Water Research 147 (2018) 362–372

Contents lists available at ScienceDirect

Water Research

journal homepage: www.elsevier.com/locate/watres

Review

Migration and potential risk of trace phthalates in bottled water: A global situation

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ARTICLE INFO

Article history:
Received 4 June 2018
Received in revised form
17 August 2018
Accepted 1 October 2018
Available online 5 October 2018

Keywords:
Phthalates
Bottled water
Migration kinetics
Risk assessment
Daily intakes
Estrogenic effects

ABSTRACT

Increasing attention has been dedicated to trace phthalates in bottled water due to the serious concerns on public health, while there is still a lack of systematic analysis and assessment of current global situation. Through analyzing five representative phthalates in bottled water over 20 countries, this work clearly revealed the phthalates-associated potential risks in both human daily intake and estrogenic effect. In the risk assessment, the kinetic models were also developed to describe and predict phthalates migration. In more than three hundred brands of bottled waters from twenty one countries, the detection frequency of the five targeted phthalates was found to be in the order of dibutyl phthalate (DBP, 67.6%), di-2-(ethyl hexyl) phthalate (DEHP, 61.7%), diethyl phthalate (DEP, 47.1%), benzyl butyl phthalate (BBP, 36.9%), and dimethyl phthalate (DMP, 30.1%). Among the countries studied relating concentrations of DEHP in bottled waters, the top five countries ranked in the order of high to low were Thailand, Croatia, Czech Republic, Saudi Arabia and China with an average level of 61.1, 8.8, 6.3, 6.2 and 6.1 μg/L, respectively. The average levels of BBP, DBP, DMP and DEP in bottled water from Pakistan were high, in which DEP and DMP were ranked 1st among all countries with the average levels of 22.4 and 50.2 μg/L, while BBP and DBP were ranked 2nd and 3rd with the average levels of 7.5 and 17.8 μg/L, respectively. The human daily intake-based risk assessment revealed that phthalates in bottled waters studied would not pose a serious concern on public health. However, the adverse estrogenic effects of phthalates in bottled water from some countries appeared to be significant. This study just shed light on global situation of phthalates in bottled water, and more efforts should be needed to systematically examine the phthalates-related safety of bottled water.

Contents

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https://doi.org/10.1016/j.watres.2018.10.002

1. Introduction

The bottled water industry has steadily expanding over decades, with the global annual productivity of 62305.3 millions of gallons in 2015 (IBWA, 2016). In general public perception, bottled water is better than tap water in many terms, e.g. taste, safety, portability, etc. (Diduch et al., 2013; Saylor et al., 2011). However, some organic compounds had been frequently detected in bottled water, among which, estrogenic phthalates have attracted increasing attention due to their potential adverse effects on public health (Al-Saleh et al., 2011; Amiridou and Voutsa, 2011; Casajuana and Lacorte, 2003; Diduch et al., 2013). Phthalates as plasticizers have been widely used in different products including wall coverings, wire, cable jacketing, toys, food packaging materials, blood bags, etc. (Bornehag et al., 2005; Farag et al., 2013; Guart et al., 2011, 2013; Haned et al., 2018; Inonas et al., 2014; Mundra et al., 2013; Pradeep and Benjamin, 2012). The global demand on phthalate-type plasticizers has been projected to increase at an average annual rate of 1.3% in 2017–2022 (CEH, 2018). Phthalates are additives commonly used to convert hard polyvinyl chloride resins into flexible and workable plastics employed in the production of chewable rubber toys and other soft-plastic products (Corea-Tellez et al., 2008; Fasano et al., 2012; Haned et al., 2018), while phthalates in plastic bottle for potable water are considered as non-intentionally added substances (NIAS) during plastic bottle manufacturing (Garcia et al., 2018; Kassouf et al., 2013). Phthalates in plastic bottle are not chemically combined, and can migrate from PE and PET bottles (McNeal et al., 2000; Schettler, 2006; Wilkinson and Lamb, 1999).

Phthalates represent a wide range of chemical compounds, such as di-2-(ethylhexyl) phthalate (DEHP), benzylbutyl phthalate(BBP), dibutyl phthalate (DBP), diethyl phthalate (DEP), dimethyl phthalate (DMP), diisodecyl phthalate (DiDP), diisononyl phthalate (DiNP), di-n-octyl phthalate (DnOP), etc. Among which, DEHP and DBP had been found to estrogenically active, which could be completely suppressed by the addition of pure anti-estrogen ICI 182,780 (Okubo et al., 2003), while BBP and DBP had been reported to be estrogenic and toxic to fishes (Hu et al., 2016; Sun and Liu, 2017; Dong et al., 2018). Moreover, the mixture of phthalates (e.g. BBP, DBP, DEHP, DiDP, DiNP, and DnOP) exhibited enhanced estrogenic activity to 17β-estradiol (E_2) (Chen et al., 2014). The exposure to phthalates can cause many negative adverse effects on health, including endocrine disruption, disorders of neurodevelopment, cardiovascular and reproductive systems (Harris et al., 1997; Engel et al., 2009; Heudorf et al., 2007; Martino-Andrade and Chahoud, 2010; Swan, 2008). According to some toxicity experiments, DEHP affected the development of male's reproductive tract, and other phthalates induced similar effects (Field et al., 1993; Jurewicz and Hanke, 2011; Pradhan et al., 2018; Kay et al., 2014). Animal studies showed that phthalate exposure may adversely affect reproductive physiology and the development of estrogen sensitive target tissues (Kay et al., 2013). Perinatal exposure of phthalates caused a series of harmful effects in the offspring, and these phthalates can induce reproductive abnormalities, including malformations in epididymis, vas deferens, seminal vesicles, prostate, external genitalia (hypospadias), cryptorchidism, testicular injury, feminization in the retention of nipples/areolae (sexually dimorphic structures in rodents), a reduced anogenital distance and altered sexual differentiation in male rats (Gray et al., 2000; Swan et al., 2005; Foster, 2006). In a case of Puerto Rico, significant levels of phthalates and their corresponding phthalate metabolites were detected from thelarche patients (Colon et al., 2000). In addition, more and more epidemiological studies found that human exposure of phthalates could induce many adverse effects. These include but not limited to increased respiratory and allergic diseases in children (Buckley et al., 2018; Lin et al., 2018; Shu et al., 2018), decline in lung function and depression in elder people (Kim et al., 2018; Lee et al., 2018), high risk on decreasing liver function and cardio-metabolism in men (Milosevic et al., 2018), and retard neurodevelopment in infants (Jurewicz and Hanke, 2011; Kim et al., 2011; Jones et al., 2018).

