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Simultaneous trace determination of acidic non-steroidal anti-inflammatory drugs in purified water, tap water, juice, soda and energy drink by hollow fiber-based liquid-phase microextraction and ultra-high pressure liquid chromatography coupled to tandem mass spectrometry

Haojie Zhang [a], Zhenxia Du [a,*], Yu Ji [a], Mei Mei [a,b]

[a] *College of Science, Beijing University of Chemical Technology, Beijing 100029, China*
[b] *Department of Earth and atmospheric sciences, University of Houston, TX 77204, USA*

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ABSTRACT

In this study, a two-phase hollow fiber liquid-phase microextraction (HF-LPME) coupling with ultra performance liquid chromatography tandem mass spectrometry (UPLC-MS/MS) method was developed for determination of four non-steroidal anti-inflammatory (NSAIDs)—salicylic acid, ibuprofen, naproxen and diclofenac in real water samples. The influencing parameters of HF-LPME sample preparation method, such as organic solvents (acceptor phase), pH of sample solution (donor phase), extraction time, stirring speed, extraction temperature and ionic strength were systematically optimized. Through the developed determination method, high enrichment factors (195–346) were achieved for the four drugs. The instrumental calibration curves of salicylic acid, naproxen, diclofenac, and ibuprofen show good linear relations ($R > 0.998$) in the concentration range of 1–500, 5–2500, 10–5000 and 5–2500 μg L^{-1}, respectively. The average recoveries of the four drugs in the low, medium and high spiked concentration levels (20–200, 50–500 and 100–1000 μg L^{-1}) were between 98–115% with relative standard deviation (RSD) values were less than 12% ($n=6$). Limits of detection (LOD) of salicylic acid, naproxen, diclofenac, and ibuprofen in water were 0.5, 0.5, 1.0, and 1.25 μg L^{-1}, respectively. The determination method has been applied for the real samples (purified water, tap water, juice, soda and energy drinks), and the results show that salicylic acid was detected in tap water and soda, the concentrations were 2.85 μg L^{-1} and 61.22 μg L^{-1} separately, the RSD values were less than 9% ($n=6$). Salicylic acid and diclofenac were detected in energy drink, the concentrations were 44.62 μg L^{-1} and 8.31 μg L^{-1}, the RSD values were less than 11% ($n=6$).

1. Introduction

Pharmaceuticals and personal care products (PPCPs) concept was first proposed by Daughton [1] in 1999 and subsequently received wide attention as an emerging class of potentially harmful environmental pollutants. The PPCPs include various classes of compounds used in veterinary, agriculture, human medicines (anti-inflammatory painkillers, antibiotics, lipid-lowering drugs, hormones, tranquilizers, etc.) and personal care products (perfume, cosmetics, hair dye, shampoo, etc.) [1,2]. Non-steroidal anti-inflammatory drugs (NSAIDs) are a group of pharmaceutical compounds which have analgesic, antipyretic and platelet-inhibitory actions. Since they are used by human and animals in large quantities and emitted into water environment, it seems that they are persistent in the aquatic system. For instance, NSAIDs are usually detected in the influent flow of wastewater treatment plants (WWTPs). It is reported that NSAIDs are only partially removed by WWTPs [3,4] when the NSAIDs environmental inputs tend to increase considerably [5]. The combination of widespread use and incomplete WWTP elimination results an extensive occurrence of NSAIDs in surface waters. Indeed, NSAIDs have been detected in water systems all over the world [6–12].

In order to assess the quality of surface water and safety of drinks, it is essential to develop sensitive, reliable and rapid methods to detect NSAIDs in surface water and drinks samples. Methods for analyzing NSAIDs in surface water are based on a very efficient enrichment and separation method coupled with a sensitive detection technique (GC–MS or LC–MS) because of trace level.

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* Correspondence to: Analysis and Testing Center, College of Science, Beijing University of Chemical Technology, No. 15, BeiSanhuan East Road, Box 89, 100029 ChaoYang District, Beijing, China. Tel./fax: +86 10 64433909.
E-mail addresses: 2011201047@grad.buct.edu.cn (H. Zhang), duzx@mail.buct.edu.cn, duzhenxia2009@gmail.com (Z. Du).

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To obtain accurate, reliable and sensitive results, a sample preparation is required prior to detection. The traditional techniques (LLE [13]) are characterized by long analytical time, manual manipulation of the extracts, large consumption of sample and reagents and generates of large amounts of waste. In recent years, various sample preparation methods, based on solvent microextraction approaches, such as liquid-phase microextraction (LPME) [14], have been developed. LPME can be divided into single-drop microextraction (SDME) [15–18], dispersive liquid–liquid microextraction (DLLME) [19–21] and hollow-fiber liquid phase microextraction (HF-LPME) [22–24]. HF-LPME is used for the microextraction of organic compounds from liquid samples. HF-LPME, a relatively new miniaturized technique, has gained considerable interest in the analytical area [25–29]. High enrichments, simplicity, rapidity and low consumption of organic toxic solvents (low microliter range) are its prominent advantages over conventional extraction techniques. Some HF-LPME procedures have been proposed for determination of NSAIDs residues in water [30–34]. However, there has almost no research about NSAIDs in drinking water and beverages.

In the present study, an HF-LPME combined with UPLC-MS/MS method was developed to determine four representative NSAIDs (salicylic acid, naproxen, diclofenac and ibuprofen) in purified water, tap water, juice, soda and energy drinks samples. In order to obtain high recovery and enrichment of the analytes, an HF-LPME sample preparation method was developed and its influencing parameters such as organic solvents (acceptor phase), pH of donor phase, extraction time, stirring speed, extraction temperature and ionic strength were optimized. To evaluate the practical applicability of the determination method, parameters such as specificity, limits of detection, linearity, enrichment factor, matrix effect and recovery were investigated under optimal extraction conditions. The method has also been satisfactorily applied to their determination in real water and drinks samples.

