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RESEARCH ARTICLE

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Separation and quantification of diazinon in water samples using liquid-phase microextraction-based effervescent tablet-assisted switchable solvent method coupled to gas chromatography-flame ionization detection

Hojatollah Kakaei[1,2] | **Seyed Jamaleddin Shahtaheri**[3,4] | **Khosrou Abdi**[5] | **Nader Rahimi Kakavandi**[1]

[1]Department of Occupational Health Engineering, School of Health, Ilam University of Medical Sciences, Ilam, Iran

[2]Health and Environment Research Center, Ilam University of Medical Sciences, Ilam, Iran

[3]Department of Occupational Health Engineering, School of Health, Tehran University of Medical Sciences, Tehran, Iran

[4]Institute for Environmental Research Center for Water Quality Research, Tehran University of Medical Sciences, Tehran, Iran

[5]Department of Nuclear Pharmaceutical Group, School of Pharmacy, Tehran University of Medical Sciences, Tehran, Iran

Correspondence
Nader Rahimi Kakavandi, Department of Occupational Health Engineering, School of Health, Ilam University of Medical Sciences, Ilam, Iran.
Email: nrahimi@razi.tums.ac.ir

Abstract

This study used a liquid-phase microextraction-based effervescent tablet-assisted switchable solvent method coupled to gas chromatography-flame ionization detection as an eco-efficient, convenient-to-use, cost-effective, sensitive, rapid, and efficient method for extracting, preconcentrating, and quantifying trace amounts of diazinon in river water samples. As a switchable solvent, triethylamine (TEA) was used. *In situ* generation of CO_2 using effervescent tablet containing Na_2CO_3 and citric acid changed the hydrophobic TEA to the hydrophilic protonated triethylamine carbonate (P-TEA-C). CO_2 removal from the specimen solution using NaOH caused P-TEA-C to be converted into TEA and led to phase separation, during which diazinon was extracted into the TEA phase. The salting-out process was helpful in enhancing extraction efficiency. In addition, a number of significant parameters that affect extraction recovery were examined. Under optimum conditions, the limit of detection and limit of quantitation were 0.06 and 0.2 ng/ml, respectively. The extraction recovery percentage and pre-concentration factor were obtained at 95 and 190%, respectively, and the precision (inter- and intra-day, relative standard deviation %, $n = 5$) was <5%.

KEYWORDS
diazinon, effervescent tablet, gas chromatography-flame ionization detection, liquid-phase microextraction, surface water sample, switchable solvent

1 | INTRODUCTION

Diazinon is an organophosphorus pesticide that is frequently employed in agricultural areas to safeguard crops and fields (Ghoraba et al., 2018; Shah & Iqbal, 2010). The World Health Organization (WHO) has classified diazinon as belonging to the relatively hazardous group (class II) in terms of toxicity (Shah & Iqbal, 2010). Diazinon is toxic for aquatic creatures and mammals (including humans) (Hosseini et al., 2021). In addition to acting as an inhibitor of cholinesterase, diazinon alters liver enzymes and other biochemical factors (Poet et al., 2004), ultimately resulting in problems with neurological, respiratory, skin, and digestive systems (Hosseini et al., 2021). A variety of

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List of Abbreviations: LPME, Liquid-phase microextraction; ETA, Effervescent tablet-assisted; SS, Switchable solvent; GC-FID, Gas chromatography- flame ionization detector; TEA, Triethylamine; P-TEA-C, Protonated triethylamine carbonate; LOD, Limits of detection; LOQ, Limits of quantitation; ER, Extraction recovery; PF, Pre-concentration factor; RSD, Relative standard deviation; WHO, World Health Organization; EA-LPME, Effervescence-assisted liquid–phase microextraction; RR, Relative recovery.