Due to potential adverse effects of phthalates on public health, many studies had examined the concentration levels of phthalates in bottled water, together with the assessment of potential risks to human health (Bach et al., 2012; Santana et al., 2014; Jeddi et al., 2015a, 2016). Meanwhile, attention had also be dedicated to migration of phthalates from plastic bottles under different storage conditions (Bach et al., 2013; Diduch et al., 2013; Guart et al., 2014a; Keresztes et al., 2013). It is still not clear about global situation of phthalates in bottled waters worldwide (Devier et al., 2013; Santana et al., 2014; Zaater et al., 2014). It should be realized that many studies had been conducted on a short-term basis, but most bottled water has at least one year quality warranty period. These in turn indicate that long-term monitoring of migration of phthalates is necessary, but it is highly challenging in reality. Given such a situation, mathematical model can provide a simple and effective option for predicting migration potential of phthalates during water storage. Therefore, this study aimed to: (1) provide phthalate levels in bottled waters produced from different countries; (2)

develop a kinetic model for migration of phthalates from plastic bottles to bottle water, accompanied with a preliminary risk assessment on human health.

2. Materials and methods

2.1. Target phthalates

According to the reported detection frequency in bottled water, five phthalates (i.e. DEHP, BBP, DBP, DEP and DMP) were selected as the target compounds in this study, and their physicochemical properties are presented in Table 1.

2.2. Data sources

The key words “bottled water”, “packaged water”, “drinking water”, “PAEs”, or “phthalates” were used to search for the relevant information on Web of Science, Scopus and Scholar Google over a period of 2000–2018. The total 59 publications from 21 countries were identified and used as the data sources for further analysis.

2.3. Migration kinetics of phthalates from plastic bottle to water

The levels of phthalates in plastic bottled water had been commonly studied on a short-term basis which is much shorter than the allowable quality guarantee period of at least one year. To properly address such a challenge lacking long-term monitoring data, kinetic models describing the migration of phthalates from plastic bottle to water were developed in this study, in which the effects of storage time and temperature were considered. The data collected from the literature were fitted to different linear, exponential, and polynomial models as shown below.

$$\text{Linear fit } C = a + b \times t \tag{1}$$

$$\text{Exponential fit } C = a \times EXP(b \times t) \tag{2}$$

$$\text{Polynomial model fit } C = a + b \times t + c \times t^2 \tag{3}$$

Showing in equations (1)–(3), C is the phthalates concentration, t is the storage time, while a,b, and c are all fitting constants.

2.4. Calculating of country average concentration of phthalates in bottled water

For convenience of comparison, the average concentration of phthalates in bottled water in each country (P_A) is calculated using the equation below.

$$P_A = \frac{\sum C_i \times n_i}{\sum n_i} \tag{4}$$

Showing in equation (4), C_i is the average concentration of phthalates in one study, while n_i is the corresponding sample size of bottled water with data summarized in Table S1. Profile of sample sizes of bottled water, and average concentrations as well as concentration ranges of phthalates in bottled water is summarized in Table S2. For bottled water without detection reported as not detected (ND), the corresponding phthalates concentration is taken as half of the limit of detection (LOD). When the phthalates concentration in bottled water is above LOD but below limit of quantification (LOQ), the calculating concentration is considered as half of the LOQ. All related work in Table S1 with reported phthalates concentrations were included in this study, except for a work from Thailand, in which the reported concentrations of DEHP, DBP, DEP, and DMP were ND-490, ND-330, ND, and ND-530 μg/L, respectively (Prapatpong and Kanchanamayoon, 2010). The reason to exclude this work is the extremely high LODs of 25–50 μg/L, in which the LODs are one or more orders of magnitude higher than those of other studies. To avoid miscalculation, the investigated result in Prapatpong and Kanchanamayoon (2010) is not included.

2.5. Risk assessment

2.5.1. Daily intake-associated risk assessment

Human daily intake of phthalates through bottled water was calculated as follows:

$$E_{IN} = \frac{C \times V}{W} \tag{5}$$

where E_{IN}, C, V, and W are human daily intake of phthalates (μg/kg bw/d), phthalates concentration in bottled water (μg/L), daily consumption of bottled water (L/d), and human body weight (kg). The consumption of bottled water by adults and children are set to be 2 and 1 L per day, respectively, with the body weight of 70 kg for adults and 20 kg for children (Montuori et al., 2008; Huang et al., 2017, 2018).