2. Experimental

2.1. Chemicals and reagents

Purified water, juice, soda and energy drink were purchased from supermarket in Beijing, China. Salicylic acid and diclofenac sodium were purchased from The China drugs and Biological Products Inspection Institute, Naproxen was purchased from The Dr. Ehrenstorfer (German), Ibuprofen was purchased from Xinhua Pharmaceutical Co., Ltd. (Shandong, China). Tributyl phosphate, n-butyl alcohol and sodium chloride were purchased from Beijing Chemical Plant. 1-Octanol was from West Long Chemical Co., Ltd. Acetone was from Beijing modern Oriental Fine Chemicals Co., Ltd. Formic acid was from Tedia Company Inc. (Ohio, USA). HPLC grade acetonitrile and methanol were obtained from Fisher Scientific (Fair Lawn, USA). HPLC-grade water was prepared by a Milli-Q-Plus ultra-pure water system from Millipore (Milford, MA, USA). The porous PVDF hollow fiber (PVDF, 1.15 mm external diameter, 0.80 mm inner diameter, 0.16 μm pore size, and 0.82 membrane porosity) was purchased from Institute of Biological and Chemical Engineering in Tianjin Polytechnic University. Mechanical crimping (1.30 mm inner diameter) was from Yueqing Baoxing Electrical Factory (Zhejiang, China).

2.2. Instrumentation

Table 1 Parameters of MS/MS detection

Compound	Confirmation ion	CV/CE	Quantification ion	CV/CE
Salicylic acid	137.1 > 65.1	17/28	137.1 > 93.0	17/15
Naproxen	229.3 > 170.1	22/15	229.3 > 185.2	22/10
Diclofenac	294.2 > 214.2	22/23	294.2 > 250.1	22/12
Ibuprofen	–	–	205.3 > 161.2	22/7

CV = cone voltage (V); CE = collision energy (eV).

Waters Acquity^{TM} ultra-high performance liquid chromatography (autosampler, binary solvent management system) and Waters Micromass Quattro Premier XE triple quadruple spectrometer (Waters, USA) equipped with an electrospray ion source were used in this study and operated by using Mass Lynx 4.1 software. The sample was separated by a Waters Acquity^{TM} CSH C_{18} column (100 mm × 2.1 mm, 1.7 μm) with gradient elution using methanol (A) and water (B) at the flow rate of 0.25 mL min^{-1}. An initial 70% component A was used in isocratic mode for 1.0 min and then a linear elution gradient was programmed from 70% to 75% A for 0.5 min. Then a linear elution gradient was programmed from 75% to 80% A for another 0.5 min. Finally, it was held for 3.0 min which allowed re-equilibration of the column to the initial conditions. The mass spectrometry detection method was performed in the multiple reaction monitoring (MRM) mode, and the electrospray ionization was under positive mode (ESI$^+$). The MRM detection parameters are shown in Table 1. The fragmentation ion with the highest response was chosen as the quantitative ion, and fragmentation ion with the second high response was selected as the confirmation ion. The nebulizer gas was set at 600 L h^{-1} at a temperature of 350 °C, the cone gas was set at 50 L h^{-1}, The source temperature was 100 °C. The capillary voltage was 3.5 kV.

2.3. Preparation and extraction procedure

Hollow fibers were cut into 3 cm pieces, washed with acetone in an ultrasonic bath and dried. The fiber was soaked with 1-octanol to impregnate the pores for 15 s, the lumen of the prepared fiber piece was filled with 15 μL 1-octanol using a HPLC syringe. Both open ends of the fiber were closed by a pair of mechanical crimping. Then the hollow fiber was immersed in the 5 mL sample solution. The sample was stirred for 20 min by means of a magnetic stirrer at 300 rpm at room temperature. Extraction system is shown in Fig. 1. After extraction, the fiber was taken out, and one of its ends was cut. Then the acceptor phase was extracted by a HPLC syringe and analyzed with the UPLC/MS system.

2.4. Preparation of real water samples

Soda sample was put in an ultrasonic bath for 20 min to remove air bubbles, juice sample and energy drink were filtered with activated carbon to remove the pigment. Then tap water, purified water, treated soda, juice and energy drink samples were filtered with 0.22 μm PES filters (MEMBRANA, Germany) to remove suspended particles, and the filtered samples were finally adjusted to pH 1.5 with formic acid.

3. Results and discussion

3.1. Optimization of experimental conditions for HF-LPME

In order to achieve fast and efficient extraction of the four NSAIDs from water and drinks, a two phase HF-LPME method was applied. The effects of pH, organic extraction solvents, extraction time, stirring speed, extraction temperature and ionic strength were investigated by the “one variable at a time” method.

3.1.1. *Choice of organic extraction solvents*

The choice of organic extraction solvents plays a key role in improving the extraction efficiency. Firstly, organic extraction solvent should have a good affinity with the fiber, and should be insoluble in water. Also, the ideal organic extraction solvent should have a low volatility and an appropriate viscosity to prevent diffusion and volatile loss. Based on the thought, three organic extraction solvents (tributyl phosphate, 1-butanol, and 1-octanol) were chosen to evaluate their influence on the extraction efficiency.

