



Research Paper

Occurrence and sorption behaviour of bisphenols and benzophenone UV-filters in e-waste plastic and vehicle fluff

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ABSTRACT

Bisphenols and benzophenone UV-filters are hazardous, high production volume chemicals. There is concern that these contaminants could leach into the environment or be recycled into new products during waste management. To investigate this, nine bisphenols and five benzophenones were quantified in Norwegian e-waste and car fluff. To understand their leachability, equilibrium passive sampling methodology, using polyoxymethylene (POM), was calibrated for these substances, many of which for the first time. This method can differentiate freely dissolved substances in the aqueous phase from those sorbed to suspended colloids and microplastics in the leachate water. Equilibrium POM partitioning was reached within 14 days of shaking; all bisphenols and benzophenone UV-filters exhibited linear isotherms (R^2 ranged from 0.83 to 1.0), when deriving POM-water partition coefficients (K_{POM}). Bisphenol A and bisphenol F displayed the highest concentrations, with maximum levels of 246,000 and 42,400 ng g^{-1} , respectively. Logarithms of waste-water partition coefficients ($\log K_{\text{waste}}$) ranged from 1.7 (benzophenone 2) to 4.5 (bisphenol P). The established K_{POM} values agreed with measured K_{waste} values (within a factor of ~ 3), unlike octanol-water partition coefficients. This indicated that POM is a better surrogate for waste plastic partitioning than octanol. Results are discussed in the context of assessing risks from waste management in a circular economy.

1. Introduction

Bisphenols and benzophenone UV-filters are structurally similar groups of hazardous chemicals that are commonly used and co-occur in many products (Asimakopoulos et al., 2016). Bisphenol A (BPA), in particular, is one of the most abundant synthetic chemicals in the world (Usman and Ahmad, 2016), due to its use as a monomer in the preparation of polycarbonate materials and epoxy resins. BPA can be found in a wide variety of products, such as food containers, thermal paper, CDs, toys, baby bottles, and the inner lining of metal cans (Asimakopoulos et al., 2016; Castro et al., 2019; Liao and Kannan, 2013; Regueiro et al., 2015). Benzophenone UV-filters, in contrast with benzophenols, are used as additives in plastic materials, to prevent the degradation of the plastic polymer through photo-oxidation (Maier and Calafut, 1998), and also as ingredients in sunscreens and cosmetics (Kim and Choi, 2014).

The increased use of these hazardous chemicals has led to their distribution in the environment and the unavoidable exposure to the

general population. Concerns about this has been raised. These two groups of substances are considered endocrine disrupting chemicals capable of promoting adverse health outcomes in humans and animals (García et al., 2015; Ghazipura et al., 2017; Suzuki et al., 2005; Usman and Ahmad, 2016). For this reason, in 2011, the European Commission regulated BPA migration in food packages to a maximum of 0.6 mg kg^{-1} (European Commission, 2011). These restrictions concerning the use of BPA promoted the gradual entry of other bisphenol analogues into the market as substitutes. However, some of these analogues also demonstrated reproductive toxicity and endocrine disrupting potential (den Braver-Sewradj et al., 2020; Zhu et al., 2020). This has caused the substitution of BPA with other bisphenols to be listed as an example of "regrettable substitution", referring to instances where one hazardous chemical is substituted by other similarly hazardous substances (Trasande, 2017). The presence of alternative bisphenols in the environment was reported during the past years, for example median concentrations of 0.38 ng g^{-1} for bisphenol AP (BPAP) were reported in sludge (Pang

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et al., 2019), as well as 4.91 and 75 ng L⁻¹ in effluent and influent wastewater, respectively (Çesen et al., 2018). Following a similar pattern as bisphenols, the widespread use of benzophenone UV-filters has led to the release of these concentrations and their derivatives into the environment, such as rivers and lakes, at concentrations reaching up to 125 ng L⁻¹ (Kim and Choi, 2014).