For human risk assessment, the reported and predicted levels of phthalates were compared to the recommended values by U.S FDA (2012) and WHO (2011) by which the MCL (maximum contaminant levels) values are set to 6 and 8 μg/L, respectively. For the purpose of comparison, the safety factor (SF) was also calculated as follows:

$$SF = \frac{RfD}{E_{IN}} \tag{6}$$

where RfD is the reference dose given by regulations or guidelines

Table 1
Basic physicochemical properties of the five target phthalates.

Compound	CAS	Abbreviation	Formula	Molecular weight	$\log K_{ow}$[a]	Water solubility (mg/L)[a]	Saturated vapor pressure (25 °C,Pa)	Estrogenic potency (EP)
di-2-(ethylhexyl) phthalate	117-81-7	DEHP	$C_{24}H_{38}O_4$	391	7.5	0.003	$8.6*10^{-4}$	3E-7[b]
Benzyl butyl phthalate	85-68-7	BBP	$C_{19}H_{20}O_4$	312	4.59	2.7	$1.1*10^{-3}$	2E-4[c]
Dibutyl phthalate	84-74-2	DBP	$C_{16}H_{22}O_4$	278	4.45	11.2	$9.7*10^{-3}$	4.1E-5[c]
Diethyl phthalate	84-66-2	DEP	$C_{12}H_{14}O_4$	222	2.38	1100	0.22	5E-7[d]
Dimethyl phthalate	131-11-3	DMP	$C_{10}H_{10}O_4$	194	1.61	4200	0.22	–

[a] Prokupkova et al., 2002.
[b] Cavanagh et al., 2018.
[c] Kim and Ryu, 2006.
[d] Harris et al., 1997.

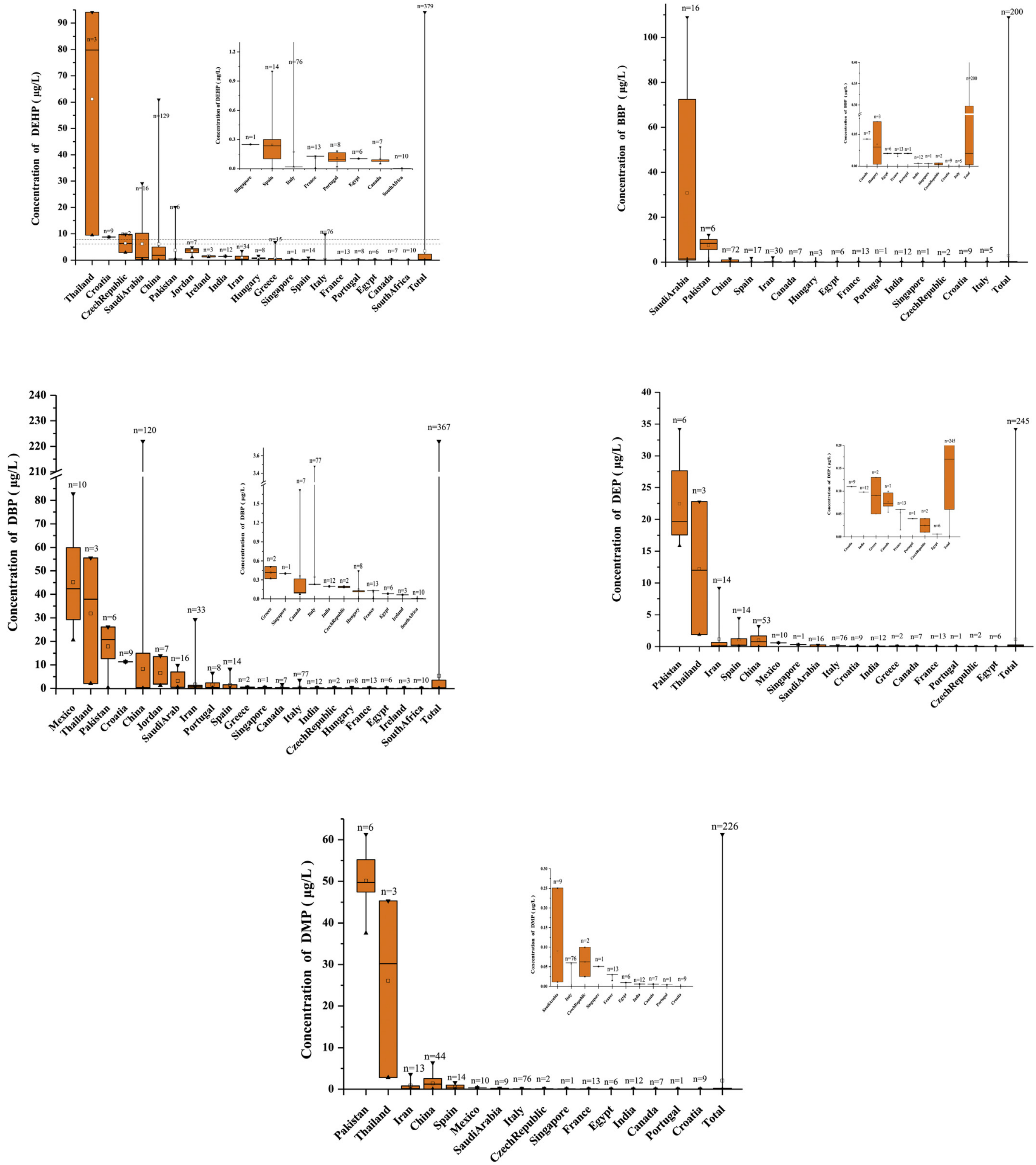

Fig. 1. Levels of the five main phthalates in bottled water among different countries (n above the column means the number of brands).

