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Does the reuse of PET bottles during solar water disinfection pose a health risk due to the migration of plasticisers and other chemicals into the water?

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ABSTRACT

Solar water disinfection (SODIS) is a simple, effective and inexpensive water treatment procedure suitable for application in developing countries. Microbially contaminated water is filled into transparent polyethylene terephthalate (PET) plastic bottles and exposed to full sunlight for at least 6 h. Solar radiation and elevated temperature destroy pathogenic germs efficiently. Recently, concerns have been raised insinuating a health risk by chemicals released from the bottle material polyethylene terephthalate (PET). Whereas the safety of PET for food packaging has been assessed in detail, similar investigations for PET bottles used under conditions of the SODIS treatment were lacking until now. In the present study, the transfer of organic substances from PET to water was investigated under SODIS conditions using used colourless transparent beverage bottles of different origin. The bottles were exposed to sunlight for 17 h at a geographical latitude of 47° N. In a general screening of SODIS treated water, only food flavour constituents of previous bottle contents could be identified above a detection limit of 1 μg/L. Quantitative determination of plasticisers di(2-ethylhexyl)adipate (DEHA) and di(2-ethylhexyl)phthalate (DEHP) revealed maximum concentrations of 0.046 and 0.71 μg/L, respectively, being in the same range as levels of these plasticisers reported in studies on commercial bottled water. Generally, only minor differences in plasticiser concentrations could be observed in different experimental setups. The most decisive factor was the country of origin of bottles, while the impact of storage conditions (sunlight exposure and temperature) was less distinct. Toxicological risk assessment of maximum concentrations revealed a minimum safety factor of 8.5 and a negligible carcinogenic risk of 2.8×10^{-7} for the more critical DEHP. This data demonstrate that the SODIS procedure is safe with respect to human exposure to DEHA and DEHP.

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1. Introduction

1.1. Water quality situation in developing countries

Water in sufficient quantity and good quality is essential for life. However, in 2002, 17% of the global population, 1.1 billion people, lacked access to sufficient water supply, and many more consume microbiologically contaminated water. This situation leads to a high risk for waterborne diseases such as diarrhoea, cholera, typhoid fever, hepatitis A, amoebic and bacillary dysentery and other diarrhoeal diseases. Each year 1.8 million people die of diarrhoea, 90% of which are children under the age of five primarily in developing countries. This is equivalent to about 4500 children dying each day (World Health Organization, 2004).

Public water supplies in developing countries often fail to produce and distribute water that is safe for consumption. Therefore, in many cases, the provision of safe drinking water has become a task of the individual household. Treating drinking water at the point of use is one option to mitigate the high incidence of diarrhoeal diseases, and can have a significant positive impact on the economic situation of the household as financial resources for medical care are saved and productivity is enhanced. Furthermore, school attendance of children is improved, and their future perspectives are enhanced therewith.

1.2. Benefits of solar water disinfection

Health agencies in developing countries have been recommending the boiling of drinking water for generations. Boiling, however, can increase the risk of respiratory diseases due to the exposure to smoke and is rarely practiced where firewood and fuel are scarce and expensive. Gilman and Skillicorn (1985) have found that boiling of drinking water would lead to an 11% increase of the total household budget of the poorest quartile of a study population in Bangladesh (Clasen et al., 2008). A range of alternative methods for household water treatment is available, such as chlorination, filtration or solar water disinfection. The suitability of these different methods and the acceptance among the population is highly site-specific and depends, inter alia, on the socio-economic conditions and the users' specific preferences.

Solar water disinfection (SODIS) is a well established and simple method for drinking water treatment. Microbiologically contaminated water is filled into transparent polyethylene terephthalate (PET) plastic bottles (or glass bottles) and exposed to full sunlight for at least 6 h (or for 2 days under mostly cloudy conditions). During the exposure, solar radiation (UV-A light and elevated temperature) destroys pathogenic bacteria, viruses as well as *Giardia* spp. and *Cryptosporidium* spp. (Acra et al., 1980; Berney et al., 2006a; Berney et al., 2006b; Gaafar, 2007; Heaselgrave et al., 2006; Joyce et al., 1996; Kehoe et al., 2004; McGuigan et al., 1998; McGuigan et al., 2006; Méndez-Hermida et al., 2005; Smith et al., 2000; Sommer et al., 1997; Wegelin et al., 1994; Boyle et al., 2008).

