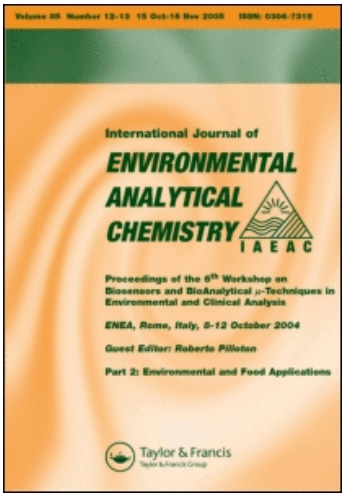

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A new method for the determination of four sulfonylurea herbicides (metsulfuron-methyl, chlorsulfuron, bensulfuron-methyl and chlorimuron-ethyl) in water samples was developed by dispersive liquid–liquid microextraction coupled with high performance liquid chromatography-diode array detector. Parameters that affect the extraction efficiency, such as the kind and volume of the extraction and disperser solvent, extraction time and salt addition, were investigated and optimised. Under the optimum conditions, the enrichment factors were in the range between 102 and 216. The linearity of the method was obtained in the range of 1.0–100 ng mL^{-1} with the correlation coefficients (r) ranging from 0.9982 to 0.9995. The method detection limits were 0.2–0.3 ng mL^{-1}. The proposed method has been successfully applied to the analysis of target sulfonylurea herbicides in river, stream and well water samples with satisfactory results.

Keywords: sulfonylurea herbicides; high performance liquid chromatography; diode array detection; dispersive liquid–liquid microextraction; water samples

1. Introduction

Sulfonylurea herbicides are widely used as selective pre- and post-emergence herbicides for the control of most broad-leaved weeds and annual grasses in many agricultural crops due to their low application rates, low toxicity to mammals and unprecedented herbicidal activity. However, due to their moderate to high mobility, fairly high water solubility and widespread use, sulfonylurea herbicides can contaminate the aquatic environment through agricultural run-off and leaching [1] and some of them have been detected in natural waters [2].

For many years, gas chromatography (GC) has been the method of choice for the determination of a wide range of pesticide residues because of its inherent high separation power and the availability of sensitive and selective detectors. However, because sulfonylurea herbicides are polar compounds with low volatility and thermal instability, they cannot be analysed directly by GC without prior dramatisation. Then, high performance liquid chromatography (HPLC) has become the main technique for their

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analysis, and conventional UV or diode array detection has been extensively used in HPLC for their determinations [3–6].

Sample preparation before instrumental analysis is usually necessary to reduce or even eliminate the interferences originally present in the sample and simultaneously to concentrate the analytes to facilitate their determinations at low levels. It is often the bottleneck for rapidly obtaining the desired results, especially for the determination of trace analytes in samples with complex matrix. For the determination of sulfonylurea herbicides, several sample preparation methods have been developed, including liquid–liquid extraction (LLE) [3,7–8], solid-phase extraction (SPE) [6,9–11], supercritical fluid extraction [12], microwave-assisted extraction [13] and molecularly imprinted SPE [14]. LLE and SPE are the typical sample preparation approaches and have been most widely used for sample preparations. However, LLE and SPE often require large volumes of toxic organic solvents, which are unfriendly to the environment, and involve laborious and time-consuming experimental procedures. In contrast, dispersive liquid–liquid microextraction (DLLME) is a novel microextraction technique recently developed by Assadi and co-workers [15], and it has been applied for the analysis of various organic pollutants in environmental samples [16–25]. DLLME is a miniaturised LLE that uses microlitre volumes of the extraction solvent. In this method, an appropriate mixture of water-immiscible extraction solvent and water-miscible dispersive solvent is rapidly injected into an aqueous sample solution with a syringe, resulting in the formation of a cloudy solution containing fine droplets of extraction solvent dispersed entirely in the aqueous phase. After centrifugation of the sample solution, the enriched analytes in the sedimented phase are determined by GC or HPLC. The aim of this work was to explore the possibility of DLLME for the fast and sensitive determination of sulfonylurea herbicides in water samples. The effect of several experimental parameters on the efficiency of the DLLME process has been thoroughly investigated, and the performance of the presented method has been compared with that of other reported sample preparation procedures. The method shows obvious excellence of rapidness, simplicity, low cost, high recovery and high enrichment factor. To the best of our knowledge, its application to the analysis of sulfonylurea herbicides has not been reported.