techniques such as immunoassay, MS, HPLC, and GC have been used for monitoring diazinon (Abu-Qare & Abou-Donia, 2001; Brun et al., 2004; Everett & Rechnitz, 1998; Hernandez et al., 2002; Vinas et al., 2002; Zulin et al., 2002). Before analysis using analytical techniques, an acceptable, reliable, sensitive, rapid, cost-effective, and eco-efficient method of sample preparation is needed for diazinon analysis in samples (Lamei et al., 2018; Rahimi Kakavandi et al., 2021). For sample preparation and pre-concentration of the samples containing pesticides, various methods such as vortex-assisted liquid-phase microextraction, dispersive liquid-phase microextraction, homogeneous liquid-phase microextraction, single-drop microextraction, and solid-phase microextraction have been utilized (Cavaliere et al., 2012; Jia et al., 2010; Tsiropoulos & Amvrazi, 2011; Yazdanfar et al., 2014; Zacharis et al., 2012). The key advantages of using liquid–phase microextraction (LPME) methods are their high pre-concentration factor (PF), ease of use, and low cost (Yilmaz & Soylak, 2016). One of the LPME techniques is effervescence-assisted liquid-phase microextraction (EA-LPME), which has been expanded in recent years (Asadi et al., 2022; Shishov et al., 2017). An effervescent reaction is the basis of the EA-LPME method (Yang et al., 2016). In this method, CO_2 is produced *in situ* when an effervescent tablet (ET) that contains both an effervescent agent and a proton donor agent dissolves in the specimen solution. The CO_2 bubbles produced help the extractant solvent disperse in the specimen solution, without the use of ultrasonic radiation, vortex agitation, or disperser solvent (Ezoddin et al., 2022; Yang et al., 2016). In recent years, switchable solvents (SS) have been suggested as green solvents (Lasarte-Aragonés et al., 2015; Rahimi Kakavandi et al., 2017). By adding or removing CO_2 from the sample, SSs can switch between two forms (water miscible and water immiscible) (Jessop et al., 2005; Yilmaz & Soylak, 2015). SSs contain organic acids or bases, including amines, amidines, or fatty acids, which, in the presence of water in its neutral state, could create a biphasic system. They become bicarbonate salt when CO_2 is added to the sample solution, creating a monophasic solution (Jessop, Heldebrant, Li, Eckert, & Liotta, 2005). The aim of this study was to separate and pre-concentrate diazinon in surface water samples using a liquid-phase microextraction-based effervescent tablet-assisted switchable solvent (LPME-ETA-SS) method and analyze using gas chromatography-flame ionization detection (GC-FID).

2 | MATERIALS AND METHODS

2.1 | Chemicals

Diazinon and ET ingredients (Na_2CO_3 and citric acid) were supplied by Sigma-Aldrich (UK, Merk Life Science UK Limited, an affiliate of Merk KGaA, Darmstadt, Germany). Triethylamine (TEA), NaOH, GC-grade methanol, internal standard (diphenylamine), and NaCl were provided by Merck (Darmstadt, Germany). To prepare the aqueous solutions, a Milli-Q device was used to purify the water utilized (Millipore, Bedford, MA, USA). The standard stock solutions of diazinon were prepared in methanol.

2.2 | Instruments and chromatographic condition

The analytes were separated and detected using the Hewlett-Packard GC model 6890 with a fused-silica capillary column (30 m × 0.25 mm i.d., and film thickness 0.25-μm ft, coated with 5% phenyl methyl polysiloxane) and a flame ionization detector. The temperature of the column was adjusted from 120°C (kept for 2 min) to 260°C (kept for 20 min) at a flow of 10°C/min. Samples were injected into the splitless inlet system at a steady temperature of 250°C. With H_2/air at 35/350, the detector's temperature was set to 300°C. Also, N_2 as carrier gas was adjusted with a steady flow of 1.3 ml/min. A Texol zero-air generator and a Packard hydrogen generator provided the necessary air and H_2 for the flame ionization detector, respectively. Chemstation software assessed all the data acquired from the GC.

2.3 | Standard solution preparation

Methanol was used to prepare diazinon standard stock solutions (1 mg/ml). To prepare the standard solutions, a river water sample was utilized. To construct the calibration curve using analysis of least-squares linear regression, the concentrations of the standards in river water samples of 0.2, 5, 50, 500, 1000, 3000, and 5000 ng/ml were utilized ($n = 3$ for each concentration). The calibration curve shows the change in the diazinon peak area ratio to the peak area of diphenylamine (as the internal standard) with the related concentration.

