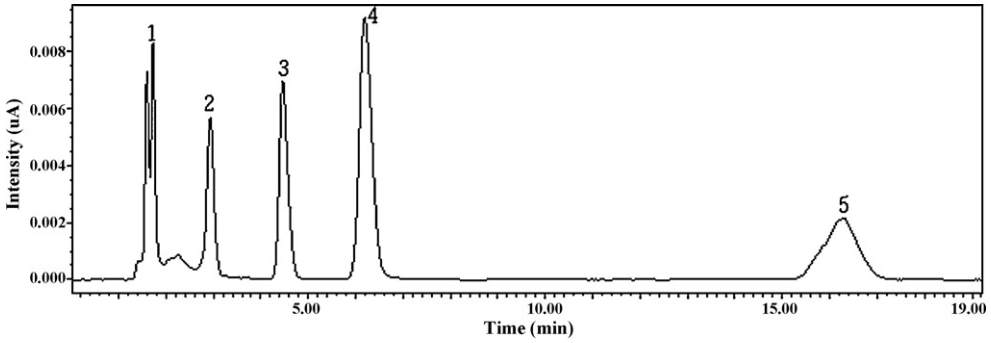

Fig. 6. HPLC of extract of spiked river water sample (25 ng/ml of each analyte) under the most favorable extraction conditions (see text). Peaks: 1: [HMIM][FAP]; 2: DHB; 3: BP; 4: BP-3; 5: 4-MBC.

Table 2
Relative recoveries and precision of IL-HF-LPME of tap water and river water spiked with UV filters at different concentration (5 ng/ml and 25 ng/ml) ($n = 3$).

Analytes	Tap water								River water							
	5 ng/ml				25 ng/ml				5 ng/ml				25 ng/ml			
	Recovery%	RSD%	EF	ER%	Recovery%	RSD%	EF	ER%	Recovery%	RSD%	EF	ER%	Recovery%	RSD%	EF	ER%
DHB	102.7	3.3	24	1	82.6	8.4	22	1	101.9	5.2	24	1	95.2	1.9	25	1.1
BP	96.9	4.2	212	8.5	89.1	7.9	201	8.1	101.1	6.6	221	8.8	96.1	5.3	216	8.7
BP-3	98.1	5.7	191	7.6	105.9	6.5	205	8.2	104.9	4.2	204	8.2	101.5	4.8	196	7.8
4-MBC	104.2	1.8	208	8.3	101.6	2.3	199	8	98.9	1.1	197	7.9	104.1	3.1	203	8.1

the present study was between 15% and 20% (w/v). Beyond 20%, the increased viscosity of the sample solution conceivably inhibited the extraction by retarding the mass transfer of the analytes. Since 3 of the analytes showed better extraction at 20% NaCl, this value was adopted as the most favorable condition.

3.6. Method validation and application

Under the optimum extraction conditions, repeatability, linearity and limits of detection (LODs) were measured under the described extraction conditions using spiked ultrapure water samples, and the results are given in Table 1. The linearity of calibration plots was investigated at the concentrations of between 0.005 (0.01 for DHB) and 1 μg/ml. All analytes exhibited good linearity with correlation coefficients of 0.993 or better. The LODs were calculated at a signal-to-noise (S/N) ratio of 3; they were in the range of 0.2–0.5 ng/ml. Previous work [18,31] has shown that the common levels for the four investigated UV filters were about 0.4–2.5 ng/ml (except for one BP-3 concentration that was lower than these values) in real water samples, which were higher than the LODs of the present work. Therefore, the developed method can be applied to routine analysis of real water samples. The repeatability of the analytical performance, expressed as relative standard deviations (RSDs), was calculated for five replicates of sample containing the analytes at 50 ng/ml. These were <8.2%. EFs ranging from 25 to 221 were obtained for these analytes. The obtained EFs were higher than those reported in previous microextraction work (except for DHB), where IL-single drop microextraction [32] or DLLME [33] were the techniques applied.

In order to check the applicability of IL-HF-LPME, the method was applied to tap water and river water samples. The water samples were subjected to IL-HF-LPME without filtration. There were, however, no target analytes detected, indicating the absence of these compounds, or they were below the LODs. Nevertheless, the proposed method was evaluated by means of relative recovery (defined as the ratio of peak areas of the spiked real water extracts to spiked ultrapure water extracts) experiments at concentration levels of 5 ng/ml and 25 ng/ml. Fig. 6 shows a chromatogram of an extract of a spiked river water sample (25 ng/ml of each analyte). For all sample solutions at different concentrations, three replicate extractions were conducted. Table 2 show that the relative recoveries varied between 82.6% and 105.9% and RSDs ($n = 3$) were in the range of 1.1–8.2%. For each analyte, the recoveries did not differ significantly when the extraction was carried out in a more complex matrix (i.e. river water). These results indicated that the proposed method was tolerant of matrices generally representative of environmental water samples.

4. Conclusions

An FAP-based IL, [HMIM][FAP], was applied for the first time as an HF-LPME extraction solvent for the preconcentration of UV filters from environmental water samples. Under the optimized IL-HF-LPME conditions, up to 216-fold enrichment factors (EFs) were achieved. Compared to other commonly used ILs in extraction processes, [HMIM][FAP] gave high EFs for the UV filters considered in this work, making it an ideal candidate for use in the HF-LPME of these contaminants. Under optimum conditions,

the proposed method provided good relative recoveries with acceptable repeatability and exhibited good linearity over the investigated concentration ranges. The technique can be applied to the routine determination of UV filters in natural water samples.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.chroma.2011.12.110.

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