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Simultaneous determination of six phthalate esters in bottled milks using ultrasound-assisted dispersive liquid–liquid microextraction coupled with gas chromatography

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ABSTRACT

A new method was developed for the simultaneous determination of six phthalate esters in bottled milks using ultrasound-assisted dispersive liquid–liquid microextraction (UA-DLLME) followed by gas chromatography–flame ionization detection (GC–FID). 0.8 mL of methanol (dispersant) and 40 μL of CCl_4 (extractant) were injected into 8.0 mL of milk solution and then emulsified the mixture by ultrasound for 2.0 min to form the cloudy solution. Under the optimum condition, the enrichment factors of the analytes ranged from 220 to 270 fold and the recovery ranged from 93.2% to 105.7%. Good linearity was observed for all analytes in a range of 0.8–51 ng g^{-1} with the correlation coefficient (r^2) ≥0.9992. The limits of detection (LODs) based on signal to noise of 3 were 0.64–0.79 ng g^{-1}. The repeatability evaluated as intra-day and inter-day precision (relative standard deviation, RSD) were less than 4.0% (n = 5). The presented UA-DLLME–GC–FID method was successfully applied to determine the six phthalate esters in different bottled milk products.

1. Introduction

Phthalate esters (PAEs) are used primarily as plasticizers in polymeric materials to increase their flexibility through weak secondary molecular interactions with polymer chains. Since they are physically bound to the polymer chains, they can be released easily from products and migrate into the food or water that comes into direct contact [1–3]. PAEs as well as their metabolites and degradation products can cause adverse effects on human health, especially on liver, kidney and testicles [4–7]. Moreover, some recent studies have revealed that PAEs may cause hormone disrupting activities [8,9,2]. Food products contaminated with PAEs has become a matter of public concern in recent years due to the use of plastics as food containers and packaging [10]. However, the reports for monitoring PAEs were mainly focused on the relatively simple samples, such as the contaminated water from its plastic packaging [11]. The penetration of PAEs from plastic packaging into complex samples, such as milk, juice, meat, etc was hardly determined due to the complicated sample matrix and low level of PAEs. Therefore, a sensitive and reliable pretreatment method for analysis of PAEs in bottled milk samples is imperative.

Until now, various pretreatment techniques have been developed to extract PAEs from different samples. Liquid–liquid extraction (LLE) as a traditional pretreatment method suffered from time-consuming and requires large amounts of organic solvent. Solid-phase extraction (SPE) as an alternative to LLE [12] owing to its high flexibility and lower consumption of organic solvent than LLE, however, the cartridge and process is relatively expensive and tedious [13]. In recent years, solid-phase microextraction (SPME) [14,15] and liquid-phase microextraction (LPME) [16,17] had been developed as a solvent-minimized sample pretreatment procedure, in which the analytes are extracted from aqueous or gaseous samples onto a solid porous hollow fiber/membrane/fused silica fiber coated with a stationary phase. From the practical point of view, most of these techniques were non-equilibrium procedures since the time required to reach this state was too long, which was owing to the small contact surface between the sample and the extractant [18,19]. Moreover, the coated fibers are generally expensive, fragile and have limited lifetimes.

Recently, Rezaee et al. [20] developed a novel microextraction technique, termed dispersive liquid–liquid microextraction (DLLME), which is based on a ternary solvent system like homogeneous liquid–liquid extraction and cloud point extraction. In this method, the appropriate mixture of extractant and dispersant is injected rapidly into an aqueous sample by syringe, and then a cloudy solution is formed, which markedly increase the contact surface between phases and reduce the extraction times with increasing enrichment factors. After extraction, the phase separation is performed by centrifugation, and the analytes in the sediment phase are determined by chromatography or spec-