(EPA, 2009). SF below 1 indicates safety concern.

2.5.2. Estrogenic activity-associated risk assessment

As shown in Table 1, some selected phthalates are estrogenic, e.g. DEHP, BBP, DBP and DEP which are very weak estrogens. In this study, the concept of estrogen equivalence (EEQ) was adopted for the purpose of comparison (Liu et al., 2009, 2010a; b):

$$EEQ = \sum EP_i \times c_i \tag{7}$$

where EP and c denote the estrogenic potency of an individual estrogenic phthalate and its corresponding concentration, respectively. EP of estrogenic compound is often determined by *in vitro* bioassays, in which E_2 with the strongest estrogenic activity among natural estrogens is often selected as the standard estrogenic compound, i.e, the EP of E_2 is arbitrarily set as 1. When the estrogenic activity of one compound is stronger than that of E_2, its EP would above 1, otherwise, the corresponding EP would below 1. Therefore, the unit of EEQ is normally represented as ng E_2/L.

3. Results and discussion

3.1. Occurrence and migration of phthalates in bottled water

3.1.1. Potential sources of phthalates in bottled water

Trace residues of phthalates in bottled water may originate from different sources, e.g. source water, production process, raw material of packaging, and even background contamination in laboratory (Schmid et al., 2008; Sax, 2010; Bach et al., 2012; Guart et al., 2014b; Szendi et al., 2018). The investigation of 131 source waters in Spain showed that all monitored phthalates including DMP, DBP, BBP, DEHP and DEP were not detected, except for a borehole source water close to an industrial area, with a detected concentration of 1.12 μg/L DEP (Bono-Blay et al., 2012). Contamination of phthalates may also come from production process, but there is no quantitative study reported yet (Salazar-Beltran et al., 2017).

Contamination of phthalates during sample preparation had also been reported, and the main source was found to be air pollution to the syringe needle, and such contamination indeed could be avoided by quick injection at the temperature below 40 °C (Frankhause-Noti and Grob, 2007; Marega et al., 2013). With the analytical methods checked with linearity of calibration curves, recovery, sensitivity, repeatability, reproducibility and blank control, false reports due to outer contamination during sample preparation are unlikely significant (Haned et al., 2018; Gonezalez-Salamo et al., 2018). On the other hand, the considerable contents of phthalates were found in plastic bottles, e.g. the detected levels of DBP and DEHP in plastic bottle were in the range of 21–72 and 393–1499 mg/kg, respectively (Otero et al., 2015), while the detected levels of DBP, DEP, and DMP were reported to be up to 63.5, 3.1 and 14.8 mg/kg, respectively (Salazar-Beltran et al., 2018). Therefore, it is reasonable to consider that the main source of phthalates in bottled water likely originated from plastic bottle instead of the other sources (Wu et al., 2014; Rastkari et al., 2017; Zaki and Shoeib, 2018). The regulations on phthalates have been established for food related plastic packaging material in which DEHP, DBP and BBP are limited to be 1.5, 0.3, and 30 mg/kg, respectively (EU, 2015). Consequently, plastic bottle should be the main source of phthalates found in bottled water. In addition, trace phthalates were also detected in some glass bottled water, which suggested that bottle cap is also a potential source (Leivadara et al., 2008; Prokupkova et al., 2002).

3.1.2. Migration of phthalates from plastic bottle to water

Several factors may influence the migration of phthalates from plastic bottle to water, e.g. storage conditions (i.e. temperature, time, sunlight intensity), pH, bottle size, etc. As seen in Table S3, the migration of phthalates in bottled water indeed resulted from the combined effects of multiple factors. In general, high temperature favors accelerate the migration (Zaater et al., 2014; Jeddi et al., 2016; Pourzamani et al., 2017), while storage time was found to be positively correlated to the migration of phthalates from plastic bottle to water (Criado et al., 2005; Keresztes et al., 2013; Guart et al., 2014b; Jeddi et al., 2016; Cincotta et al., 2018). In addition, the intensity of sunlight may also play an important role during migration because it may affect the degradation degree of phthalates (Lertsirisopon et al., 2009). Similarly, pH also contributed to the observed migration of phthalates in bottled water, with the contradictory results reported in the literature. Bosnir et al. (2007) and Martine et al. (2013) found that acidic condition promoted the diffusion of phthalates. On the contrary, the majority of studies seemed to suggest that the higher pH is favorable for the migration of phthalates from plastic bottle (Biscardi et al., 2003; Montuori et al., 2008; Keresztes et al., 2013; Martine et al., 2013; Rastkari et al., 2017). In addition, there is no concrete evidence showing the effect of bottle size or more importantly the surface to volume ratio on the migration of phthalates, e.g. positive or no correlation between them had been reported (Montuori et al., 2008; Keresztes et al., 2013).

3.1.3. A global picture of trace residues of phthalates in bottled water

The trace residues of phthalates had been reported in bottled water of over three hundreds brands worldwide (Table S3). The detection frequency of the five selected phthalates in all the brands of the bottled water was in the order of DBP (67.6%), DEHP (61.7%), DEP (47.1%), BBP (36.9%) and DMP (30.1%). The highest concentrations of DEHP, BBP, DBP, DEP, and DMP in all the bottled waters studied were found to be 94.1, 109.0, 222.0, 34.2 and 61.3 μg/L, respectively. According to the recommendations of WHO (2011) and U.S FDA (2012) for DEHP (i.e. 8 and 6 μg/L, respectively), about 13.9% of the 379 brands of bottled water could not meet the WHO's recommended limit, while 14.2% failed to comply with the U.S FDA's limit. For better understanding of global situation, a concentration profile of phthalates in bottled waters from various

Table 2
Estimated human exposure of phthalates via drinking bottled water.

