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RESEARCH ARTICLE

Several environmental endocrine disruptors in beverages from South China: occurrence and human exposure

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Abstract
Environmental endocrine disruptors (EEDs) in beverages may enter the human body by ingestion and thus may represent a potential health risk. In this study, phthalates, bisphenol A, and its analogues, parabens, benzophenone-type UV filters, and triclosan (TCS) were analyzed in beverage samples ($n = 116$) collected from local markets in Guangzhou, South China. Twelve of 30 target compounds were found in > 50% samples, and for the first time, TCS was found in a majority of beverages from China (~ 80%). Among all analytes, concentrations of total phthalates (median = 14.4 ng/mL) were generally two orders of magnitude higher than other target EEDs, and concentrations of total benzophenone-type UV filters (0.02 ng/mL) and TCS (0.01 ng/mL) were the lowest. Among all targets, phthalates were predominant, accounting for > 99% of the total EEDs, and dimethyl phthalate was frequently detected in beverages (> 60%). In addition, we estimated the daily intake (EDI) of EEDs for Chinese populations of different age groups based on the daily consumption of beverages. The EDIs of total EEDs were the highest for toddlers (mean = 14,200 ng/kg-bw/day) followed by children and teenagers (3420 ng/kg-bw/day), adults (1950 ng/kg-bw/day), the elderly (1740 ng/kg-bw/day), and infants (70 ng/kg-bw/day). Compared to all food categories, EEDs from beverage consumption accounted for ~ 0.1% (parabens) to 20% (phthalates) of total exposure from diet. However, intakes of phthalates, bisphenols, and TCS from beverages were comparable to those from other potential sources (food, dust, personal care products, cloth, and medicines). Furthermore, the cumulative risks of EEDs by beverage consumption were not high, which indicated that EEDs in beverages might not represent a potential human health risk for Chinese populations.

Keywords Phthalate esters · Parabens · Bisphenols · Triclosan · Beverages · Human exposure

Introduction

Phthalates, bisphenol A (BPA) and its analogues, parabens, and benzophenone-type UV filters are several groups of typical environmental endocrine disruptors (EEDs), which may have adverse effects on human health, such as interfering with the body's endocrine system and producing adverse developmental, reproductive, neurological, cardiovascular, metabolic, and immune effects in humans (Casalscasas and Desvergne 2011; Schug et al. 2011). These compounds are widely used as plasticizers or antiseptics in daily consumer products, such as plastics, thermal papers, can linings, and personal care products (PCPs). These EEDs are ubiquitous in our surrounding environment at wide concentration levels, usually from 1.0 to 10,000 ng/g. For example, the EEDs were frequently detected in environmental media in China (detection frequency 50–100%), including indoor dust (Wang et al. 2012), foodstuffs (Liao and Kannan 2014a), PCPs (Guo et al. 2014), and even various papers (Liao and Kannan 2011b).

The EEDs mentioned above are traditionally considered harmless due to their relatively low toxicities. The EEDs' existing reference doses for daily exposure are generally orders of magnitude higher than the values estimated from their concentration levels in potential sources and exposure models. For example, our previous study indicated that the daily exposure doses of diethyl phthalate (DEP) and dibutyl phthalate

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(DBP) were 3.8 and 12.2 μg/kg-bw/day, respectively, for a Chinese population (Guo et al. 2012), but the reference doses (*RfDs*) of DEP and DBP are 800 and 100 μg/kg-bw/day, respectively, as suggested by the United States Environmental Protection Agency (U.S. EPA) (Aylward et al. 2009). However, recent epidemiological studies have found that exposure to those EEDs was associated with some disease. For instances, phthalate exposure was positively associated with premature breast development in girls (Colón et al. 2000); paraben exposure may have a potential relationship to breast cancer (Golden et al. 2005; Casalscasas and Desvergne 2011), and triclosan (TCS) and BPA are weakly estrogenic, associated with changes in the levels of reproductive hormone or low birth weight in infants (Chen et al. 2016). By 2016, the European Chemical Agency has published 169 very high concern substances according to the "Registration, Evaluation, Authorization and Restriction of Chemicals" regulation, and phthalates, such as DBP, diethylhexyl phthalate (DEHP), di-iso-butyl phthalate (DIBP), di-*n*-hexyl phthalate (DNHP), and butyl benzyl phthalate (B_ZBP), were included. In addition, although most of those EEDs are quickly metabolized after entering the human body, since we are exposed to them in daily life, they can be considered as "persistent" to a certain extent. Therefore, human exposure status, related source diagnosis, and health risk assessment of these "low toxic but persistent" EEDs have recently become heavily researched topics again.

Diet is a potential source for human exposure to EEDs, and its contribution to total exposure varies depending on specific contaminants. Our previous studies indicated that diet was the predominant source of high molecular phthalates, e.g., DEHP (Guo et al. 2012). Additionally, diet was the major source of human exposure to BPA, hundreds of times that of nondietary sources (Geens et al. 2012), but diet only contributed a small amount to human exposure to parabens (0.5–1.3%) (Liao et al. 2013b). Several studies reported the occurrence of those EEDs in foodstuffs in China, e.g., phthalates (Guo et al. 2012), parabens (Liao et al. 2013a), bisphenol analogues (Liao and Kannan 2014a), and TCS (Yang et al. 2014). However, as a part of food, data of EEDs in beverages from China were limited (only several samples were collected in previous studies), and little data for benzophenone-type UV filters in foodstuffs were reported (Shen et al. 2009), although the benzophenone-type UV filters may be accumulated in biota (Gago-Ferrero et al. 2012; Gago-Ferrero et al. 2015).

Considering the importance of diet to EED exposure and the limited data for EEDs in beverages in China, in the present study, we analyzed several groups of EEDs (including phthalates, parabens, bisphenol analogues, benzophenone-type UV filters, and TCS) in 116 beverage samples collected in South China. With these data, we aimed to determine the contaminant status of those EEDs in beverages and to estimate human exposure doses from beverages for Chinese populations.