2. Experimental

2.1 *Reagents and materials*

Metsulfuron-methyl (MSM), chlorsulfuron (CS), bensulfuron-methyl (BSM) and chlorimuron-ethyl (CME) were purchased from Agricultural Environmental Protection Institution in Tianjin (Tianjin, China). Chloroform ($CHCl_3$), dichloroethane ($C_2H_4Cl_2$), dichloromethane (CH_2Cl_2), tetrachloride ethylene (C_2Cl_4), carbon tetrachloride (CCl_4) and chlorobenzene (C_6H_5Cl) were purchased from Beijing Chemical Reagents Company (Beijing, China). Acetone, acetonitrile, tetrahydrofuran (THF), sodium chloride (NaCl), hydrochloric acid (HCl), ethanol and methanol were from Sinopharm Chemical Reagent Co. Ltd. (Beijing, China). Double-distilled water was used for the preparation of aqueous solutions.

River water was collected from Yimu River, stream water from Baoding in summer, and well water from Wumazhang (Baoding, China), respectively. All the solvents and water samples were filtered through a 0.45 μm membrane to remove suspended particles prior to the analysis by the proposed method.

A mixture stock solution containing MSM, CS, BSM, and CME at 10.0 μg mL^{-1} was prepared in methanol. A series of standard solutions were prepared by mixing an appropriate amount of the stock solution with double-distilled water in a 10 mL volumetric flask. All the standard solutions were stored at 4°C in the dark.

2.2 *Instruments*

The HPLC system, assembled from modular components (Waters, Milford, MA, USA), consisted of an in-line degasser, a 600E pump, and a diode array detection (DAD) detector. A Millennium^{32} workstation (Waters) was utilised to control the system and for the acquisition and analysis of the data. The injection loop volume is 20.0 μL. A Centurysil C_{18} column (4.6 i.d. × 250 mm, 5.0 μm) from Dalian Johnsson Separation Science Technology Corporation (Dalian, China) was used for separations. The mobile phase was a mixture of acetonitrile-water (50 : 50 v/v), the pH of which was adjusted to 3.0 with 1 mol L^{-1} HCl, at a flow rate of 1.0 mL min^{-1}. The DAD monitoring wavelengths were chosen at 225 nm for MSM and CS, and 240 nm for BSM and CME, respectively.

2.3 *Dispersive liquid–liquid microextraction procedure*

For the DLLME, an aliquot of 5.00 mL of water sample was placed in a 10 mL glass test tube with conical bottom. Then 1.0 mL of acetone (as dispersive solvent) and 60.0 μL of chlorobenzene (as extraction solvent) were injected rapidly into the sample solution with a 1.0 mL syringe. After vortexing for 5 s, a cloudy solution that consisted of very fine droplets of chlorobenzene dispersed into the aqueous sample was formed, and the analytes were extracted into the fine droplets. After centrifugation at 3500 rpm for 5 min, the chlorobenzene phase was sedimented at the bottom of the centrifuge tube. The sedimented phase was completely removed into a 0.50 mL centrifugal tube, and evaporated to dryness under a stream of nitrogen at room temperature. The residue was reconstituted in 15.0 μL acetonitrile, and 10.0 μL was injected into the HPLC system for analysis.