Parameter	DEHP	BBP	DBP	DEP	DMP
Maximum concentration (μg/L)	94.1	109.0	222.0	34.23	61.34
Mean concentration ± SD (μg/L)	3.42 ± 8.94	2.89 ± 13.99	5.35 ± 15.46	1.15 ± 4.00	2.07 ± 8.86
Daily exposure (adults, μg/kg bw/d)[a]	0.10–2.69	0.08–3.11	0.15–6.34	0.03–0.98	0.06–1.75
Daily exposure (children, μg/kg bw/d)[a]	0.17–4.70	0.14–5.45	0.27–11.1	0.06–1.71	0.10–3.10
Drinking water guidelines (μg/L)	6.0[b]/8.0[c]	–	–		–
Tolerable daily intake (TDI, μg/kg bw)	50.0[d]	500.0[e]	10.0[f]	–	–
Reference dose (RfD, μg/kg bw/d)[g]	20.0	200.0	100.0	800.0	–
Safety factor (adults, based on maximum concentration)	7.4	64.3	15.8	816.3	–
Safety factor (children, based on maximum concentration)	4.7	36.7	9.0	467.8	–

[a] Based on mean and maximum concentration reported in bottled water.
[b] U.S FDA, 2012.
[c] WHO, 2011.
[d] EFSA, 2013a.
[e] EFSA, 2013b.
[f] EFSA, 2013c.
[g] EPA, 2009.

Table 3
Kinetic models on migration of phthalates from plastic bottle with prolonged storage.

Compounds	Temperature	Fitting function						Ref
		Linear fit	R^2	Exponential fit	R^2	2^{nd} order polynomial fit	R^2	
DEHP	−18	C = 0.2926 + 0.00116*t	0.8830	C = 0.2928*exp(0.00368*t)	0.8987	C = 0.2994–2.051*10-4*t+2.971*10-5*t2	0.9939	(Jeddi et al., 2016)
	0	C = 0.2860 + 0.00207*t	0.9813	C = 0.2873*exp(0.00625*t)	0.9912	$C = 0.2908 + 0.00112*t+2.074*10^{-5}*t^2$	1.000	(Jeddi et al., 2016)
	4–8	C = 0.3355 + 0.00407*t	0.7804	C = 0.3433*exp(0.00915*t)	0.7299	$C = 0.3048 + 0.01016*t-1.326*10^{-4}*t^2$	0.9021	(Jeddi et al., 2016)
	25	C = 0.2986 + 0.00523*t	0.9731	C = 0.3088*exp(0.01228*t)	0.9385	$C = 0.2840 + 0.00813*t-6.31019*10^{-5}*t^2$	1.000	(Jeddi et al., 2016)
	40	C = 0.5682 + 0.0162*t	0.9900	C = 0.6096*exp(0.01703*t)	0.9343	$C = 0.5393 + 0.02195*t-1.252*10^{-4}*t^2$	0.9622	(Jeddi et al., 2016)
	outdoor	C = 0.2685 + 0.00699*t	0.9302	C = 0.2854*exp(0.01611*t)	0.8999	$C = 0.2601 + 0.00867*t-3.649*10^{-5}*t^2$	0.8703	(Jeddi et al., 2016)
	4	C = 0.102 + 0.00178*t	0.9320	C = 0.1197*exp(0.00812*t)	0.8688	$C = 0.09486 + 0.00227*10^{-4}*t-3.965*10^{-6}*t^2$	0.8824	(Zaki and Shoeib, 2018)
	25	C = 0.1350 + 0.00154*t	0.8422	C = 0.1573*exp(0.00527*t)	0.7056	$C = 0.104 + 0.00344*t-8.000*10^{-7}*t^2$	1.0000	(Zaki and Shoeib, 2018)
	40	C = 0.1132 + 0.00276*t	0.9750	C = 0.1458*exp(0.00932*t)	0.8879	$C = 0.09865 + 0.00376*t-8.081*10^{-6}*t^2$	0.9828	(Zaki and Shoeib, 2018)
DBP	−18	$C = 0.07513 + 1.9159*10^{-4}*t$	0.8293	C = 0.07515*exp(0.00243*t)	0.8411	$C = 0.07643 -6.6921*10^{-5}*t+5.6276*10^{-6}*t^2$	0.9468	(Jeddi et al., 2016)
	0	$C = 0.07385 + 6.6992*10^{-4}*t$	0.7065	C = 0.07389*exp(0.00782*t)	0.7535	$C = 0.0802 -5.9261*10^{-4}*t+2.748*10^{-5}*t^2$	0.9230	(Jeddi et al., 2016)
	4–8	C = 0.08681 + 0.00173*t	0.9224	C = 0.09086*exp(0.0133*t)	0.8722	$C = 0.07951 + 0.00318*t-3.159*10^{-5}*t^2$	0.9640	(Jeddi et al., 2016)
	25	C = 0.08933 + 0.00125*t	0.9527	C = 0.09125*exp(0.01051*t)	0.9326	$C = 0.08691 + 0.00173*t-1.048*10^{-5}*t^2$	0.9310	(Jeddi et al., 2016)
	40	C = 0.2020 + 0.00469*t	0.8639	C = 0.2157*exp(0.01443*t)	0.7879	$C = 0.1723 + 0.01058*t-1.284*10^{-4}*t^2$	0.9849	(Jeddi et al., 2016)
	outdoor	C = 0.08336 + 0.00141*t	0.8869	C = 0.08551*exp(0.01225*t)	0.8839	$C = 0.08429 + 0.00123*t+4.015*10^{-6}*t^2$	0.7767	(Jeddi et al., 2016)
	4	$C = 0.083 + 7.524*10^{-4}*t$	0.9989	C = 0.08757*exp(0.00577*t)	0.9795	$C = 0.08241 + 7.93182*10^{-4}*t-3.28283*10^{-7}*t^2$	0.9986	(Zaki and Shoeib, 2018)
	25	$C = 0.08443 + 7.988*10^{-4}*t$	0.9962	C = 0.09212*exp(0.00506*t)	0.9568	$C = 0.0824 + 0.0008*t-3.0*10^{-7}*t^2$	0.9995	(Zaki and Shoeib, 2018)
	40	C = 0.0864 + 0.00122*t	0.9887	C = 0.09716*exp(0.00734*t)	0.9333	$C = 0.08126 + 0.00158*t-2.854*10^{-6}*t^2$	0.9983	(Zaki and Shoeib, 2018)
DEP	4	C = 0.008 + 1.048*t	0.8095	C = 0.00901*exp(0.00687*t)	0.7260	$C = 0.00682 + 1.864*10^{-4}*t-6.566*10^{-7}*t^2$	0.7521	(Zaki and Shoeib, 2018)
	25	C = 0.00743 + 1.238*t	0.9460	C = 0.0075*exp(0.0081*t)	0.8666	$C = 0.006 + 0.0002*t-5*10^{-7}*t^2$	1.0000	(Zaki and Shoeib, 2018)
	40	C = 0.0088 + 1.752*t	0.8740	C = 0.01105*exp(0.00817*t)	0.7339	$C = 0.00616 + 3.573*10^{-4}*t-1.465*10^{-6}*t^2$	0.9963	(Zaki and Shoeib, 2018)
DBP	25*	C = −0.47409 + 0.3364*t	0.9544	C = 0.49434*exp(0.14002*t)	0.9646	$C = 0.00995 + 0.11282*t+0.01228*t^2$	0.9962	(Cincotta et al., 2018)

