



Ferrofluid-based liquid-phase microextraction

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ABSTRACT

A new mode of liquid-phase microextraction based on a ferrofluid has been developed. The ferrofluid was composed of silica-coated magnetic particles and 1-octanol as the extractant solvent. The 1-octanol was firmly confined within the silica-coated particles, preventing it from being lost during extraction. Sixteen polycyclic aromatic hydrocarbons (PAHs) were used as model compounds in the development and evaluation of the extraction procedure in combination with gas chromatography–mass spectrometry. Parameters affecting the extraction efficiency were investigated in detail. The optimal conditions were as follows: 20 mL sample volume, 10 mg of the silica-coated magnetic particles (28 mg of ferrofluid), agitation at 20 Hz, 20 min extraction time, and 2 min by sonication with 100 μ L acetonitrile as the final extraction solvent. Under optimal extraction conditions, enrichment factors ranging from 102- to 173-fold were obtained for the analytes. The limits of detection and the limits of quantification were in the range of 16.8 and 56.7 pg mL^{-1} and 0.06 and 0.19 ng mL^{-1} , respectively. The linearities were between 0.5–100 and 1–100 ng mL^{-1} for different PAHs. As the ferrofluid can respond to and be attracted by a magnet, the extraction can be easily achieved by reciprocating movement of an external magnet that served to agitate the sample. No other devices were needed in this new approach of extraction. This new technique is affordable, efficient and convenient for microextraction, and offers portability for potential onsite extraction.

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1. Introduction

Despite the tremendous evolution in analytical science in the past few decades, complex samples often cannot be analyzed directly because of the possible low concentration of target analytes, potential matrix interference and incompatibility of the analytes with detection systems [1]. As a result, sample preparation is unavoidable in most analytical procedures and imperative in those involving complex matrices. Much effort has been devoted to the development of miniaturized sample preparation methods, which are recognized as simple, cost-effective as well as environmentally friendly [2–4].

Solid-phase microextraction (SPME) is one example of an established commercial microextraction technique [5–8]. In recent years, an alternative solvent-minimized sample preparation approach, liquid-phase microextraction (LPME), has gained considerable attention [9–22]. LPME commonly uses a water-immiscible solvent at the low microliter range as an extractant. It overcomes many of the disadvantages of traditional liquid–liquid extraction, and has been clearly demonstrated to be simple, rapid, cost-effective and suitable for real world applications. It also eliminates

the possibility of carry-over between runs that may occur in the case of SPME.

Single-drop microextraction (SDME) was the first mode of LPME to emerge [9–11]. In this procedure, an organic droplet is suspended at the tip of a microsyringe needle for extraction. It is a facile, cost-effective technique for sample preparation. However, since the droplet is held by the needle tip, its stability during extraction can be problematical.

The drawbacks of SDME led to the development of hollow fiber or silica monolith protected liquid-phase microextraction [12–18]. In these techniques, organic solvents are confined within the pores or channels of the fiber or monolith during extraction. Since the solvents are protected, extraction efficiency can be easily enhanced by increasing the agitation of the sample and larger extraction solvent volumes.

Very recently, another mode of LPME, dispersive liquid–liquid microextraction (DLLME) has been developed [19–22]. In this technique, an organic extractant (that is water-immiscible) and disperser solvent (that is water-miscible) are quickly injected into an aqueous sample to form an emulsified solution. Since the extractant is highly dispersed in the aqueous phase, extraction can be achieved within a few seconds. However, there is some inconvenience in retrieving the organic phase. Classical DLLME avoids this problem with the use of extraction organic solvents with densities higher than water, such that, after extraction, the extractant can be sedimentated by centrifugation. This means that the

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method needs an additional sedimentation process, which is an inconvenience.

The different modes of LPME demonstrate that the convenient retrieval of organic solvents after extraction is of crucial importance in such techniques. In the present report, for the first time, a new LPME technique based on a ferrofluid is proposed. The ferrofluid is composed of silica-coated magnetic particles and organic solvent (1-octanol). By the reciprocating movement of an external magnet adjacent to the ferrofluid in the vial, the latter was agitated to enhance the extraction. No stirring bar or any other agitation devices were necessary. After extraction, the ferrofluid could be easily recovered for analysis by gas chromatography–mass spectrometry.

