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Application of electro-enhanced solid-phase microextraction for determination of phthalate esters and bisphenol A in blood and seawater samples

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ABSTRACT

The electro-enhanced solid-phase microextraction (EE-SPME) method was developed for the determination of endocrine disruptor compounds such as phthalate esters and bisphenol A in human blood and seawater samples. After EE-SPME, samples were analyzed by gas chromatography–mass spectrometry (GC–MS). In this approach, commercial SPME fiber was used in direct-immersion mode with an applied potential to extract di-ethyl phthalate, di-butyl phthalate, benzyl butyl phthalate and bisphenol A. The applied potential facilitates and enhances the extraction efficiency of the target analytes. Various experimental conditions influencing performance of the EE-SPME such as extraction time, applied potential and ionic strength were optimized. Under the optimum conditions, EE-SPME was more efficient than a conventional SPME approach. Very good linearity was observed for all analytes in a range between 1 and 100 $\mu g\,L^{-1}$ with correlation of determination (R^2) between 0.963 and 0.996. The limits of a detection based signal-to-noise of 3 were from 0.004 to 0.15 $\mu g\,L^{-1}$. The reproducibility of EE-SPME was evaluated and the relative standard deviations were between 1.0% and 5.0% ($n=9$). The proposed method was applied to human blood samples stored in transfusion bags and seawater. Results showed that the proposed EE-SPME was simple and suitable for trace level analysis.

1. Introduction

Phthalate esters (PAEs) are used as plasticizers in the manufacturing process of plastics, polyvinyl chloride and polyethylene materials to improve their flexibility and transparency. These plasticizers do not have a strong interaction with polymer chains and easily leach at harsh conditions [1–6]. Bisphenol A (BPA) is a chemical produced in large quantities for use primarily as a flame retardant and stabilizer in the production of polyvinyl chloride, polycarbonate plastics, rubber, and epoxy resins [7–9].

PAEs and BPA are classified as endocrine disruptor chemicals (EDC) which are able to cause abnormalities in invertebrate, fish, avian, reptilian, and mammalian species [10]. The carcinogenic toxicity of EDCs is known even at very low concentrations; their mode of action mimics estrogenic activity and may affect the health and reproduction systems of humans as well as wildlife [8,11–16]. Various mechanisms have been proposed in the literatures on the disruption activities of EDCs, for example, (i) by binding to receptors and mimicking or antagonizing the effects of the endocrine hormones [17–19], (ii) by affecting the concentration of hormones through the altering of their synthesis or metabolism of natural hormones [20], (iii) by interfering with the signal between the different components of the hypothalamus–pituitary–endocrine gland axes [21] and (iv) modifying the number of hormone receptors in a cell [22,23]. Studies have shown that BPA concentration at a level of 0.23 $ng\,L^{-1}$ will exhibit the estrogenic affect [24]. The United States Environmental Protection Agency has proposed a maximum concentration level for benzyl butyl phthalate (BBP) in drinking water of 100 $\mu g\,L^{-1}$ [6].

The leaching of BPA and PAEs from different industrial products such as plastic packaging and stored canned food have hardly been determined due to the complicated sample matrix and low concentrations [5,25–28]. In recent years, considerable attention has been given to the leaching effects of PAEs and BPA due to its high toxicity for humans [13,29–34].

In this regard different preconcentration techniques have been developed to extract EDCs from aqueous samples which include liquid–liquid extraction (LLE) and solid-phase extraction (SPE) [35–42]. However, LLE and SPE require larger volumes of organic solvents and multi-step extractions, thus these techniques are not suitable for trace level determination of EDCs in water and food samples [7]. Liquid phase microextraction (LPME) [43,44] and dispersive liquid–liquid microextraction (DLLME) [2,45] have been used for the extraction of PAEs from aqueous samples. Recently, the low density solvent-based vortex-assisted surfactant-enhanced-emulsification

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liquid–liquid microextraction (LDS-VSLLME) technique was developed by Zhang and Lee for the determination of phthalate esters in bottled water samples in which multistep complex extraction procedures were reported [46]. For the phthalates analysis, single step analytical methods should be preferred due to the risk of contamination from glassware. Additionally, the selection of suitable solvents for the extraction of polar analytes such as PAEs and BA is a challenging task in LPME, DLLME and LDS-VSLLME [45,46]. Stir bar sorptive extraction (SBSE) is an another solvent minimized method used for the extraction of BPA from waste water [8], seawater [47], and milk samples [7], and also for the determination of PAEs in water samples [48].