Methods and materials

Standards

Nine standard solutions of phthalates, including dimethyl phthalate (DMP), DEP, DBP, DIBP, B_ZBP, DEHP, DNHP, dicyclohexyl phthalate (DCHP), and di-*n*-octyl phthalate (DNOP), and their corresponding deuterated (d_4) internal standards were purchased from AccuStandard Inc. (New Haven, CT, USA) and/or C/D/N Isotopes (Pointe-Claire, Quebec, Canada). Six paraben standards and nine bisphenol analogues, including methyl paraben (MeP), ethyl paraben (EtP), *n*-propyl paraben (PrP), *n*-butyl paraben (BuP), benzyl paraben (BzP), and heptyl paraben (HepP) and BPA, 4-hydroxyphenyl sulfone (BPAF), 4,4′-(1-phenylethylidene)-bisphenol (BPAP), 4, 4′-methylenedi phenol (BPF), 4-hydroxyphenyl sulfone (BPS), and 4,4′-cyclohexylidene bisphenol (BPZ), 2,2-bis (4-hydroxy-3-isopropylphenyl) propane (BPG), 2,2-bis(2-hydroxy-5-biphenylyl) propane (BPPH), and bis(4-hydroxyphenyl)-diphenylmethane (BPBP), as well as TCS, were purchased from AccuStandard (New Haven, CT, USA). Five benzophenone-type UV filter standards, including 2,4-dihydroxy benzophenone (BP-1), 2,2′,4,4′-tetrahydroxy benzophenone (BP-2), 2-hydroxy-4-methoxy benzophenone (BP-3), 2,2′-dihydroxy-4-methoxy benzophenone (BP-8), and 4-hydroxy benzophenone (4-HBP), were purchased from Sigma-Aldrich (St. Louis, MO, USA). Isotope-labeled ^{13}C-MeP, ^{13}C-BuP, ^{13}C-BPA, ^{13}C-BPS, and ^{13}C-TCS were purchased from Cambridge Isotope Laboratories (Andover, MA). Hexane, methanol, acetonitrile, and HPLC grade water were purchased from J.T. Baker (Phillipsburg, NJ, USA). All standards were > 95% or > 99% pure.

Sample collection and preparation

A total of 116 beverage samples were purchased from local supermarkets in Guangzhou, South China during September 2014. The beverage samples were grouped into four categories, including bottled water ($n = 17$), soft drinks ($n = 53$), juice ($n = 31$), and protein drinks ($n = 15$). Further details of the beverage information are shown in Table S1 ("SI" designates figures and tables in the Supporting Information thereafter). All samples were stored in dark cabinets at room temperature. We hypothesized that contamination of packaging materials (if any) could migrate into beverages during storage, and the worst human exposure situation was when the beverages were consumed when almost past their shelf life. As the longest shelf lives of beverages were almost 24 months (Table S1), all samples were prepared and analyzed for target EEDs from May to September 2016.

Beverage samples (~ 10 g) were ultrasonically extracted with hexane (10 mL) in Teflon tubes at 25 °C for 20 min after spiking with deuterated compounds as internal standards. After shaking another 30 min, the beverage was centrifuged at 4000 rpm for 10 min, and the hexane layer was transferred to a clean glass flask. The sample was re-extracted two times. All the extractions were combined and then concentrated to 0.5 mL for instrumental analysis of phthalates. After determination of phthalates, the solution was concentrated to near dryness under a gentle stream of nitrogen and settled in 10% acetonitrile in water to 0.5 mL for other EED analysis.

Instrumental analysis

For phthalate analysis, the method was similar to our previous study (Guo et al. 2012). Briefly, phthalates were determined using gas chromatography (Agilent Technologies 7890) coupled with mass spectrometry (Agilent Technologies 5977) in the selected ion-monitoring (SIM) mode. A fused-silica capillary column (HP-5 ms; 30 m × 250 μm I.D.; 0.25 μm film thickness) was used for separation. The reporting limits (RLs) were calculated from the lowest concentrations of the calibration curve and a nominal sample weight of 1.0 g. The RL was 10.0 ng/g for DNOP and DEHP and 2.0 ng/g for other phthalates.

The other target EED analysis method was similar to our previous study (Zhang et al. 2018). Briefly, target analytes were quantified with a Shimadzu Nexera-XZ LC system (Shimadzu Corporation Inc., Kyoto) coupled with an AB-Sciex 5500 triple quadruple mass spectrometer (ESI-MS-MS; Applied Biosystems, Foster City, CA). A Betasil C18 column (2.1 mm × 100 mm, 5 μm; Thermo Electron Corporation, Waltham, MA) was used for separation. The instrument was set in multiple reaction monitoring (MRM) negative ionization modes for target EEDs. The RL was 0.1 ng/g for EEDs except for phthalates. The details of instrumental analysis are shown in SI and Tables S2 and S3.

Quality assurance and quality control

To reduce background, all glassware was washed with a soap solution, hot water, and HPLC grade water after leaching with soap solution overnight and dried at 120 °C for 2 h in the oven. Then, the glassware was baked at 450 °C for 4 h in the muffle furnace, wrapped with clean aluminum foil, and kept in a furnace until use. For each batch of ten samples, three procedural blanks (pre-extracted water was used as sample) were processed. Trace levels of phthalates, parabens, TCS, bisphenol analogues, and benzophenone-type UV filters were detected in procedural blanks (Table S4). Reported concentrations were subtracted by the average value of blanks in each batch. In addition, the mean recovery of deuterated phthalates ranged from 77 to 119% and was 94, 99, 114, 73, and 78% for $^{13}C_6$-MeP, $^{13}C_6$-BuP, $^{13}C_{12}$-BPA, $^{13}C_{12}$-BPS, and $^{13}C_{12}$-TCS, respectively. The recoveries of spiked native phthalates, parabens, bisphenols, and TCS in blank spikes ($n = 12$) were 82–113, 74–116, 62–95, and 70%, respectively. Concentrations lower than RLs were set as zero for statistical analysis.

