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RESEARCH ARTICLE

Analysis and contamination levels of ten phthalic acid esters (PAEs) in Chinese commercial bubble tea: a comparison with commercial milk

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Abstract

Phthalic acid ester (PAE) contamination in popular drink bubble tea has been hardly studied in the world. In this work, a liquid–liquid extraction following solid phase extraction (LLE-SPE)-UPLC-MS/MS method was first established for trace determination of ten PAEs in bubble tea. The developed method was validated with respect to linearity ($R^2 > 0.992$), low limit of detections (LODs, 0.49–3.16 μg/L), and satisfactory recoveries (61.8–127.6%) with a low relative standard derivations (RSDs, 1.1–16.4%), which was also validated for commercial milk. Six out of ten PAEs, i.e., diethylhexyl phthalate (DEHP), dibutyl phthalate (DBP), diisobutyl phthalate (DIBP), diethyl phthalate (DEP), dihexyl phthalate (DHP), and diphenyl phthalate (DPP) were detected in Chinese bubble tea with concentrations ranging from not detection (ND) to 53.43 μg/L, while DEHP, DBP, DIBP, DEP, and dimethyl phthalate (DMP) were detected in commercial milk with concentrations ranging from ND to 110.58 μg/L. The respective average concentrations of DEHP in Chinese bubble tea and commercial milk were 19.40 and 23.46 μg/L, which were over two times that in drinking water quality standards of several countries including Israel, Korea, Oman, and Singapore (i.e., 8 μg/L). Calculated with human estimated daily intake (EDI), the average EDIs of five out of seven PAEs in bubble tea were higher than those in commercial milk. For example, the calculated EDI of DIBP in bubble tea was 5 times that in commercial milk, while their respective corresponding EDIs of DBP and DEHP were over 2.4 and 1.6 times. Based on estrogen equivalence (EEQ) with the unit of ng E2/L, the average EEQs of the ten PAEs in Chinese bubble tea and commercial milk were 14.26 and 17.06 ng E2/L, which were 52.8 and 62.3 times the observed effect concentration that could cause egg mortality of zebrafish. It is evident that the potential estrogenic effect of PAEs in bubble tea and commercial milk cannot be negligible. Given the fact that PAE contamination in bubble tea has been hardly investigated, such study is urgently to be performed in a global view.

Keywords Bubble tea · Commercial milk · Human daily intake · Phthalates · Ultra-high performance liquid chromatography-Tandem mass spectrometry · Risk assessment

Introduction

Phthalic acid esters (PAEs) as industrial additives have been increasingly employed (Lee et al. 2019). The world annual usage of PAEs has been supposed to increase by 1.3% annually from 2017 to 2022 (Luo et al. 2018), while the total global production of phthalates in 2018 was reported to be approximately 5.5 million tons (Sahoo and Kumar 2023; Zhang et al. 2022). As not chemically bonded to polymeric materials, PAEs can be migrated from the surface of different industrial products during production, usage, and discharge, posing potential risk to human health (Arfaeinia et al. 2019; De-la-Torre et al. 2022; Sungur et al. 2014). Extensive usage and leachable nature contributed to its ubiquity in air and dust (Ma et al. 2014; Yang et al. 2020),

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aqueous (Hajiouni et al. 2022), soil and sediment (Arfaeinia et al. 2019; Takdastan et al. 2021), biota (He et al. 2020), and food and drink (Kargarghomsheh et al. 2023; Mehraie et al. 2022; Li et al. 2022; Rezaei et al. 2021).

PAEs were widely determined in human serum and adipose tissue, while mono-PAEs were also widely determined in human urine, which illustrated that the vast majority of people around the world are being exposed to PAEs (Liou et al. 2014). Many adverse effects on humans due to exposure of PAEs have been found including endocrine disruption, damage to reproductive, cardiovascular, developmental and immune systems, testicular toxicity, and carcinogenicity (Caldwell 2012; Martino-Andrade and Chahoud 2010; Wang et al. 2014). Due to their potential toxicity, the United States Environmental Protection Agency (US EPA) and the European Union (EU) had listed dibutyl phthalate (DBP), di-(2-ethylhexyl) phthalate (DEHP), diethyl phthalate (DEP), dimethyl phthalate (DMP), di-n-octyl phthalate (DOP), butyl benzyl phthalate (BBP) as priority pollutants. To protect human health, China, the USA, and EU have restricted the use of some PAEs in food packaging material including DEHP, DMP, DEP, DBP, and DOP (Huang and Wang 2016; Jeddi et al. 2015; Wei et al. 2022). Meanwhile, DEHP, a widely used PAE, has been listed in the drinking-water quality standard of several countries including China, Canada, Australia, Israel, Japan, the USA, while DBP and DEP have also been included in the Chinese drinking-water quality standard (Liu et al. 2021).