Solid-phase microextraction (SPME) is a solventless polymer sorption technique [49]. SPME is relatively simple: samples are extracted based on the partitioning between the polymeric sorbent and target analytes [49,50]. There are three modes of extraction by SPME: (i) direct immersion-SPME in which SPME fibers are exposed directly to the sample solution [51], (ii) headspace-SPME, where the SPME fibers are suspended on the headspace of the heated sample to extract volatile target compounds [52] and (iii) membrane protected-SPME, in which a porous polymeric membrane is used as a protective sleeve to extract polar analytes from complex samples [53].

Xin et al. [54] and Noushin et al. [55] developed functionalized-SPME fiber assisted microextraction for the determination of phthalate ester and bisphenol A. Additional fiber modification and longer extraction time were required to achieve better extraction efficiency. Recently, to enhance the performance of SPME, electrical potential was applied to pencil lead fibers for the extraction of the drug methamphetamine in an aqueous sample [56]. In this method pencil lead was conditioned at high temperature (600 °C) for a long time (60 min) before each run. To overcome these challenges and improve the conductivity of the SPME, fibers were functionalized with electrically conductive materials such as multi-walled carbon nanotubes/nafion to determine basic drug in urine samples [57].

In EE-SPME, faster transport of charged analyte from samples toward the surface of the fiber via electrophoresis was observed, which increased the enrichment of analytes on SPME fiber [57]. In our study, for the first time, a single-step EE-SPME method was developed using commercial SPME fiber (without any modification) for the extraction of phthalates and bisphenol A. The extraction performance of the EE-SPME was compared with the conventional SPME methods.

2. Experimental

2.1. Chemicals and materials

A mixture of PAEs and bisphenol A standards were purchased from Sigma-Aldrich (St. Louis, MO, USA). This mixture containing diethyl phthalate (DMP), di-n-butyl phthalate (DBP), butyl benzyl phthalate (BBP), and bisphenol A (BPA) (Fig. 1) at 1000 μg mL^{-1}, was prepared in dichloromethane. A working standard solution was prepared daily by appropriate dilution of stock solution of EDCs in the same solvent. Physical and chemical properties of target analytes are shown in Table 1. Analytical grade solvents were purchased from Supelco (Bellefonte, PA, USA). Double deionized water was obtained from a Milli-Q system (Millipore, Bedford, MA, USA). Sodium hydroxide, sulfuric acid and sodium chloride were obtained from Merck (Darmstadt, Germany). To avoid any carryover of EDCs, all laboratory glassware was washed with concentrated hydrochloric acid and rinsed with deionized water and acetone and dried out in the laboratory oven at 100 °C for 1 h. A manual SPME fiber holder and 30 μm polydimethylsiloxane (PDMS) fibers were also obtained from Supelco (Bellefonte, PA, USA). Prior to use, the fibers were conditioned in the GC injection port in accordance with manufacturer's recommendation. A variable voltage DC power supply was used. Two silver cable wires clipped at each end and a 10 cm long inert metallic wire with a diameter of 0.5 mm were used to complete the electrical circuit.