All data are reported on a wet weight basis. Data analysis was conducted by SPSS (Version 22), and a value of $p < 0.05$ was set for statistical significance. Nonparametric tests (Kruskal-Wallis *H* Test and Mann-Whitney *U* Test) were used to compare concentration differences for all target compounds among different beverage groups.

Results and discussion

Detection frequency and concentration of EEDs

Among the 116 samples, at least one individual target compound was found in each beverage. As shown in Fig. 1, DMP (73%), DEP (52%), DIBP (54%), B_ZBP (56%), BPZ (53%), BPG (50%), BPPH (66%), BPF (77%), BPA (54%), MeP (76%), EtP (54%), PrP (65%), BP-8 (50%), and TCS (77%) were frequently detected, with frequencies > 50%, whereas DCHP, DNOP, BPAF, BuP, HePp, BP-1, and BP-2 were rarely detected, with frequencies < 30%. The details for the detection frequencies of target EEDs are shown in Table S5. The detection frequencies of phthalates, parabens, and bisphenols in beverages in the present study were very similar to previous food surveys in China (Guo et al. 2012; Liao et al. 2013a; Liao and Kannan 2014a). For example, DMP, DEP, DBP, DIBP, BzBP, and DEHP were frequently detected phthalates (> 60%), DCHP and DNOP were less detected phthalates (< 16%) in food samples (Guo et al. 2012), and MeP (100%) and EtP (50%) were the most abundant parabens in four beverages from China (Liao et al. 2013a). In addition, for the first time, we found that TCS was frequently detected (~ 80%) in beverages in China. The existence of BPS and BPF in beverages was interesting, as they were BPA analogues to replace BPA in some usage in recent years. As BPF and BPS were observed in many environmental media and commercial products (Wu et al. 2018), it was not surprising that they were detected in beverages.

The detection frequencies of EEDs varied among different beverage categories (Table S5). For example, DBP and DEHP were detected in less than half of bottled water, soft drinks, and juice but found in almost all protein drinks, and similar trends were also observed for other EEDs among categories. However, the detection frequency of DMP and BPF in bottled water was higher or similar to that in other beverages. In a recent beverage study (69 plastic bottled non-alcoholic samples) in China, different from our results, the authors reported that DBP and DEHP were found in almost all samples, but DMP was found in 34% of samples (Yang et al. 2017).

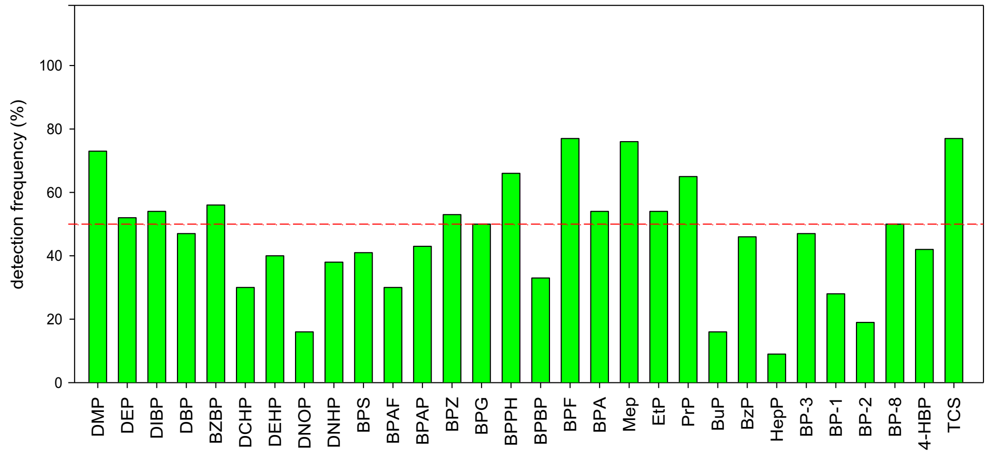

Fig. 1 Detection frequency of target EEDs in beverages from South China (100%). (Red line represents the 50% mark)

The sum of concentrations of nine phthalates, six parabens, nine bisphenol analogues and five benzophenone-type UV filters was defined as $\sum_9$Phthalate, $\sum_6$Paraben, $\sum_9$Bisphenol, and $\sum_5$BP, respectively. In all beverages, concentrations of $\sum_9$Phthalate (median value = 14.4 ng/mL) were the highest (generally two orders of magnitude higher than other target EEDs), followed by $\sum_6$Paraben (0.14 ng/mL) and $\sum_9$Bisphenol (0.18 ng/mL), and the concentrations of $\sum_5$BP (0.02 ng/mL) and TCS (0.01 ng/mL) were the lowest (Fig. 2 and Table S6). As a result, phthalates were predominant, accounting for > 99% of the total EEDs in all beverage categories, except for the protein drink (> 70%). Levels of EEDs were significantly higher in protein drinks than in other categories, except for phthalates (Fig. 3), for which no difference was observed among the four beverage categories. In addition, the concentrations of $\sum_6$Paraben in juice samples were higher than in soft drinks and bottled water ($p > 0.05$), and the concentrations of $\sum_9$Bisphenol, $\sum_5$BP, and TCS were not significantly different.