Food safety and quality have received increasing attention in recent years among Chinese population due to the booming economic growth. Bubble tea generally refers to a beverage containing a tea base, milk, and chewy tapioca starch balls, which has been a popular drinking for young Chinese population. The Chinese bubble tea market had increased at an average rate of over 20% in 2014–2018, with an annual sale of 50 billion of Chinese yuan, i.e., proximately 7 billion USD (Prospective Industry Research Institute 2022). There have been many studies in contaminations of PAEs in bottled water and milk (Dobaradaran et al. 2020; Herrero et al. 2021; Korkmaz and Kuplulu 2019; Liu et al. 2016; Selvaraj et al. 2016; Tang et al. 2019; Zelenkin et al. 2018). However, as a popular drink, the contamination situation of PAEs in bubble tea has not been studied yet, which could be an important exposure source to human. Therefore, the main objectives of this work were (1) to develop a sensitive analytical method for trace determination of PAEs in bubble tea, (2) to investigate contamination situation of PAEs in Chinese commercial bubble tea in which a comparison with milk was performed, and (3) to investigate human daily intake based on both concentration and potential estrogenic effects.

Materials and methods

Standards and reagents

Ten PAEs were selected as the targeted compounds, which included DEHP, DBP, DEP, DMP, BBP, DOP, diisobutyl phthalate (DIBP), dihexyl phthalate (DHP), diphenyl phthalate (DPP), and dibenzyl phthalate (DBzP). Their basic physiochemical properties as well as estrogenic potencies are shown in Table 1. DEHP, DBP, DEP, DMP, BBP, and DOP were purchased from Dr. Ehrenstorfer Gmbh (Augsburg, Germany). DIBP, DHP, DPP, and DBzP were bought from Sigma-Aldrich (USA). Deuterated diethyl hexyl phthalate (DEHP-D4) was used as the internal standard (IS),

Table 1 Ten target compounds and their basic information

Compound (abbr.)	Formula	CAS	Solubility (mg/L, 25 °C)	Saturated vapor pressure (Pa, 25 °C)	log K_{ow}	Estrogenic potency (EP)
Dimethyl phthalate (DMP)	$C_{10}H_{10}O_4$	131–11-3	5220	0.263	1.66	9.804E-6[a]
Diethyl phthalate (DEP)	$C_{12}H_{14}O_4$	84–66-2	591	6.48E-2	2.70	5.747E-6[a]
Dipentyl phthalate (DPP)	$C_{18}H_{26}O_4$	131–18-0	1.29E-2	2.8E-5	3.27	5.97E-7[a]
Dibenzyl phthalate (DBzP)	$C_{22}H_{18}O_4$	523–31-9	–	–	–	1.95E-6[a]
Butyl benzyl phthalate (BBP)	$C_{19}H_{20}O_4$	85–68-7	3.8	2.49E-3	4.73	7.752E-6[b], 2E-4[c]
Diisobutyl phthalate (DIBP)	$C_{16}H_{22}O_4$	84–69-5	6.2	–	4.11	4.52E-6[d]
Dibutyl phthalate (DBP)	$C_{16}H_{22}O_4$	84–74-2	9.9	4.73E-3	4.90	1.01E-6[a], 4.1E-5[c]
Dihexyl phthalate (DHP)	$C_{20}H_{30}O_4$	84–75-3	0.24	6.93E-6	6.82	9.18E-6[d]
Di-2-ethylhexyl phthalate (DEHP)	$C_{24}H_{38}O_4$	117–81-7	3E-3	8.6E-4	7.50	1.735E-6[a], 7.1E-4[d]
Di-n-octyl phthalate (DOP)	$C_{24}H_{38}O_4$	117–84-0	2.49E-3	2.52E-5	8.01	1.7E-4[d]

[a] Céspedes et al. 2004

[b] Zhang et al. 2011

[c] Kim and Ryu 2006

[d] Luo et al. 2020

which was purchased from ANPEL Laboratory Technologies (Shanghai, China) Inc. The purity of each PAE was over 97%. Acetonitrile (ACN), n-hexane, acetic acid, acetone, and methanol (MeOH) were obtained from Fisher Scientific (USA). All solvents were HPLC grade. Sodium chloride was obtained from Sinopharm Chemical Reagent (Shanghai, China), which was purified in a muffle furnace at 450 °C for 6 h. Stock standard solutions of all eleven standards were prepared with MEOH at a concentration of 20 mg/L and stored in a refrigerator at – 20 °C. The mixed working standard solution (ten PAEs) or the IS (DEHP-D4) were prepared with ACN at a concentration of 2 mg/L by diluting the stock standard solution. The brown screw-capped glass tubes were used to hold all the stock standard and working standard solutions. The ultrapure water (18.2 MΩ·cm) was used for cleaning and preparing for mobile phase A.

Sample collection and pretreatment

In total, seventeen bubble tea samples belonging to 17 different brands were purchased via a Chinese popular app called Meituan. To give a comparison, twenty-one commercial milk samples were purchased from a campus supermarket and campus milk reservation service provider in South China University of Technology, Guangzhou, China, which belonged to 14 national famous brands. The sample information of bubble teas and commercial milks were listed in Table 2. The gathered commercial milks were all pasteurized milk whose shelf lives were normally within 1 month under the storing temperature recommended by their manufacturers. Once the bubble tea and commercial milk samples were delivered to the lab, they were immediately extracted with liquid–liquid extraction and then solid phase extraction (LLE-SPE).