2. Experimental

2.1. Reagents and materials

Analytical-grade ferrous chloride ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$), polyvinyl alcohol (PVA), hydrogen peroxide (H_2O_2 , 30% w/w), sodium hydroxide (NaOH), ammonia solution (25%, v/v) and tetraethoxysilane (TEOS) were purchased from Sigma-Aldrich (St. Louis, MO, USA). HPLC-grade ethanol and acetonitrile were obtained from Fisher (Loughborough, UK). The 1-octanol was purchased from Merck (Darmstadt, Germany). The PAH standards (naphthalene (Nap), acenaphthylene (Acp), acenaphthene (Ace), fluorene (Flu), phenanthrene (Phe), anthracene (Ant), fluoranthene (Flt), pyrene (Pyr), chrysene (Cry), benz[a]anthracene (BaA), benzo[b]fluoranthene (BbF), benzo[k]fluoranthene (BkF), benzo[a]pyrene (BaP), indeno[1,2,3-cd]pyrene (InP), dibenz[a,h]anthracene (DBA), benzo[g,h,i]perylene (BPe)) were bought from Supelco (Bellefonte, PA, USA). Ultrapure water was produced on a Nanopure (Barnstead, Dubuque, IA, USA) water purification system.

2.2. Synthesis of silica-coated magnetic particles

In a three-necked round bottom flask, 90 mL of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ (0.24 mol L^{-1}) solution and 120 mL of PVA (10%, w/w) solution were mechanically stirred in a 50°C water bath for 10 min. Then, 30 mL of H_2O_2 (0.24 mol L^{-1}) and 120 mL of NaOH (3 mol L^{-1}) solutions were added in sequence. Stirring was maintained for 2 h. The as-obtained black particles were magnetically collected and washed with water three times. The particles were dried in an oven at 60°C before use.

The particles (0.2 g) were then mechanically dispersed in a mixture of ethanol (50 mL), water (4 mL) and ammonia solution (3 mL, 25% w/w). Under stirring, TEOS (2 mL) was added dropwise. After 6 h, the particles were collected and calcinated at 200°C for 2 h under a nitrogen atmosphere.

2.3. Characterization of the silica-coated magnetic particles

The silica-coated magnetic particles were studied by a JSM-6701F scanning electron microscope (SEM) (JEOL, Tokyo, Japan) equipped with an energy dispersive X-ray spectroscopy (EDS) accessory to observe their morphology and to analyze their elemental composition. The porous structure of the silica-coated magnetic particles was characterized by a Beckman Coulter (Miami, FL, USA) SA 3100 Plus instrument. The sample was evacuated in vacuum at 120°C for 3 h to remove any physically adsorbed substances before analysis. The specific surface area values were calculated according to the BET (Brunauer–Emmett–Teller) equation at P_5/P_0 (relative pressure) to be between 0.05 and 0.2. The pore parameters (pore volume and pore diameter) were evaluated from the adsorption

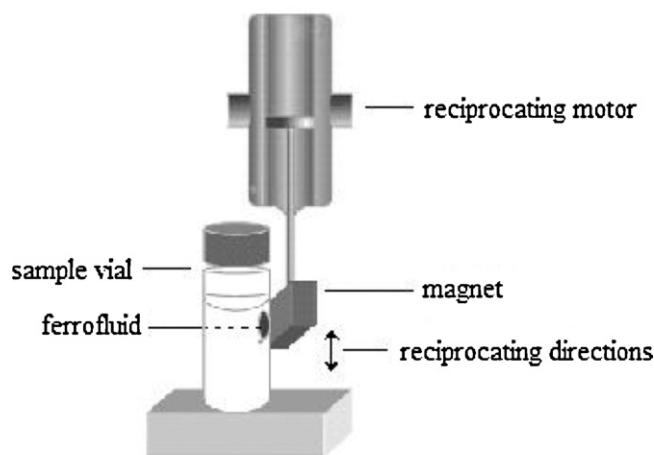


Fig. 1. Schematic of the ferrofluid-based microextraction system.

branch of the isotherm based on the Barrett–Joyner–Halenda model [23].