An important emission source of these contaminants into the environment is waste handling facilities, such as recycling facilities, where substantial concentrations of BPA can be measured (Morin et al., 2017). Two waste fractions of particular concern are Waste Electronic and Electrical Equipment (WEEE), also known as “e-waste”, which is a complex mixture of materials, plastics, metals, metalloids, and chemicals, as well as Automotive Shredder Residue (ASR), which comprises all non-metallic, non-easily removable parts of discarded vehicles, such as plastics, textiles, dirt and a variety of other materials, which are commonly referred to collectively as “vehicle fluff” or “car fluff” (Nourreddine, 2007). These two matrices are two different types of predominantly plastic waste that contain a multitude of contaminant classes and plastic additives (Robinson, 2009; Morin et al., 2017). Reported concentrations of BPA in such e-waste display concentrations above 70,000 µg kg⁻¹, and 1.4% of the total BPA concentration is generally leached out when this waste is submerged in water (Morin et al., 2015). Landfill leachate concentrations of BPA have been reported to range from 0.01 to 107 µg L⁻¹ in Sweden (Kalmykova et al., 2013); 0.07 to 228 µg L⁻¹ in Japan (Asakura et al., 2004) and have an average of 27.6 µg L⁻¹ in China (Huang et al., 2021). In Norway, a compilation of screening data from 2002 to 2012 reported median concentrations of BPA of 400 ng g⁻¹ in leachate sediments and 8.3 ng g⁻¹ in fresh water sediments (Arp, 2012). Regarding bisphenol analogues, landfill leachate concentrations of bisphenol AF (BPAF) (18.6 ng L⁻¹), BPAP (5.00 ng L⁻¹), bisphenol BP (BPBP) (0.72 ng L⁻¹), bisphenol E (BPE) (97.9 ng L⁻¹), bisphenol F (BPF) (3840 ng L⁻¹), bisphenol P (BPP) (28.2 ng L⁻¹), bisphenol S (BPS) (62.0 ng L⁻¹), bisphenol TMC (BPTMC) (437 ng L⁻¹) and bisphenol Z (BPZ) (11.5 ng L⁻¹) were reported in China (Huang et al., 2021). To the best of our knowledge, actual concentrations of bisphenol analogues (other than BPA) and benzophenone UV-filters are reported in e-waste and vehicle fluff in this study for the first time, even though there are a number of published biomonitoring studies associated to e-waste handling facilities (Song et al., 2019; Zhang et al., 2020).

The main motivation of the circular economy model in Europe is to use resources more efficiently and to minimize waste, in part by developing suitable recycling processes (Burlakovs et al., 2019; Kacprzak et al., 2017). However, the acceptability of recycling waste materials containing hazardous substances such as bisphenols and benzophenone UV-filters is still a matter of discussion. The sorption properties of these chemicals can play a key role on their fate during waste management and possible recycling processes. The leachability of bisphenols and benzophenone UV-filters from waste can constitute a reliable indicator about the ability of the contaminants to dissolve from waste and potentially be released into their surroundings when in an aqueous environment. The leachability can also indicate which substances are most likely to end up in recycled products.

To address these concerns, the first objective of this study was to measure the concentrations of bisphenols and benzophenone UV-filters in e-waste and vehicle fluff. The second objective was to develop a methodology to measure freely dissolved phase concentrations of bisphenols and benzophenone UV-filters, when these wastes reach equilibrium with surrounding water (similarly to when they are either in a landfill, occur as litter in fresh/marine water, or are washed during a recycling process). For this second objective, a method using the polyoxymethylene (POM) passive sampler was developed by quantifying POM-water kinetic uptake rates and partition coefficients for these substances for the first time. The final objectives were to describe the measured waste-water partitioning behaviour of the bisphenols and benzophenone UV-filters to e-waste plastic and vehicle fluff based on modelling predictions, and to develop a general approach for assessing

their partitioning behaviour in plastic rich waste materials.