Both detection frequencies and concentrations of EEDs in beverages may partly reflect their potential contamination sources. Several previous studies demonstrated that some EEDs could migrate from containers into food, such as phthalates in plastic containers or BPA in PVC stretch film, as they were not chemically bonded with those materials (Bosnir et al. 2007; Lópezcervantes and Paseirolosada 2003). Another study conducted by Kondyli reported that concentrations of DNOP in meat were 2 to 80 mg/kg, which indicated a migration rate of 0.12 to 4.8 mg/dm^2 from its plasticizer packaging after 8 days of storage at 4 °C (Kondyli et al. 1992). Additionally, a considerable amount of DEHP and DBP was reported as having migrated from packaging materials into food during storage; their concentrations were ≤ 0.2 to 11.11 and 0.58 to 28.20 mg/kg after 28 days of storage (Soňa and Alžbeta 2015). Therefore, we believe that the migration of EEDs from packaging materials may be an important source of EEDs in our beverages. For example, high detection frequencies and concentrations of EEDs were usually found in protein drinks, in which the migration of EEDs from their packaging materials may be more rapid than other beverages due to the presence of lipid or protein. Notably, as our samples were stored at room temperature for 2 years, the EED concentrations may be different from when beverages were produced because of degradation or migration from packaging. For example, photodegradation of BPA in riboflavin photosensitization can be an efficient way to decrease concentration of BPA in beverages (Ha et al. 2009). A study also reported migration of DEHP from plastic packaging in bottled water was increased with high temperature and long duration of storage time (Jeddi et al. 2015). In addition to migration from packaging materials, several target analytes were reported as added to beverages as additives, e.g., parabens. In China, the maximum allowed usage of parabens as additives in beverages is 200–250 μg/g (National Standard of the People's Republic of China, Food Additives, GB 1886.31-2015). However, we did not obtain extremely high concentrations of parabens in our study. Previous studies reported that parabens were stable in acidic solutions, and hydrolysis occurred above pH 7 (Haman et al. 2015). Most of beverages are acid, and no significantly high

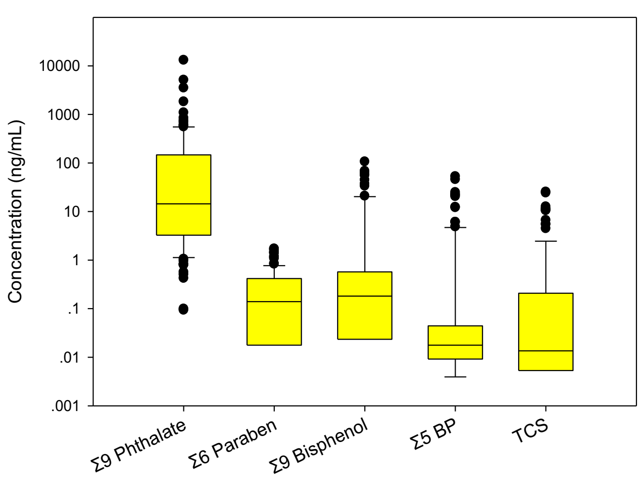

Fig. 2 Concentrations of EEDs in beverages from South China (ng/mL)

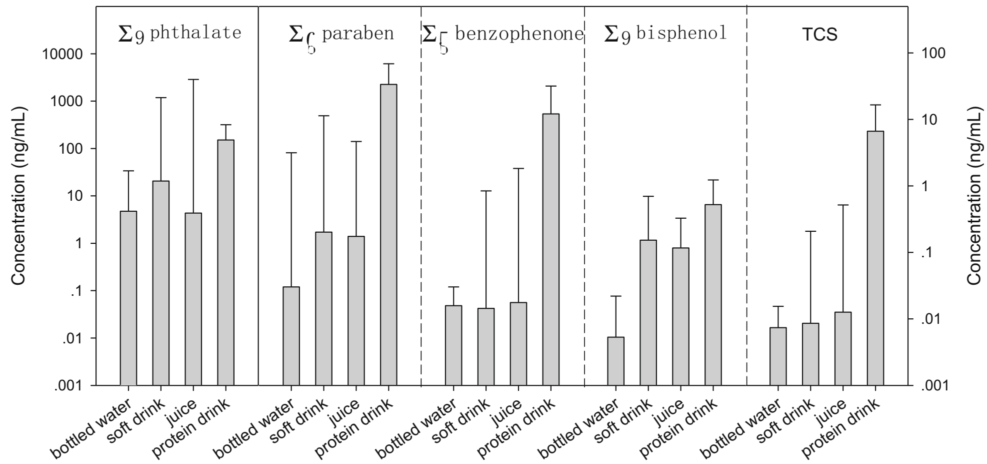

Fig. 3 Concentrations of EEDs in different categories of beverages from South China (ng/mL)

levels of parabens in beverages or food from China were reported in other research (Liao et al. 2013a), so we speculated that the degradation of parabens in beverages may not be serious, and most of parabens were not intentional added as preservatives in our beverages.

Based on categories of beverages, the composition profiles of individual target compounds for each chemical group (expressed as a percentage of the total) were calculated (Fig. 4). For nine phthalates, DMP was predominant in bottled water and juice (> 70%), and DMP was comparable to DEP, DBP, DEHP, and BzBP in soft and protein drinks (~ 20%). For nine parabens, the composition profiles were almost similar in soft drinks, juice, and protein drinks, where MeP was the most abundant compound (~ 40%), followed by BzP, EtP, and PrP (~ 10–20%). The profiles of parabens in bottled water were different from other beverages, for which more MeP (~ 70%) and no BzP were observed. However, the profiles changed a great deal for BPA analogues among the four categories. As shown, BPF was the predominant analogue followed by BPA and BPPH in bottled water and juice, but BPPH and BPS accounted for more than BPF and BPA in soft drink and protein drinks. For five UV filters, BP-3 and BP-8 were predominant in all samples, except protein drinks, in which 4-HBP was the most abundant.