2.4 *Calculation of enrichment factor and extraction recovery*

In order to evaluate the effect of different experimental parameters on the performance of DLLME, the terms of the enrichment factor (*EF*) and extraction recovery (*R*%) were used according to the Equations (1) and (2) as follows [15–17]:

$$EF = \frac{C_{\mathrm{rec}}}{C_0} \tag{1}$$

where *EF*, C_{rec} and C_0 are the enrichment factor, the analyte concentration in the final reconstituted solution in the extraction and the initial analyte concentration in the aqueous samples, respectively.

$$R\% = \frac{V_{\mathrm{rec}} C_{\mathrm{rec}}}{C_0 V_{\mathrm{aq}}} \times 100 \tag{2}$$

where *R*%, V_{rec} and V_{aq} are the extraction recovery, the volume of the final reconstituted solution and the volume of the aqueous sample, respectively.

3. Results and discussion

3.1 *Optimisation of the DLLME procedure*

In order to optimise the DLLME procedure, 5.0 mL double-distilled water spiked with 100 ng mL^{-1} each of the four sulfonylurea herbicides was used to study the extraction performance of the DLLME under different experimental conditions. All experiments were performed in triplicate, and the means of the results were used for optimisation.

3.1.1 *Selection of extraction and dispersive solvent*

The selection of appropriate extraction and dispersive solvents is a critical factor in the DLLME process. In the selection of extraction solvent, some requirements must be considered: it should have a higher density than water, a low solubility in water, high extraction capability for the target analytes, and should form a stable two-phase system in the presence of a dispersive solvent when injected into an aqueous solution. Based on these criteria, CCl_4, $CHCl_3$, $C_2H_4Cl_2$, CH_2Cl_2, C_2Cl_4 and C_6H_5Cl were selected for the study. On the other hand, the disperser solvent should be miscible with both water and the extraction solvent, and could form a cloudy state when injected with the organic extractant into water. So the selection of a dispersive solvent is limited to solvents such as acetone, methanol, THF, ethanol and acetonitrile. Due to a limited number of organic extractants, all combinations of using CCl_4, $CHCl_3$, $C_2H_4Cl_2$, CH_2Cl_2, C_2Cl_4 or C_6H_5Cl (50 μL) as extractant with acetone, acetonitrile, methanol, THF or ethanol (1.0 mL) as dispersive solvent were investigated. As a result, in the case of $C_2H_4Cl_2$ and CH_2Cl_2 as extraction solvents, a two-phase system was not formed with any dispersive solvents studied. For $CHCl_3$, a two-phase system was not observed either with methanol or ethanol as dispersive solvent. Based on the above results, C_6H_5Cl, CCl_4, $CHCl_3$ and C_2Cl_4 were chosen as potential extraction solvents for further study. Figure 1 illustrates the effect of the extraction solvents on the recoveries with the use of ethanol as disperser solvent. As can be

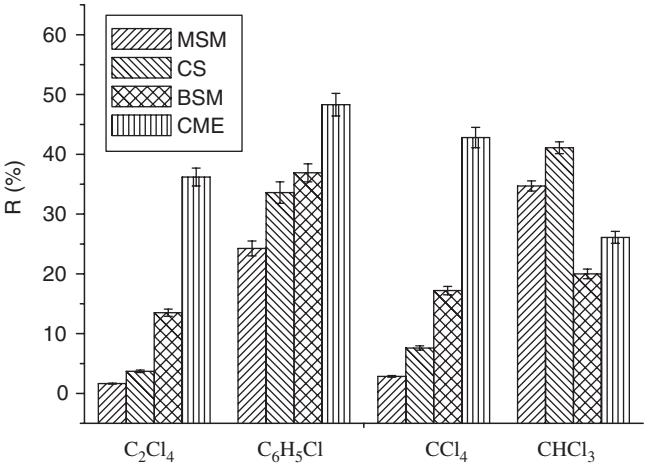

Figure 1. Effect of different extraction solvents on the extraction recovery of the sulfonylureas. Extraction conditions: sample volume, 5.0 mL; dispersive solvent, 1.0 mL ethanol; extraction solvent volume, 50 μL.

seen in Figure 1, C_6H_5Cl gives the highest extraction efficiency for the target analytes among the four solvents investigated. Therefore, C_6H_5Cl was selected as the extraction solvent.