2. Experimental

2.1. Chemicals and materials

The list of bisphenols and benzophenones were chosen based on the availability of reference standards and literature considerations presented in the introduction above about their presence in diverse products. Standards of bisphenol A (BPA, 99%), AF (BPAF, 99%), AP (BPAP, 99%), B (BPB, 98%), F (BPF, 98%, 4,4'-methylenediphenol), M (BPM, 99%), P (BPP, 99%), S (BPS, 98%), and Z (BPZ, 99%), and benzophenone-1 (BzP-1, 99%), -2 (BzP-2, 97%), -3 (BzP-3, 98%), and -8 (BzP-8, 98%), and 4-hydroxybenzophenone (4-OH BzP, 98%) were purchased from Sigma-Aldrich (St. Louis MO, USA). Isotopically labelled analogues (ISs), namely BPA-¹³C₁₆ (99%), BPAF-¹³C₆ (99%), BPB-¹³C₆ (99%), BPF-¹³C₆ (99%, 4,4'-Methylenediphenol), and BPS-¹³C₆ (98%) were purchased from Cambridge Isotope Laboratories (Andover MA, USA). Chemical structures, molecular weight, and octanol-water partition coefficients (log K_{ow}) of the above substances are presented in Table S1. Methanol (MeOH; LC-grade) was purchased from (Darmstadt, Germany). LC grade solvents of ethyl acetate, hexane, and acetone were supplied from VWR Chemicals (Oslo, Norway). Ammonium hydroxide solution (ACS reagent), ammonium acetate (≥98%) and sodium azide (NaN₃; neat) were purchased from Sigma-Aldrich (Steinheim, Germany). Water was purified with a Milli-Q grade water purification system (Q-option, Elga Labwater, Veolia Water Systems LTD, U.K.). Polyoxymethylene (POM) was purchased as sheets of 76 µm thickness (CS Hyde, USA).

2.2. POM passive sampler calibration

An equilibrium passive sampling method (EPS) using POM was developed to measure the freely dissolved concentration at equilibrium, C_{water} (µg L⁻¹). This approach avoids quantifying analytes that may be sorbed to dissolved, suspended, colloidal phases, such as nanoplastics or suspended microplastics, in the aqueous phase. The EPS-POM method was previously developed for BPA (Morin et al., 2015), but was further developed here by measuring POM-water partition coefficients, K_{POM} (Eq. 1) for other bisphenols and benzophenone UV-filters.

$$K_{POM} (\text{L kg}^{-1}) = C_{POM} / C_{water} \quad (1)$$

where C_{POM} is the equilibrium concentration in the POM phase (µg kg⁻¹).

POM samplers were cut into pieces weighing 0.10 g (± 0.05) and rinsed into an orbital shaker (VWR Standard Orbital Shaker, Model 5000) with a 50 mL solution of acetone:hexane (1:1 v/v) and shaken at 60 rpm for 24 h to pre-clean them. The process was repeated with 50 mL MeOH and followed by 50 mL Milli-Q water (which was performed three consecutive times). After cleaning, the POM strips were dried overnight on a surface of aluminium foil at room temperature and stored in clean amber vials.

A kinetic experiment was designed to obtain an understanding of bisphenol and benzophenone UV-filter uptake kinetics into POM passive samplers within the experimental setup applied here. For this purpose, a solution of 100 ng mL⁻¹ of target analytes, 10 ng mL⁻¹ of ISs and 1 g L⁻¹ NaN₃ (biocide) was prepared in Milli-Q water. Thirty-three mL of the obtained solution were transferred into 18 amber vials. Fifteen out of 18 vials contained 0.1 g of POM, while the other three were references (without POM). All the samples were shaken with the orbital shaker in the dark at 60 rpm in triplicates for 3, 7, 14, 21, and 28 days, respectively. POM strips were then removed, dried at room temperature under the laboratory fume hood and extracted with 10 mL of ethyl acetate. Losses of tested analytes to the air phase during drying was assumed to be minimal. The extraction was carried out in the darkness using the

orbital shaker at 60 rpm for 7 days, and the extract was concentrated to 250 μL under a gentle nitrogen stream and diluted with MeOH to 1 mL. Based on the results of the kinetic experiment (presented below) a shaking time of 21 days was considered sufficient to reach equilibrium between POM and Milli-Q water. Equilibrium K_{POM} values were then determined at four fortified concentrations (1, 10, 20, and 100 ng mL^{-1}), with a shaking duration of 21 days and using the aforementioned procedure. The values were compared with estimated K_{POM} values from the UFZ-LSER database (Ulrich et al., 2017), as presented in Table S2.