Occurrence of EEDs in beverages reported worldwide

Many previous studies have determined phthalates in beverages worldwide, such as reports from China (Guo et al. 2012), Belgium (Fierens et al. 2012), Canada (Page and Lacroix 1995), Italy (Montuori et al. 2008), the USA (Schecter et al. 2013), and Spain (Casajuana and Lacorte 2003) (Table 1). As shown, phthalates were detected in those beverages from different countries, and DMP, DBP, DINP, and DEHP were

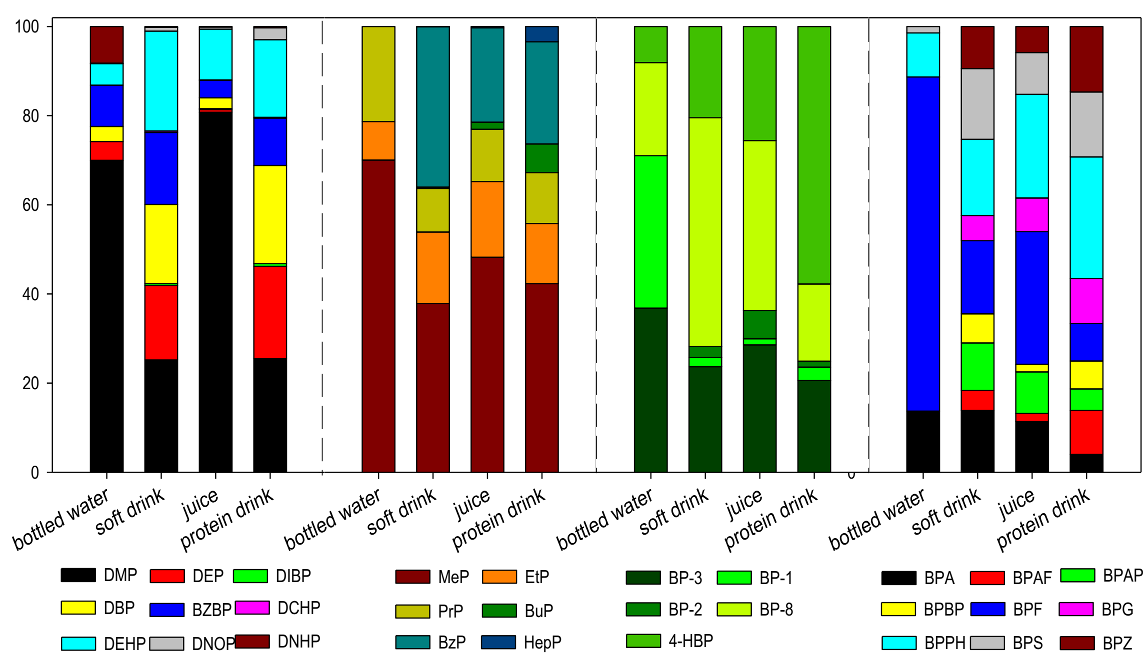

Fig. 4 Composition profiles of EEDs in different categories of beverages from South China (100%)

Table 1 Concentrations of environmental endocrine disruptors in beverages from different countries

Country	Beverage type	Year	Number	Environmental endocrine Disruptors	Ref.
Belgium	Beer/soft drinks/juices/water	2009–2010	85	Phthalates (ng/g, median): DMP = 0.1; DEP = 0.1; DiBP = 0.1; DBP = 0.1; BzBP = 0.1; DEHP = 0.1; DCHP = nd; DnOP = nd	Fierens et al. (2012)
Canada	Bottled water/soft drinks/fruit juices/beer/wine	1987–1989	80	Phthalates (μg/g, range): DEP = nd–0.09; DBP = nd; DEHP = 0.02–0.07	Page and Lacroix (1995)
China	Bottled water/soft drinks/wine/beer	2011	17	Phthalates (ng/g, range): DMP = nd–97; DEP = nd–13.3; DIBP = 0.011–107; DBP = nd–557; DNHP = nd–0.21; BzBP = nd–0.43; DCHP = nd–0.15; DEHP = nd–73.1; DNOP = nd	Guo et al. (2012)
Italy	Water	2007	142	Phthalates (μg/L, mean): DMP = 0.07; DEP = 0.17; DIBP = 0.20; DEHP = 0.02	Montuori et al. (2008)
Japan	Bottled water/beer/ soft drinks/juices/wine	1998	23	Phthalates (μg/mL, mean): DBP = 0.034 ± 0.059; DEHP = 0.032 ± 0.028	Yano et al. (2002)
Korea				Phthalates (μg/mL, mean): DBP = 0.023 ± 0.042; DEHP = 0.018 ± 0.22	
Norway	Soft drinks/bottled water/juice	2012	11	Phthalates (ng/g, range): DMP = nd–0.060; DEP = nd–0.070; DiBP = 0.060–0.88; DnBP = 0.34–0.95; BzBP = nd–0.19; DEHP = 0.17–0.74; DCHP = nd–0.070; DnOP = nd–0.12; DiNP = nd–3.2 Bisphenol analogues (ng/g, range): BPA = nd–0.37	Sakhi et al. (2014)
Portugal	Mineral bottled water	2006	–	Phthalates (μg/L, mean): DMP = nd; DEP = 0.04; DBP = 0.35; DEHP = 0.17	Carmona et al. (2014)
Spain	Drinking water	2002	18	Phthalates (μg/L, mean): DMP = 0.002; DEP = 0.254; DBP = 0.047; DEHP = 0.164	Casajuana and Lacorte (2003)
Turkey	Soda/lemonade/cola/mineral water	2014	10	Phthalates (μg/mL, range): DMP = 0.028–0.238; DEP = 0.015–0.271; DBP = 0.034–0.747; DEHP = nd–2.352; DINP = nd–1.878	Ustun et al. (2015)
USA	Tea/soda/bottled water/juice/sports drink	2011	8	Phthalates (ng/g, mean): DMP = 0.13; DEP = 0.1; DIBP = 0.29; DBP = 0.7; DNHP = 0.10; BBzP = 0.10; DCHP = 0.10; DEHP = 3.89; DNOP = 0.50	Schecter et al. (2013)
China	Juice/liquor/coffee drink	2012	4	Parabens (ng/L, mean): BzP = 0.011; BuP = 0.009; EtP = 0.283; HepP = 0.005; MeP = 0.524; PrP = 0.007; total = 0.839	Liao et al. (2013a)
Spain	Tap water	2012	8	Parabens (ng/mL, mean): MeP = 12; Etp = < 0.3; PrP = 9; BuP = 28	Carmona et al. (2014)
	Bottled mineral water		11	Parabens (ng/L, mean): MeP = 40; EtP = 2; PrP = 23; BuP = 26	
Spain	Tap water	2009	1	Parabens (ng/L, mean): MeP = 40; EtP = < 2.3; PrP = < 1.9; BuP = < 1.8; BzP = < 2.3	Blanco et al. (2009)
USA	Bottled water/soft drinks/wine, beer/juice	2008, 2011, 2012	33	Parabens (ng/g, mean): BzP = 0.016; BuP = 0.007; EtP = 8.53; MeP = nd; PrP = 4.74; total = 0.882	Liao et al. (2013b)
Canada	Beer/soft drinks/juice/wine/bottled coffee	2008	5	Bisphenol analogues (ng/g, mean): BPA = 0.37	Cao et al. (2011)
Canada	Soft drinks/tea/beer/soda/tonic water	2007	72	Bisphenol analogues (μg/L, range): BPA = 0.18–1.2	Cao et al. (2009)
China	Juice/liquor/coffee drink	2012	4	Bisphenol analogues (ng/g, median): BPA = 7.84; BPAF = < 0.01; BPAP = < 0.01; BPB = 0.01; BPF = 0.03; BPP = 0.01; BPS = < 0.01; BPZ = 0.03; total = 7.93	Liao and Kannan (2014a)