With C_6H_5Cl as extraction solvent, the use of acetonitrile, acetone or ethanol as dispersive solvent could produce a two-phase system. The effect of different dispersive solvents (acetonitrile, acetone and ethanol) on the extraction recovery of the target analytes is shown in Figure 2. As can be seen from Figure 2, acetone gives the best extraction efficiency. Therefore, acetone was selected as the dispersive solvent.

3.1.2 *Effect of extraction solvent volume*

In order to examine the effect of the extraction solvent volume on the performance of the DLLME procedure, the volume of C_6H_5Cl was changed in the range of 20 to 100 μL in 20 μL intervals, with other experimental conditions being kept unchanged. Figure 3 shows the variation of extraction recovery versus the extraction solvent volume. It can be seen from Figure 3 that, as the volume of C_6H_5Cl was increased, the extraction recovery was first increased until 60 μL, and then remained almost constant between 60 and 100 μL for all the target analytes. From the obtained results, 60 μL of C_6H_5Cl was chosen as the optimal volume for the extraction solvent.

3.1.3 *Effect of disperser solvent volume*

For the optimisation of the disperser solvent volume, the experiments were performed by using different volume of acetone (0.4, 0.6, 0.8, 1.0, 1.2 and 1.4 mL) and fixed volume of C_6H_5Cl (60 μL). According to Figure 4, the extraction efficiency of the herbicides is increased first by increasing the volume of acetone before 1.0 mL, and then decreased by further increasing the volume of acetone. It seems that, at a low volume of acetone, a cloudy state is not formed well, thereby, the recovery is low. At higher volume of acetone,

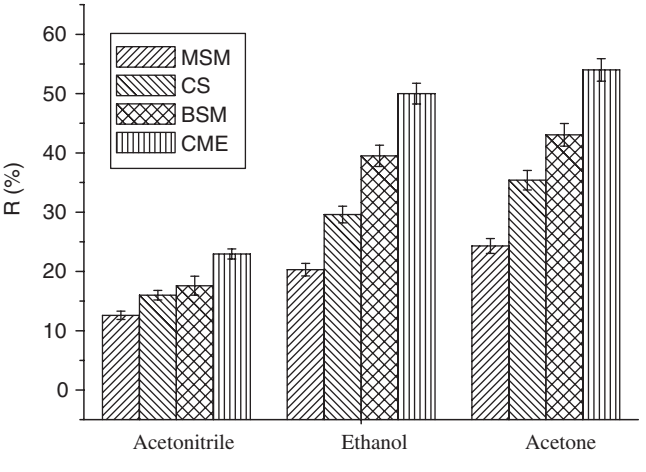

Figure 2. Effect of different dispersive solvents on the extraction recovery of the sulfonylureas. Extraction conditions: sample volume, 5.0 mL; dispersive solvent volume, 1.0 mL; extraction solvent, 50 μL C_6H_5Cl.