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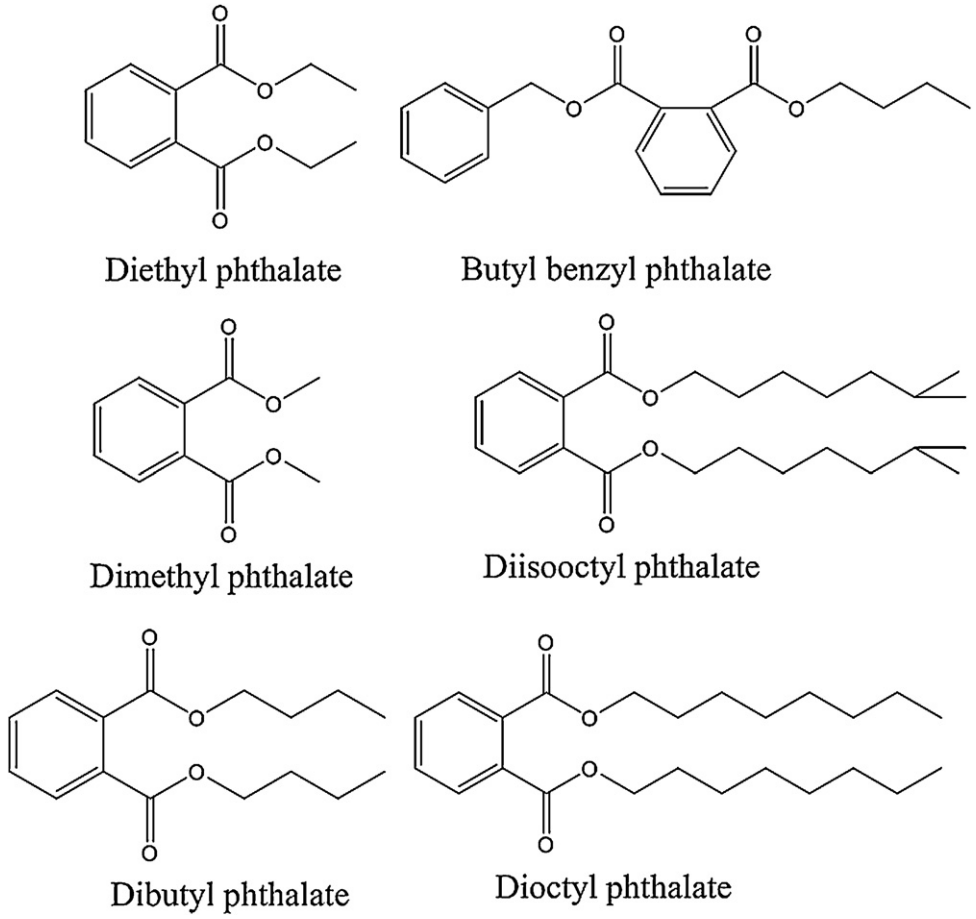

Fig. 1. Molecular structures of the six PAEs.

trometry methods. As the advantages of simplicity, rapidity, low cost, and enrichment factors, the DLLME method had been widely applied for the determination of polycyclic aromatic hydrocarbons [21], organphosphorus pesticides [22,23], chlorobenzenes [24], trihalomethanes [25], chlorophenols [26], and metals ions in aqueous samples [27–30]. However, it still suffered from low repeatability and lack of special selectivity, so it application for complex samples than water was rarely.

This work presented the first attempt to apply the ultrasound-assisted DLLME for extraction and concentration of six PAEs in bottled milk products. After suitable pretreatment procedure and optimization of DLLME for complex samples, the bottle milk sample could be rapidly extracted and analyzed by GC–FID, which is markedly increased the extraction efficiency and reduced the equilibrium time. This method offers a good alternative for routine analysis due to its simplicity and at the same time reliability.

2. Experimental

2.1. Reagents and standards

Dimethyl phthalate (DMP), diethyl phthalate (DEP), dibutyl phthalate (DBP), butyl benzyl ester (BBP), diisooctyl phthalate (DIOP), dioctyl phthalate (DNOP) were obtained from Huaxin Chemical Reagent Co. (Baoding, China) (Fig. 1). Chloroform ($CHCl_3$), tetrachloroethane ($C_2H_2Cl_4$), tetrachloroethylene (C_2Cl_4), chlorobenzene (C_6H_5Cl), and carbon tetrachloride (CCl_4) were purchased from Tianyi Chemical Co. Ltd. (Tianjin, China). All the other reagents used in the experiment were of the highest grade available. The stock solutions were prepared in acetone at a concentration of 0.04 mg mL^{-1}. The working standard solutions were prepared by diluting the stock solution with ultrapure water to get different concentrations in a range of 0.016–1.0 μg mL^{-1}. All the glassware used in the study was previously washed with acetone before using.

2.2. Instrumentation

A gas chromatograph (Shimadzu GC-2014) equipped with a split/splitless injector and a flame ionization detector (FID) (Shimadzu, Japan). High-purity nitrogen (99.999%) was used as the carrier gas, a GH-300 high-purity hydrogen generator and GA-2000A air pump (Beijing ZXHL Technology Development Co. Ltd., China) were used to supply hydrogen and oxygen at the rate of 40 mL min^{-1} and 400 mL min^{-1}, respectively. The capillary column was KB-1 (30 m × 0.25 mm × 0.25 mm, 100% dimethylpolysiloxane as stationary phase, Kromat Co. Delran, NJ, USA) and its column flow rate was set at 1.7 mL min^{-1} with a split ratio of 10. The column temperature programming was as follows: the initial temperature was 150 °C for 2.0 min, and then increased to 285 °C at a rate of 25 °C min^{-1} and held at 285 °C for 10.0 min. The injection port and detector were maintained at 290 °C and 300 °C, respectively. The chromatograms of the six PAEs are shown in Fig. 2. An ultrasonic cleaner (KQ3200E, Kunshan Ultrasonic Instrument, Jiangsu, China) set at 40 kHz was used to emulsify the solutions (20 °C) and a centrifuge (0406-1, Medical Devices, Shanghai, China) was used to accelerate the separation of sediment phases.