2.4 | ET preparation

For the preparation of ETs initially, sodium carbonate and citric acid were placed in a desiccator after being dried for 2 h at 90°C in an oven. Then, 550 mg of sodium carbonate and 900 mg of citric acid were weighed and ground in a pounder to acquire a homogeneous powder. After the two were mixed, the homogeneous mixture was compressed at 10 tons for 10 s using a tablet press machine to create an ET. The tablets were then placed in a desiccator after being stored in a plastic zip-tie bag.

2.5 | Microextraction process

In a 15-ml conical glass test tube, 10.0 ml of the sample solution consisting of 20 ng/ml of diazinon and 20 ng/ml of diphenylamine as the internal standard was taken. Then 300 μl of TEA (extractant solvent) and one ET were added. Many bubbles formed at the bottom of the tube in this stage. By dissolution of the ET, CO_2 bubbles were produced *in situ*, and TEA was converted into protonated triethylamine carbonate (P-TEA-C), which was then dispersed homogeneously throughout the aqueous specimen. Then 1.5 ml of NaOH (10 M) solution was added, and a cloud formed in the tube. The addition of NaOH solution at this stage caused the separation of the phases as

P-TEA-C (hydrophilic) was converted into TEA (hydrophobic). Simultaneously, diazinon was absorbed and extracted as fine droplets of TEA. The tube was centrifuged at 4000 rpm for 5 min. Then, using a micro syringe, the upper phase was removed carefully, transferred to a microtube, and evaporated to dryness under a gentle flow of nitrogen. Then the remnant was dissolved in 50 μl of methanol, and finally, 2 μl was injected into the gas chromatography-flame ionization detector.

2.6 | Collection and preparation of samples

The river water samples were obtained from the Seimare River (Darreh Shahr, Iran). A 0.45-μm pore-size filter was utilized to filter out any interfering particles before the extraction process.

2.7 | Validation of method

Method validation was performed using optimal parameters in accordance with the principles of validation of the bioanalytical method outlined in the FDA guidelines (FDA, U., 2018). Key analytical parameters such as linearity, limit of detection (LOD), limit of quantification (LOQ), precision, selectivity, accuracy, extraction recovery percentage (ER%), and PF in this microextraction method were assessed.

3 | RESULTS AND DISCUSSION

3.1 | ET combination

A proton donor agent, a source of CO_2, and an active disperser composition have all been attributed to the ET. In addition, the dispersion of the extractant in the specimen solution was enhanced by CO_2 bubbles, which elevated the mass transfer of analytes.

An ideal composition of the ET is necessary to produce sufficient CO_2 bubbles. An ET with the proper composition can produce sufficient bubbles and expedite the dispersion of the extractant solvent in the specimen solution. Indeed, a suitable effervescence with a prolonged time reaction could be perfect because it provides more gas bubbles and extends the period that organic droplets (extractant solvent) stay in contact with the gas bubbles. A large quantity of the effervescent agent, however, reduces mass transfer and ER by increasing the viscosity of the solution and ionic strength (Asadi et al., 2022). Therefore, the quantity of Na_2CO_3 (effervescent agent) and citric acid (proton donor agent) was examined in the ER; 300–1500 mg of citric acid was present in the ET containing 550 mg of Na_2CO_3. Figure 1 shows that an increase in the quantity of citric acid results in an increase in the ER of up to 900 mg. The optimum quantity of citric acid was, therefore, determined to be 900 mg. In addition, 250–750 mg of Na_2CO_3 was present in the ET containing 900 mg of citric acid (Figure 2). Using 550 mg of Na_2CO_3 resulted in the highest ER. Therefore, in continuing studies, 550 mg of the effervescent agent was utilized.