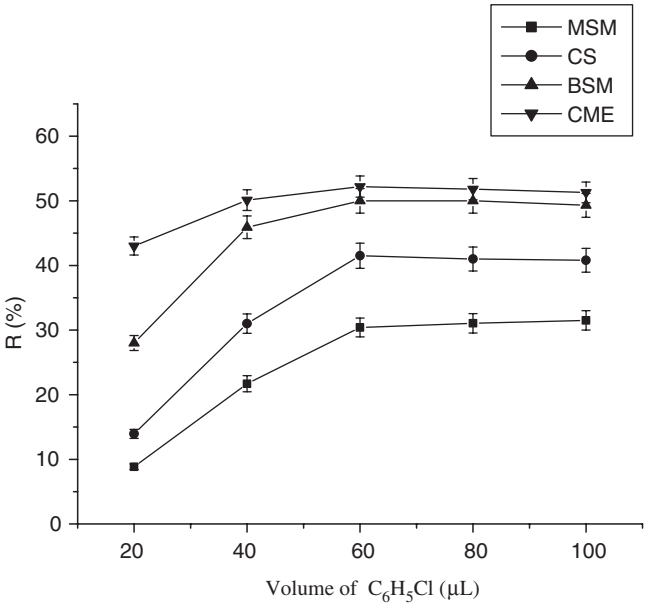

Figure 3. Effect of the volume of the extraction solvent (C_6H_5Cl) on the extraction recovery of the sulfonylureas. Extraction conditions: sample volume, 5.0 mL; dispersive solvent, 1.0 mL acetone; extraction solvent, C_6H_5Cl.

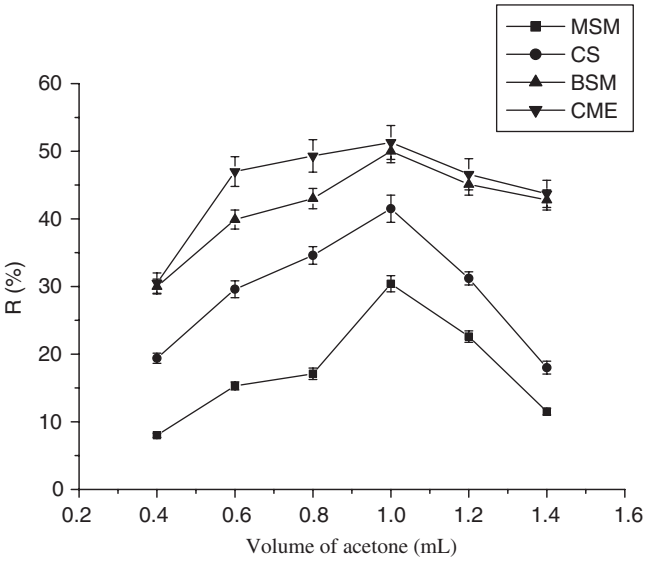

Figure 4. Effect of the volume of the dispersive solvent (acetone) on the extraction recovery of the sulfonylureas. Extraction conditions: sample volume, 5.0 mL; extraction solvent, 60 μL C_6H_5Cl.

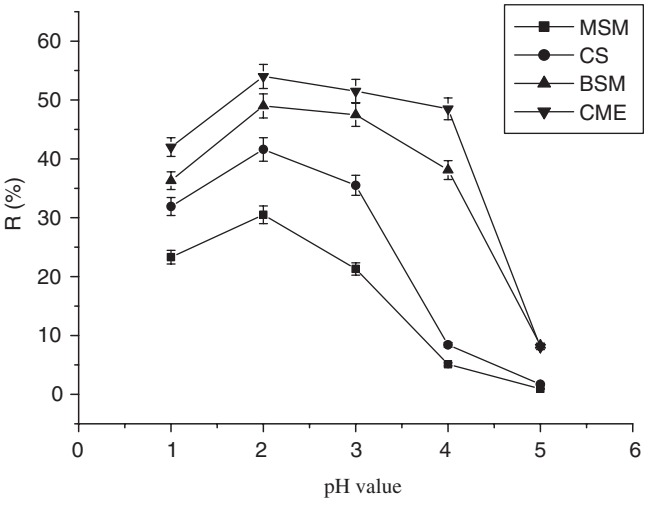

Figure 5. Effect of pH value on the extraction recovery of the sulfonylureas. Extraction conditions: sample volume, 5.0 mL; extraction solvent, 60 μL C_6H_5Cl; dispersive solvent, 1.0 mL acetone.

the solubility of the herbicides in aqueous solutions increases, therefore, the extraction efficiency decreases due to the decrease of distribution coefficient. Based on the experimental results, 1.0 mL of acetone was chosen for the study.