The results (Fig. 2) show that salicylic acid, ibuprofen, and diclofenac obtained higher extraction efficiency in 1-octanol, while naproxen got better extraction in tributyl phosphate. 1-octanol possesses an active hydrogen atom (hydroxyl group), and is capable of specific interaction with oxygen bearing species (the four NSAIDs have the common structure of carboxyl group), resulting in the formation of mutual hydrogen bonds, which make it easier to extract the four NSAIDs. In addition, 1-octanol has other better characteristics, such as larger viscosity and less volatility, those attributed to lower solvent loss. Therefore, 1-octanol was chosen as extraction solvent for further experiments.

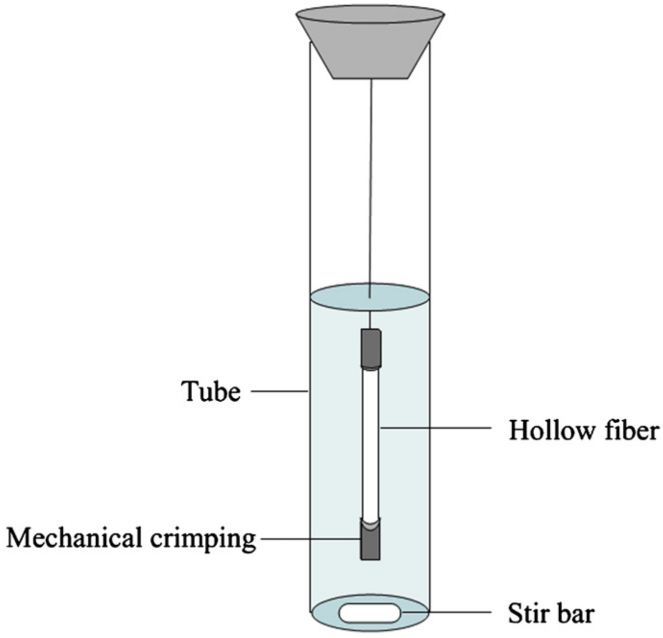

Fig. 1. The equipment of extraction system.

3.1.2. *PH of donor phase*

For two-phase HF-LPME, the pH value of sample solution (donor phase) is important for extraction efficiency. It can change the partition coefficient of analytes between the sample solution and extraction solvent. Five pH values (ranges from 1.5 to 3.5) were investigated to study their influence on the extraction efficiency. Fig. 3 shows that the extraction efficiency is the highest when the pH value is 1.5. Thus, pH 1.5 was selected as optimum for donor phase. The results can be explained by the principle that, the lower the pH value, the more inhibited the ionization of the four drugs. The pKa values of salicylic acid, naproxen, diclofenac and ibuprofen are 2.98, 4.15, 4.50 and 5.20, respectively. Thus, they are in a less ionized condition at the lower pH than at a higher pH. At low pH value, all drugs will be in the neutral form, which facilitates the extraction from donor phase. Therefore, low pH value benefits to extract four drugs in sample to the organic solvent immersed in the hollow fiber membrane.

3.1.3. *Stirring rate*

As for HF-LPME, stirring can continuously update the sample solution outside the fiber membrane and increase the contacting frequencies between analytes and extraction solvent therefore it can shorten extraction equilibrium time and enhance the extraction efficiency. However, if stirring speed is too fast, air bubbles is prone to form outside the fiber membrane. In addition, fast stirring speed can also speed up the extraction solvent evaporation and affect the extraction accuracy and reproducibility. From the standpoint, stirring rates in the range of 300–l500 r min^{-1} were investigated. Results are shown in Fig. 4. When the stirring rate is

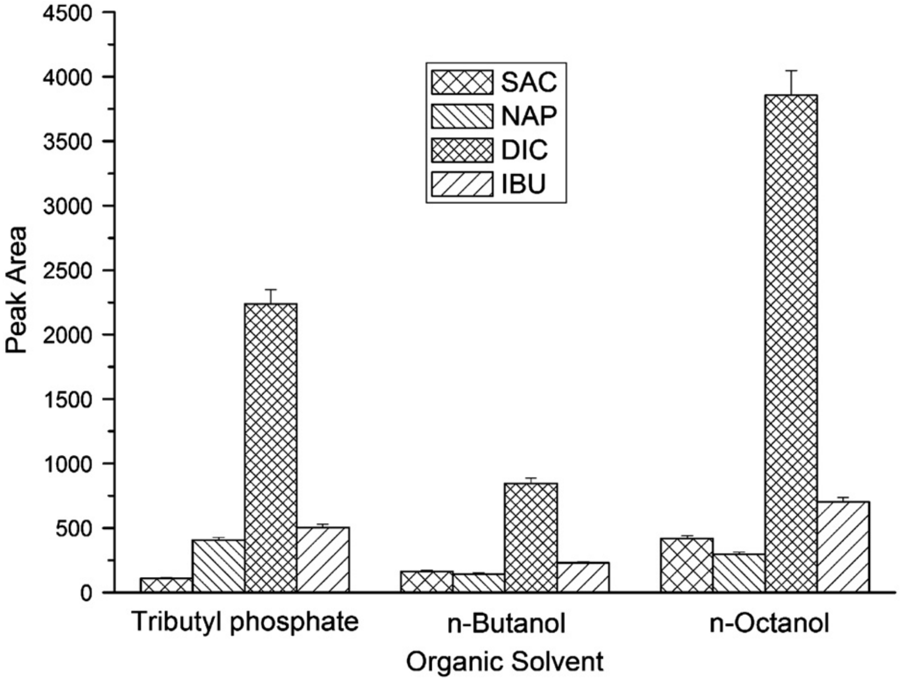

Fig. 2. Effect of organic solvents, 300 rpm, 20 °C, 30 min, pH=2.5, no NaCl added.