2.3. Waste sampling

Waste samples were collected from recycling and waste handling facilities located in the South Eastern part of Norway during a previous sampling campaign (Morin et al., 2015). The sampling locations included four facilities that defragment and sort e-waste, and/or vehicle waste, and one industrial waste sorting facility; their names are anonymised (Table S3) using the same nomenclature as in the previous studies that analysed these samples (Arp et al., 2020; Morin et al., 2015). Waste samples were collected by representatively subsampling large piles of a specific waste type, where a minimum of 10 spatially and visually diverse locations within the waste pile were sampled by hand (using nitrile gloves) and into polyurethane bags (VWR Norway). The samples included 3 different types of plastic cable granulates, 3 samples of plastic residue from crushed small electric waste sorted based on their exceedance of brominated flame retardant content thresholds (as determined by the waste facility), 4 vehicle fluff samples obtained at four car recycling facilities (one sample per facility), and 2 samples of municipal waste sorted for combustion (covering two size fractions, fine particles 2–16 mm and medium-sized particles >16 mm) from a municipal waste-to-energy plant for comparison (Table S2). The content of the samples was visually heterogeneous, including several types of materials in addition to the visually predominant plastic. Once in the laboratory, samples were ground and sieved through a 4 mm sieve. The grinding was carried out using a hand-powered malt mill (Bryggeland, Norway), a mortar and a pestle, and by directly sieving through a mesh (Morin et al., 2015). After processing the samples, they were stored under dark, dry conditions at 4°C until analysis. More information about the samples is presented in Table S3.

2.4. Waste concentrations

Ground and sieved (< 4 mm) waste subsamples (1.0 ± 0.2 g) were fortified with internal standards (ISs) and a solution of ammonium acetate (300 μL , 1 M) was added. Thereafter, 3 mL of ethyl acetate were added, and vortex-mixing for 1 min was performed. The samples were ultrasonicated for 45 min and preserved in the refrigerator overnight. Thereafter, samples were centrifuged at 3500 rpm for 10 min and the supernatants were collected. This process was performed 3 consecutive times, and consequently, a total of 3 supernatant fractions were obtained per sample and combined. 2 mL of Milli-Q water were added to the combined extract and centrifuged again (3500 rpm for 10 min). The obtained supernatant was collected, concentrated to a volume of 250 μL under a gentle stream of nitrogen, and a solution of MeOH:Milli-Q water (1:1 v/v) was added to a final volume of 1 mL.

2.5. Waste-water partitioning coefficients

The waste-water partitioning coefficients (K_{waste}) were determined following Eq. (2).

$$K_{\text{waste}} (\text{L kg}^{-1}) = C_{\text{waste}}/C_{\text{water}} \quad (2)$$

For this, a lab-scaled batch experiment of leaching was developed for the four samples displaying the highest concentrations of bisphenols and benzophenone UV-filters to ensure detection for as many analytes as

possible. The liquid/solid ratio (L/S) for these experiments was 33 L kg^{-1} , where 1 g of waste sample was added to a clean amber vial containing 0.1 g of POM strips, thereafter, fortified with ISs, and 1 g L^{-1} NaN_3 (biocide) in Milli-Q water. The obtained mixture was shaken under darkness for 21 days at 60 rpm. After shaking, the POM strips were removed and submitted for extraction as described above (Section 2.2.) to determine C_{POM} ; the freely dissolved bisphenol and benzophenone UV-filter concentrations were then converted into C_{water} using the previously calibrated K_{POM} value (Section 2.2.). It is assumed by this approach that the K_{POM} values are not dependent on the water chemistry after adding the waste samples, which is a reasonable assumption as K_{POM} values for neutral substances are not affected by salt ions, dissolved organic matter or colloids, which is its chief advantage over total water extracts concentrations (Endo et al., 2011), and salting-out effects are also likely negligible (Endo et al., 2012). The waste samples (solid state) were dried after shaking in an oven at 37 °C for 60 min and kept at room temperature overnight. Then, 0.1 g of dried waste sample were extracted with 3 mL of ethyl acetate, as described above to determine C_{waste} .

2.6. Instrumental analysis

The chromatographic separation was carried out using an Acquity UPLC I-Class system (Waters, Milford, U.S.) coupled to a triple quadrupole mass analyser (QqQ; Xevo TQ-S) with a ZSpray ESI ion source (Waters, Milford, U.S.). The LC column used was a Kinetex C18 EVO (50 \times 2.1 mm, 1.7 μm) connected to a Phenomenex C18 guard column (2.0 \times 2.1 mm). The column temperature was set at 30 °C. The mobile phase consisted of solvent (A) 0.1% v/v ammonium hydroxide in Milli-Q water and (B) MeOH. The flow rate was 300 $\mu\text{L min}^{-1}$ and the injection volume was 4 μL . The elution gradient started with 75% A, (0 min), decreased to 25% A within (0.1–3.6 min), then further decreased to 1% A (3.7–4.2 min), and reverted to 75% A (4.3–4.4 min). Analytes were ionized under negative mode (ESI-). Nitrogen was employed as drying gas in the ionization source (350 °C, 650 L hour^{-1}). The capillary was maintained at 1.5 kV, cone at 50 V and source offset at 30 V. Two transitions were recorded per compound (Table S4) considering a time window of 60 s around their retention time. The instrumental limits of quantification (LOQs) were calculated as the lowest concentration of analyte that can be quantified with a signal-to-noise ratio of 10. LC-MS/MS data was acquired with the MassLynx v4.1 software, while quantification processing was performed with TargetLynx (Waters, Milford, U.S.).