3.1.4 *Effect of sample solution pH*

The pH of sample solution affects the extraction performance greatly. For acidic sulfonylurea herbicides, the sample solution should be rather acidic to effectively deionise the analytes and consequently reduce their solubility within the sample solution [26]. The effect of sample pH in the range from 1.0 to 5.0 on the extraction of the sulfonylurea herbicides was investigated (Figure 5). The results indicated that the best extraction efficiency was observed at pH 2.0. Consequently, the pH of the sample solution was selected as 2.0 for the subsequent studies.

3.1.5 *Effect of extraction time*

Extraction time is another important parameter affecting the extraction efficiency in DLLME as in most extraction procedures. In the present study, the influence of extraction time was investigated in the time range between 0.1 and 10 min. The results indicated that the extraction time has no impact on the extraction recoveries. This could be attributed to the fact that equilibrium state can be achieved very quickly in DLLME so that the extraction time required can be very short. The extremely short extraction time required by DLLME is one of the big advantages for the technique.

3.1.6 *Effect of salt addition*

The effect of addition of the salt on the extraction efficiency was studied by adding NaCl (0–6%, w/v) into the aqueous solution. At the NaCl concentration higher than 6%,

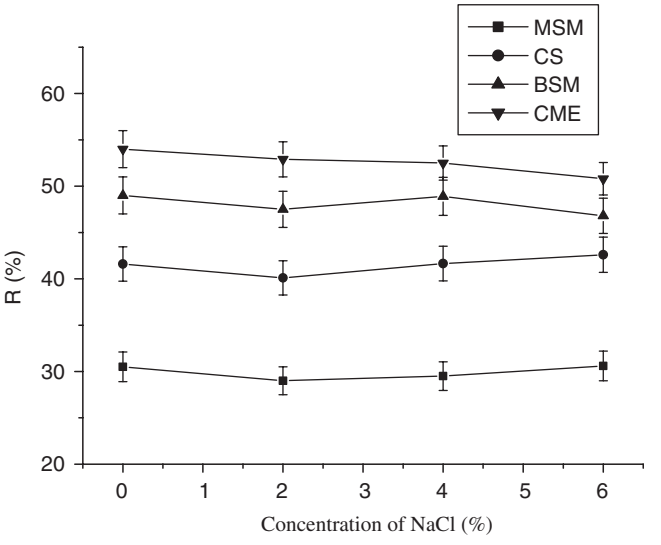

Figure 6. Effect of salt addition on the extraction recovery of the sulfonylureas. Extraction conditions: sample volume, 5.0 mL; extraction solvent, 60 μL C_6H_5Cl; dispersive solvent, 1.0 mL acetone.

the extraction solvent phase could not be sedimented at the bottom of the centrifuge tube, but went to the upper layer in the tube. Figure 6 demonstrates the extraction recovery versus concentration of NaCl. It can be seen that no significant effect on the extraction recoveries for any of the target analytes is observed when different amounts of NaCl are added into the aqueous solution. Hence, NaCl was not added in all subsequent experiments.

Under the above-optimised experimental conditions, the enrichment factors of DLLME for MSM, CS, BSM, and CME were 102, 138, 186, and 216, respectively.

3.2 *Evaluation of method performance*

Under the above-optimised conditions, the proposed method was validated by linearity, precision, the limits of detection (LOD) and the limits of quantification (LOQ). A series of working solutions containing each of MSM, CS, BSM, and CME at five concentration levels of 1, 5, 20, 40, 80, 100 ng mL^{-1} were prepared for the establishment of the calibration curve. For each concentration point, three parallel extractions and analyses were performed. The characteristic calibration data are listed in Table 1. Linearity was observed in the range of 1–100 ng mL^{-1} with the correlation coefficient (r) ranging from 0.9982 to 0.9995. The LOD ($S/N = 3$) and the LOQ ($S/N = 6$) for the four sulfonylureas were ranged from 0.2 to 0.3 ng mL^{-1} and 0.4 to 0.6 ng mL^{-1}, respectively. The repeatability study was carried out by five parallel experiments at the concentration of 10 and 50 ng mL^{-1} for each of the sulfonylureas under the optimal conditions. The resultant repeatabilities expressed as relative standard deviations (RSDs) varied from 3.3% to 6.5%. These results show that the proposed method has a high sensitivity and good repeatability.