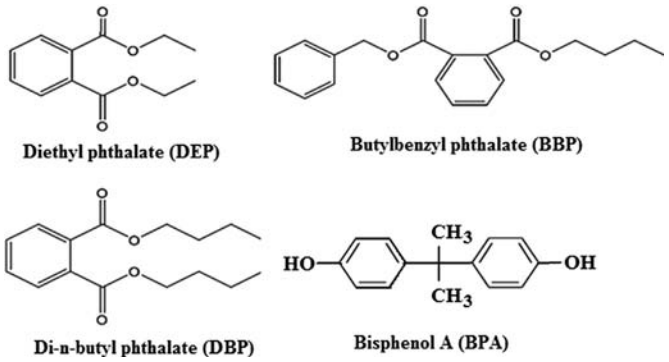


Fig. 1. Molecular structures of three PAEs and bisphenol A.

Table 1
Physical properties of selected EDCs (three PAEs and BPA) [10].

	DEP	DBP	BBP	BPA
Molecular weight (g mol^{-1})	222.24	278.34	312.36	228.29
Density (g mL^{-1})	1.12	1.043	1.0	1.2
Melting point (°C)	−40.5	−35	61.3	159
Boiling point (°C)	295	340	92.5	220
Water solubility (g L^{-1})	1.1	13	3	0.0027

2.2. Blood and seawater samples

Stored blood samples were collected from a blood bank at a local hospital at Al-Khobar, Saudi Arabia. Seawater samples were collected from the coastal area of Al-Khobar in pre-cleaned glass bottles. Blood samples were treated with anticoagulant and stored at 4 °C. Samples were directly extracted using EE-SPME without any further pre-treatment.

2.3. EE-SPME

A 10 mL sample solution spiked with EDCs was placed in a volumetric flask with a magnetic stir bar. The SPME fiber and inert metallic wire were inserted in the sample solution. Both the metallic wire and SPME holder were connected via cable wires to the DC power supply. A positive voltage (+32 V) was applied to the SPME fiber and a negative (−32 V) potential was applied to the inert metallic wire as shown in Fig. 2. The SPME fiber was immersed in the sample solution. Then the sample was agitated at 800 rpm for 20 min. After the extraction, the fiber was thermally desorbed in the GC–MS injection port for 3 min at 290 °C.

2.4. GC–MS analysis

Analyses were carried out using a gas chromatogram (Agilent Technologies, 6890N GC) coupled with a mass spectrometer (Agilent Technologies, 5975B MSD). An HP-1 methyl siloxan column (Agilent 19091Z-213; 30 m × 320 μm I.D. × 1 μm thickness) was used. High purity helium (> 99.999%) was used as a carrier gas and the samples were analyzed in a constant flow at 1.2 mL min^{-1}. The oven temperature program used for the analyses was as follows: the initial temperature was 55 °C held for 15 min which was then increased to 250 °C at 6 °C min^{-1} and held for 2 min. Samples were analyzed in

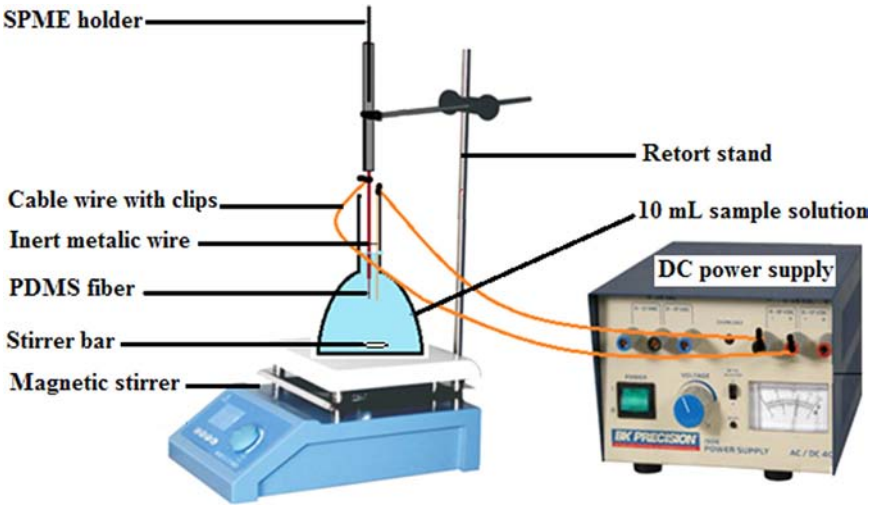


Fig. 2. Schematic of EE-SPME.