2.4. Sample preparation

A stock solution (containing $5 \mu\text{g mL}^{-1}$ of each PAH) was prepared in methanol and stored in a refrigerator. Water samples were prepared by spiking deionized water with analytes at known concentrations to study extraction performance under different conditions.

Genuine environmental water samples were collected from a local river. The samples were not filtered prior to analysis. They were analyzed directly or after being spiked with PAH standards at concentrations of 10 or 25 ng mL^{-1} , separately.

2.5. Extraction procedures

2.5.1. Preparation of the ferrofluid

The silica-coated magnetic particles (10 mg) and 1-octanol (100 μL) were mixed in a vial by sonication for 30 min. A magnet was held next to the bottom of the vial to attract the particles and the supernatant 1-octanol was recovered by a pipette. Thereafter, with the magnet still kept in situ, an aliquot of water (0.5 mL) was added into the vial to rinse the possible excess 1-octanol from the particles' surface. This step was repeated to obtain the ferrofluid. It was found that 78 μL of 1-octanol was recovered and that confined within the interstices and pores of the particles had a volume of 22 μL . The ferrofluid thus obtained weighed 28 mg.

2.5.2. Extraction

With the magnet kept in situ, 20 mL of sample solution was added to the vial containing the ferrofluid. The vial was then sealed and the magnet was connected to a YYCJ reciprocating motor (Ningbo, China), which allowed it to move in an up-and-down fashion on the outside of the vial (see Fig. 1). The reciprocating frequency was set at 20–80 Hz. Attracted by the moving magnet, the ferrofluid shifted accordingly, agitating the aqueous sample solution to facilitate the extraction process. Extraction was carried out for 20 min. The ferrofluid was subsequently moved to the bottom of the vial. The sample solution was discarded simply by decanting it. Thereafter, the magnet was removed and 100 μL of acetonitrile was introduced to the vial to desorb the 1-octanol and the PAHs by sonication. Finally, the magnet was again placed next to the vial and the supernatant was collected into an Eppendorf tube by a pipettor for analysis.

2.6. Gas chromatographic–mass spectrometric (GC–MS) analysis

Analysis was carried out on a Shimadzu QP2010 (Kyoto, Japan) GC–MS system with a DB-5ms fused silica capillary column (30 m × 0.25 mm I.D., film thickness 0.25 μm) (J&W Scientific, Folsom, CA, USA). Helium was employed as the carrier gas at a flow rate of 1.7 mL min⁻¹. The extractant injection volume was 1 μL and the injector temperature was set at 280 °C. The GC oven temperature was initially held at 70 °C for 2 min and then programmed to 190 °C at 15 °C min⁻¹. After being kept at 190 °C for 1 min, the temperature was programmed to 260 °C at 10 °C min⁻¹. Finally it was programmed to 285 °C at 5 °C min⁻¹ and held for 5 min. The GC/MS interface was maintained at 300 °C. The solvent cut time was 6 min (to bypass the solvent peak). All injections were in splitless mode. Selective ion monitoring mode was adopted for quantitative determination of the analytes (Nap, *m/z* 128, 129, 127, 102; Acp, *m/z* 152, 153, 151; Ace, *m/z* 153, 154, 152; Flu, *m/z* 166, 165, 167; Phe, *m/z* 178, 179, 176; Ant, *m/z* 178, 179, 176; Flt, *m/z* 202, 200, 203, 101; Pyr, *m/z* 202, 200, 101, 203; Cry, *m/z* 228, 226, 229; BaA, *m/z* 228, 226, 229, 227, 252; BbF, *m/z* 252, 250, 253, 126; BkF, *m/z* 252, 126, 250; BaP, *m/z* 252, 253, 250, 126; InP, *m/z* 276, 277, 138; DBA, *m/z* 278, 276; Bpe, *m/z* 276, 277, 138). The masses monitored by the detector were set as follows: 6–8 min, *m/z* 128, 129, 127, 102; 8–9.5 min, *m/z* 152, 153, 151, 154; 9.5–10.8 min, *m/z* 166, 165, 167, 139; 10.8–13 min, *m/z* 178, 176, 179, 152; 13–16 min, *m/z* 202, 203, 200, 101; 16–20 min, *m/z* 228, 226, 229, 227, 252; 20–23 min, *m/z* 253, 252, 250, 126; and 23–28 min, *m/z* 276, 278, 277, 138.