Table 1. The linear ranges, correlation coefficient, detection limits and enrichment factors of the method.

Herbicides	Linear range ($ng\,mL^{-1}$)	Correlation coefficient (r)	LOD ($ng\,mL^{-1}$)	LOQ ($ng\,mL^{-1}$)	RSD (%)	*EF*
MSM	1–100	0.9984	0.2	0.4	5.2	102
CS	1–100	0.9995	0.2	0.4	6.5	138
BSM	1–100	0.9982	0.3	0.6	3.3	186
CME	1–100	0.9985	0.3	0.6	4.0	216

3.3 *Water samples analysis*

The accuracy and applicability of the proposed method was evaluated by determining the four sulfonylurea herbicides in river, stream and well water samples. No sulfonylurea herbicides residues were found in stream and well water at the quantification level of the method. Only MSM residue was found in river water at a concentration of $1.2\,ng\,mL^{-1}$. For the determination of the recoveries of the method, these samples were spiked with the standard solutions of the target analytes at three concentration levels. For each concentration level, five replicate experiments were performed. The results of recoveries for these water samples are listed in Table 2. For all the four sulfonylurea, the recoveries were in the range from 85.0% to 103.5%, and the RSDs fell in the range from 3.0% to 6.6%, respectively. Figure 7 gives the typical chromatograms of the river water sample before and after being spiked with the four sulfonylurea herbicides at each concentration of $10\,ng\,mL^{-1}$ (230 nm). It can be seen from the chromatograms that there are no interference peaks from the sample matrix for the determination of the herbicides.

3.4 *Comparison of DLLME with other sample preparation techniques*

The extraction efficiency of the presented DLLME method was compared with other reported methods such as LLE, SPE, SFE, and MASE from the viewpoint of the extraction time, LOD, RSD and linear range. As listed in Table 3, the extraction time for DLLME is very short because the extraction equilibrium is reached extremely quickly (a few seconds) due to the large surface area between the extraction solvent and the sample solution. Only a few minutes are needed before instrumental analysis. However, the extraction times for LLE, SPE, MASE, and SFE range from 10 to 150 min. For DLLME, precision, expressed as RSD, is comparable with that of the extraction methods mentioned above. The volume of sample solution required for DLLME is small (5.0 mL) owing to the high enrichment factor. Furthermore, the DLLME process does not require special instrumentations and consumes much less toxic organic solvent. All these results reveal that the DLLME is simple, rapid and convenient for the sample preparation of the sulfonylurea herbicides from water samples.

4. Conclusion

In this paper, a simple, rapid and sensitive DLLME extraction technique coupled with HPLC-DAD detection has been newly developed for the determination of sulfonylurea

Table 2. The determinations of herbicides residues in water samples and recoveries of spiked water samples.