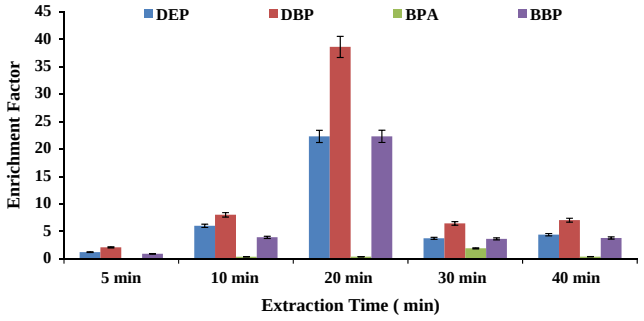


Fig. 3. Effect of absorption time of SPME mode on the enrichment factor of target compounds.

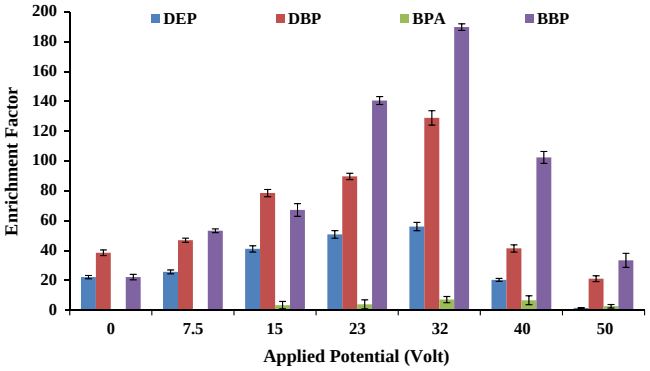


Fig. 4. Effect of voltage on EE-SPME (extraction time 20 min).

splitless mode. For qualitative determinations, the MSD was operated in full-scan mode from *m*/*z* 50 to 550. For quantitative determinations, the MSD was operated in selected ion monitoring (SIM) mode.

3. Result and discussion

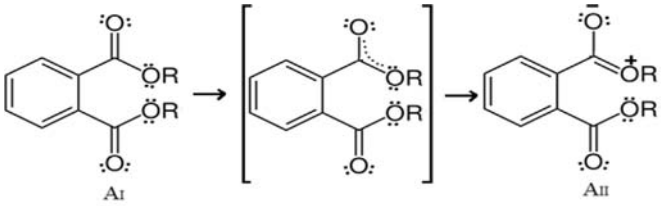
3.1. Extraction time of SPME (without potential)

The optimum absorption time can be obtained when no additional increases in peak areas with further time of extraction are found [50]. The influence of extraction time on the SPME enrichment factor was investigated with the time varying from 5 to 40 min at room temperature and samples being stirred at 800 rpm. Fig. 3 shows the enrichment of PAEs and BPA using direct immersion-SPME (without potential). The enrichment factor for the PAEs and BPA slowly increased as the extraction time varied from 5 to 20 min and tended to reach equilibrium at 20 min. Based on the results, 20 min was selected for the further investigation of applied potential and salt addition studies.

3.2. Effect of applied voltage on SPME

The effect of applied potential on EE-SPME was investigated by plotting analyte enrichment factor as a function of applied potential. Potential varying from 7.5 to 50 V was applied for the SPME method (extraction time was optimized as 20 min). Fig. 4 shows that the enrichment factor for the PAEs and BPA obviously increased as the potential varied from 7.5 to 32 V and then decreased.

The ester groups in the phthalate esters have a partial double bond character due to the delocalization of electrons, as shown in the resonance structures (A_I and A_{II}). The applied potential may enhances the charge formation on the phthalate ester and expedite the extraction process via electrokinetic migration. Without applied potential, the extraction process was slow and only small amount of analytes were extracted by the same SPME fiber (Fig. 4).