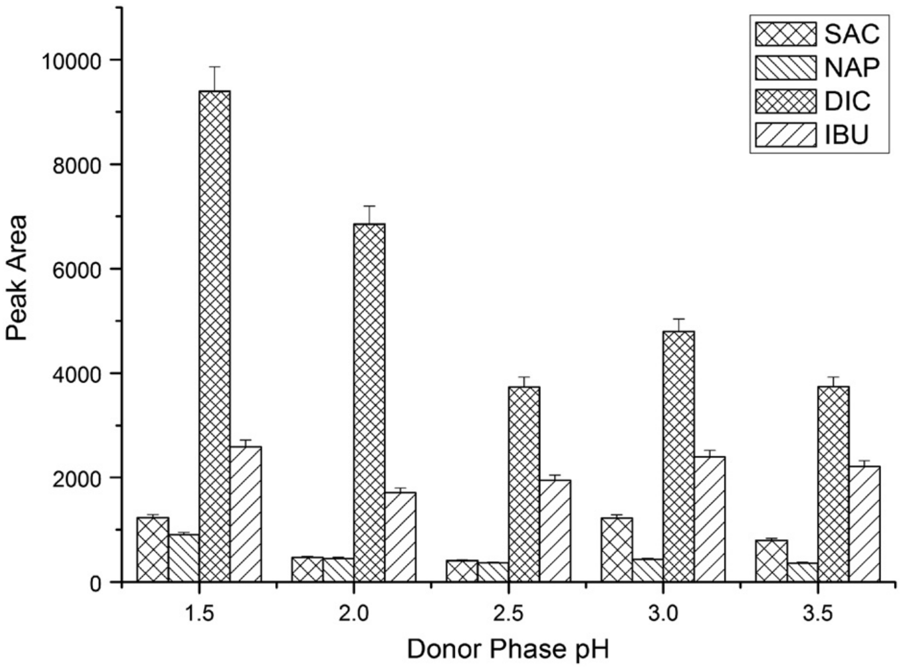

Fig. 3. Effect of pH value of donor phase, 300 rpm, 20 °C, 20 min, 1-octanol as acceptor phase, no NaCl added.

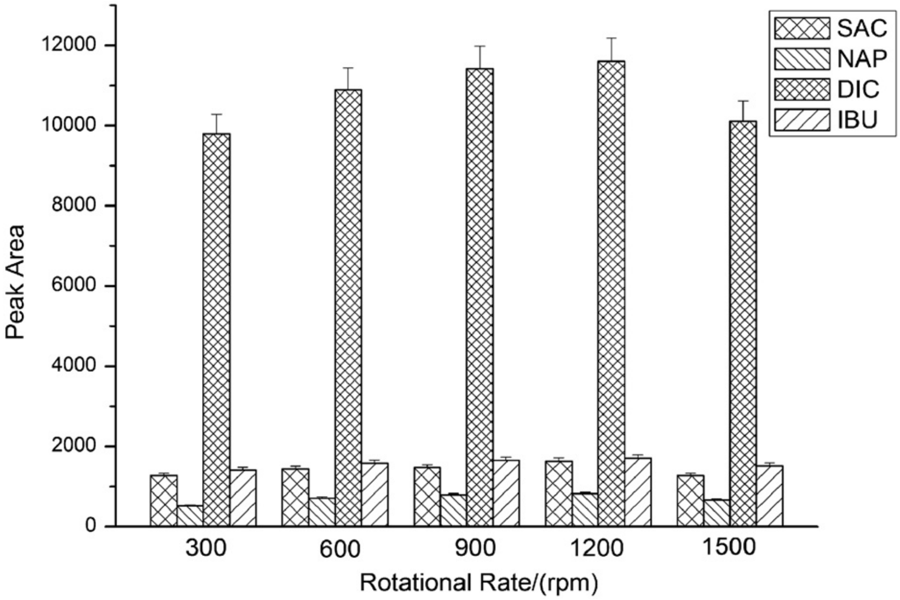

Fig. 4. Effect of stirring rate, 20 °C, 30 min, pH = 1.5, 1-octanol as acceptor phase, no NaCl added.

1200 r min^{-1}, extraction efficiencies of analytes are the highest. Therefore, the optimum magnetic stirring rate is 1200 r min^{-1}.

3.1.4. Extraction time

The HF-LPME is based on analytes' partition equilibrium between the sample and organic solvent (receptor). The extraction time is an important factor which affects the extraction efficiency. Extraction time was optimized to obtain better extraction efficiencies. Theoretically, when extraction time is longer, analytes' partition equilibrium between the sample and organic solvent is more complete. From results in Fig. 5, the extraction efficiencies increased rapidly by increasing the extraction time up to 30 min and then remained relatively constant. It is possible that after 30 min it could reach a maximum value of the amount extracted, and the variation in the values obtained after 30 min could be due to the actual reproducibility of the methodology. Therefore, 30 min was the best for extraction time.

3.1.5. Extraction temperature

The impacts of temperature on the extraction efficiency are in two ways, on the one hand, with the increase of temperature, the mass transfer rate of analytes in solution will increase; on the other hand, the acceptor loss will increase. Five points between 20–40 °C were chosen to test. Fig. 6 shows that higher the temperature, lower the extraction efficiency. This may be because in this experimental system, the acceptor loss has greater impact than the mass transfer rate of analytes in solution. Consequently, 20 °C was the optimum extraction temperature.