3. Results and discussion

3.1. Characteristics of the silica-coated magnetic particles

Previous research indicated the magnetic particles synthesized in this way are composed of nanoarrays of Fe₃O₄ [24]. It is known that Fe₃O₄ is not stable, especially under moist conditions. The particles are apt to be oxidized into Fe₂O₃ and thus lose magnetism gradually, which is detrimental to their applications. To increase the stability of such particles, surface modification or doping with inert ingredients may be effected. In the present study, the magnetic particles were coated with inert silica to increase their stability.

The SEM analysis showed that the silica-coated magnetic particles are spherical in shape, narrowly distributed and well dispersed, with a particle size of ca. 90 nm. The EDS analysis reveals an atom ratio of Fe:Si:O (22.5:9.1:68.3), suggesting the silica has been successfully coated onto the particle surface. The normal and silica-coated Fe₃O₄ particles were placed in aqueous solution to investigate their respective magnetic stability. It was observed that, after two weeks, the Fe₃O₄ particles showed no response to magnetism, while the silica-coated particles remain magnetic, indicating that surface modification increased their magnetic stability.

The nitrogen adsorption isotherm of the silica-coated magnetic particles shows a capillary condensation at medium relative pressures, suggesting the pores are mainly in the mesoporous range. The BET surface area, pore volume and average pore size were found to be 127 m² g⁻¹, 0.12 cm³ g⁻¹ and 4.2 nm, respectively.

3.2. Extraction optimization

3.2.1. Organic solvent selection for the ferrofluid

To achieve satisfactory LPME, several criteria on selecting the organic solvent phase or extractant should be met. First of all, the solvent should be immiscible with aqueous solution (except when the headspace mode is employed). Secondly, the target analytes should have good solubility in the selected solvent to ensure high extraction performance. Additionally, the solvent should have

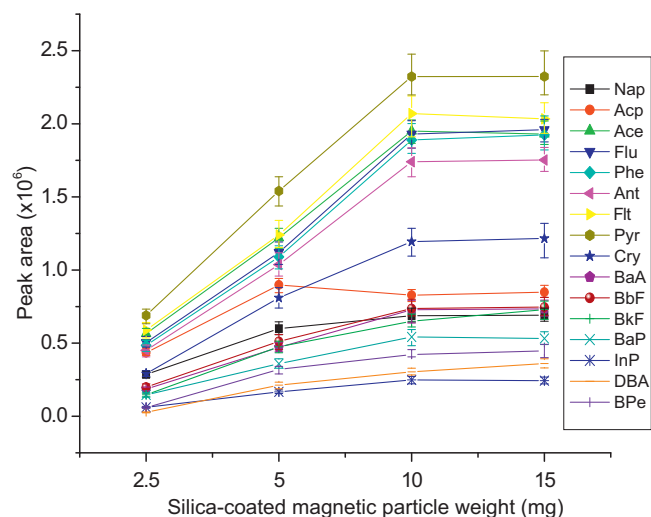


Fig. 2. The influence of silica-coated magnetic particle amount used in preparing the ferrofluid on the extraction performance for the PAHs.

a low vapor pressure to prevent loss during extraction, particularly sample agitation. According to these criteria, 1-octanol is a suitable extractant that has been widely used in many LPME applications. Moreover, it conceivably also has a particular affinity with the silica-coated magnetic particles because of the terminal hydroxyl moiety. By sonication of the silica-coated magnetic particles and 1-octanol, the ferrofluid can be easily established. The ferrofluid maintained its integrity upon the introduction of a magnet. Although the 1-octanol is lighter than water, under continuous magnetization, the solvent was firmly confined within the interstices and pores of the silica-coated magnetic particles (no leakage was observed), which guaranteed a successful liquid extraction process.

3.2.2. Optimization of the extraction process

Several parameters, including silica-coated magnetic particle weight, agitation frequency, extraction time, sonication (desorption) time, were evaluated to determine their most suitable values. A spiked water sample (20 mL) containing all the PAHs each at a concentration of 50 ng mL⁻¹, was used for the extraction.