The extraction method was based on Bai et al. (2014) that used for extraction of PAEs in milk with some modifications. In brief, 1-mL bubble tea or commercial milk was added to one 5-mL centrifuge glass tube and 100 ng IS was added to the tube. After gentle mixing with hand for three times, 0.1-g sodium chloride and 3-mL acetonitrile with 5% acetic acid solution were subsequently added to the tube, which was vortexed for 1 min, following with ultra-sonication for 20 min. The tube was centrifuged for 3 min at a speed of 4000 r/min, and the upper layer was transferred into another glass tube. The remainder was re-extracted with the same extraction steps as mentioned above. The two upper layers were combined together for further cleanup with SPE using a Si/PSA GLASS Cartridge (500 mg/500 mg, 6 mL), which was purchased from ANPEL Laboratory Technologies (Shanghai) Inc. In the SPE extraction, the aqueous sample was loaded into the cartridge under vacuum, which was preconditioned with 5-mL dichloromethane, 5-mL acetonitrile, and 5-mL ultrapure water. After the loading, acetonitrile (5 mL) and

Table 2 Information of the commercial milk samples and bubble tea samples included in this study

Sample	Brand	Packaging	Shelf life	Sample	Brand	Packaging	Shelf life
CM-01-C	#01	Carton package	30d	BT-15-PP	#15	PP cup	/
CM-01-G-	#01	Glass bottle	14d	BT-16-PP	#16	PP cup	/
CM-01-PET	#01	PET bottle	21d	BT-17-PP	#17	PP cup	/
CM-02-C	#02	Carton package	30d	BT-18-PP	#18	PP cup	/
CM-02-G	#02	Glass bottle	14d	BT-19-PP	#19	PP cup	/
CM-03-C	#03	Carton package	28d	BT-20-PP	#20	PP cup	/
CM-03-G	#03	Glass bottle	5d	BT-21-PP	#21	PP cup	/
CM-04-C	#04	Carton package	30d	BT-22-PP	#22	PP cup	/
CM-04-G	#04	Glass bottle	7d	BT-23-P	#23	Paper cup	/
CM-05-G	#05	Glass bottle	7-14d	BT-24-P	#24	Paper cup	/
CM-05-C	#05	Carton package	21d	BT-25-P	#25	Paper cup	/
CM-06-G	#06	Glass bottle	7d	BT-26-P	#26	Paper cup	/
CM-06-C	#06	Carton package	25d	BT-27-P	#27	Paper cup	/
CM-07-G	#07	Glass bottle	7d	BT-28-P	#28	Paper cup	/
CM-08-C	#08	Carton package	14d	BT-29-P	#29	Paper cup	/
CM-09-C	#09	Carton package	14d	BT-30-P	#30	Paper cup	/
CM-10-C	#10	Carton package	30d	BT-31-P	#31	Paper cup	/
CM-11-PET	#11	PET bottle	21d				
CM-12-PET	#12	PET bottle	21d				
CM-13-PET	#13	PET bottle	30d				
CM-14-PET	#14	PET bottle	25d				

CM commercial milk, *BT* bubble tea, *PET* polyethylene terephthalate, *PP* polypropylene, / not provided acetone (1 mL) were used as the elution solvents to elute the ten target compounds from the cartridge. The eluate was evaporated to dryness under a gentle stream of high purity nitrogen (> 99.99%) at 35 °C. Finally, the eluent was re-dissolved with 1-mL acetonitrile. The sample was added to one brown glass vial for UPLC-MS/MS analysis. Each sample was performed in triplicate.

Sample analysis by UPLC-MS/MS

The ten PAEs were analyzed with a Shimadzu LC-20AD XR series (Kyoto, Japan) coupled to an AB Sciex API 4000 triple quadrupole mass spectrometer (Applied Biosystems, Foster City, CA, USA), which was equipped with an ESI ionization source. The chromatographic separations were achieved with an UHPLC BEH C18 column (2.1 × 100 mm, 1.7 μm). Mobile phase (A) was ultrapure water with 0.1% formic acid and 5 mM ammonium acetate. Mobile phase (B) was acetonitrile. The mobile phase flow rate was set to 0.4 mL/min. The gradient elution program was started at 55% B and continued for 1 min, which then linearly rose to 85% B within 1.5 min. Subsequently, it linearly increased to 97% B within 3 min, which was kept for 2 min. Finally, it linearly returned to 55% B within 0.1 min and maintained for 2.4 min. The entire separation time was 10 min for each injection. The column temperature was maintained at 40 °C, and the sample injection volume was 10 μL. The tandem mass spectrometer (MS/MS) was performed in positive ESI mode with the following operational parameters: MS ion source temperature, 600 °C; ion spray voltage, − 4500 V; curtain gas pressure, 40 psi; collision gas (N2) pressure, 9 psi; gas 1, 85 psi; gas 2, 80 psi. Multiple reaction monitoring (MRM) mode was adopted for quantitative analysis. The optimal MRM parameters for all chemicals are presented in Table 3.

Risk assessment

Daily intake-associated risk assessment

Human daily intake of phthalates via the ingestion of bubble tea or milk can be estimated based on the method of Luo et al. (2018) as shown in Eq. (1):

$$EDI_i = \frac{C_i \times V}{bw} \tag{1}$$

where EDI_i is the estimated human daily intake (EDI) for an individual PAE (ng/kg-bw/day); C_i is measured concentration of the corresponding PAE in bubble tea or commercial milk (μg/L); V is the human daily ingestion volume (L/day/person) of bubble tea or milk; and bw (kg) was the reference body weight for an adult. The latest dietary guidelines for Chinese residents recommend a daily intake of 300–500 g of milk or dairy products (Chinese Dietary Guidelines 2022). The size of bubble tea cup is generally between 350 and 1000 mL (Henan Shuangjiang Paper Plastic Packaging Co. 2022). According to the European Food Safety Agency, micro-pollutants in food should be overestimated to ensure consumer health (Luo et al. 2020), thus, 1000 and 500 mL were used as the V values for bubble tea and milk. Seventy kilogram was used as the reference *bw* value (Huang et al. 2017).