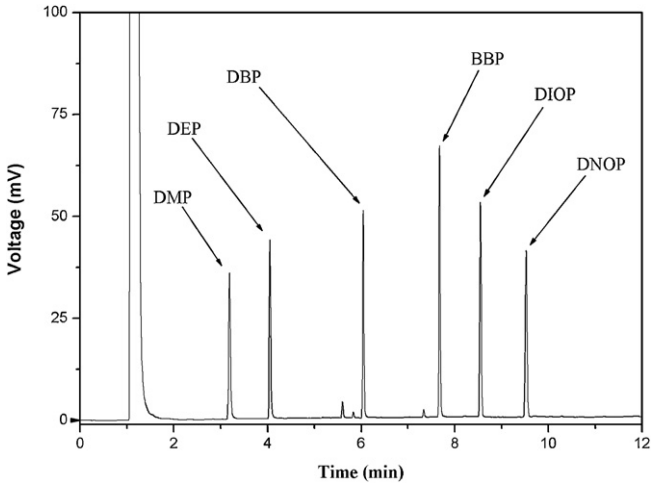

Fig. 2. Chromatograms of the six phthalate esters.

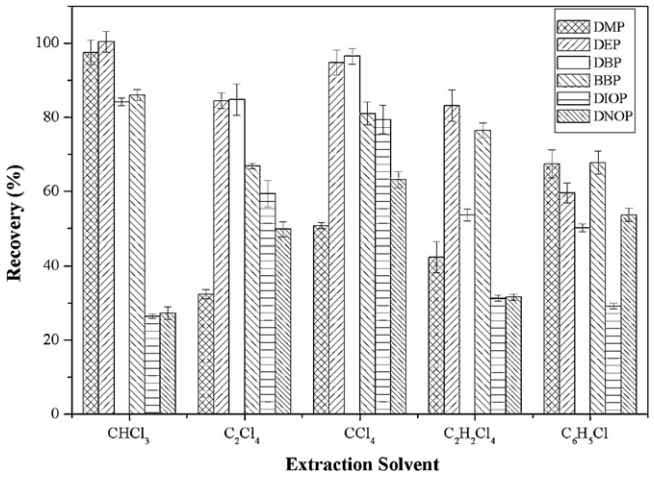

Fig. 3. Effects of extraction solvent on ERs of PAEs.

2.3. Sample pretreatment and DLLME procedure

40 g of the bottled milk samples purchased from local markets of Baoding were mixed with 6.7 mL of 16% (w/v) trichloroacetic acid solution and centrifuged at 4000 rpm for 10 min. The supernatants were further mixed adequately with 4.0 mL of 4% (w/v) lead acetate solution. After centrifuging at 4000 rpm for 10 min, the supernatant (8.0 mL) was placed into a 10.0 mL conical centrifuge tube and mixed with 6% NaCl (w/v). 0.8 mL methanol and 40 μL CCl_4 was injected rapidly into the solution and then the mixture was gently shaken for several seconds and further emulsified by ultrasound for 2.0 min to get the cloudy solution. Finally, the phase separation was performed by a rapid centrifugation at 4000 rpm for 5.0 min and 1.0 μL of sediment phase was injected into GC for further analysis. Enrichment factor (EF) and extraction recovery (ER) were employed for the evaluation of the proposed UA-DLLME. The EF was defined as the ratio between the concentration of analyte in the sediment phase (C_{sed}) and the initial concentration of analyte (C_0) in the sample: $EF = C_{sed}/C_0$. The ER was defined as the percentage of the total analyte (n_0) that was extracted to the sediment phase (n_{sed}):

$$ER = \frac{n_{sed}}{n_0} \times 100 = C_{sed} \times V_{sed}/C_0/V_{aq} \times 100$$

where V_{sed} and V_{aq} are the volume of sediment phase and sample solution, respectively.