SODIS is presently applied by more than 2 million people in 31 countries for the treatment of their drinking water. Epidemiological assessments and health evaluations in SODIS projects reveal a significant positive health impact among user families. Health impact studies conducted in Kenya, Bolivia and India revealed that the diarrhoea incidence of children below 5 years was reduced by 24% (Conroy et al., 1996) to 40% (Hobbins, 2003; Rose et al., 2006) through the regular consumption of SODIS treated water. During a cholera outbreak in a Maasai community, Conroy et al. (2001) found a 86% reduction of cholera cases among SODIS users.

Like boiling, chlorination and filtration, SODIS is not suitable for the treatment of chemically polluted water. In comparison with other methods for point-of-use water treatment, the advantages of SODIS lie in the very low up-front and maintenance costs, the independence from specific materials (other than PET bottles) and related supply chains, the fresh taste of the water after treatment, and the fact that the water is protected from re-contamination in the SODIS bottles. Disadvantages include the relatively long treatment time (6 h to two days), the dependence on a minimum dose of sunlight (SODIS is not recommended under conditions of continuous rainfall), the relatively small volumes of treated water and the decreased disinfection efficiency in the case of highly turbid water.

1.3. Possible health risks of SODIS

Concerns about health risks related to SODIS are based on the suspicion that toxic substances could migrate from the PET bottle into the drinking water. Particularly, fears were fuelled by a press report published on 10 February 2003 in Source Weekly on the migration of organic compounds from reused PET bottles. This report was based on data taken from an investigation of organic chemicals trespassing to water in PET bottles reused for drinking water (Lilya, 2001). The authors identified several organic compounds in the water and observed increased migration rates with duration of reuse. The plasticiser di(2-ethylhexyl)adipate (DEHA) turned out as the toxicologically most relevant compound, exceeding acceptable carcinogenic risk levels in the water.

Migration of components of PET such as degradation products of the polymer, catalyst residues and other contaminants has been broadly and profoundly investigated in the past. Migrating compounds comprise production residues as well as hydrolysis and thermal degradation products of PET itself, additives, and, in the case of recycled or reused PET bottles, constituents of beverages previously contained in the bottles, such as flavour compounds. The most important degradation products present in PET are aldehydes such as formaldehyde and acetaldehyde (Nawrocki et al., 2002; Mutsuga et al., 2006). In the context of recycled and reused PET the presence of contaminants from previous contents may pose a problem (Nerìn et al., 2003; Franz et al., 2004).

The influence of environmental factors and storage time on the release of chemical substances from PET has been investigated in several studies. Photolytic formation of diffusing substances may also play a role. In sunlight exposure tests Wegelin et al. (2001) have shown that PET degradation products such as terephthalate monomers and dimers are primarily formed at the outer surface of the bottles. Evaluation of the water samples stored in the bottles revealed higher formaldehyde and acetaldehyde levels after

longer storage times but no differences between water samples with and without exposure to sunlight. In a bioassay with *Allium*, Evandri et al. (2000) observed cytogenetic activity after 8 weeks storage of mineral water in PET bottles. The observed effects were enhanced by sunlight exposure and slightly increased storage temperatures. However, no chemical compounds responsible for these effects were identified. Biscardi et al. (2003) and Criado et al. (2005) found di(2-ethylhexyl)phthalate (DEHP) and dibutyl phthalate, respectively, in water stored in PET bottles for several months; however, these effect-based investigations revealed no mutagenic and carcinogenic components at critical concentrations. From the literature on leaching of PET it becomes evident that time is a dominant factor governing the release of organic substances (Wegelin et al., 2001; Lilya, 2001; Nawrocki et al., 2002). This was also observed in the case of antimony which is present in PET due to its use as polymerization catalyst (Shotyk and Krachler, 2007). A substantial migration of antimony from PET to water only takes place under conditions not typically occurring in the SODIS process, i.e. exposure at 80 °C for several days (Westerhoff et al., 2008).