Estrogenic activity-associated risk assessment

Potential estrogenic effects of PAEs in bubble tea or milk were evaluated based on chemically calculated estrogen equivalence (EEQ) (Liu et al. 2009; 2010), i.e.,

$$EEQ = \sum EP_i \times C_i \tag{2}$$

where EP_i and C_i mean the estrogenic potency of an individual PAE and its corresponding average concentration

Table 3 Optimized MRM parameters for the ten target compounds and IS analyzed by UPLC-MS/MS

Compound	Retention time (min)	Precursor ion (m/z, Da)	Product ions (quantifier/qualifier, m/z, Da)	Declustering potential (V)	Collision energy (eV)	Collision cell exit potential (V)
DMP	1.63	195.2	163.1/133.1	60	14/32	14/8
DEP	2.47	223.2	149.1/71.2	55	24/42	8/7
DPP	3.02	319.2	225.0/77.1	70	11/53	17/7
DBzP	3.08	347.1	91.1/181.1	103	22/12	16/10
BBP	3.15	313.2	91.0/149.2	69	29//17	9/8
DIBP	3.18	279.2	149.2/57.1	89	18/30	15/10
DBP	3.22	279.3	149.1/57.2	58	18/35	15/9
DHP	4.58	335.4	149.2/85.4	128	19/25	14/8
DEHP	6.79	319.4	149.1/71.2	82	23/33	8/12
DOP	7.34	319.5	149.1/71.2	81	18/35	14/6
DEHP-D4	6.78	395.5	153.1/10.9	80	25/29	8/3

in bubble tea or commercial milk (μg/L), respectively. 17β-estradiol (E2), the strongest natural estrogenic compound, is often selected as the standard estrogenic compound, for which its EP is arbitrarily set to 1. Hence, the unit of EEQ is defined as ng E2/L. As can be seen in Table 1, some PAEs showed different EP values from different studies. To avoid possible underestimation, the maximum reported EP of each PAE was selected for the EEQ calculation.

Quality control and quality assurance

To avoid possible contamination during sample preparation and analysis, a stringent cleaning procedure was performed as follows: all glassware used in the experiment were carefully cleaned with detergent and thoroughly rinsed with ultrapure water except for the 2-mL brown glass sample vial, and they were immersed in acetone and sonicated for 30 min. Finally, they were thoroughly rinsed with ultrapure water, dried in oven at 100 °C for at least 3 h before use. All solvents employed were checked by UPLC-MS/MS before use to ensure that only those solvents with the least trace residues of PAEs were used. Three blank controls with ultrapure water were performed with the same pretreatment procedure for bubble tea or milk sample as described above. Injections of blank control samples were performed for every 12 real samples, and the average concentrations of blank control samples were obtained for background subtraction. Each sample was performed in triplicate.

Data analysis

The concentrations marked as not detected (ND) were set to zero for statistical analysis. Nonparametric statistical tests were applied to assess the statistical significance because the data were not normally distributed (Shapiro-Wilk test; $p < 0.05$). The concentration levels of PAEs in commercial milk and bubble tea were compared using the Mann – Whitney test. Statistical analyses and all figures were created with Origin 2018. The statistical significance was set at $p < 0.05$.

Results and discussion

Performance of the developed methods

The developed LLE-SPE-UPLC-MS/MS method was validated with respect to linearity, sensitivity, recovery, and precision. Standard calibration curves were established based on seven gradient standard concentrations ranging from 5 to 500 μg/L. Satisfactory linearity ($R^2 > 0.99$) for each target compound based on the internal standard method was obtained (Table 4). LODs and limit of quantifications (LOQs) were calculated based on three times and ten times of the standard deviation (SD), in which the SD values were obtained from the seven repeated injections with the lowest concentration of 5 ng/mL used for the standard calibration curve in blank bubble tea or milk sample. The two kinds of blank samples were obtained by passing the bubble tea or milk sample to a SPE cartridge, through which the background PAEs could be removed. As shown in Table 4, the LODs and LOQs of all ten target PAEs in bubble tea and milk samples were 0.64–3.71 and 2.13–12.39 μg/L, respectively. Due to the presence of background contamination, DBP and DEHP showed higher LODs and LOQs. Compared to milk sample, the ten target PAEs in bubble tea sample showed relative higher LODs and LOQs, and the difference might derive from more complex matrix in bubble tea. Recovery experiments were carried out by spiking each PAE with three known concentrations of 50, 100, and 200 μg/L into real bubble tea or milk sample. The same real bubble tea or milk sample without spiking was also analyzed, and the analyzed values were subtracted for the recovery calculation. The recoveries of the 10 target compounds ranged

Table 4 Linearity, coefficient of determination, LOD, and LOQ for the analysis of PAEs in milk and bubble tea