2.7. Quality assurance and quality control (QA/QC)

QA/QC protocols were implemented during the sample preparation and analysis to avoid any contamination or false-positive quantification. For this purpose, the glassware was cleaned with MeOH before and after use. One procedural blank was prepared simultaneously for every 5 samples to detect potential contamination during sample preparation. Signals in the procedural blanks were subtracted from the respective sample signal as they were assumed to be contamination valid for all samples. During the analysis, a mixture of standards of 20 ng mL^{-1} was injected at the beginning and at the end of the sequence, to account for potential instrumental drift. Analysis of solvent blanks were performed for every 10 samples along the sequence to check for sample carryover during analysis. Data analysis did not include non-detects (NDs).

2.8. Method performance

The trueness of the method was assessed through absolute recoveries (R %) and matrix effects (ME %), calculated as described by Matuszewski et al. (Matuszewski et al., 2003). Absolute recoveries of the target analytes were evaluated at a fortification level of 100 ng mL^{-1} . Obtained values are compiled in Table S5, demonstrating values of R % between 68% and 117% and ME (%) between 97% and 223%. Linearity was

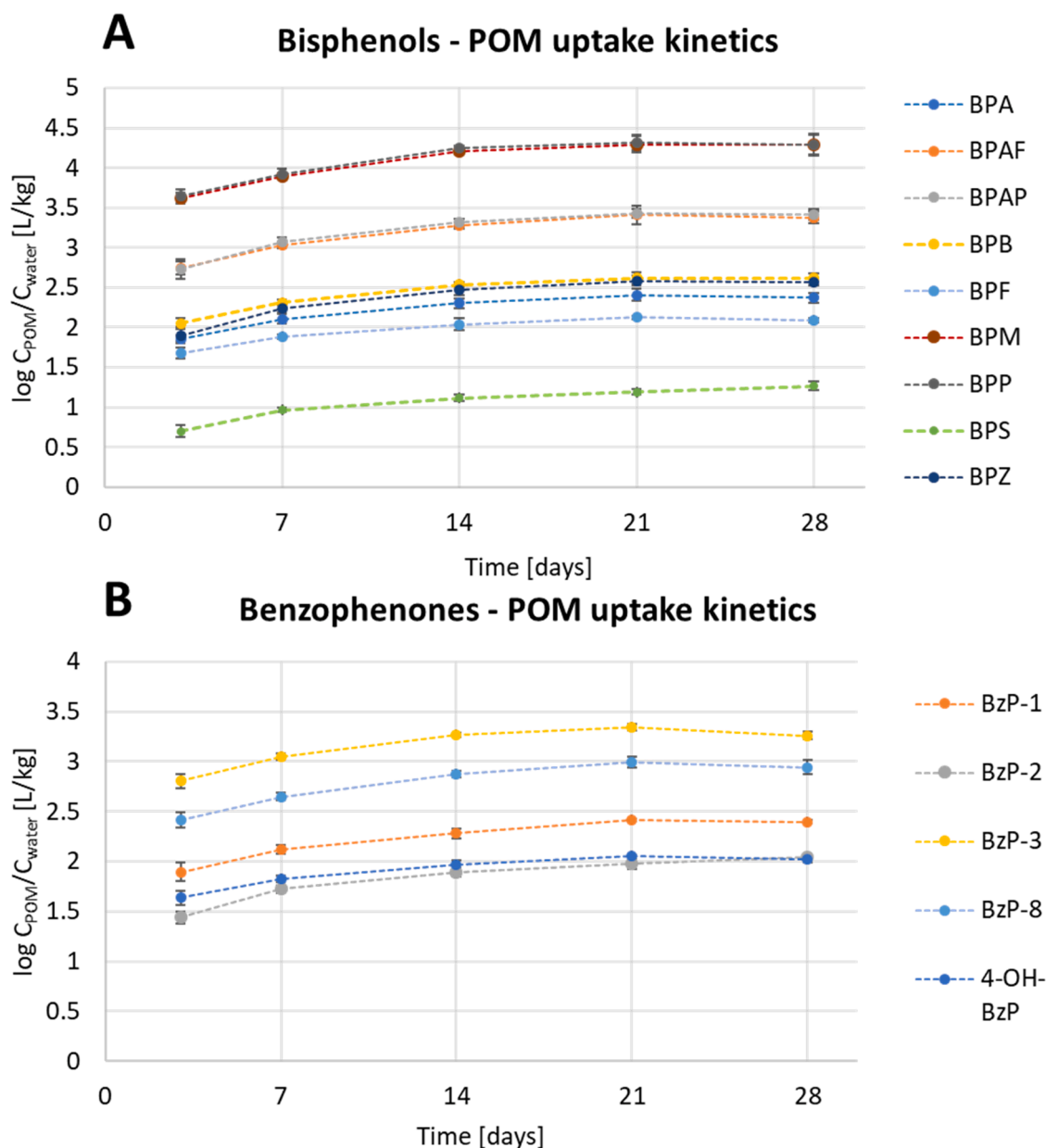


Fig. 1. Polyoxymethylene (76 μm) uptake kinetics for bisphenols (A) and benzophenones (B) as a function of time. Equilibrium partitioning is typically considered to occur when the $C_{\text{POM}}/C_{\text{water}}$ ratio plateaus, or achieves 80% of its maximum value, which generally appears at 14 days.

evaluated with standard solutions prepared in the range from 0.1 to 50 ng mL^{-1} , providing correlation coefficients (R^2) > 0.99 for all the target analytes. Repeatability and reproducibility in the responses of the compounds were studied by the injection of a 10 ng mL^{-1} standard solution 6 times and in 3 consecutive days, respectively. Instrumental limits of quantification (iLOQs) ranged between 0.1 and 0.2 ng mL^{-1} . Obtained R^2 , repeatability and reproducibility (expressed as RSD%), iLOQs as well as precursor and product ions of the target analytes, collision energies and cone voltage values for the LC-MS/MS analysis are summarized in Table S4. Positive identification for a target analyte was accepted when the variation of its relative retention time (RRT) was < 0.1 min and the variation of its corresponding ion ratio ($Q2/Q1$) was $\pm 30\%$ (SANTE, 2021).