*: The migration of 6-month-old water samples were performed at 60 °C for 144 and 288 h, which represented a storage time of 12 and 18 months, respectively.

countries is presented in Fig. 1. The top five countries ranked in the order of high to low for DEHP, were Thailand, Croatia, Czech Republic, Saudi Arabia and China, with the average levels of 61.1, 8.8, 6.3, 6.2 and 6.1 μg/L, respectively. These average values were all above the recommended limit of 6 μg/L by U.S FDA. The average level of DEHP in bottled water in Pakistan was 3.8 μg/L, which was ranked 6th and not extraordinarily high. However, the corresponding average levels of BBP, DBP, DMP, and DEP were remarkably high, in which the average levels of DEP and DMP were ranked 1st among all countries and were over 20 even 50 times higher than those of other countries, with the average levels of 22.4 and 50.2 μg/L, respectively. Meanwhile, the average levels of BBP and DBP in Pakistan were ranked 2nd and 3rd with 7.5 and 17.8 μg/L, respectively. The country with the reported highest average level of BBP was Saudi Arabia with 30.8 μg/L (Al-Saleh et al., 2011; Mousa et al., 2013), while the country with the reported highest average level of DBP was Mexico with 45.1 μg/L (Salazar-Beltran et al., 2017). It is surprising as these countries with extremely high average levels of BBP, DBP, DMP or DEP, showed no remarkable high average concentrations of DEHP.

3.2. Risk assessment of phthalates in bottled water

3.2.1. Human daily intake

The maximum and average daily intakes of adults and children were calculated according to the maximum and average concentrations of phthalates in bottled waters from various countries. As shown in Table 2, the calculated maximum daily intakes of DEHP, BBP, DBP, DEP, and DMP were 2.7, 3.1, 6.3, 1.0, and 1.8 μg/kg bw/d for adults, respectively, and 4.7, 5.5, 11.1, 1.7, and 3.1 μg/kg bw/d for children. According to the recommended reference dose values, the SF calculated for the selected phthalates with the maximum reported concentrations were all far above 1 for adults and children, implying that the phthalates concentrations reported in bottled water should not pose serious safety concern. This indeed is in good agreement with previous studies showing that bottled water was safe for human consumption (Montuori et al., 2008; Erythropel et al., 2014; Guart et al., 2014a; Jeddi et al., 2015b).