Chemical	Calibration equation	Coefficient of determination (R^2)	Milk		Bubble tea	
			LOD (μg/L)	LOQ (μg/L)	LOD (μg/L)	LOQ (μg/L)
DMP	$y = 1.8998x + 0.2444$	0.9988	0.78	2.57	0.64	2.13
DEP	$y = 1.4584x + 0.2938$	0.9974	0.49	1.63	0.73	2.44
DPP	$y = 0.5804x + 0.088$	0.9982	0.84	2.86	0.79	3.21
DBzP	$y = 0.4602x - 0.0101$	0.9936	0.69	2.31	0.86	2.92
BBP	$y = 0.5934x - 0.0262$	0.9945	1.27	4.24	1.31	4.36
DIBP	$y = 0.1813x + 0.0656$	0.9992	0.91	3.04	1.27	4.23
DBP	$y = 0.2369x + 0.0178$	0.9966	1.54	8.46	2.69	8.99
DHP	$y = 0.7859x - 0.0984$	0.9958	1.01	3.37	0.82	2.74
DEHP	$y = 0.0926x + 0.0425$	0.9922	3.16	10.55	3.71	12.39
DOP	$y = 1.2232x + 0.2437$	0.9978	0.97	3.24	0.87	2.89

from 61.8 to 118.6% with RSDs ranging from 0.6 to 16.4% (Table 5). The developed analytical method for the ten target compounds in the bubble tea or milk sample showed satisfactory recovery and precision, in which recovery efficiency with 50.0–120.0% and RSD below 20.0% was regarded as acceptable (Tang et al. 2020; Yuan et al. 2019; Wan et al. 2022). All analytical parameters in terms of linearity, sensitivity, recovery, and precision suggested that the developed method is reliable and appropriate for trace determination of the ten PAEs in bubble tea or milk sample.

PAEs in bubble tea and commercial milk

Concentration levels of the ten PAEs in bubble tea and commercial milk samples were shown in Fig. 1, while their original data was provided in Table S1. It could be seen that six out of ten PAEs including DEHP, DIBP, DBP, DEP, DHP, and DPP were detected in the bubble tea samples. Among them, DEHP was the most frequently detected PAE with a detection frequency of 70.6%, followed by DIBP with 47.1%, DBP with 29.4%, DEP with 17.7%, DHP with 11.8%, and with DPP the least with 5.9%. The concentrations of DEHP ranged from 8.64 to 53.43 μg/L in the detected 12 samples with an average concentration of 19.40 μg/L, and the average concentration was over two times that in drinking-water quality standards (i.e., 8 μg/L) in several countries, including Israel, Korea, Oman, Singapore (Liu et al. 2021), which suggested relatively heavy contamination of DEHP in bubble tea. The respective maximum and average concentrations of DBP were 83.6 and 11.6 μg/L, while the respective corresponding concentrations of DIBP were 22.4 and 4.7 μg/L. DEP, DHP, and DPP were only detected in no more than 3 samples and their concentrations ranged from ND-3.76, ND-3.48, to ND-3.31 μg/L, respectively.

For commercial milk samples, five out of ten PAEs including DEHP, DBP, DIBP, DEP, and DMP were detected in commercial milk samples. Similar to bubble tea, DEHP and DBP were the two most frequently detected PAEs, in which their respective concentrations ranged from ND-110.58 to ND-37.19 μg/L with respective average concentrations of 23.46 and 9.5 μg/L. The other three detected PAEs were DIBP, DEP, and DMP, and their respective maximum concentrations were 7.58, 7.13, and 5.14 μg/L. The average concentrations of DEHP, DBP, DIBP, DEP, and DMP in commercial milk samples were 23.46, 9.50, 1.87, 1.49, and 0.89 μg/L, respectively. To give a comparison, related investigations on PAEs in milk samples all over the world in the last decade were summarized. As shown in Table 6, PAEs varied greatly in different milk samples, but DEHP was no doubt the most frequently detected PAE, and on most occasions, it shared the highest maximum concentration.

Table 5 Recoveries of ten target analytes in different real samples

Matrix	Spiking concentration (μg/L)	Recovery efficiencies (%, mean (RSD), $n=3$)									
		DMP	DEP	DPP	DBzP	BBP	DIBP	DBP	DHP	DEHP	DOP
Ultrapure water	50	88.7 (2.3)	89.9 (5.4)	98.4 (3.1)	114.3 (1.8)	116.2 (3.0)	104.6 (8.3)	84.9 (7.4)	104.8 (3.2)	97.6 (5.7)	75.4 (5.3)
	100	93.2 (1.2)	96.2 (7.1)	91.8 (1.6)	79.1 (2.9)	105.8 (2.1)	89.4 (5.9)	67.2 (6.7)	75.9 (3.4)	79.4 (2.4)	84.7 (5.4)
	200	90.3 (3.6)	95.1 (3.9)	102.4 (3.3)	81.5 (1.4)	127.6 (3.4)	99.4 (6.1)	116.3 (3.4)	118.6 (5.4)	91.5 (8.3)	97.1 (2.4)
Commercial milk	50	72.3 (5.8)	74.2 (8.2)	113.4 (4.9)	94.8 (1.7)	111.8 (4.6)	84.6 (11.5)	99.1 (8.4)	73.4 (5.6)	61.8 (14.3)	66.8 (5.9)
	100	77.6 (3.4)	75.4 (6.4)	88.1 (2.7)	116.5 (3.7)	76.7 (8.4)	104.7 (9.7)	107.8 (16.4)	90.7 (3.4)	104.6 (6.4)	69.4 (3.7)
	200	86.6 (4.7)	98.7 (7.6)	92.4 (1.6)	78.6 (2.8)	108.4 (3.3)	114.3 (8.4)	108.3 (6.4)	116.6 (3.9)	79.5 (5.1)	75.4 (8.4)
Bubble tea	50	75.7(6.8)	67.6 (10.8)	94.7 (2.5)	76.1 (2.6)	63.9 (2.8)	76.6 (14.8)	114.5 (7.4)	89.4 (10.4)	104.5 (8.7)	68.4 (9.8)
	100	68.5(5.4)	74.9 (8.3)	86.9 (3.4)	113.7 (1.1)	90.4 (5.1)	115.1 (15.7)	93.4 (14.5)	118.6 (4.1)	116.1 (5.8)	75.1 (8.1)
	200	80.4(3.1)	61.8 (5.5)	90.7 (3.0)	91.6 (3.7)	88.6 (3.9)	95.1 (11.6)	104.8 (13.7)	97.4 (6.3)	115.4 (8.3)	63.7 (5.6)