1.4. *Goals of this work*

The objective of this work was to quantify migration of plasticisers as well as to identify and characterize additional organic compounds released from PET during the SODIS process. Possible health risks associated with consumption of SODIS treated water were assessed. For this purpose, the migration of organic components from colourless, transparent PET bottles as used for SODIS (used PET bottles from Honduras, Nepal, and Switzerland) was studied under realistic conditions. Concentrations of the plasticisers DEHA and DEHP were determined in water with and without application of SODIS. Furthermore, the water was analysed to identify unknown components. Based on the gathered data, the health risk resulting from the intake of such components in SODIS water was assessed.

2. Materials and methods

The investigation was based on the following approach: In a first step, pure (distilled) water was filled into PET bottles of different origin, and the bottles were stored under different conditions (typical SODIS conditions, SODIS conditions with additional heating, storage at room temperature without exposure to sunlight). Apolar components were then extracted with *n*-hexane and qualitatively and quantitatively analysed in the extract using gas chromatography/high resolution mass spectrometry (GC/MS). In particular, plasticisers DEHA and DEHP were quantified. To account for possible cross-contamination by plasticisers, procedural blank samples were included in the investigation.

Colourless PET beverage bottles of various origins (see Table 1) suitable for solar disinfection of water by the SODIS protocol were pre-rinsed and filled with pure water (water for chromatography, Merck, Darmstadt, Germany). Bottles are denominated as 'used' if they were not yet used for SODIS or as 'reused' if they were already used for SODIS treatment of water. Exposure to sunlight was conducted on two consecutive sunny and cloudless days (2 and 3 June 2003) at Dübendorf, Switzerland (geographical latitude of 47° 24′ 15″ N). Bottles were exposed in horizontal position at a maximum ambient temperature of 34 °C. Some of the bottles were put half-way in a water bath kept at 60 ± 5 °C. Control bottles were kept in the shade at room temperature (25 °C). The solar radiation was measured using a pyranometer with a spectral range of 305–2800 nm (CM 3, Kipp & Zonen, Delft, The Netherlands). Total time of exposure to sunlight was 17 h at a horizontal solar radiation varying between 194 and 845 W/m^2 depending on the sunset. Total residence time of the water in the PET bottles was 48 hours.

After exposure, 100 mL aliquots of the water were spiked with the internal standards (0.5 μg $^{13}C_6$-DEHA CLM-4675-1.2 and 2.5 μg ring-D_4-DEHP DLM-1368 from Cambridge Isotope Laboratories, Andover, USA) and extracted with 1 mL of *n*-hexane. Three method blanks were prepared using pure water (see above) instead of water from PET bottles.

Analyses were carried out on a MAT 95 mass spectrometer (Thermo Finnigan MAT, Bremen, Germany) coupled to a gas chromatograph HRGC Mega 2 series (Fisons Instruments, Rodano, Italy). Samples of 2 μL were injected in splitless mode (splitless time 20 s) at an injector temperature of 260 °C. GC separation was carried out on a 20 m × 0.28 mm glass capillary coated with a DB-5 analogue stationary phase (PS 086, Fluka, Buchs, Switzerland) with a film thickness of 0.15 μm using hydrogen at 50 kPa as carrier gas. The following temperature programs were used for single ion monitoring (SIM) of DEHA and DEHP and full-scan measurements, respectively: 60 °C (1 min), 20 °C/min up to 220 °C, 4 °C/min up to 260 °C (5 min) and 60 °C (1 min), 8 °C/min to 260 °C (5 min). The mass spectrometer was operated in electron ionisation (EI) mode at 70 eV electron energy. In full-scan measurements, mass spectra were recorded in a mass range of m/z 33–500. In SIM, DEHA, DEHP and the respective isotope labelled surrogates were detected by recording the two most abundant characteristic fragment ions at a mass resolution of 8000.

Quantification was based on signal areas in the mass chromatograms. Authentic DEHA and DEHP used as references were purchased from Fluka (Buchs, Switzerland). Detection limits based on a signal-to-noise ratio of 3:1 were 0.005 μg/L for quantitative determination of DEHA and DEHP, and approximately 1 μg/L for qualitative full-scan GC/MS analysis. Unknown compounds discovered in full-scan GC/MS runs were identified by comparison with reference spectra from the ''Wiley Registry of Mass Spectral Data, 6th Ed.'' using the software MassLib (MPI Mülheim/Ruhr, Germany).