Table 1 (continued)

Country	Beverage type	Year	Number	Environmental endocrine Disruptors	Ref.
China	Soft drinks/cola/tea/energy drink	2012	8	Bisphenol analogues (μg/mL, range): BPA = nd–0.86	Li et al. (2012)
Italy	Energy drink	2016	40	Bisphenol analogues (ng/mL, mean): BPA = 1.2; BPF = 0.82; BPB = nd	Gallo et al. (2017)
New Zealand	Beverages	2003–2004	4	Bisphenol analogues (ng/g, value): BPA = < 10	Thomson and Grounds (2005)
Portugal	Soft drinks/beer	2009	30	Bisphenol analogues (ng/mL, range): BPA = nd–4.7; BPB = nd–0.17	Cunha et al. (2011)
Spain	Soft drinks/soda/beer/ tea	2011	11	Bisphenol analogues (ng/mL, range): BPS = nd; BPF = nd–0. 218; BPE = nd; BPA = nd–0.607; BPB = nd	Alabi et al. (2014)
Spain	Tonic water/sports drink/tea/soda/beer	2012	10	Bisphenol analogues (ng/mL, range): BPF = nd–0.26; BPA = nd–0.68; BPZ = nd–0.09	Gallart-Ayala et al. (2011)
Turkey	Juice/milk	2012	12	Bisphenol analogues (ng/g, range): BPA = 42.3–156	Sungur et al. (2014)
USA	Bottled water/soft drinks/fruit juice/beer/wine	2008–2012	31	Bisphenol analogues (ng/g, mean): BPA = 0.235; BPAF = 0.006; BPAP = 0.005; BPB = 0.013; BPF = 0.025; BPP = 0.025; BPS = 0.007; BPZ = 0.025; total = 0.34	Liao and Kannan (2013)
China	Milk (carton, plastic)	2009	18	Benzophenones (ng/g, range): benzophenone = nd–2.84	Shen et al. (2009)
Italy	Milk/fruit juice/wine	2008	40	Benzophenones (μg/L, range): benzophenone = 5–217	Sagratini et al. (2008)
China	Mineral water/pure drinking water	2009	21	Bisphenol analogues (ng/L, median): BPA = 82.4; triclosan (ng/L, median) = 1.5 ng/L	Li et al. (2010)
Taiwan	Tap water/drinking fountain water	2011–2013	87	Triclosan (ng/L, range): nd–103 (ng/L)	Yang et al. (2014)
USA	Drinking water	2012	8	Triclosan (ng/L, median): 1.4 = ng/L	Padhye et al. (2014)

frequently detected congeners. The occurrence of phthalates in beverages is usually attributed to the migration of phthalates from packaging materials (Cao 2010; Rudel et al. 2011). The migration of phthalates from packaging materials may also be partly reflected by the relatively high concentrations in beverages collected earlier when phthalates were not regulated in packaging materials. For example, the concentration of DEHP was 20 to 70 ng/g in beverages collected in 1987–1989 in Canada (Page and Lacroix 1995), and concentrations of DBP and DEHP were ~ 20–30 ng/mL in beverages collected in 1998 in Korea and Japan (Yano et al. 2002). In addition, we also found that phthalate concentrations in protein drinks, including milk and lactic acid drinks, were higher than those in other categories. A recent study has reported that amounts of phthalates in whole milk packed in plastics were higher than those in glass or metal, indicating leaching of phthalates from packaging materials (Lin et al. 2015). Therefore, concentrations of phthalates in beverages depended on food characteristics, store condition, and packaging materials (Gartner et al. 2009). Due to the migration of EEDs into food, the usage of EEDs in food packaging materials was limited by legislation, such as phthalates in food packaging in European countries (EU 2007/19/EC). In China, packaging for lipid food or baby food is not allowed to add phthalates, and in polyvinylidene chloride plastic, the maximum amount of DIBP or DBP is 10% (National Standard of the People's Republic of China, GB 9685-2008).