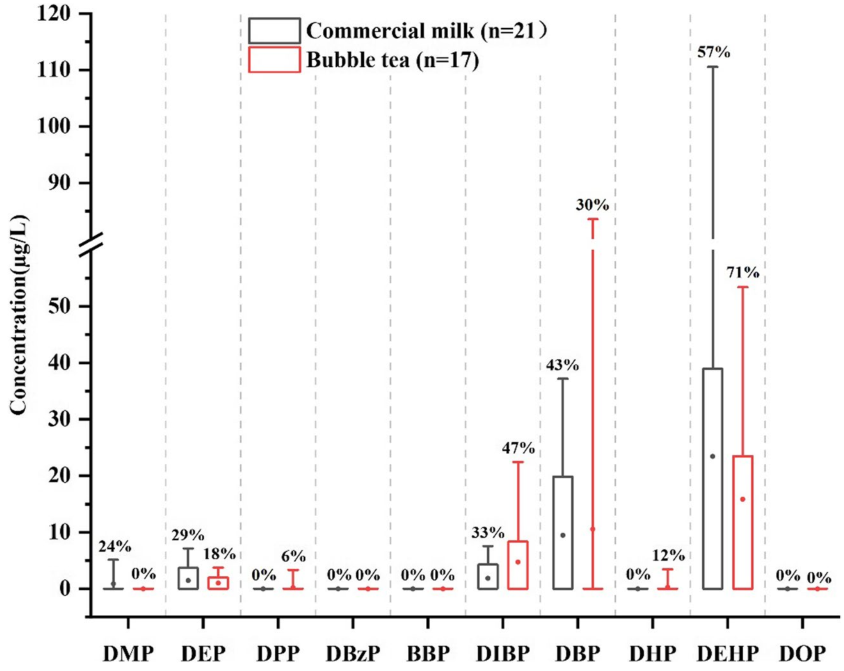

Fig. 1 Concentrations of ten PAEs in commercial milk and bubble tea (the mean values are plotted as filled color circles; the percentage above the column means the detection frequency)

Table 6 Concentrations of PAEs of commercial milk in recent studies (μg/L)*

Country	Sample	Package	DMP	DEP	DIBP	DBP	DEHP	Reference
United States	2	Plastic	0.1[a]	0.17[a]	0.2[a]	1.5[a]	48.6[a]	Schecter et al. (2013)
Spain	42	Metallic aluminum bag, carton packaging, HDPE, metal pail, PET	ND-4.17	ND-25.77	–	ND-6.61	–	Herrero et al. (2021)
Norway	3	Cardboard box	< 0.1	< 1.5	< 0.5	< 0.5	19	Sakhi et al. (2014)
Turkey	5	Printed PE, HDPE	ND-0.58	ND-0.94		1.1–5	0.41–62.6	Tuncel and Senlik (2016)
Saudi Arabia	3	Plastic bottle, tetra pack	ND-25.1	ND	ND	6.3–32.3	ND-10.2	Sajid et al. (2016)
Iran	4	PE packages	ND	ND	ND	72–79	187–201	Farajzadeh et al. (2012)
Iran	24	PET bottle	ND-0.01	ND-0.01	–	0.11–0.56	0.20–0.62	Dobaradaran et al. (2020)
India	7	LDPE/LLDPE	ND-1.35	0.43–54.3	–	3.6–22.6	33.8–656	Selvaraj et al. (2016)
China	3	Plastic, glass, metal	ND	ND	4.8–23.9	ND	6.6–79.3	Lin et al. (2015)
China	5	Plastic bottle	ND-6.4	ND	–	ND-5.21	ND-2.36	Yan et al. (2011
China	14	PE bag	ND-0.76	ND-0.87	ND-1.34	ND-74.1	ND-375	Liu et al. (2016)
China	21	Glass bottle, carton package, PET bottle	ND-5.14	ND-7.13	ND-7.58	ND-37.19	ND-110.58	This study

*The unit was all transformed to μg/L assumed that the density of all milk samples was 1 g fresh weight/mL for convenience of comparison (Selvaraj et al. 2016)

[a] Indicated as mean concentration

ND not detected

Differences were seen in bubble tea and commercial milk. For example, DEHP, DBP, DIBP, and DEP were the top four PAEs ranked from high to low based on average concentration in both bubble tea and commercial milk samples. However, the average concentrations of DEHP and DEP in commercial milk samples were higher than those in bubble tea. On the other hand, the opposite results were observed for DBP and DIBP. Nevertheless, no statistical differences were observed for the four PAEs ($p > 0.05$). Neither were the concentrations of the total ten PAEs (Fig. 2).