It should be realized that most investigations were primarily based on short-term monitoring which may not adequately reflect the situation of prolonged storage of bottled water with a typical guarantee of at least one year. Hence, the development of

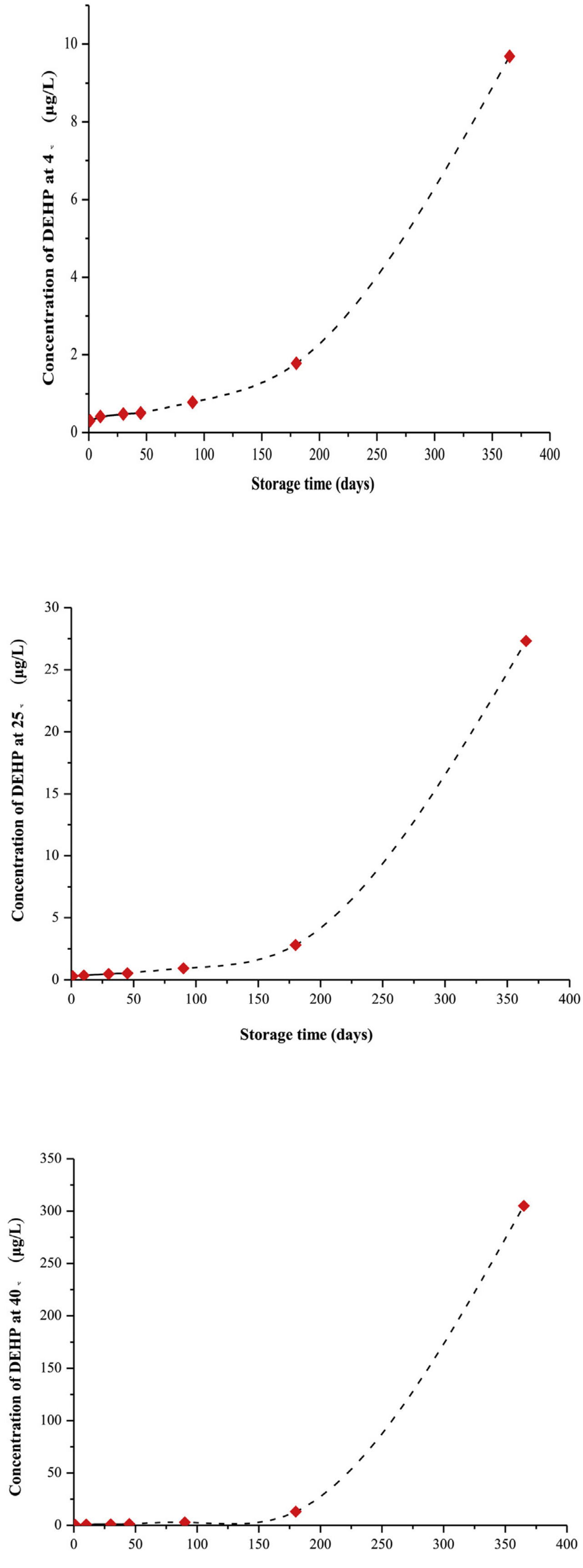

Fig. 2. Monitored and predicted concentrations of phthalates from plastic bottle at different storage temperature. Solid line: monitored concentration; Dot line: predicted concentration. Raw data taken from Jeddi et al. (2016).

appropriate kinetic model for migration of phthalates during storage is necessary and helpful. The data collected from the literature with regard to the release of phthalates from plastic bottle at different storage times and temperatures (Jeddi et al., 2016; Zaki and Shoeib, 2018) were fitted to different kinetic models as summarized in Table 3. All models showed excellent fittings, but polynomial fitting has evident defect. For example, the predicted values of phthalates such as DEHP would turn to be negative when the storage time is over 158 d under the storage temperature of 25 °C, which is not reasonable. However, it is difficult to identify whether linear or exponential fit is more appropriate solely based on the information in Table 3. Fortunately, the long term monitoring data over 184 d clearly suggested that the exponential model can provide the best fitting (Abboudi et al., 2017). Therefore, the exponential model was used to predict the possible concentrations of phthalates in bottled water against the storage time.

With data from Jeddi et al. (2016), the DEHP concentration was predicted to reach 10 μg/L after 284 days of storage at 25 °C (Fig. 2) versus 0.522 μg/L at the storage time of 45 days. In fact, after 180 days of storage, the predicted DEHP concentration already exceeded the recommended guidelines by U.S FDA (2012) (i.e. 6 μg/L) and WHO (2011) (i.e. 8 μg/L). These clearly suggest that phthalates in plastic bottled water would be accumulated significantly with prolonged storage, eventually exceeding the recommended limits even within the water quality guarantee period. Therefore, the quality of bottled water may change during the warranty period and the long-term monitoring of phthalates in plastic bottled water is essential.

Migrations of phthalates from plastic bottles to water had been reported to be positively correlated to temperature (Zaki and Shoeib, 2018). This is consistent with the data in Fig. 2. For the bottled water stored at 40 °C and 4–8 °C, the respective times required for DEHP to reach a predicted concentration of 10 μg/L were found to be 165 and 368 days in the case of Jeddi et al. (2016), i.e. low storage temperature can greatly decrease the migration of phthalates into water. Therefore, it appears from the above discussion that the quality warranty period of plastic bottle water should be carefully be re-examined in consideration of the potential migration of phthalates migrated from plastic bottle to water over water storage.