R: -C_2H_5 , - C_4H_9 , -benzyl group

Furthermore during extraction, the tip of the SPME holder needle was actually immersed in the sample solution, together with the PDMS SPME-fiber. Thus, a complete electrical circuit was established between the needle and the platinum wire electrode, as shown in Fig. 2. The BPA is relatively polar than PAEs, application of positive potentials made the fiber coating positively charged and therefore enhanced the extraction of deprotonated BPA and PAEs via electrophoresis and complementary charge interaction [57]. At higher potential >32 V, bubble formation on the SPME fiber reduced the active surface area of the polymer

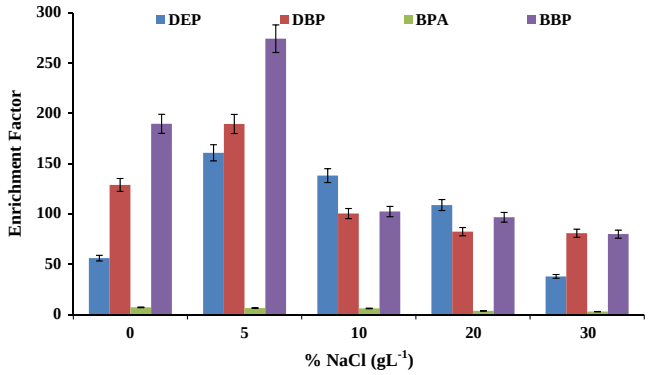


Fig. 5. Effect of % NaCl added to the sample solution (extraction time 20 min; applied voltage 32 V).

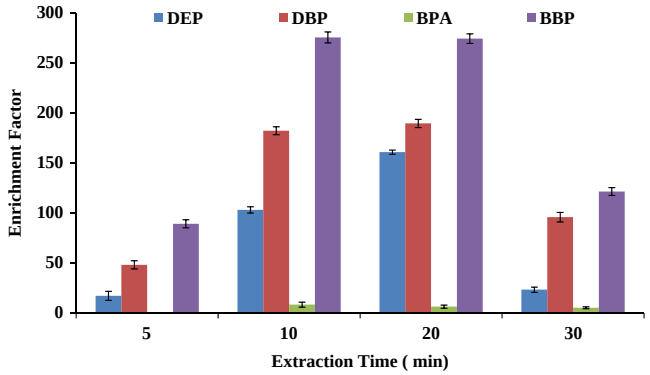


Fig. 6. Effect of absorption time (applied voltage 32 V, 5% NaCl).

Table 2
Features of the EE-SPME method.

Compound	Linearity range (μ gL^{-1})	R^2	Equation	RSDs ($n=9$)[a]	LODs ($S/N=3$)
DEP	1.0–100	0.991	$y=3E-05x+2.4087$	4.5	0.15
DBP	1.0–100	0.996	$y=1E-05x-1.7262$	1	0.004
BBP	1.0–100	0.98	$y=2E-05x+1.9972$	3.3	0.1
BPA	2.0–100	0.963	$y=1.6E-05x-23.6$	5	0.096

Linear range, correlation of determination (R^2), linear equations, relative standard deviations (% RSDs), and limits of detections (LODs) of PAEs and BPA by EE-SPME/GC–MS.

[a] Under repeatability condition.

coating. Thus, an optimum applied potential of 32 V was selected for further analysis.