Fig. 2 shows the influence of the weight of the silica-coated magnetic particles used in preparing the ferrofluid, on the extraction of PAHs. It can be observed that the peak areas for most of the analytes increased as the weight increased from 2.5 to 10 mg; after 10 mg, the signals reached a plateau. The result demonstrates that, in the given conditions, 10 mg of the silica-coated magnetic particles could confine sufficient 1-octanol to achieve high extraction performance. The further increase in weight gave no additional increase in extraction.

Fig. 3 displays the effect of reciprocating frequency on the extraction of the PAHs. The extraction performance decreased as the frequency increased. As might be expected, high frequency agitation may cause the 1-octanol to leach out from the nanoparticles (ferrofluid) because of mechanical considerations. In such a case, the 1-octanol cannot be totally recovered after extraction. It is noteworthy that although the agitation frequency for the ferrofluid was relatively low, under the given conditions, enrichment factors ranging from 102- to 173-fold could be obtained for the PAHs.

Fig. 4 shows the extraction time profiles. It can be observed that as the extraction time was increased up to 20 min, the peak areas increased significantly; extraction beyond 20 min did not contribute to additional increase in analytical signals. It is therefore reasonable to select an extraction time of 20 min.

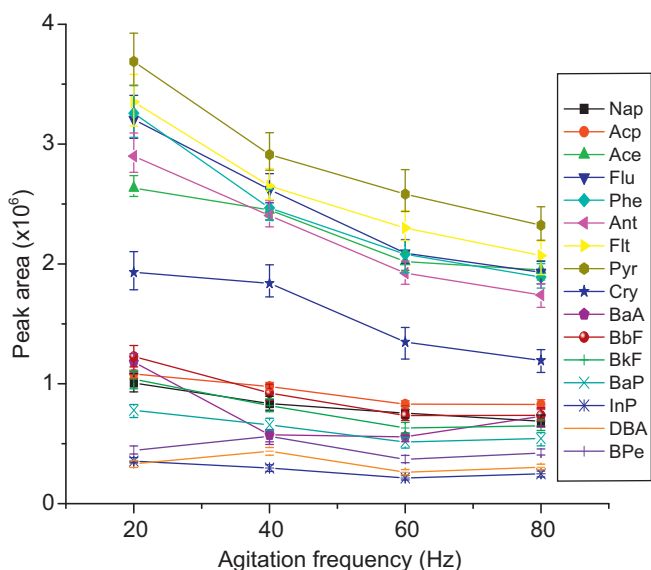


Fig. 3. The influence of agitation frequency on the extraction of PAHs.

It has been reported that silica may adsorb PAHs [18]. In the present study, since the magnetic particles were coated with a silica layer, the adsorption of PAHs by silica alone was investigated. The particles were used directly for extracting PAHs under the same conditions as when the ferrofluid was used. However, the GC–MS trace (not shown) did not show any analyte signals, indicating that the PAHs were extracted by the 1-octanol rather than by the particles.

The 1-octanol as well as the PAHs confined within the ferrofluid was released by desorption with acetonitrile. The desorption time was investigated from 1 to 8 min. As shown in Fig. 5, the peak areas of the PAHs increased as the desorption time was increased to 2 min. Beyond 2 min, the analytical signals remained largely invariant, indicating that desorption was completed after just 2 min.

Based on the above discussion, the most suitable extraction conditions were based on the use of 10 mg of the silica-coated magnetic particles (28 mg of ferrofluid), 20 Hz of agitation, 20 min for the extraction and 2 min by sonication with 100 μ L acetonitrile as the final extraction solvent.