Similar to our study, MeP, PrP, BuP, and BzP of parabens and BPA and BPAF of bisphenols were also frequently detected in beverages collected from other countries, such as Spain (Carmona et al. 2014), the USA (Liao and Kannan 2013; Liao et al. 2013b), Canada (Cao et al. 2009), Italy (Gallo et al. 2017), and Portugal (Cunha et al. 2011) (Table 1). In those reports, parabens and bisphenols were frequently detected in beverages, with concentrations of MeP, BuP, and BPA several times higher than others. However, studies of TCS and benzophenone-type UV filter in beverages were limited. The concentrations of $\sum_5$BP were nd–52.8 ng/mL in the present study in the range of the beverages collected from Italy in 2008 (5–217 ng/mL) (Sagratini et al. 2008). In our study, the mean concentrations of TCS were 0.01, 0.08, 0.16, and 8.73 ng/mL in bottled water, soft drinks, protein drinks, and juice, respectively. TCS was also detected in drinking water from Taiwan (nd–103 ng/mL) (Yang et al. 2014) and the USA (median = 1.4 ng/mL) (Padhye et al. 2014).

Human exposure to EEDs from beverages

Daily exposure doses of EEDs from the consumption of beverages were estimated for Chinese populations of different age groups with the following equation (U.S. EPA 2011) based on their mean, median, and 95th concentration values.

$$\mathrm{EDI} = \frac{C\mathrm{w} \times Q\mathrm{w} + C\mathrm{b} \times Q\mathrm{b}}{\mathrm{BW}} \times R\mathrm{uptake} \qquad (1)$$

where EDI (ng/kg-bw/day) represents the estimated daily intakes of EEDs from beverages, C_w and C_b (ng/g) represent the concentration of target EEDs in drinking water and beverages, respectively, Q_w and Q_b (g/day) represent the average amount of daily ingestion of drinking water and beverages, respectively, R represents the gastrointestinal uptake factor, and BW (kg) represents body weight. For EDI calculation, considering the diet characteristics of different age groups, we grouped the population into five age groups: infants (< 1 year), toddlers (1–5 years), children and teenagers (6–17 years), adults (18–59 years), and the elderly (≥ 60 years). For the daily consumption of water and beverages, we used values reported in the exposure handbook of China (Duan 2013). We assume human exposure to EEDs from both water and beverage consumption but water only for infants. The details of all parameters used for EDI calculation are shown in Table S7.

Generally, values of EDIs were much higher calculated from the mean than those from the median concentrations of EEDs in beverages (Table 2). For all EEDs, toddlers were exposed to the highest levels by the consumption of beverages (based on mean concentrations), followed by children and teenagers, and infants were exposed to the lowest levels. The EDIs of EEDs were very similar for adults and the elderly. However, the EDIs of EEDs for infants were lower than toddlers but higher than other age groups if calculated from median concentrations of EEDs in beverages. Similar to their concentration distributions, the EDI of phthalates were usually 2 to 4 orders of magnitude higher than those of other EEDs.

The mean EDIs of Σ_9Phthalate were 13,700, 3290, 1890, 1860, and 699 ng/kg-bw/day for toddlers, children and teenagers, adults, the elderly, and infants, respectively. The EDIs of Σ_9Phthalate from beverages were much lower than the RfDs suggested by the U.S. EPA (800, 100 and 20 μg/kg-bw/day for DEP, DBP and DEHP, respectively) (Aylward et al. 2009) and lower than the tolerable daily intake (TDI) proposed by the European Food Safety Authorities (EFSA, 10 and 50 μg/kg-bw/day for DBP (EFSA 2005a) and DEHP (EFSA 2005b), respectively). The low exposure levels of phthalates indicated that beverage consumption was safe if considering only phthalate concentrations in beverages. Similar to phthalates, the EDIs of Σ_9Bisphenol for all age groups were lower than the RfDs of BPA (50 μg/kg-bw/day) recommended by EFSA (2006) and the U.S. EPA (2002), and the median EDIs of Σ_9Bisphenol were 15.5, 4.26, 2.79, 2.65, and 2.40 for toddlers, children and teenagers, adults, the elderly, and infants, respectively.

As a part of the diet, beverage consumption accounted for ~ 20% of total phthalate exposure (233 vs. 1150 ng/kg-bw/day) (Guo et al. 2012), ~ 0.1% of total paraben exposure (0.90 vs. 1010 ng/kg-bw/day) (Liao et al. 2013a), and ~ 0.4% of total bisphenol exposure (2.79 vs. 646 ng/kg-bw/day) (Liao and Kannan 2014a) from the diet in Chinese adults, respectively (Table 3). Compared with other potential exposure sources, intakes of phthalates from beverages were comparable to those from PCPs (Guo et al. 2014) and dust (Guo

Table 2 Estimated daily intakes of several environmental endocrine disruptors from beverages for Chinese population of different age groups (ng/kg-bw/day)

		Σ_9Phthalate	Σ_6Paraben	Σ_9Bisphenol	Σ_5BP	TCS	Total
Infants	Mean[a]	699	0.63	2.40	1.01	0.57	70
	Median	276	0.31	2.40	0.92	0.43	280
	95th	1880	1.67	4.47	2.21	1.00	1880
Toddlers	Mean	13,700	12.7	394	94.6	53.2	14,200
	Median	1030	6.60	15.5	1.54	1.03	1050
	95th	30,100	53.6	2940	770	396	34,300
Children and Teenagers	Mean	3290	3.06	89.1	21.5	12.1	3420
	Median	323	1.58	4.26	0.65	0.37	329
	95th	7370	12.5	660	173	89.0	8300
Adults	Mean	1890	1.75	47.2	11.50	6.46	1950
	Median	233	0.90	2.79	0.55	0.30	238
	95th	4290	6.95	346	91.1	46.8	4780
The elderly	Mean	1680	1.55	40.2	9.85	5.54	1740
	Median	230	0.80	2.65	0.58	0.30	235
	95th	3860	6.08	294	77.4	39.7	4270

[a] Estimated daily intakes calculated from mean, median, or 95th concentrations of EEDs in beverages