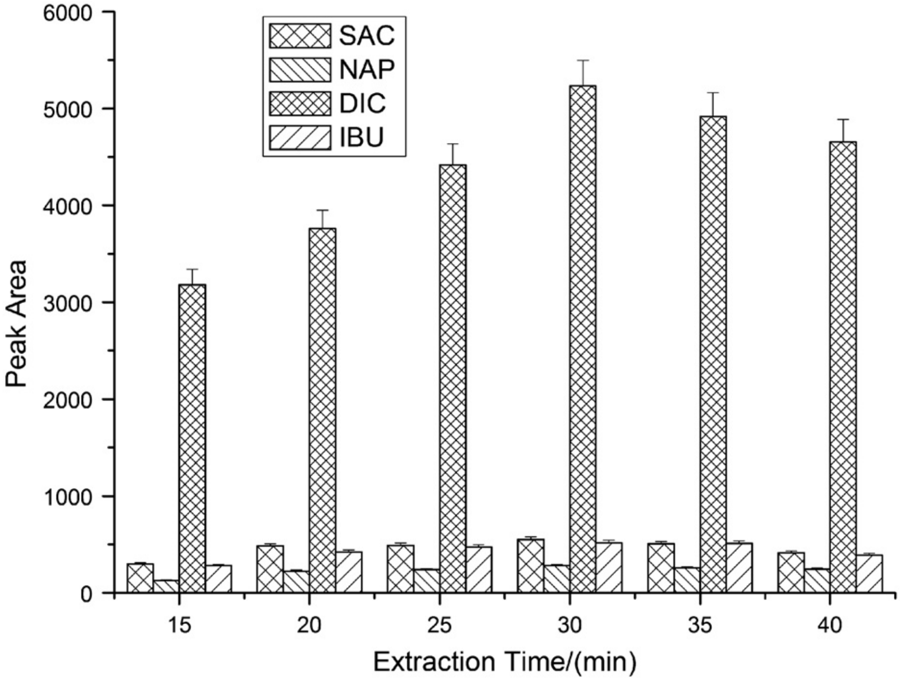

Fig. 5. Effect of extraction time, 300 rpm, 20 °C, pH = 1.5, 1-octanol as acceptor phase, no NaCl added.

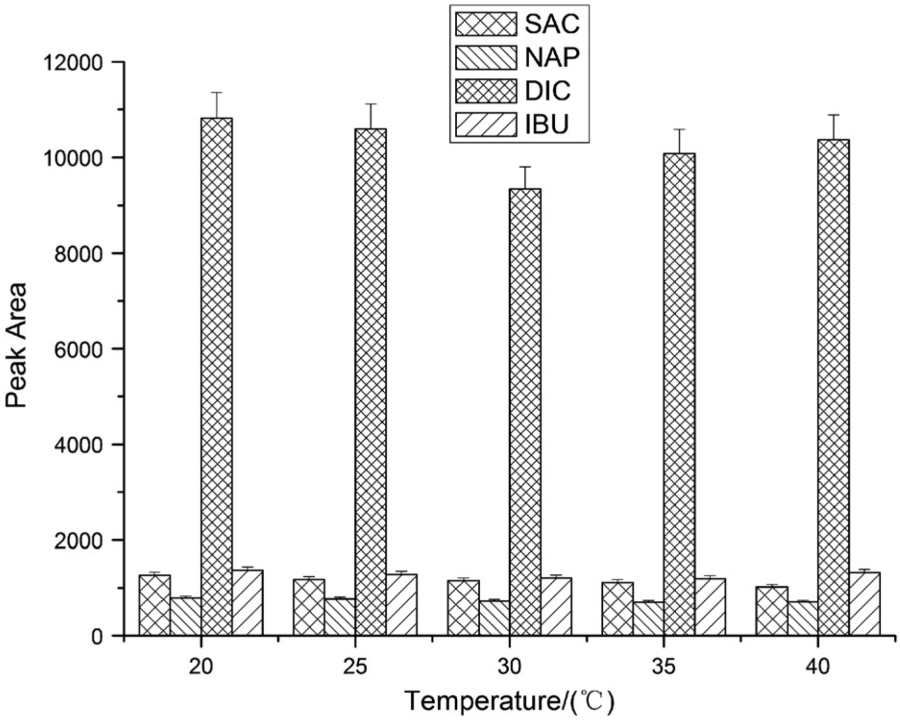

Fig. 6. Effect of extraction temperature, 1200 rpm, 30 min, pH = 1.5, 1-octanol as acceptor phase, no NaCl added.

3.1.6. *Influence of ionic strength*

The effect of salt addition on the extraction efficiency of drugs by the HF-LPME method was examined by adding of NaCl to 5 mL aqueous samples at 0, 0.05, 0.10, 0.15 and 0.20 (w/v).

On the one hand, the addition of salt to the sample will lead to a higher ionic strength in the sample and decrease the solubility of four drugs in the aqueous solution. On the other hand, electrostatic interaction will resist organic solvent extraction. Therefore, the effect of salt on extraction is indefinite. Fig. 7 shows that the addition of NaCl decreased the extraction efficiencies of the four drugs. Hence, NaCl was not added in further study.

3.2. *Method validation*

3.2.1. *Selectivity*

The specificity of the method was evaluated with respect to different water samples by extracting and analyzing blank samples. Fig. 8 is the chromatogram of blank and spiked