Fig. 6 displays the GC–MS total ion current traces of a PAH standard solution (50 ng mL⁻¹ of each analyte) (a), and the PAHs (also

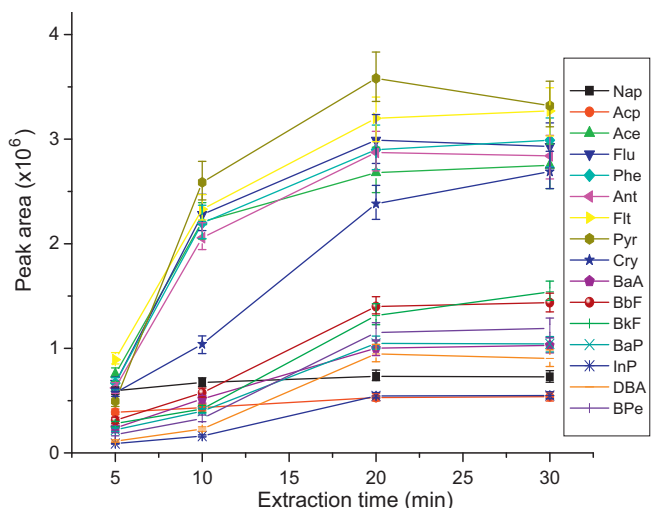


Fig. 4. The extraction time profiles for PAHs.

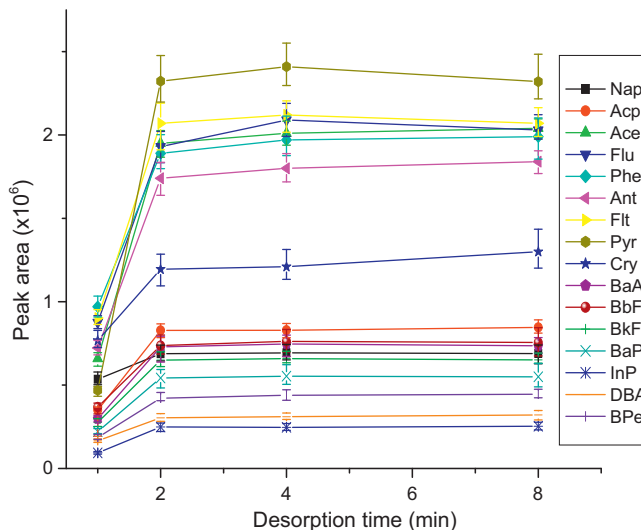


Fig. 5. The influence of sonication time on desorption of the PAHs.

at individual concentrations of 50 ng mL⁻¹) in a water sample after extraction under the optimized conditions (b). The enrichment is obvious, demonstrating the good extraction performance of the proposed method.

3.3. Method evaluation

A series of experiments with regard to the linearity, limits of detection (LODs), limits of quantification (LOQs), repeatability and recoveries was performed to validate the proposed method under the developed working conditions. The recoveries were obtained by calculating the ratio of the amount of the analytes in the ferrofluid to that in the water solution prior to extraction. The results are listed in Table 1. The linearity of the method was tested over a range of 0.5 and 100, or 1 and 100 ng mL⁻¹, depending on the analytes, and calibration curves were plotted. A statistical regression model was applied and regression coefficients (r^2) were calculated to be higher than 0.982 for all analytes. The LODs for the PAHs, calculated at a signal-to-noise (S/N) ratio of 3, ranged from 16.8 to 56.7 pg mL⁻¹. The LOQs, calculated at S/N = 10, ranged from 0.06 to 0.19 ng mL⁻¹.

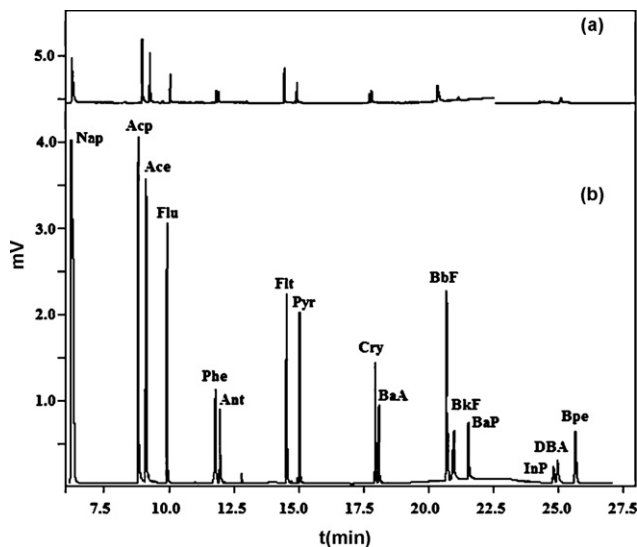


Fig. 6. The GC–MS total ion current traces of a PAH standard solution (50 ng mL⁻¹ of each analyte) (a), and the PAHs (also at 50 ng mL⁻¹ of each) in a water sample after extraction under the optimized conditions (b).