The main sources of PAEs in bubble tea or commercial milk include migration from package materials, contamination during production, or product itself. In the commercial milk sample labeled as CM-02, the respective

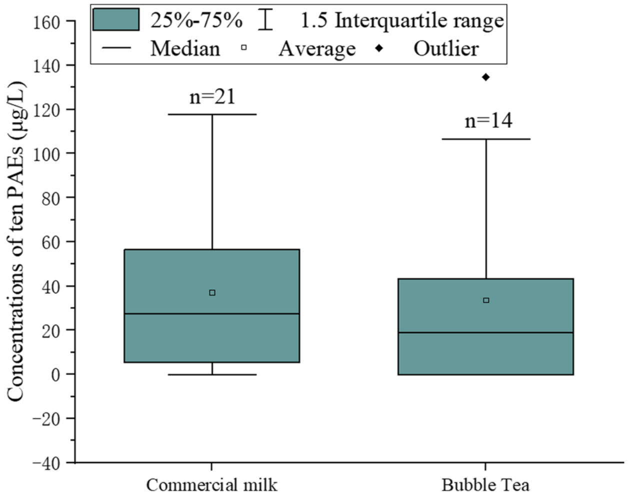

Fig. 2 Difference of total ten PAEs in commercial milk and bubble tea samples ($p > 0.05$) with Mann-Whitney test

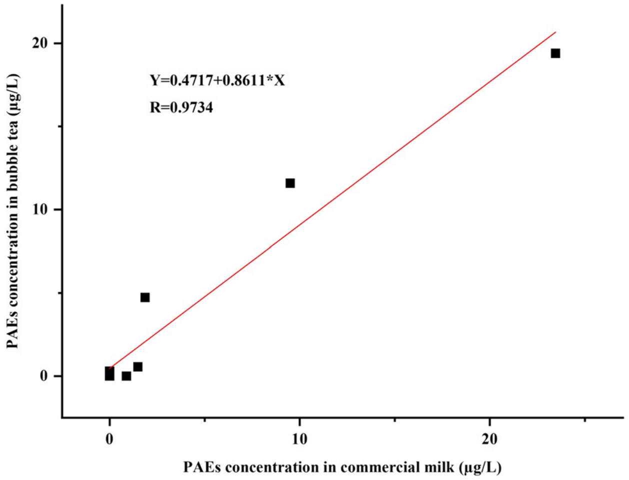

Fig. 3 The relationship between ten PAEs concentration in commercial milk and bubble tea

concentrations of DEP, DBP, DEHP, and DBP were 5.86, 6.77, 12.17, and 16.28 μg/L in carton package, while their corresponding concentrations were 3.67, 4.59, 8.89, and 10.54 μg/L in glass package (Table S1). Similar results were observed in milk sample labeled as CM-03 and CM-06. The above results suggested that PAE migration from package material might be one important source, which agreed well with the work of Herrero et al. (2021). However, it is interesting to find that PAEs in bubble tea and milk samples showed excellent linearity (Fig. 3). As all these samples shared different package materials, it suggested that the most important source of PAEs was likely the product itself or contamination during production.

Health risk assessment

Human daily intake

To evaluate health risk of PAEs in bubble tea and milk samples, the average and maximum concentrations as shown in Fig. 1 and Table S1 were adopted to calculate human's EDI and EEQ. As shown in Table 7, the average EDIs of five out of seven PAEs in bubble tea were higher than those in commercial milk. For example, the calculated EDI of DIBP in bubble tea was 5 times that in commercial milk, while their respective corresponding EDIs of DBP and DEHP were over 2.4 and 1.6 times. The calculated EDI of DEP in bubble tea was similar to that in commercial milk. DPP and DHP were detected in bubble tea but not in commercial milk. The reason can be explained by the serious problem of food additives and preservatives, which have been reported to be harmful to human health (Herrero et al. 2021; Wu et al. 2022). DMP was detected in commercial milk sample but not in bubble tea, the reason was unknown. It should be noted that many young adults likely consume two or more cups of bubble tea per day, thus the actual exposure via bubble tea is likely higher than the calculated EDIs (Huang et al. 2022; Wu et al. 2022). The European Food Safety Authority (ESFA) has established tolerable daily intakes (TDIs) for some PAEs, e.g., 50, 500, 10, and 50 μg/kg bw/d for DMP, DEP, DBP, and DEHP, respectively (Dobaradaran et al. 2020). It was evident that the corresponding EDIs of the seven detected PAEs in bubble tea or commercial milk were far below those of their recommended TDIs. However, when compared to bottled water across 20 countries, as summarized in Luo et al. (2018), the respective average EDIs of DEHP in bubble tea and commercial milk were 2.8 and 1.7 times that in bottled water. The above fact suggests that PAEs in bubble tea and commercial milk are two important exposure sources to human. Compared to lots of investigations on PAEs in bottled water worldwide, PAEs in bubble tea has been hardly investigated, which should be paid with more attention.