3. Results and discussion

3.1. Optimization of the UA-DLLME condition

3.1.1. Selection of extraction solvent

In the UA-DLLME method, there are several factors that would significantly affect the extraction efficiency, such as the type and volume of extractant and dispersant, extraction and ultrasonic time, and pH of the solution. According to the principles of DLLME, the selection of an appropriate organic extraction solvent is the most important in DLLME process since the target analytes should be efficiently extracted and the remaining matrix components should be retained in the matrix. The extraction solvent should be higher density than water and high extraction capability for the target compounds and low solubility in water [31,32]. Therefore, $CHCl_3$, $C_2H_2Cl_4$, C_6H_5Cl, C_2Cl_4 and CCl_4 were investigated by spiking 100 μL of each extraction solvent and 0.6 mL methanol into 8.0 mL milk samples to achieve the sediment phase at the bottom of conical tube. The results in Fig. 3 revealed that CCl_4 presented the highest ERs among the five extractants with a small dosage. Therefore, CCl_4 was selected as the extractant for this work.

3.1.2. Selection of disperser solvent

As the dispersant of UA-DLLME, it should be quite miscible in both the organic phase (extraction solvent) and the aqueous phase (sample solution), so that it can disperse the droplets of extraction solvent into the aqueous phase and increase the surface area between the phases for the mass transferring of target compounds, accordingly improve the extraction efficiency. Thereby, methanol, ethanol, acetonitrile, acetone, isopropanol and tetrahydrofuran were studied by applying 0.6 mL of each disperser solvent containing 100 μL CCl_4 into 8.0 mL sample solution. Fig. 4 showed that the best ERs was obtained using methanol as the disperser solvent, which may be due to its higher dispersing capability for the extractant and relatively less loss for the analytes. Therefore, methanol was selected as the dispersant for further work.

3.1.3. Effect of extractant volume

In order to investigate the effect of extraction solvent volume on extraction efficiency, different volumes of CCl_4 (20–140 μL at 20 intervals) contained in 0.6 mL of methanol were applied to the DLLME procedure. The results in Fig. 5 showed that the recoveries of PAEs increased with the increasing volume of extraction solvent from 20 to 40 μL and then almost kept constant even fur-

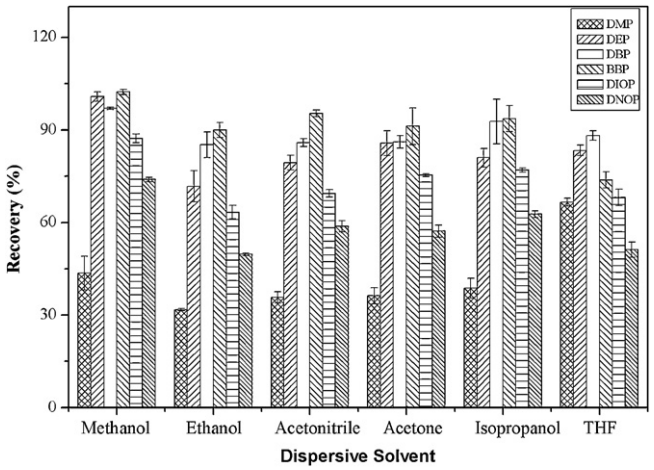

Fig. 4. Effects of disperser solvent on the ERs of PAEs in DLLME.

Table 1
Features of the UA-DLLME method.

Linear equation of analytes		r^2	LOD/ng g^{-1}	LOQ/ng g^{-1}	RSD (%)	EF (fold)
DMP	$y = 2.40 \times 10^5 + 1.33 \times 10^3$	0.9995	0.79	3.69	4.0	226
DEP	$y = 5.74 \times 10^5 + 8.75 \times 10^2$	0.9997	0.75	2.94	3.1	254
DBP	$y = 7.50 \times 10^5 + 1.17 \times 10^3$	0.9993	0.77	2.54	3.0	258
BBP	$y = 9.80 \times 10^5 + 2.62 \times 10^2$	0.9992	0.66	2.17	3.0	270
DIOP	$y = 7.28 \times 10^5 + 1.17 \times 10^3$	0.9997	0.64	2.93	2.8	220
DNOP	$y = 5.65 \times 10^5 + 1.12 \times 10^3$	0.9996	0.76	2.52	3.6	229

ther increased the volume of CCl_4 to 140 μL, which was due to the completed extraction equilibrium. By increasing the volume of CCl_4 from 20 to 140 μL, the volume of the sediment phase was increased accordingly, therefore, the enrichment factor decreased from 102–299 to 30–59 folds. At smaller volume of the extraction solvent, higher enrichment factor was obtained. However, when the volume of CCl_4 was 20 μL, the sediment phase was hard to remove by microsyringe and the reproducibility reduced drastically. Considering the enrichment factor, droplet volume, reproducibility, and extraction recovery, 40 μL of extraction solvent was used in subsequent experiments.