		River water ($n=5$)			Stream water ($n=5$)			Well water ($n=5$)		
Herbicides	Spiked ($ng\,mL^{-1}$)	Found ($ng\,mL^{-1}$)	R^b (%)	RSD (%)	Found ($ng\,mL^{-1}$)	R^b (%)	RSD (%)	Found ($ng\,mL^{-1}$)	R^b (%)	RSD (%)
MSM	0	1.2			nd[a]			nd[a]		
	6	7.4	102.8	5.8	5.8	96.7	5.4	5.7	95.0	6.6
	20	20.2	95.3	4.1	20.7	103.5	6.5	18.9	94.5	3.9
	60	54.9	89.7	3.5	61.2	102	4.7	57.4	95.7	3.0
CS	0	nd[a]			nd[a]			nd[a]		
	6	5.8	96.7	6.1	5.7	95	6.3	6.2	103.3	5.6
	20	18.5	92.5	5.2	18.2	91.0	4.5	19.1	95.5	4.1
	60	60.7	101.2	3.7	58.2	97	3.1	58.5	97.5	3.4
BSM	0									
	6	5.5	91.7	4.3	5.3	88.3	6.0	5.8	96.7	5.7
	20	17.8	89.0	3.9	18.5	92.5	4.7	18.3	91.5	4.1
	60	57.1	95.1	3.2	55.5	92.5	3.6	51.8	86.3	3.8
CME	0									
	6	5.2	86.7	5.8	5.1	85.0	5.3	5.2	86.7	6.6
	20	17.5	87.5	4.2	18.0	90.0	4.5	18.7	93.5	4.7
	60	57.1	95.1	3.6	56.7	94.5	3.2	52.6	87.7	3.5

Notes: nd[a]: not detected; R^b: recovery of the method.

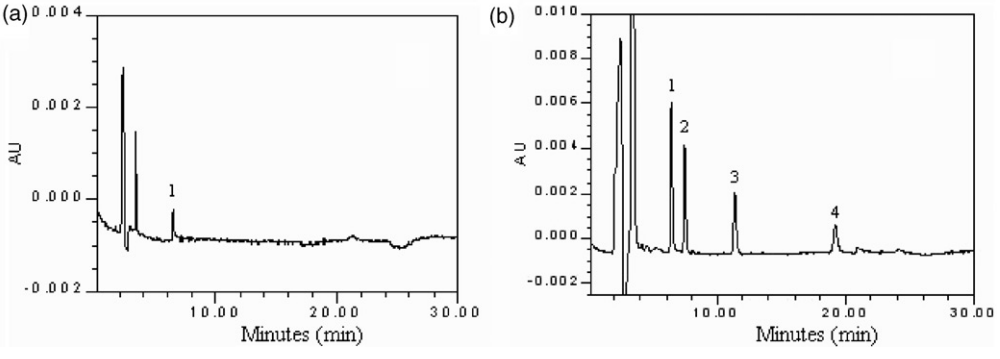

Figure 7. Chromatograms of river water before (a) and after spiked with sulfonylurea herbicides at each concentration of $10\,ng\,mL^{-1}$ (b). Monitoring wavelength: 230 nm; (1) MSM, (2) CS, (3) BSM, (4) CME.

herbicides in water samples. Enrichment factors were between 102 and 216 and the recoveries were acceptable for the pesticide residues analysis. Compared with other conventional extraction methods, this method offers advantages such as rapidity, simplicity, ease of operation, high enrichment factor, and friendliness to the environment. The DLLME combined with HPLC-DAD method is suitable for the analysis of sulfonylurea herbicides in water samples.

Table 3. Comparison of DLLME with other sample preparation techniques for the determination of the sulfonylureas.

Methods	Linearity ($ng\,g^{-1}$)	LOD ($ng\,g^{-1}$)	RSD (%)	Extraction time (min)	References
LLE-HPLC-UV	20–5000	–	7–10	150	[7]
LLE-HPLC-MS^2	0.05–10	–	–	100	[8]
SPE-HPLC-UV	60–1200	7	3.5–4.4	140	[7]
SPE-HPLC-UV	250–10,000	5	3.8–8.5	60	[3]
SPE-HPLC-MS	0.05–2	0.007–0.048	4.0–22	90	[2]
SPE-HPLC-MS	100–10,000	0.6–3.5	0.02–12.8	130	[9]
SPE-HPLC-MS^2	5–500	0.4–1.2	<4	120	[6]
SFE-HPLC-UV	60–1200	35	2.5–3.2	40	[7]
MASE-HPLC-UV	5–250	–	1–9	10	[13]
DLLME-HPLC-UV	1–100	0.2–0.3	3.3–6.5	a few seconds	This method

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