3. Results and discussion

The results of the qualitative survey for unknown components possibly released from PET are compiled in Table 1. All detected compounds are terpenes which most likely are present as residues of flavouring constituents of the originally bottled beverages (e.g. limonene in a cola beverage bottle). No further organic components could be found above the

Table 1 – PET beverage bottles used in the exposure experiment (used: used beverage bottle not yet used for SODIS; reused: used beverage bottle reused for SODIS)

Bottle no.	Country of origin	Size (L)	(Re-)Use status	Exposure	Component	DEHA [μg/L]	DEHP[μg/L]
1	Honduras	2	Reused	Sun 60 °C	Limonene *Carvone*	0.046	0.33
2	Honduras	2	Reused	Sun ambient	n.d.	0.025	0.29
3	Honduras	1.5	Reused	Shade	n.d.	0.024	0.19
4	Honduras	2	Used	Sun 60 °C	Myristicine Limonene Safrol *Carvone* *Terpinene*	0.044	0.35
5	Honduras	2	Used	Sun ambient	Myristicine Limonene Safrol *Carvone* *Terpinene*	0.023	0.31
6	Nepal	1	Reused	Sun 60 °C	n.d.	0.022	0.44
7	Nepal	1	Reused	Sun ambient	n.d.	0.016	0.18
8	Nepal	1	Reused	Shade	n.d.	0.015	0.24
9	Nepal	1	Used	Sun 60 °C	n.d.	0.012	0.71
10	Nepal	1	Used	Sun ambient	n.d.	0.016	0.38
11	Switzerland	1	Reused	Sun 60 °C	n.d.	0.017	0.15
12	Switzerland	1	Reused	Sun ambient	n.d.	0.021	0.30
13	Switzerland	1	Reused	Shade	n.d.	0.021	0.14
14	Switzerland	1	Used	Sun 60 °C	n.d.	0.021	0.16
15	Switzerland	1	Used	Sun ambient	n.d.	0.010	0.10
Blank 1	–		–	–	–	0.015	0.024
Blank 2	–		–	–	–	0.021	0.14
Blank 3	–		–	–	–	0.017	0.18

Tentative mass spectral assignment of components and concentrations of DEHA and DEHP in water from PET bottles treated under SODIS conditions (components listed in italic are present at levels very close to the detection limit; n.d.: no components above detection limit of approx. 1 μg/L identified).

detection limit of the qualitative GC/MS screening method in the order of 1 μg/L.

Similar findings are reported for recycled PET which can contain residues of foodborne compounds (Franz et al., 2004; Nerìn et al., 2003), including limonene, a flavour component in soft-drinks, which sorbs well into PET (Safa and Bourelle, 1999).

One of the objectives of the present investigation was the quantitative determination of the plasticisers DEHA and DEHP. The concentrations given in Table 1 and in Fig. 1 are without exception above the quantification limit of 0.005 μg/L (signal-to-noise ratio 3:1). However, these concentrations are still close or equal to concentrations measured in samples of pure water used as procedural blanks. From the data no distinct correlation between DEHA and DEHP levels in the water samples can be discerned. Comparison of the average DEHA and DEHP concentrations in water exposed under different conditions reveals only minor differences:

3.1. *Country of origin*

Concentrations of DEHA in water from Honduras bottles (used and reused for SODIS) are slightly but significantly (Student's *t*-test, $p > 0.95$) higher than the levels in the other samples (bottles from Nepal, Switzerland, and blank samples). Likewise, DEHP levels in bottles from Honduras (used und and reused for SODIS) are significantly higher than in Swiss bottles and blank samples. However, as bottles of different brands were analysed, the relatively high levels in bottles from Honduras cannot further be traced back to a specific bottle type.