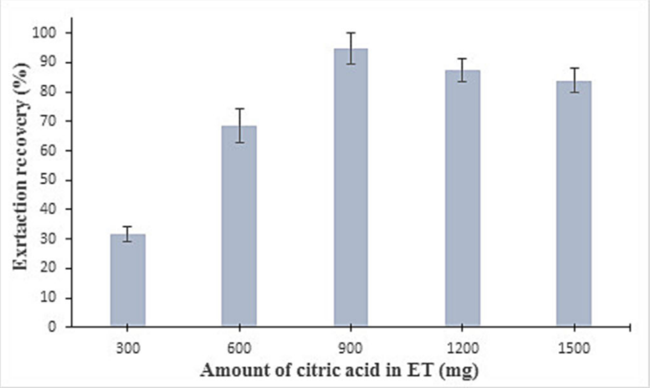

FIGURE 1 Effect of quantity of citric acid on extraction recovery of diazinon. Composition of tablet: Na_2CO_3 (550 mg) and citric acid ($n = 5$).

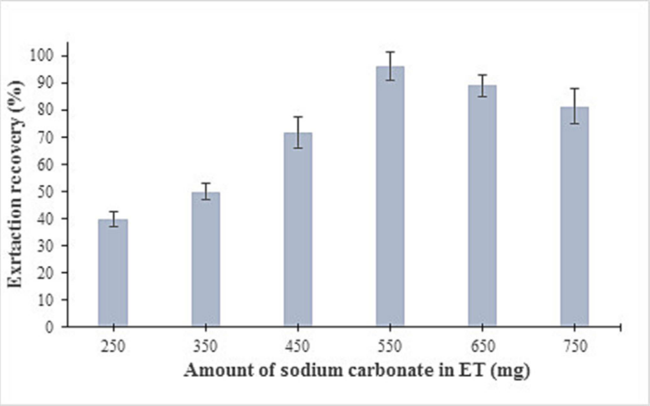

FIGURE 2 Effect of quantity of Na_2CO_3 on extraction recovery of diazinon. Composition of tablet: citric acid (900 mg) and Na_2CO_3 ($n = 5$).

3.2 | Impact of extractant solvent volume

The SSs should possess particular properties to achieve an effective ER, which are as follows: (a) switching between hydrophilic and hydrophobic forms, and vice versa, by adding or removing CO_2; (b) high solubility in water for the hydrophilic form and poor solubility in water for the hydrophobic form; (c) lower density than water; and (d) the potential for analyte extraction from various specimens. As TEA was considered to have these properties, it was chosen as the extractant solvent to prepare the SS.

To acquire the optimum volume of TEA on diazinon ER, 100–700 μl of TEA was used. The ER percentage increased up to 300 μl of TEA, as shown in Figure 3, but then it began to gradually decrease. As the volume of TEA increased, the afloat phase volume also increased and the PF decreased. Therefore, 300 μl of TEA was used as the volume of extractant solvent to achieve the highest ER.

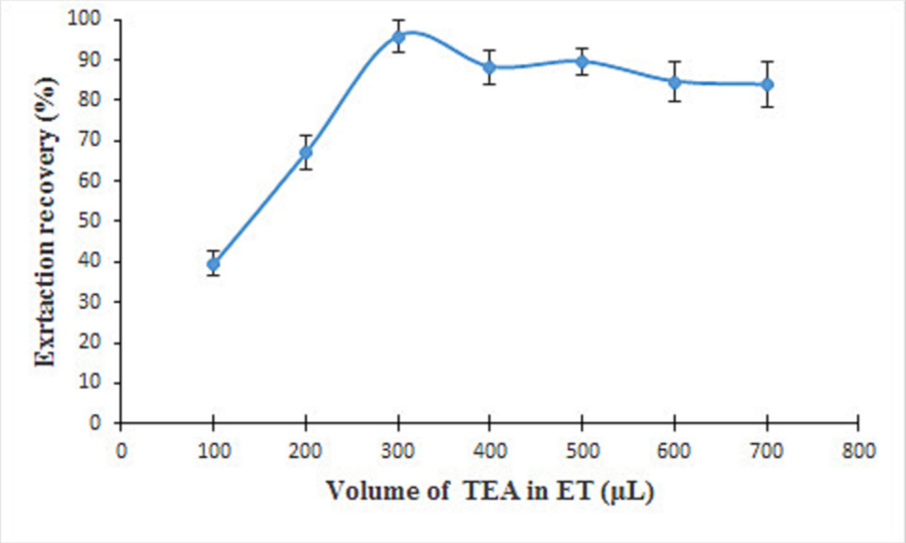

FIGURE 3 Effect of the volume of TEA (triethylamine) on extraction recovery of diazinon (10 ng/ml of diazinon, sample volume: 10 ml, NaCl: 1.5 g, 10 M of NaOH volume: 1.5 ml, $n = 5$).