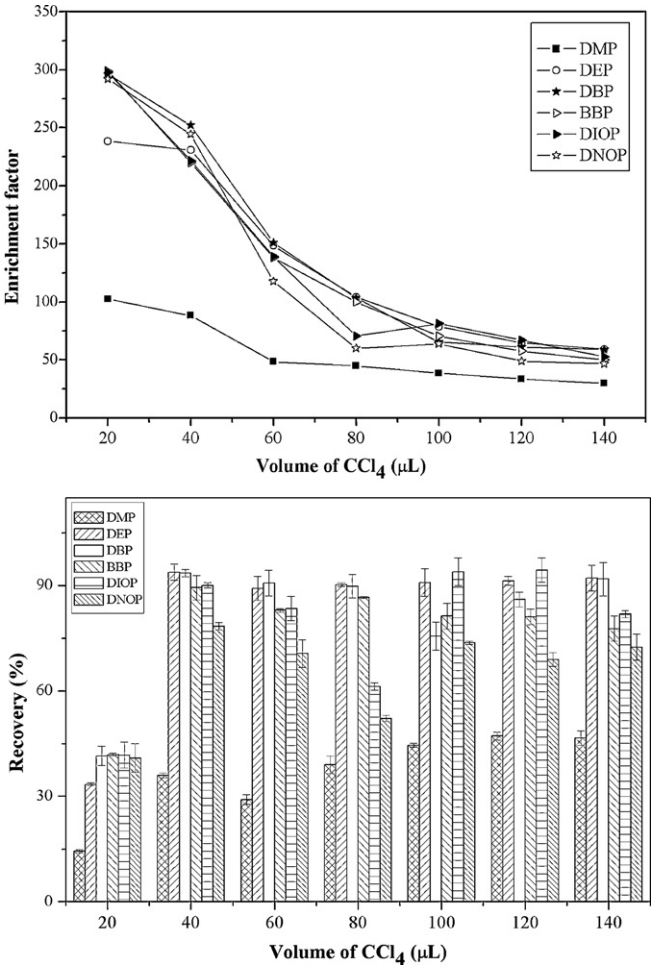

Fig. 5. Effect of extraction solvent volume on EFs and ERs of PAEs.

3.1.4. Effect of dispersant volume

The volume of disperser solvent is a crucial parameter that has an important effect on extraction efficiency. Commonly,

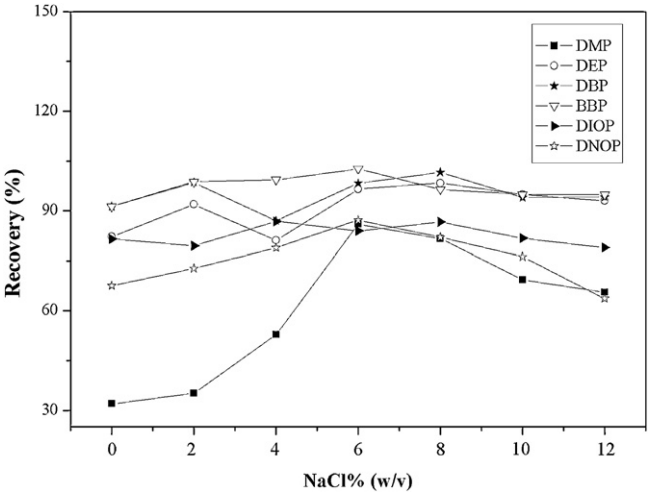

Fig. 6. Effect of salt concentration on ERs of PAEs.

Table 3
The concentration of PAEs in different bottled milk samples (ng g^{-1}).

Milk samples	DMP	DEP	DBP	BBP	DIOP	DNOP
Brand #1	nd	nd	1.61	nd	nd	nd
Brand #2	nd	nd	4.18	nd	2.01	nd
Brand #3	6.40	nd	nd	nd	nd	nd
Brand #4	nd	nd	3.57	nd	nd	nd
Brand #5	nd	nd	5.21	nd	2.36	nd

nd: not detected.

Table 2
Comparison of the presented work with other reported methods.

Sample matrix (mL)	Method	Linearity (μg L^{-1})	Recovery (%)	LOD (μg L^{-1})	RSD (%)	Ref.
Water/5	SPME–GC–MS	0.02–10	–	0.02–0.005	5.6–7	[33]
Water/10	MSPE–GC–MS	0.1–100	91.1–113.4	0.006–0.068	3.4–11.7	[34]
Bottled water/10	SPME–GC–MS	0.1–20	–	0.085–0.003	0.78–10	[35]
Bottled wine/4	SPME–GC–MS	0.03–9.6	81–100	0.4004–0.0214	0.24–5.7	[36]
Water/3.5	SPME–GC–MS	0.02–10	–	0.17–0.006	4.2–14.2	[37]
Aqueous sample/12	MISPME–GC–MS	0.01–10	94.54–105.34	0.0208–0.0022	1.50–8.04	[38]
Mineral water/4	SPME–GC–MS	0.001–10	0–116	0.05–0.001	4–10	[39]
Cow milk/5 g	SPME–GC–MS	–	82–104	3.3–0.31 (ng g^{-1})	5–10	[40]
Milk/9.8 g	DLLME–GC	0.8–51	93.2–105.7	0.79–0.64 (ng g^{-1})	2.8–4.0	Present