Table 1

Linear range, regression data, limits of detection (LODs), limits of quantification (LOQs) and recoveries of PAHs of the ferrofluid-based liquid-phase microextraction method.

Analyte	Linear range (ng mL ⁻¹)	r ²	LOD (pg mL ⁻¹)	LOQ (ng mL ⁻¹)	RSD ^a (% , n = 3)	Recovery (%)
Nap	1–100	0.9926	49.1	0.16	4.4	66.4
Acp	1–100	0.9897	41.2	0.14	5.5	83.1
Ace	0.5–100	0.9934	24.5	0.08	6.3	89.5
Flu	0.5–100	0.9953	23.2	0.08	4.7	89.2
Phe	0.5–100	0.9921	19.6	0.07	8.5	90.1
Ant	0.5–100	0.9936	22.7	0.08	5.9	91.7
Flt	0.5–100	0.9945	17.9	0.06	7.3	90.2
Pyr	0.5–100	0.9976	22.1	0.07	3.9	93.3
Cry	0.5–100	0.9932	16.8	0.06	6.2	84.2
BaA	1–100	0.9976	27.5	0.1	6.7	77.3
BbF	1–100	0.9931	36.1	0.12	4.9	69.5
BkF	1–100	0.9911	34.2	0.11	5.2	66.1
BaP	1–100	0.9861	28.9	0.1	6.6	82.4
InP	1–100	0.9832	41.5	0.14	8.1	77.3
DBA	1–100	0.9831	56.7	0.19	11.3	64.1
BPe	1–100	0.9823	43.2	0.14	9.1	59.2

^a Calculated from the sample spiked at LOQ levels.**Table 2**

PAHs determined in river water by ferrofluid-based liquid-phase microextraction with GC-MS.

Analyte	Quantity (ng mL ⁻¹)	RSD (% , n = 3)	Analyte	Quantity (ng mL ⁻¹)	RSD (% , n = 3)
Nap	3.76	8.4	Cry	0.19	8.4
Acp	2.14	7.8	BaA	n.d	–
Ace	1.57	5.2	BbF	n.d	–
Flu	2.16	6.1	BkF	n.d	–
Phe	0.98	5.8	BaP	n.d	–
Ant	0.84	11.6	InP	n.d	–
Flt	0.57	7.2	DBA	n.d	–
Pyr	0.91	9.3	BPe	n.d	–

n.d = not detected.

The results are comparable with those from previous reports, where a solvent bar (in LPME mode) or a polydimethylsiloxane fiber (in SPME mode) was used for analysis of the PAHs [18,25].

The repeatability of the peak areas was studied for three replicate experiments by spiking ultrapure water with the PAHs at the LOQ concentrations. The relative standard deviations (RSDs) for the PAHs were below 11.3%, illustrating the satisfactory repeatability achieved by the procedure.

3.4. Analysis of river water

To eliminate possible matrix effects, the standard addition method was adopted for the quantitative determination of the PAHs in river water. Three aliquots of the river sample were analyzed in parallel, with the results presented in Table 2. The RSDs were generally satisfactory (<12%). Several PAHs were detected in the river samples, indicating the procedure developed was suitable for genuine environmental applications.

4. Conclusion

In the present study, a new liquid–liquid microextraction (LLME) technique based on the application of a ferrofluid was developed. The ferrofluid was composed of silica-coated magnetic particles and adsorbed 1-octanol. During extraction the 1-octanol was firmly confined within the pores and interstices of the particles. By the reciprocating movement of a magnet adjacent to the ferrofluid, the latter shifted accordingly, accelerating the extrac-

tion process. It is noteworthy that no additional stirring or any other agitation processes were necessary in the extraction. Thus, the procedure is suitable for onsite sample preparation. Under the optimal extraction conditions, the limits of detection were as low as in the pg mL⁻¹ range for polycyclic aromatic hydrocarbons. Good linearity and repeatability were also achieved. The procedure was applied to river water samples and found to be a feasible approach. In general, the ferrofluid-based LPME is a portable, simple and effective method for sample preparation.

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