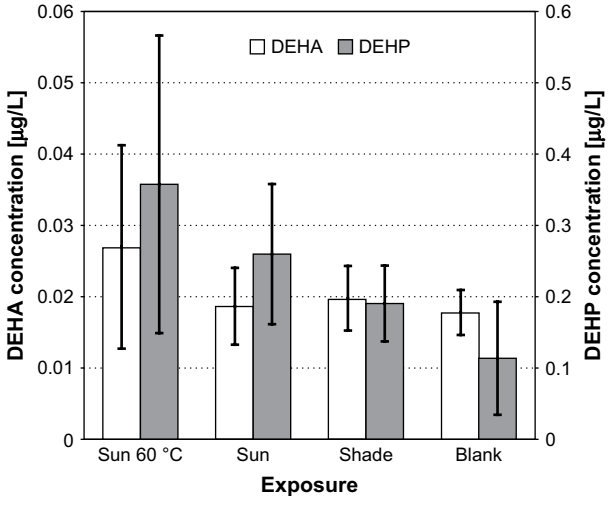

Fig. 1 – Average DEHA and DEHP concentrations in water from PET bottles stored under various conditions (error bars indicate standard deviation).

3.2. Different (re-)use status of bottles

DEHA and DEHP concentrations are slightly but not significantly (Student's *t*-test, $p > 0.95$) higher in bottles reused for SODIS than in used bottles and blank samples.

3.3. Exposure

Average concentrations of DEHA and DEHP in water from bottles exposed to sunlight and heated to 60 °C, bottles exposed to sunlight at ambient temperature, bottles kept in the shade and blank samples show a slight decreasing tendency (Fig. 1). However, only DEHP levels in bottles exposed to sunlight at 60 °C were significantly (Student's *t*-test, $p > 0.95$) higher than the respective blank concentrations.

In summary, the only noticeable distinctive parameters of the DEHA and DEHP levels in the water were the country of origin of the PET bottles and increased temperatures as present in the SODIS process.

While no literature data on plasticiser migration upon the SODIS process is available, a series of reports on possible sources of plasticiser residues in bottled water have been published (Peñalver et al., 2000; Lilya, 2001; Casajuana and Lacorte, 2003; Serôdio and Nogueira, 2006; Bošnir et al., 2007; Montuori et al., 2008). According to these studies, residues of plasticisers in samples of bottled water can be due to various sources: (i) Water may be contaminated in the bottling plant, (ii) plasticisers may migrate from the bottle material to the water, or (iii) widely used plasticisers such as DEHP may be introduced into the sample as cross-contamination during the analytical procedure. Therefore, analytical data on DEHA and DEHP levels in bottled water have to be scrutinised by rigorous consideration of quality assurance aspects concerning e.g. analytical method blanks. The most comprehensive discussion of these issues has been presented by Montuori et al. (2008). These authors have compared own data on phthalates in water packed in PET bottles with corresponding literature data. Maximum DEHP concentrations were in the range of 1 μg/L with detection limits of up to 0.6 μg/L (Peñalver et al., 2000; Casajuana and Lacorte, 2003; Kayali et al., 2006; Serôdio and Nogueira, 2006). Bošnir et al. (2007) reported significantly higher DEHP concentrations of up to 50 μg/L. The situation for DEHA is similar: Peñalver et al. (2000) and Serôdio and Nogueira (2006) found DEHA levels not exceeding 0.15 μg/L whereas Lilya (2001) reported DEHA concentrations of up to 32.8 μg/L. However, the fact that DEHP could not be quantified due to blank problems in this latter study gives rise to questions concerning a possible contribution of DEHA introduced in the analytical procedure. In the light of these data, the levels of up to 0.046 μg DEHA/L and 0.71 μg DEHP/L reported here (see Table 1) are in the same order of magnitude as concentrations detected in commercial mineral water from PET bottles. Therefore, no unusual exposure to these plasticisers is expected by consumption of SODIS treated water.

3.4. Toxicological risk assessment

All substances detected in the qualitative survey (Table 1) are flavour components which are originating from the previous content of the bottles. As typical food constituents they are not of toxicological importance, and the toxicological assessment of the results in this study can therefore be limited to the plasticiser compounds DEHA and DEHP. Toxicological data for chronic exposure to DEHA and DEHP in drinking water are considered in the Guidelines for Drinking-water Quality of the WHO (World Health Organization, 2006), the Integrated Risk Information System (IRIS) of the US EPA (EPA Integrated Risk Information System, 1993; EPA Integrated Risk Information System, 1994) and the National Primary Drinking Water Standards (Office of Water, 2003) of the US EPA (Table 2). The assessment is based on a worst case scenario supposing permanent consumption of water exhibiting the maximum DEHA and DEHP concentrations of the present study (see Table 1).