SPME: solid-phase microextraction; MSPE: micro solid-phase extraction; MISPME: molecularly imprinted-solid phase microextraction; DLLME: dispersive liquid–liquid microextraction.

Table 4
Recovery of PAEs in bottled milk sample ($n = 3$).

Analytes	Sample/ng g^{-1}	Spiked amount/ng g^{-1}	Detected amount/ng g^{-1}	Recovery (%)	RSD (%)
		4.85	4.59	94.5	
DMP	nd	24.26	22.76	93.8	4.0
		60.67	61.25	100.9	
		4.85	5.00	103.0	
DEP	nd	24.26	24.40	100.6	1.7
		60.67	60.61	99.9	
		4.26	5.92	101.2	
DBP	1.61	21.31	23.15	101.1	1.5
		53.27	54.14	98.6	
		4.55	4.42	97.1	
BBP	nd	21.31	22.52	105.7	4.5
		56.89	56.41	99.1	
		4.02	4.17	103.7	
DIOP	nd	20.10	19.50	97.0	3.4
		50.27	50.06	99.6	
		3.99	3.72	93.2	
DNOP	nd	19.94	19.90	99.8	5.3
		49.86	51.60	103.5	

it is expected that as little as possible is used to achieve the highest EF and the lowest toxicity for environment; on the other hand, at the lower volumes of disperser solvent, tiny droplet formation may not be effective thereby lowering the extraction efficiency. Therefore, various volumes of disperser solvent (0, 0.2, 0.4, 0.6, 0.8, 1.0 and 1.2 mL) were applied to 8.0 mL sample solution, respectively. The results exhibited that with the increase of methanol volume from 0 to 0.8 mL, the ERs increased gradually. It seemed that at low volumes of methanol, the cloudy state was not formed well and therefore resulted in the low recovery. At the volume of methanol higher than 0.8 mL, the PAEs solubility in water increased obviously, which decreased the distribution coefficient between extraction solvent and sample solution. Considering the extraction efficiency, the enrichment factor and volume of sediment phase, 0.8 mL methanol was chosen for further work.

3.1.5. *Effect of ionic strength and the pH of sample*

For investigating the influence of ionic strength on the extraction efficiency of DLLME, different amount of NaCl (0–12%, w/v) were investigated (Fig. 6). With increasing amount of NaCl from 0% to 12%, the volumes of sediment phase increased from 27.6 to 33.6 μL due to the decreased solubility of extraction solvent in sample solution. Moreover, the increase of NaCl from 0 to 6% (w/v) led to an obviously increase in ERs for DMP and DNOP from 32% to 86% and 67% to 87%, respectively. The salting-out effect decreased the solubility of the analytes in water and therefore increased the concentration of analytes in the sediment phase. However, the ERs slightly decreased when the amount of NaCl changed from 8% to 12%. For the other PAEs, the ERs were only slightly changed under the ranged salt concentration. Considering all the factors, 6% of NaCl was selected in the further experiments. The effect of pH was evaluated in a range of 3.0–9.0 and the results demonstrated that the extraction efficiencies were nearly constant within the selected pH, so the sample solution not conducted pH adjustment.

3.2. *Evaluation of UA-DLLME–GC method*

3.2.1. *Features of the method*

To evaluate the proposed UA-DLLME–GC method, the linearity, precision, recovery, the limits of detection (LOD) and limits of quantification (LOQ) were investigated. Calibration curves were constructed using the areas of the chromatographic peaks measured at seven increasing concentrations, in the range of 0.8–51 ng g^{-1}. Good linearity was observed for all analytes throughout the concentration range, and the regression equations were shown in Table 1. LOD based on $S/N = 3$ were ranged from 0.64 to 0.79 ng g^{-1}. Precision and accuracy were determined by analyzing five replicates of spiked samples at three concentration levels on the same day and three different days. The relative stan-