Table 3 Daily exposure doses of EEDs from various potential sources for Chinese adults (median value, ng/kg-bw/day)

	Beverages	Diet	PCPs	Dust	Cloth	OTC medicines	Paper currency
Phthalates	233	1148[a]	742	186	–[b]	5.66	–
Parabens	0.90	1010	305,000	0.2	–	–	0.004
Bisphenols	2.79	646	1.17	0.78	–	–	$0.028 \times 10^{-3,\ d}$; $9.79 \times 10^{-4,\ e}$
Benzophenones	0.55	–	16.0[f]	0.062	$(6.39–7.90) \times 10^{-3,\ c}$	–	–
TCS	0.30	–	0.26	0.11	–	–	–

OTC medicines over-the-counter medicines

[a] Data source: diet (phthalates (Guo et al. 2012), parabens (Liao et al. 2013a), and bisphenols (Liao and Kannan 2014a)), PCPs (phthalates and paraben (Guo et al. 2014), bisphenols and TCS (mean value) (Liao and Kannan 2014b), benzophenones (Liao and Kannan 2014d) (mean value)), dust (phthalates (Guo and Kannan 2011), parabens (mean value) (Wang et al. 2012), bisphenols (Liao et al. 2012a), benzophenones (Wang et al. 2013), and TCS (Ao et al. 2017)), cloth (benzophenones (Xue et al. 2017)), over-the-counter medicines (phthalates (Jia et al. 2017)), and paper currency (parabens (mean value) (Liao and Kannan 2014c), and bisphenols (Liao and Kannan 2011a; Liao et al. 2012b))

[b] Not available (–)

[c] Data for infants

[d] Data for BPS

[e] Data for BPA

[f] Data for BP-3

and Kannan 2011) but considerably higher than that from over-the-counter medicines (Jia et al. 2017). Intakes of parabens or bisphenols from beverages were comparable to that of dust (Liao et al. 2012a; Wang et al. 2012) and higher than that of paper currency (Liao and Kannan 2011a; Liao et al. 2012b; Liao and Kannan 2014c) but much lower than those from PCPs (Guo et al. 2014; Liao and Kannan 2014b). In addition, intakes of TCS (0.30 ng/kg-bw/day) from beverages were on the same level as dust (0.11 ng/kg-bw/day) (Ao et al. 2017). Therefore, beverage consumption is an important source of human exposure to phthalates, bisphenols, and TCS.

Cumulative risk assessment

To evaluate the cumulative effects of compounds with similar toxic mechanisms, we calculated the hazard quotient (HQ) and hazard index (HI) for several target EEDs (Benson 2009). The value of HI < 1.0 was used, as no adverse health effects are expected as a result of exposure caused by EEDs. A potential risk is indicated when HI ranges from 1.0 to 100, and it is not safe when the HI value > 100. At the same time, an HQ > 1.0 is identified as a higher intake above the suggested dose. The details for the calculation methods are shown in SI.

Table 4 Values of HQs and HIs of phthalates and BPA from beverage consumption calculated for different age groups for Chinese population

	Infants	Toddlers	Children and teenagers	Adults	The elderly
$HQ_{DEP\ RfD}$	< 0.001	0.002	< 0.001	< 0.001	< 0.001
$HQ_{DBP\ RfD}$	< 0.001	0.016	0.004	0.005	0.002
$HQ_{DIBP\ RfD}$	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
$HQ_{DEHP\ RfD}$	0.002	0.116	0.026	0.041	0.012
$HQ_{BPA\ RfD}$	< 0.001	< 0.001	0.012	< 0.001	< 0.001
HI_{RfD}	0.002	0.134	0.042	0.047	0.014
$HQ_{DBP\ TDI}$	0.002	0.156	0.036	0.055	0.017
$HQ_{DIBP\ TDI}$	< 0.001	0.005	0.001	0.002	< 0.001
$HQ_{DEHP\ TDI}$	0.001	0.046	0.011	0.016	0.005
$HQ_{BPA\ TDI}$	< 0.001	< 0.001	0.012	< 0.001	< 0.001
HI_{TDI}	0.003	0.207	0.06	0.073	0.022
$HQ_{DBP\ RfD\ AA}$	< 0.001	0.016	0.004	0.005	0.002
$HQ_{DIBP\ RfD\ AA}$	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
$HQ_{DEHP\ RfD\ AA}$	0.001	0.077	0.018	0.027	0.008
$HQ_{BPA\ RfD\ AA}$	< 0.001	< 0.001	0.012	< 0.001	< 0.001
$HI_{RfD\ AA}$	0.001	0.093	0.034	0.032	0.01

Our results showed that values of HIs for all EEDs were lower than 1.0, indicating that human exposure to EEDs from beverages was tolerable and safe for Chinese populations (Table 4). In addition, the values of HQs for all EEDs were from < 0.001 to 0.156, which indicated the intakes were much lower than their maximum acceptable doses, reflecting a low potential of exposure to EEDs from beverages affecting human health. In addition, the values of HQs and HIs for toddlers and adults were almost two times those of other age groups. However, the cumulative risks of target EEDs were actually underestimated because cumulative risk assessment should consider the simultaneous exposure to all chemicals, and only several target EEDs were considered in our study.

Conclusions

In this study, the occurrence of several EEDs was determined in popular beverages collected from South China. Among all target EEDs, DMP, DEP, DIBP, B_ZBP, BPZ, BPG, BPPH, BPF, BPA, MeP, EtP, PrP, BP-8, and TCS were frequently found in beverages. Our results suggested that phthalates were the predominant EEDs in all beverages with concentrations that were orders of magnitude higher than other EEDs. The daily intakes of EEDs from beverages were lower than their respective maximum acceptable doses suggested by various agencies, indicating a low potential health risk from EEDs in beverages. Compared to other potential sources (food, dust, PCPs, cloth, and medicines), beverage intake is an important source for human exposure to phthalates, bisphenols, and TCS.

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