WHO guideline values are defined for a daily *per capita* consumption of 2 L of drinking water by an adult of 60 kg body weight and exploiting 1% of the tolerable daily intake. Both DEHA and DEHP exhibit low short-term toxicity and have been classified as 'not classifiable as to its carcinogenicity to humans' (Group 3) by the International Agency for Research on Cancer (IARC) (IARC, 2000). DEHA and DEHP concentrations measured in this study both exploit less than 10% of the respective guideline values (Table 2).

Compared to IRIS reference doses for chronic oral exposure (RfD), daily intake of maximum detected DEHA and DEHP levels in water is far below maximum safe doses, as indicated by the calculated safety factors (see Table 2). Starting from the unit risk, the carcinogenic risk posed by the more critical concentration of DEHP is distinctly below the accepted risk level of 10^{-6}. Finally, maximum detected DEHA and DEHP concentrations are distinctly below maximum contaminant levels (MCL) of the US National Primary Drinking Water Standards.

In an integrated health risk assessment, other health effects associated with low exposure levels besides carcinogenic risks have to be considered. For phthalates including

Table 2 – Health risk from oral exposure to DEHA and DEHP containing maximum detected levels after SODIS treatment in PET bottles exposed to sunlight (see Table 1) based on WHO guideline values for drinking water quality, EPA Integrated Risk Information System (IRIS) and US National Primary Drinking Water Standards

	DEHA	DEHP
Detected maximum concentration [μg/L]	0.046	0.71
Daily intake via drinking water (60 kg body weight, 2 L/d) [μg/kg·d]	0.0015	0.024
WHO Guideline value [μg/L]	80	8
Calculated safety factor	1.8×10^3	11
IRIS RfD chronic non-carcinogenic effect risk [mg/kg·d]	6×10^{-1}	2×10^{-2}
Calculated safety factor	3.9×10^5	8.5×10^2
USEPA Drinking water unit risk [per μg/L]	3.4×10^{-8}	4.0×10^{-7}
Calculated carcinogenic risk	1.6×10^{-9}	2.8×10^{-7}
Maximum Contaminant Level (MCL) [μg/L]	400	6
Calculated safety factor	8.7×10^3	8.5

DEHP, subtle developmental effects have been observed in male infants which significantly correlated with the prenatal exposure of the mothers to phthalates, urinary levels of these women being in the range of the female population of the United States (Swan et al., 2005). From these data, a median daily exposure of 1.32 μg DEHP/kg · d was estimated (Marsee et al., 2006). The maximum daily intake of DEHP based on the concentrations measured in this study (Table 2) corresponds to 2% of the total daily exposure of the mothers in the respective studies.

In summary, the higher observed levels and the lower toxicological limit values imply a more critical situation for DEHP. However, any of the criteria applied in Table 2 reveals the maximum detected DEHP concentration of 0.046 μg/L as safe. These results are in line with the more general finding that chemical micropollutants in drinking water can be considered a minor problem in comparison to the possible risks of microbial contamination (van Leeuwen, 2000), and that the contribution of drinking water to the total dietary exposure from most chemicals is very low (van Dijk-Looijaard and van Genderen, 2000).

4. Conclusions

In an overall assessment of SODIS, benefits and risks have to be weighed up. SODIS is an efficient and simple treatment process for the solar disinfection of water. Generally, chemical micropollutants in drinking water are considered as a minor problem in comparison to the possible risks of microbial contamination (van Leeuwen, 2000), and exposure balances indicate that the contribution of drinking water to the total dietary exposure from most chemicals is very low (van Dijk-Looijaard and van Genderen, 2000). In this sense, the present survey and literature data on chemicals migrating from PET bottles to drinking water confirm that the SODIS treatment process is safe and does not trigger the migration of hazardous contaminants at critical levels as insinuated by isolated reports.

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