Potential estrogenic effects of PAEs

Endocrine disrupting compounds (EDCs) can pose adverse effects on fishes and other animals even at environmentally relevant concentrations, which include intersex, infertility, mortality, and disruption to mating behavior (Liu et al. 2017; Tang et al. 2022). To protect human's health, some EDCs have been listed as the restricted items in the drinking water quality standards of some countries. For example, bisphenol A, DEHP, DEP, and DBP have been listed in the Chinese drinking-water quality standard, while E2 and some other EDCs have been listed in the Japanese drinking-water quality standard (Yuan et al. 2017; 2018). To assess the potential

Table 7 Human EDIs of PAEs via bubble tea and commercial milk

Matrix	Parameter	DMP	DEP	DPP	DIBP	DBP	DHP	DEHP
/[a]	TDIs (ng/kg bw/d)[b]	5×10^4	5×10^5	/	/	10^4	/	5×10^4
Commercial milk	Concentration (μg/L)[c]	0.89–5.14	1.49–7.13	–	1.87–7.58	9.50–37.19	–	23.46–110.58
	EDI (ng/kg bw/d)[c]	6.36–36.71	10.64–50.93	–	13.36–54.14	67.86–265.64	–	167.57–789.86
	EEQ (ng E2/L)[c]	0.00–0.05	0.00–0.04	–	0.01–0.03	0.39–1.52	–	16.66–78.51
	$\text{EEQ}_{\text{total}}$ (ng E2/L)[c]	17.06–80.15						
Bubble tea	Concentration (μg/L)[c]	–	0.56–3.76	0.19–3.31	4.7–22.41	11.58–83.59	0.30–3.48	19.40–53.43
	EDI (ng/kg bw/d)[c]	–	8.00–53.71	2.71–47.29	67.14–320.14	165.43–1194.14	4.29–49.71	277.14–763.29
	EEQ (ng E2/L)[c]	–	0.00–0.02	0.00–0.00	0.02–0.10	0.47–3.43	0.00–0.03	13.77–37.94
	$\text{EEQ}_{\text{total}}$ (ng E2/L)[c]	14.26–41.52						

[a] not applicable or available

[b] the data of TDIs was cited from Dobaradaran et al. (2020)

[c] Data presented as average—maximal concentration

estrogenic effects of PAEs in bubble tea and commercial milk, the average and maximum chemically calculated EEQ levels in bubble tea and milk sample were calculated and summarized in Table 7. The average EEQ levels in bubble tea and commercial milk were 14.26 and 17.06 ng E2/L, while their corresponding respective maximum EEQ levels were 41.52 and 80.15 ng E2/L. Based on the work of Soares et al. (2009), the minimum observed EEQ level of 0.27 ng E2/L could cause the egg mortality in the late gastrulation and/or early organogenesis stage of zebrafish, which suggested that the average EEQ levels in bubble tea and commercial milk were 52.8 and 63.2 times that of the observed effect concentration that could do harm to zebrafish. Moreover, a seven-year long lake experiment illustrated that chronic exposure of fathead minnow to 17α-ethynyl estradiol (EE2) at EEQ level of 7.5–9 ng/L could lead to feminization of male fathead minnow (Kidd et al. 2007; Wang et al. 2020). However, the average chemically calculated EEQ levels in bubble tea and commercial milk were about 2 times that could cause feminization of male fathead minnow. The above facts suggest that the potential estrogenic effects of PAEs in bubble tea and commercial milk should be paid with attention. It should be pointed out that DEHP contributed to over 91% of the total EEQ among the monitored ten PAEs in bubble tea and commercial milk. To decrease the potential estrogenic effect, reduction contamination of DEHP in bubble tea and commercial milk is the most effective way.

Conclusion

This work first established a LLE-SPE-LC–MS/MS method for trace determination of ten PAEs in bubble tea and then investigated their contamination level in seventeen brands of bubble tea in China, along with the contamination situation of the ten PAEs in commercial milk samples. Results of this work suggested that six PAEs including DEP, DPP, DIBP, DBP, DHP, and DEHP were detected in bubble tea, while five PAEs including DEHP, DBP, DIBP, DEP, and DMP were detected in commercial milk. Among them, DEHP was remarkably contaminated in both bubble tea and commercial milk, and its respective average concentrations were over 2.4 and 2.9 times of the regulated concentration in drinking-water quality standard of many countries including Israel, Korea, Oman, and Singapore. The respective average chemically calculated EEQ values of PAEs in bubble tea and commercial milk were 14.26 and 17.06 ng E2/L, which were 52.8 and 62.3 times that could cause egg mortality of zebrafish. The above fact suggests that the potential estrogenic effects of PAEs in bubble tea and commercial milk cannot be ignored. The limitation of this work is that this work only covered 17 bubble tea brands and 14 commercial milk brands, and more brands are necessary to be included so as the overall contamination situations of PAEs in Chinese bubble tea and commercial milk can be more accurately estimated.

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Author contribution De-kang Huang: data analysis and original draft preparation. Ze-hua Liu: supervisor, funding support, and writing review. Yi-ping Wan: sample preparation. Zhi Dang: writing review.

Data availability Original data is provided in supplementary materials.

Declarations

Ethical approval Not available.

Consent to participate All authors have given consent to their contribution.

Consent for publication All authors have agreed with the content and all have given explicit consent to publish.

Competing interests The authors declare no competing interests.

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