3.2.2. *Potential estrogenic effect of phthalates*

Estrogenic compounds (ECs) at environmentally relevant concentrations can adversely affect fishes and other animals, causing vitellogenin, intersex, infertility, and disruption to mating behaviors, mortality, etc. (Liu et al., 2017). Nowadays, some ECs have been strictly regulated in the drinking water quality standards of many countries, e.g. bisphenol A, DEHP, DEP, and DBP have been regulated in the Chinese drinking water quality standard, while E_2 has been regulated in the latest Japanese drinking water quality standards (Yuan et al., 2016, 2017, 2018). To evaluate the potential estrogenic effects of phthalates, average EEQ levels in bottled waters from four different countries were calculated and summarized in Table 4. Bottled water from each of these countries had the highest average concentration of a specific phthalate. The highest average EEQ was found in bottled water from Saudi Arabia, followed by Pakistan and Mexico, with the lowest average EEQ of 1.328 ng E_2/L in bottled water from Thailand. So far, the potential estrogenic effects of phthalates in bottled water have hardly been examined yet. However, the calculated average EEQ levels in the selected four countries were reasonably high and could not be neglected. Even at the low EEQ exposure level of 0.27 ng E_2/L, the egg mortality was observed in the late gastrulation and/or early organogenesis in zebrafish (Soares et al., 2009). As can be seen in Table 4, the calculated average EEQ levels in the bottled waters from the

Table 4
Estrogenic effects of phthalates in plastic bottled water.

Country	Phthalates	Ranking among countries	Average concentration (μg/L)[a]	EP[b]	EEQ (ngE2/L)
Thailand	DEHP	1	61.1	3E-7	0.018
	DBP	2	31.8	4.1E-5	1.304
	BBP	–[c]	–	2E-4	–
	DEP	2	12.2	5E-7	0.006
	Total phthalates				1.328
Saudi Arabia	DEHP	5	6.2	3E-7	0.002
	DBP	7	3.1	4.1E-5	0.127
	BBP	1	30.8	2E-4	6.160
	DEP	8	0.2	5E-7	0.000
	Total phthalates				6.289
Mexico	DEHP	–	–	3E-7	–
	DBP	1	45.1	4.1E-5	1.849
	BBP	–	–	2E-4	–
	DEP	5	0.6	5E-7	0.000
	Total phthalates				1.849
Pakistan	DEHP	6	3.8	3E-7	0.001
	DBP	3	17.8	4.1E-5	0.730
	BBP	2	7.5	2E-4	1.500
	DEP	1	22.4	5E-7	0.011
	Total phthalates				2.242

[a] Average concentration of phthalates in plastic bottled water in each country.
[b] Selected from Table 1.
[c] Not available.

selected countries indeed were significantly at the level of 1.328 ng E_2/L or above, which were 4.9–time higher than that causing adverse estrogenic effects on zebrafish as reported by Soares et al. (2009). Consequently, this and previous studies all point to a fact that phthalates in the bottled water would likely pose serious concern on their potential estrogenic effects on human health. It should also be realized that EEQ levels of phthalates may increase significantly with prolonged water storage. As ECs with the same EEQ values may induce different adverse effects on human health, it should be cautious in interpretation of the calculated high EEQ values of phthalates in bottled water. Given the huge annual consumption of bottled water globally, it should be stressed that the potential risks of phthalates as estrogenic compounds will need to be better understood and properly regulated. In this aspect, information available is still very limited. Thus, extensive research efforts are definitely needed towards the safety of bottled water in relation to estrogenicity of phthalates.

4. Implications to safety of bottled water

So far, there is lack of a holistic analysis of phthalates concentrations in bottled waters across different countries. As shown above, this study probably for the first time systematically looked into the five representative phthalates in bottled waters from 21 countries. About 13.9% of the 379 brands of bottled water could not meet the WHO's recommended limit, while 14.2% failed to comply with the U.S FDA's limit. In addition, the concentrations of DBP and BBP in bottle waters from some countries were found to be remarkably high. This in turn implies that both DBP and BBP in bottled water should be carefully regulated in the near future as their estrogenic potencies are more than 130 higher than that of DEHP. By analyzing the migration kinetics of phthalates from plastic bottle to water, it was shown that the potential concentrations of migrated phthalates tended to increase in a significant manner with prolonged water storage during the water warranty period. Although the safety factor-based risk assessment indicated that the levels of phthalates in all the reported bottled waters were acceptable in term of water safety, surprisingly the calculated EEQ levels revealed that phthalates in bottled waters from some countries may induce the adverse estrogenic effects on human health.

5. Conclusions

This work provided a comprehensive picture of phthalates concentrations in global bottled waters, accompanied with a risk assessment based on human daily intakes and estrogenic effects. The following concluding remarks can be drawn from this study.

1) The highest concentrations of the five selected phthalates (i.e. DBP, BBP, DEHP, DMP, and DEP) in bottled waters among 21 countries were found to be 222.0, 109.0, 94.1, 61.3, and 34.2 μg/L, respectively. Given their relative strong estrogenic potencies, DBP and BBP should be also regulated in a way similar to DEHP. In fact, recommended limits for target phthalates should need to be established urgently for bottled water.
2) Migrations of phthalates from plastic bottles to water could be described well by an exponential-type model which was able to predict the effects of storage temperature and time on release of phthalates. Lower storage temperature and shorter storage time are preferable for mitigating potential release of phthalates from plastic bottle to water, thus reducing human daily intakes of phthalates through water consumption.
3) The human daily intake-based risk assessment showed that phthalates in bottled water are at safe level, but the high EEQ values of phthalates in bottled water from some countries seemed alarming as phthalates at such levels might induce adverse estrogenic effects to human beings.

Acknowledgement

This work was financially supported by The Key Program of National Natural Science of China (No. 41330639); the Program for National Natural Science Foundation of China (No. 21107025; No. 21577040; U1501234); Special funds for public welfare research and capacity building in Guangdong Province (No. 2015A020215003); Guangzhou Science and Technology Program, Guangdong Province, China (No. 201510010162) as well as the funding for network platform construction on odorous compounds in water (No. YGSYBMKY17-N11).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.watres.2018.10.002.

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