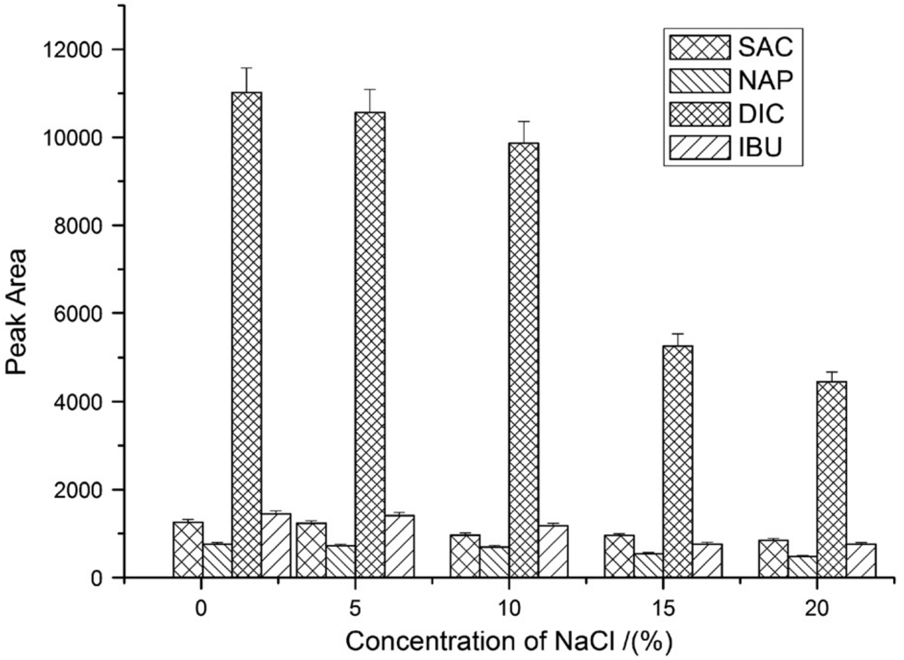

Fig. 7. Effect of ionic strength, 20 °C, 1200 rpm, 30 min, pH = 1.5, 1-octanol as acceptor phase.

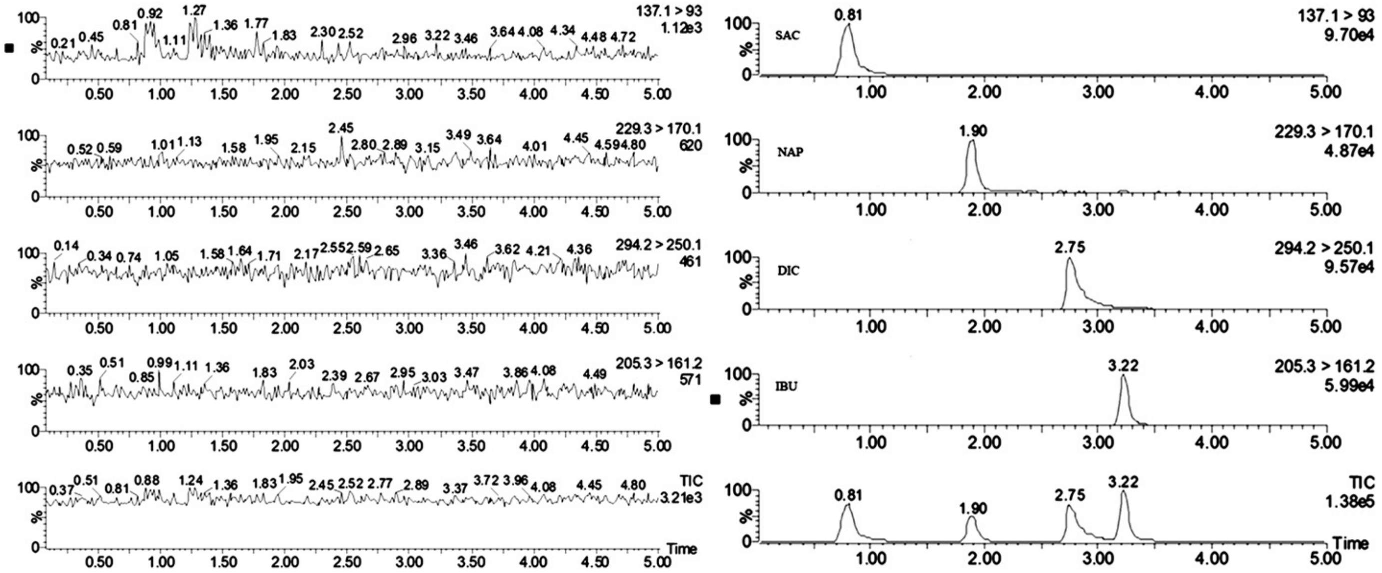

Fig. 8. The chromatograms of four drugs after HF-LPME in blank and spiked sample.

sample, it shows that the blank sample has no interference in drugs detection.

3.2.2. LOD, LOQ, linearity and enrichment factor (EF)

A number of performance parameters as the figures of merit such as linearity, limit of detection (LOD), limit of quantification (LOQ), enrichment factor (EF), repeatability, reproducibility and percent of extraction were evaluated for extraction of drugs from the aqueous solutions under the optimum conditions and results are shown in Table 2. LODs and LOQs are calculated as the minimum concentration of an analyte giving peak with signal to noise ratio of at least 3:1 and 10:1, respectively. LODs range from 0.50 to 1.25 μg L^{-1} and LOQs from 2.00 to 5.00 μg L^{-1}. Linearity correlation of the method was studied with external calibration. Ultrapure water (drugs-free) was spiked with drugs to provide standards with different concentrations. A calibration curve was constructed by using analysis of standard mixtures of the analytes at five concentration levels. Correlation coefficients for all compounds were greater than 0.998 (Table 2).

EF was calculated based on the following equation:

$$EF = C_{ap}/C_{dp} \qquad (1)$$

where C_{ap} and C_{dp} are concentrations of the analyte in the acceptor phase and in the donor phase, respectively.

3.2.3. Matrix affect, method precision and recovery experiment

LC-ESI-MS has become the premier technique for a large number of different analytical tasks, ranging from pesticides determination in fruit and vegetables to drug residues in sewage

Table 2
Method linearity, MLOD, MLOQ and enrichment factor (EF).