3.3. Effect of salt addition

To increase the ionic strength and decrease the analyte solubility in the aqueous samples NaCl is often added [48,52,58–61]. The effect of NaCl on the extraction was evaluated from 0% to 30% (w/v). For this, an extraction time of 20 min and an applied potential of 32 V were used. Fig. 5 shows the enrichment factors were highest at 5% of NaCl for all analytes. Increases in the overall ionic strength $>5\%$ of NaCl led to the decrease of the enrichment factors. This could be due to a decrease in the diffusion coefficient of analytes through the increasing viscosity of

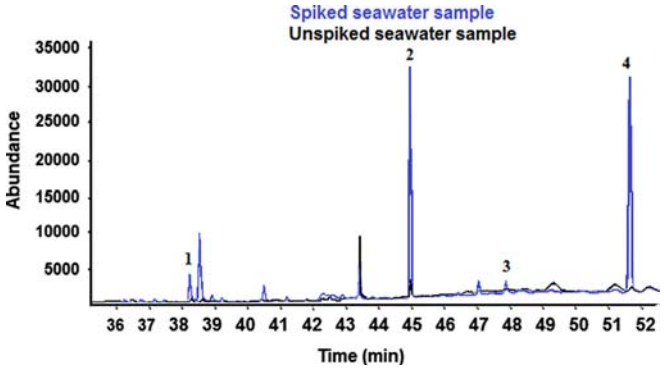


Fig. 7. EE-SPME extracted chromatograms of spiked (20 μg L^{-1} of each analyte) and unspiked seawater samples. Peak identifications: 1—DEP, 2—DBP, 3—BPA, and 4—BBP.

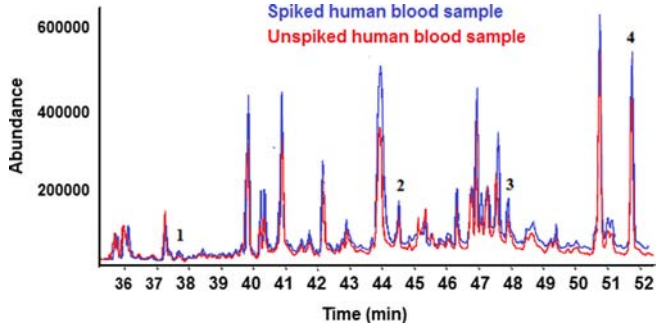


Fig. 8. EE-SPME extracted chromatograms of spiked (20 μg L^{-1} of each analyte) and unspiked blood samples. Peak identifications: 1—DEP, 2—DBP, 3—BPA, and 4—BBP.

Table 3
Comparison of EE-SPME/GC–MS with other reported methods for the determination of PAEs and BPA in liquid samples.

Method	Fiber	Sample	Extraction time (min)	LR (μg L^{-1})	LODs (μg L^{-1})	% RSDs	Ref.
PAEs							
SPME/HPLC-DAD	PDMS	Water	20	–	1.0–2.5	5.0–20	[63]
SPME/GC–MS	PA	Water	90	0.02–10	0.02–0.17	4.2–5.9	[48]
EE-SPME/GC–MS	PDMS	Water	20	1–100	0.004–0.15	1.0–4.5	Present
BPA							
SWCNTs[a]-SPME/GC–MS	Modified	Canned food	40	0.3–60 (μg Kg^{-1})	0.1 (μg Kg^{-1})	–	[55]
SPME/GC–MS	PDMS	Milk	30	1–10	0.01–0.1	4.1–5.8	[7]
SPME/GC–MS	PDMS/DVB	Water	60	0.03–195	0.04–1.0	6–9	[8]
EE-SPME/GC–MS	PDMS	Water	20	2–100	0.096	4.2–5	Present

LR: linearity range; LOD: limits of detection; and % RSDs: relative standard deviations.

[a] Single wall carbon nanotubes.

Table 4
Relative recovery of EDCs from seawater and human blood samples by EE-SPME-GC–MS.

EDCs	Human blood				Seawater			
	Concentration ($\mu g\ L^{-1}$)		% Recovery ($n=3$)	% RSDs ($n=3$)	Concentration ($\mu g\ L^{-1}$)		% Recovery ($n=3$)	% RSDs ($n=3$)
	Real sample	After spiked with 20 ($\mu g\ L^{-1}$)			Real sample	After spiked with 20 ($\mu g\ L^{-1}$)		
DEP	54.5	73.6	95	2.4	6.98	25.66	93.4	5.4
DBP	28.6	47.5	94.4	6.7	7.9	26.03	90.4	3.7
BPA	ND	17.4	87.1	15.4	ND	14.78	73.9	6.2
BBP	24	42.7	93.4	8.0	36.5	54.42	89.6	2.7

the aqueous sample [2,3,12,62]. On the basis of the results, 5% NaCl was added to the aqueous sample for subsequent experiments.