3.3 | Effect of volume of NaOH

In this microextraction method, NaOH was used to induce the separation of phases. Therefore, the switchable solvent's hydrophilic form (P-TEA-C) was converted to the hydrophobic phase (TEA) utilizing sodium hydroxide (10 M) as a phase transfer mediator, and simultaneously, diazinon was extracted to the TEA phase from the sample solution. To determine the optimal volume of NaOH solution (10 M), 0.5–3 ml was used. The acquired data showed that 1.5 ml of 10-M NaOH had the maximum ER, and then, the ER remained constant (Figure 4). In NaOH volumes less than 1.5 ml, the cloudy appearance was not observed in the solution completely, so the separation of phases did not occur completely. Therefore, for use in subsequent experiments, 1.5 ml of NaOH was considered the optimum volume.

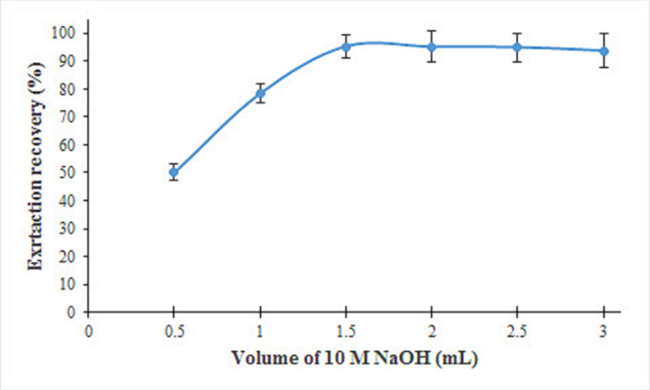

FIGURE 4 Effect of NaOH volume on extraction recovery of diazinon [10 ng/ml of diazinon, sample volume: 10 ml, NaCl: 1.5 g, TEA (triethylamine) volume: 300 µl, $n = 5$].

3.4 | Effect of NaCl volume on extraction

By increasing the speed of fine droplet formation of TEA and decreasing analyte solubility in the aquatic phase, addition of salt (salting-out process) to the specimen solution can improve the phase separation process. To determine the effect of the quantity of NaCl on ER, 0.5–3.0 g of NaCl was added to the solution. The ER improved significantly when the quantity of NaCl was increased from 0 to 1.5 g, but then it decreased due to the high NaCl concentration (Figure 5). The best theory for this effect is that adding a small amount of salt could improve the mass transfer of analytes to the extractant solvent, which would increase the performance of enrichment. In contrast, a high quantity of salt might increase the viscosity of the extraction system, which can reduce the analyte movement and decrease the ER. The results show that 1.5 g of NaCl was selected as the optimum quantity for all further experiments.

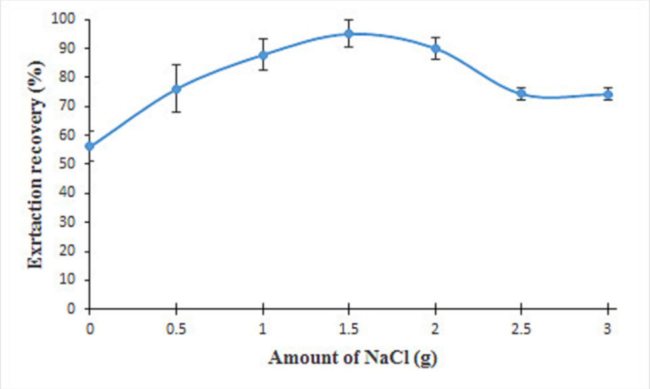

FIGURE 5 Effect of NaCl quantity on extraction recovery of diazinon [10 ng/ml of diazinon, sample volume: 10 ml, 10 M of NaOH volume: 1.5 ml, TEA (triethylamine) volume: 300 µL, $n = 5$].