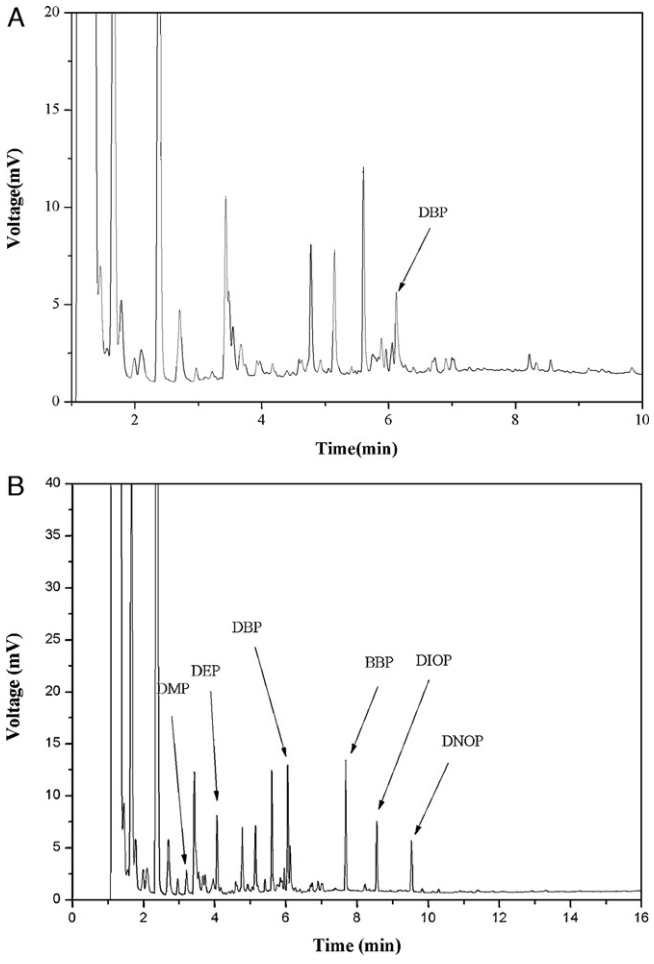

Fig. 7. Chromatogram of milk (A) and spiked milk samples (B) (sample volume, 8.0 mL; CCl_4 volume, 40 μL; methanol volume, 0.8 mL; 6% NaCl (w/v), ultrasonic time, 2.0 min; centrifuging time, 5.0 min; injection volume, 1.0 μL; spiked concentration 20 ng g^{-1}).

dard deviations (RSDs) were in the range 1.5–2.7% for intra-day precision and 2.8–4.0% for inter-day precision, respectively. The enrichment factors for the six PAEs ranged from 220 to 270 fold. The comparison of the presented UA-DLLME–GC method with other reported methods for PAEs determination was shown in Table 2.

3.2.2. Real milk samples analysis

To demonstrate the potential of the proposed UA-DLLME for the selective clean up and concentration of PAEs in real samples, five different brands of plastic bottled milk products were purchased from local markets and dealt with the proposed method. The results in Table 3 shows that trace levels of PAEs were observed in all samples, which indicated the penetration of PAEs from plastic packaging into milk samples was existed. To investigate the effect of sample matrix and the accuracy of the proposed UA-DLLME method for real samples analysis, recovery experiment was carried out by spiking three different levels of target analytes into the samples and the results are shown in Table 4 (Fig. 7). The recoveries ranged from 93.2 to 105.7% with the RSD less than 5.3%, which demonstrate the feasibility of the UA-DLLME–GC method for the determining of PAEs in bottled milk products.

4. Conclusion

In this study, a simple UA-DLLME–GC method for the determination of six PAEs in plastic bottled milk products has been developed. After optimization of DLLME for complex samples, the bottled milk sample could be rapidly extracted and analyzed by GC–FID, which markedly increased the extraction efficiency and reduced the equilibrium time. Under the optimum condition, the enrichment factors for the PAEs ranged from 220 to 270 fold and the recoveries of six PAEs at three spiked levels were in the range of 93.2–105.7%. Adequate repeatability, high recoveries and enrichment factors demonstrated that the method is feasible for quantitative analysis of phthalate esters in real milk samples, and could be used in routine analysis.