Compound	Linear range ($\mu g\ L^{-1}$)	Calibration curve	R	MLOD ($\mu g\ L^{-1}$)	MLOQ ($\mu g\ L^{-1}$)	EF
Salicylic acid	1–500	$Y=166.17X+19.26$	0.999	0.5	2.0	195
Naproxen	5–2500	$Y=81.22X+40.00$	0.998	0.5	2.5	337
Diclofenac	10–5000	$Y=266.50X+3.55$	0.999	1.0	3.0	350
Ibuprofen	5–2500	$Y=99.76X+40.83$	0.999	1.25	5.0	346

Table 3
Results of matrix effect.

Compound	k_1	k_2	Matrix effect (%)
Salicylic acid	27.19	24.58	90.04
Naproxen	5.66	6.24	110.24
Diclofenac	3.21	3.48	108.41
Ibuprofen	1.42	1.35	95.07

Table 4
Relative recoveries and precision of the compounds.

Compound	Spiked level ($\mu g\ L^{-1}$)	Recovery[a] (%)
Salicylic acid	5	56.75 ± 5.25
	20	45.62 ± 0.80
	100	33.58 ± 12.48
	25	98.17 ± 1.45
Naproxen	100	103.70 ± 1.42
	500	98.93 ± 2.24
	50	101.86 ± 3.76
Diclofenac	200	101.65 ± 1.99
	1000	87.87 ± 3.45
	25	100.88 ± 4.20
Ibuprofen	100	115.23 ± 1.94
	500	100.03 ± 0.69

[a] Average recovery (%) ± standard deviation ($n=6$).

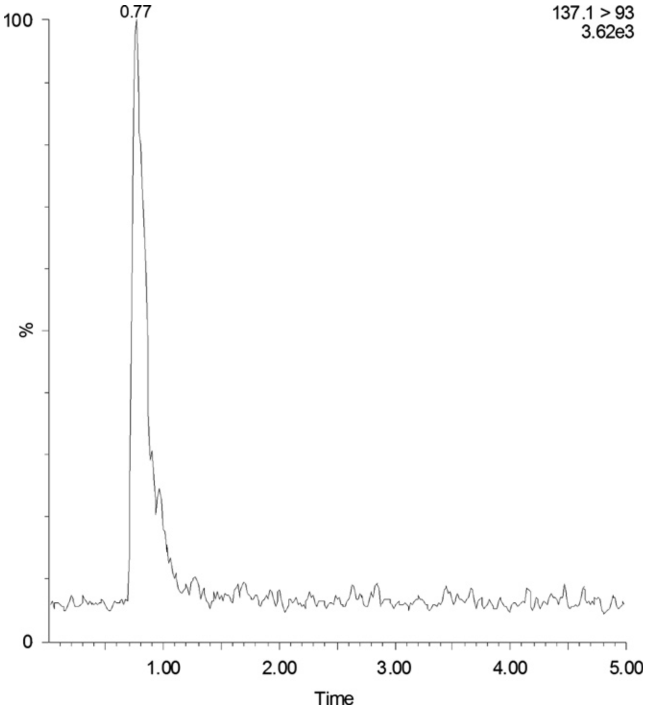

Fig. 9. The chromatogram of salicylic acid in tap water sample.

sludge. However, a major drawback of LS-ESI-MS is the so-called matrix effect—ionization suppression or enhancement of the analyte of interest by other compounds present in the sample and co-eluting with the analyte. The matrix effect, if present, can cause large uncertainties in quantification using the LC-ESI-MS technique. In this work, the ME was valued by matrix-matched standard method. Tropicana was selected for evaluation of matrix effect. Tropicana were spiked with four drugs to give different sample concentrations. A calibration curve was constructed using analysis of standard mixtures of the analytes at different concentrations.

ME was investigated by comparing calibration curve slope in Tropicana samples (k_2) to that of ultrapure water sample (k_1). ME was calculated by the following equation:

$$\mathrm{ME}=(k_2/k_1)100\% \tag{2}$$

The ME results are shown in Table 3, ranging between 90% and 110%, indicate that matrices do not have a significant influence on the signal suppression/enhancement. This illustrates that the HF-LPME devices provide clear extracts without matrix effect during UPLC-MS/MS determination.

Recoveries of the analytes were measured by spiking blank water samples with four drugs at three different concentrations, salicylic acid (5.0, 20.0, and 100.0 $\mu g\ L^{-1}$), naproxen and ibuprofen (25.0, 100.0, and 500.0 $\mu g\ L^{-1}$) and diclofenac (50.0, 200.0, and 1000.0 $\mu g\ L^{-1}$). Samples were extracted by the optimized sample preparation method. After UPLC-MS/MS analysis, recoveries results are shown in Table 4. The recoveries (R) for salicylic acid, naproxen, diclofenac and ibuprofen are 34–57%, 98–104%, 88–102% and 100–115%, respectively. The relative standard deviations (RSD) are lower than 12% based on the peak areas for six replicate runs. Among these four drugs, the recovery of salicylic acid is lower than 70%. Two reasons could explain this result, one is that salicylic acid has good water solubility, so it has lower solubility in organic solvent than other drugs. The other reason is that the pKa of salicylic acid is 2.98, which is the lowest among the analytes. Thus, when the pH value of donor phase is 1.5, a part of salicylic acid is likely to be in the ionic form. So, it is harder to extract it from the donor phase to the acceptor phase than other drugs. As a result, the recovery of salicylic acid is low.