3.5 | Method validation

3.5.1 | Selectivity

Figure 6 shows the standard solution chromatograms of diazinon (400 ng/ml) and diphenylamine (as an internal standard) (200 ng/ml), a sample of spiked river water (2 ng/ml), and a blank sample of river water. Diazinon and diphenylamine had retention times of 8.7 and 6.2 min in river water samples, respectively. The high selectivity of this microextraction technique is demonstrated by the nonexistence of any interfering substances in river water blank specimens at this retention time.

3.5.2 | Linearity

The standard calibration curve exhibited a linear behavior in the range of 0.2–5000 ng/ml (95% confidence interval of slope: 4.95–5.44, *P*-value <0.05). Utilizing this LPME technique, the correlation coefficient (R^2) was 0.9983, and the equation was $y = 5.2954x + 10.259$. Slope

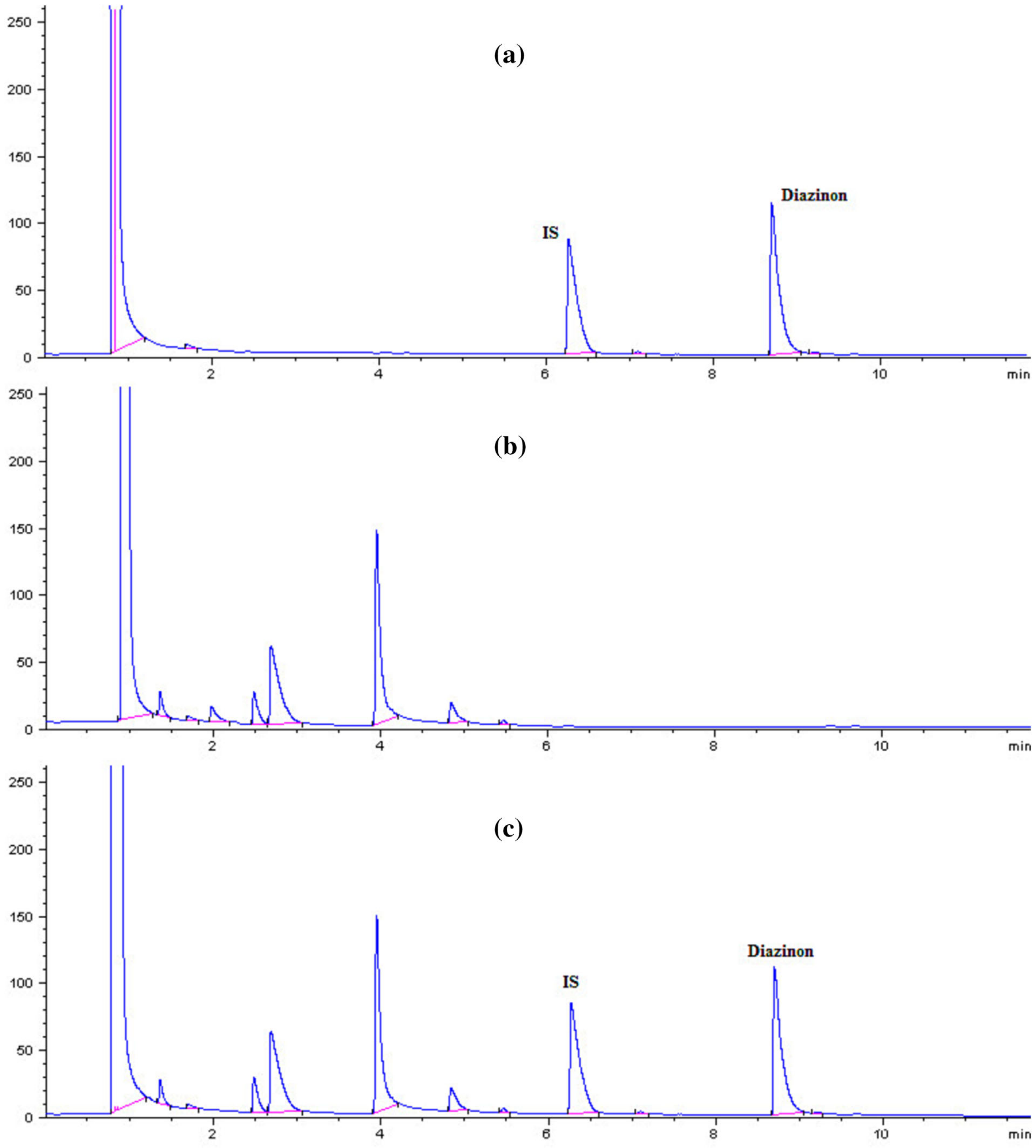

FIGURE 6 GC-FID (gas chromatography-flame ionization detection) chromatograms of (a) standard solution of diazinon (400 ng/ml), (b) blank river water sample (after microextraction procedure), and (c) spiked river water sample by 2 ng/ml of diazinon after microextraction procedure.

and intercept averages were calculated over 3 days. The evaluation of the resulting calibration curve equations under identical conditions did not reveal a significant difference between them (*P*-value >0.05). Thus, on diverse days the curves were the same.

3.5.3 | LOD and LOQ

LOD and LOQ, two key parameters used in method validation, specify the analyte's lowest concentration that an analytical technique can reliably detect and quantify. To measure the LOD and LOQ, signal-to-noise ratios of 3 and 10 were used, respectively. Table 1 presents the LOD and LOQ values as 0.06 and 0.2 ng/ml, respectively.

3.5.4 | PF and ER%

The measurement parameters of the extraction technique are PF and ER%. The diazinon concentration ratio of the final phase of organic solvent (C_f) to the sample solution's initial concentration (C_i) was measured to calculate PF (C_f/C_i). In addition, ER% was determined using Equation (1), which is given as follows:

$$\mathrm{ER} = \mathrm{PF} \times \frac{V_f}{V_i} \times 100 \tag{1}$$

V_f represents the final volume of the organic extractant, and V_i represents the sample solution's initial volume. Based on these calculations, PF and ER% were obtained as 190 and 95%, respectively. Table 1 presents the analytical performance characteristics of diazinon in the river water sample using the LPME-ETA-SS method.