3.4. Extraction profile of EE-SPME

In the electro mediated extraction techniques, application of a potential to SPME was expected to offer faster extraction rates [56]. To determine the optimum extraction time of EE-SPME, different durations of 5, 10, 20, and 30 min were studied. Fig. 6 shows that EE-SPME provides a higher enrichment factor for all the analytes when compared to the conventional SPME (Fig. 3). From the result, 20 min extraction was selected as an optimum time. The lower enrichment factor at 30 min is most likely due to bubbles observed on the fiber at longer extraction times, which inhibit and reduce target analyte absorption; this has been reported previously [56].

4. Analytical performance of the EE-SPME method

To evaluate this method, the linear range, repeatability and limits of detection (LODs) were investigated under the optimized condition. The results are summarized in Table 2. Very good linearity was observed over the concentration range of 1–100 $\mu g\ L^{-1}$ for PAEs and BPA with favorable correlation of determination (R^2) ranging from 0.963 to 0.996. The enrichment factor for the BBP was highest; its average was approximately 274. The repeatability study was carried out by extracting the spiked water samples at different concentration levels of (1, 5, 10, 20, 40, 60, and 100 $\mu g\ L^{-1}$), and the percentage relative standard deviations (RSDs) were between 1.0% and 5.0% ($n=9$). The LODs, based on a signal-to-noise ratio (S/N) of 3, ranged from 0.004 to 0.15 $\mu g\ L^{-1}$. Performance of EE-SPME was compared with those of the other methods reported in the literature and the results are shown in Table 3. Results of PAEs clearly indicate that EE-SPME performance is superior to the conventional SPME, and comparable with LDS-VSLLME [46]. Results obtained for BPA were comparable with previously reported literature (Table 3). The advantages of the EE-SPME/GC–MS over the other methods include high enrichment factor as well as being relatively fast and simple [56–57].

5. Real samples analysis

To demonstrate the feasibility of the EE-SPME/GC–MS method, the optimized conditions were applied to human blood samples (stored in transfusion bags in a local hospital blood bank) and seawater. Ten millimeters of each, seawater and blood sample, were used for the EE-SPME extraction. Figs. 7 and 8 shows the unspiked and spiked extraction chromatograms of seawater and blood samples. PAEs were detected in all samples, the highest concentration of 54.5 $\mu g\ L^{-1}$ of DEP being detected in blood samples, whereas 36.5 $\mu g\ L^{-1}$ of BBP was detected in seawater samples. BPA was not detected in either sample. To assess the matrix effect of the EE-SPME, real samples were spiked with 20 $\mu g\ L^{-1}$ of target analytes and extraction recoveries were calculated (Table 4). Recoveries for PAEs in seawater and blood samples ranged from 89.6% to 95%, while for BPA the range was 73.9–87.1%. BPA has lower solubility in water than other target analytes. Thus it might strongly bind to particles in the sample matrices. The matrix effect influences the lower recoveries of BPA in both samples.

6. Conclusion

In this study, for the first time, an electro-enhanced solid-phase microextraction was developed to determine the concentration of three phthalate esters and bisphenol A in seawater and human blood samples. Various experimental conditions influencing EE-SPME were optimized. The combination of EE-SPME with GC–MS enables PAEs and BPA compounds to be determined at ultra-trace level concentrations. Application of the proposed method reveals the trace level contamination of phthalates and BPA in transfusion blood bags and seawater samples. However, further studies with larger samples are required to better understand the leaching profile of these compounds in blood samples.

Acknowledgments

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