R was calculated by the following equation:

$$\begin{aligned} R &= \left(n_{a,\ \mathrm{final}}/n_{s,\ \mathrm{initial}}\right)100\% \\ &= (V_\mathrm{a}/V_\mathrm{s})(C_{a,\ \mathrm{final}}/C_{s,\ \mathrm{initial}})100\% \end{aligned} \tag{3}$$

where $n_{\mathrm{s,initial}}$ and $n_{\mathrm{a,final}}$ are the number of moles of analyte present in the initial sample and finally collected in the acceptor solution, respectively. V_a is the volume of the acceptor solution, V_s is the volume of sample, $C_{\mathrm{a,final}}$ is the final concentration of analyte in the acceptor solution, and $C_{\mathrm{s,initial}}$ is the initial analyte concentration within the sample.

3.3. *Analysis of real samples*

The developed two-phase HF-LPME–UPLC-MS/MS method was applied to real water samples. Results are shown in Fig. 9–11. They show that salicylic acid with the detection levels of 2.85 $\mu g\ L^{-1}$ ($n=6$, RSD=8.52%) is detected in tap water and 61.22 $\mu g\ L^{-1}$ ($n=6$, RSD=5.23%) in soda; Salicylic acid and diclofenac with the detection levels of 44.62 $\mu g\ L^{-1}$ ($n=6$, RSD=10.68%) and 8.31 $\mu g\ L^{-1}$($n=6$, RSD=7.52%) are detected in energy drink respectively.

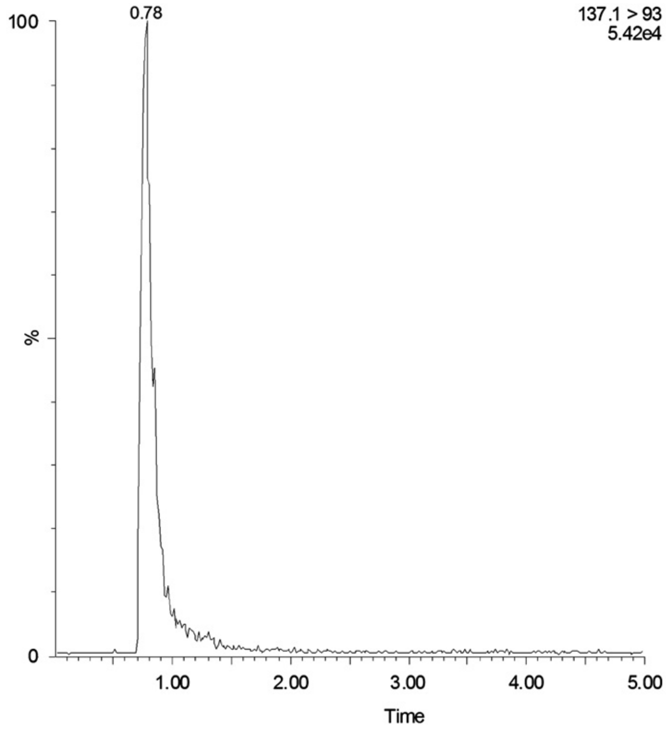

Fig. 10. The chromatogram of salicylic acid in soda sample.

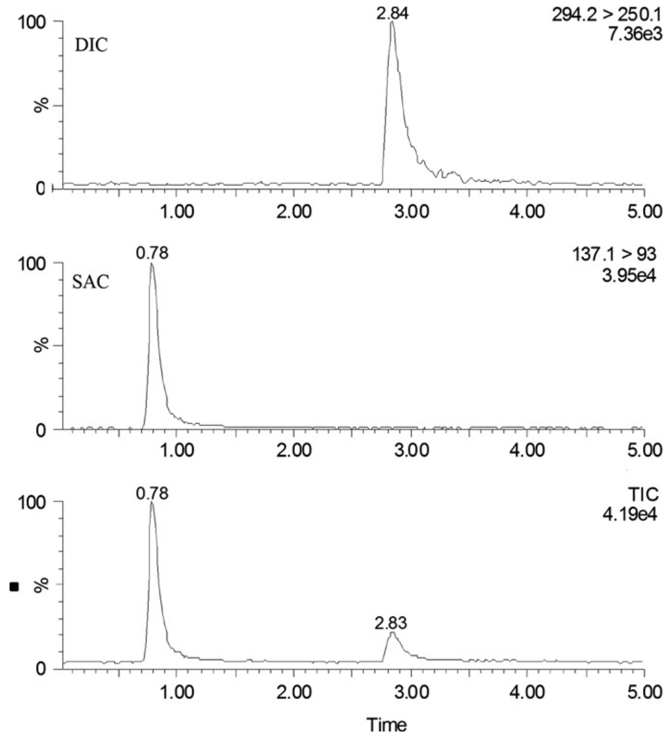

Fig. 11. The chromatogram of salicylic acid and diclofenac in energy drink.

4. Conclusions

A two-phase HF-LPME procedure combined with UPLC-MS/MS method was developed for the analysis of trace level of four non-steroidal drugs (salicylic acid, naproxen, diclofenac and ibuprofen) in water samples and drinks. Various influencing parameters including organic solvents, pH of donor phase, extraction time, stirring speed, extraction temperature and ionic strength of HF-LPME sample preparation method were optimized. The results demonstrated that the proposed HF-LPME method has many advantages such as environmental friendly, easy to operate and has high enrichment efficiency, reliability and repeatability. The HF-LPME sample preparation method coupled with rapid, sensitive, accurate and confirmative UPLC-MS/MS detection method can be applied for determination of NSAIDs at trace levels in different kinds of real water samples.

Novelty Statement

1. A two-phase HF-LPME procedure combined with UPLC-MS/MS method was developed for the analysis of trace level of four non-steroidal anti-flammatory drugs—salicylic acid, naproxen, diclofenac andibuprofen.
2. The determination method has been applied in the real water samples (purified water, tap water, juice, soda and energy drink).

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