3.5.5 | Accuracy and precision

Intra- (within 1 day) and inter-day (between three separate days) precision was evaluated at four diverse concentrations of diazinon in the river water sample ($n = 5$). In addition, using the spiked samples, some recovery examinations at varied diazinon concentrations were conducted to examine accuracy (Table 2). The intra- and inter-day precision for samples by diverse concentrations was 3–5% for relative standard deviation (RSD%) and 93–96% for accuracy.

3.5.6 | Analysis of real sample

By examining the spiked samples at various concentration levels in river water samples (0, 1, 10, 15, and 20 ng/ml), the developed technique was assessed to monitor diazinon in real specimens. Equation (2) was used to compute the relative recovery percentage (RR%):

$$\mathrm{RR}\% = \frac{C_{\mathrm{found}} - C_{\mathrm{real}}}{C_{\mathrm{added}}} \times 100 \tag{2}$$

In Equation (2), C_{found} represents the concentration of diazinon after the spiking of a specific quantity of standard to a real sample, C_{real} represents the concentration of diazinon in the real specimen before the spiking of the standard, and C_{added} represents the quantity of standard added to the real specimen. For the real specimens spiked, the RR% was in the range of 93–96% (Table 3). Good RR and acceptable RSD show that this method can accurately determine diazinon in real samples. All data related to this article are collected in the supplementary material.

3.6 | Comparing this method to other previously reported methods

Findings from the developed method evaluated in this study contrasted with some characteristic data from previously published methods to detect and determine diazinon in Table 4. Low LOD and LOQ, acceptable RSD and RR%, and high PF were attained, as is

TABLE 1 Analytical performance characteristics of LPME-ETA-SS method.

Compound	Sample	LOD (ng/ml)	LOQ (ng/ml)	Linear range (ng/ml)	R^2	RSD% ($n = 5$)	Pre-concentration factor
Diazinon	River water	0.06	0.2	0.2–5000	0.9983	4.25	190

Note: LOD, limit of detection; LOQ, limit of quantitation; LPME-ETA-SS, liquid-phase microextraction-based effervescent tablet-assisted switchable solvent; RSD, relative standard deviation.