Acknowledgements

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References

[1] J.F. Jen, T.C. Liu, J. Chromatogr. A 1130 (2006) 28.
[2] C.A. Staples, D.R. Peterson, T.F. Parkerton, W.J. Adams, Chemosphere 35 (1997) 667.
[3] D. Balafas, K.J. Shaw, F.B. Whitfield, Food Chem. 65 (1999) 279.
[4] H. Zhang, X.Q. Chen, X.Y. Jiang, Anal. Chim. Acta 689 (2011) 137.
[5] P. Liang, J. Xu, Q. Li, Anal. Chim. Acta 609 (2008) 53.
[6] P. Serodio, M.S. Cabral, J.M.F. Nogueira, J. Chromatogr. A 1141 (2007) 259.
[7] J.D. Li, Y.Q. Cai, Y.L. Shi, S.F. Mou, G.B. Jiang, Talanta 74 (2008) 498.
[8] H. Farahani, P. Norouzi, R. Dinarvand, M.R. Ganjali, J. Chromatogr. A 1172 (2007) 105.
[9] S. Jobling, T. Reynolds, R. White, M.G. Parker, J.P. Sumpter, Environ. Health Perspect. 103 (1995) 582.
[10] P. Liang, J. Xu, Q. Li, Anal. Chim. Acta 609 (2009) 53.
[11] H. Yan, B. Liu, J. Du, K.H. Row, Analyst 135 (2010) 2585.
[12] L.H. Zhang, X.Z. Wu, Anal. Chem. 79 (2007) 2562.
[13] C. Mahugo-Santana, Z. Sosa-Ferrera, E. Torres-Padrón, J.J. Santana-Rodríguez, Trends Anal. Chem. 30 (2011) 731.
[14] M.F. Alpendurada, J. Chromatogr. A 889 (2000) 3.
[15] A. Penalver, E. Pocurull, F. Borrull, R.M. Marce, Trends Anal. Chem. 18 (1999) 557.
[16] E. Psillakis, N. Kalogerakis, Trends Anal. Chem. 22 (2003) 565.
[17] K.E. Rasmussen, S. Pedersen-Bjergaard, Trends Anal. Chem. 23 (2004) 1.
[18] G. Shen, H.K. Lee, Anal. Chem. 74 (2002) 648.
[19] F. Ahmadi, Y. Assadi, M.R.M. Hosseini, M. Rezaee, J. Chromatogr. A 1101 (2006) 307.
[20] M. Rezaee, Y. Assadi, M.R. MilaniHosseini, E. Aghaee, F. Ahmadi, S. Berijani, J. Chromatogr. A 1116 (2006) 1.
[21] M.H. Mallah, F. Sheminrani, M.G. Maragheh, Environ. Sci. Technol. 43 (2009) 1947.
[22] S. Berijani, Y. Assadi, M. Anbia, M.R. MilaniHosseni, E. Aghaee, J. Chromatogr. A 1123 (2006) 1.
[23] F. Ahmadi, Y. Assadi, M.M.R. Hosseni, M. Rezaee, J. Chromatogr. A 1101 (2006) 307.
[24] R. Rahnama Kozani, Y. Assadi, F. Shemirani, M.R. Milani Hosseni, M.R. Jamali, Talanta 72 (2007) 387.
[25] R. Rahnama Kozani, Y. Assadi, F. Shemirani, M.R. Milani Hosseni, M.R. Jamali, Chromatographia 66 (2007) 81.
[26] N. Fattahi, Y. Assadi, M.R. Milani Hosseni, E.Z. Jahromi, J. Chromatogr. A 1157 (2007) 23.
[27] E.Z. Jahromi, A. Bidari, Y. Assadi, M.R. MilaniHosseni, M.R. Jamali, Anal. Chim. Acta 585 (2007) 305.
[28] N. Shokoufi, F. Shemirani, Y. Assadi, Anal. Chim. Acta 597 (2007) 349.
[29] P.X. Baliza, L.S.G. Teixeira, V.A. Lemos, Microchem. J. 93 (2009) 220.
[30] F.P. Pereira, I. Lavilla, C. Bendicho, Spectrochim Acta Part B 64 (2009) 1.
[31] T.A. Kokya, K. Farhadi, J. Hazard. Mater. 169 (2009) 726.
[32] P. Liang, E. Zhao, F. Li, Talanta 77 (2009) 1854.
[33] K. Luks-Betlej, P. Popp, B. Janoszka, H. Paschke, J. Chromatogr. A 938 (2001) 93.
[34] J.R. Meng, J. Bu, C.H. Deng, X.M. Zhang, J. Chromatogr. A 1218 (2011) 1585.
[35] X.L. Cao, J. Chromatogr. A 1178 (2008) 231.
[36] J.D. Carrillo, M.P. Mart ınez, M.T. Tena, J. Chromatogr. A 1181 (2008) 125.
[37] A. Penalver, E. Pocurull, F. Borrull, R.M. Marce, J. Chromatogr. A 872 (2000) 191.
[38] J. He, R. Lv, H.J. Zhan, H.Z. Wang, J. Cheng, K. Lu, F.C. Wang, Anal. Chim. Acta 674 (2010) 53.
[39] G. Prokupková, K. Holadová, J. Poustka, J. Hajšlová, Anal. Chim. Acta 457 (2002) 211.
[40] Y.L. Feng, J.P. Zhu, R. Sensenstein, Anal. Chim. Acta 538 (2005) 41.