TABLE 2 Inter- and intra-day reproducibility of the method for spiked river water samples ($n = 5$).

		Inter-day measured value		Intra-day measured value	
Compound	Spiked concentration (ng/ml)	Accuracy (%)	Precision (%)	Accuracy (%)	Precision (%)
Diazinon	0.5	93.56	4.79	94.10	4.31
	5	94.28	4.13	94.42	4.38
	10	95.75	3.15	95.07	3.35
	25	95.62	3.51	94.98	3.70

TABLE 3 Analytical results for determination of diazinon in water samples.

		Diazinon		
Sample	Spiked (ng/ml)	Found (ng/ml)	RR%	RSD%
Distilled water	0.0	ND	–	–
	1	0.937	93.7	4.8
	10	9.464	94.64	3.3
	15	14.22	94.8	4.7
	20	19.16	95.8	4.6
River water	0.0	ND	–	–
	1	0.956	95.6	4.9
	10	9.432	94.32	4.1
	15	14.22	94.8	3.6
	20	18.78	93.9	4.7

Note: ND, not detected; RR, relative recovery; RSD, relative standard deviation.

TABLE 4 Comparison of the presented method with other reported methods.

Methods	LOD (ng/ml)	RSD%	PF	RR%	Sample	Reference
CCSLLE-GC-FID	2	2	575	92	Aqueous	Farajzadeh et al. (2016)
SDLLME-SFO-HPLC–UV	2	5.2	180	58	Apple, pear	Pirsaheb et al. (2015)
MSPE-GC-FID	0.15	6.9	135	67.5	Water, fruit, juice	Targhoo et al. (2018)
MDSPE-DLLME-GC-FID	0.15	5	682	68	Fruit, juices	Farajzadeh and Mohebbi (2018)
LPME-ETA-SS-GC-FID	0.06	<5	190	95	River water sample	This work

Note: CCSLLE, counter current salting-out homogeneous liquid–liquid extraction; GC-FID, gas chromatography-flame ionization detection; LOD, limit of detection; LPME-ETA-SS, liquid-phase microextraction-based effervescent tablet-assisted switchable solvent; MDSPE-DLLME, magnetic dispersive solid-phase extraction-dispersive liquid–liquid microextraction; MSPE, magnetic solid-phase extraction; PF, pre-concentration factor; RR, relative recovery; RSD, relative standard deviation; SDLLME-SFO, sonication and dispersive liquid–liquid microextraction based on the solidification of floating organic drop.

evident. In addition, these analytical performances are comparable to or show more acceptable results than in previously reported procedures.

4 | CONCLUSION

This study developed an LPME-ETA-SS method coupled to GC-FID as an eco-efficient, convenient-to-use, sensitive, inexpensive, rapid, and efficient method for extracting, preconcentrating, and determining diazinon in river water specimens. The basis of this procedure is the addition of an effervescent tablet to the specimen solution, which contains citric acid as a proton donor and Na_2CO_3 as an effervescent agent. The effervescent tablet's dissolution changed TEA as the extraction solvent from a water-immiscible liquid to a water-miscible liquid by forming CO_2 bubbles in the specimen solution. The extractant solvent was also dispersed in the specimen solution by the effervescent agent. ET and NaOH were used in this study as they are safe, inexpensive, and easily accessible in a laboratory. The salting-out process was helpful in decreasing the phase separation time and enhancing extraction efficiency. The use of ETA-SS as a green solvent is advantageous because a low quantity of extraction solvent (300 μl) is required and it is eco-friendly. In addition, tablet preparation was inexpensive and simple. Therefore, the presented analytical method is suitable to be employed as a functional technique for the detection and determination of diazinon in the environmental matrix.

AUTHOR CONTRIBUTIONS

N.R.K. proposed the idea. N.R.K. and H.K. were the study supervisors and performed the study, literature search, and drafting of the article. S.J.S. and K.A were advisors. S.J.S. and K.A. assisted with some experimental parts of the study. The final version was read and approved by all authors.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

ORCID

Khosrou Abdi https://orcid.org/0000-0003-2836-2575

Nader Rahimi Kakavandi https://orcid.org/0000-0002-9328-1408

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SUPPORTING INFORMATION

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