Regarding POM experiments, the recoveries of the target analytes were calculated with fortified target analyte solutions (at a concentration of 100 ng mL^{-1}) after 28 days of shaking (Table S6). Analytes spiked into the blank control (without addition of POM, 100 ng mL^{-1} , 28 days of shaking, $n = 3$) were used as method recovery blank to correct for

mass losses of bisphenols and benzophenones UV-filters in the kinetic uptake experiment. The effect of mass loss was considered during establishing K_{waste} values when the mass recovery was < 60%. The obtained results are compiled in Table S6, demonstrating the suitability of POM as passive sampler. Recoveries > 67% were obtained for all the studied compounds except for BPZ, BzP-3 and BzP-8, whose values remained < 60%. For these three substances, a corrected concentration ($C_{\text{corrected}}$) was used since the deviation from the real value of 100% was higher than 30% (RSD) (SANTE, 2021). The same approach was followed for the calculation of C_{waste} and C_{water} :

$$C_{\text{corrected}} = C_{\text{measured}} / \% \text{Recovery} \quad (3)$$

These $C_{\text{POM,corrected}}$ was used for the interpretation of the kinetic uptake and K_{POM} partition coefficients for these three substances. Thus, the first order uptake rate constant, k , can be calculated with the concentrations in POM and water at the sampled time (t) points ($C_{\text{POM},t}$ and $C_{\text{water},t}$, respectively), using the following equation:

Table 1

First-order uptake rate constants, $\log K_{\text{POM}}$, isotherm R^2 based on the initial spiking amounts from 1 to 100 ng mL⁻¹, and comparison with UFZ-LSER predicted $\log K_{\text{POM}}$ (with values outside the application domain in *italic*).

Substance	POM uptake rate, k (day ⁻¹) (n = 3)	$\log K_{\text{POM}} (L_{\text{water}} \text{ kg}_{\text{POM}}^{-1})$ (n = 4)	Isotherm R^2	UFZ-LSER $\log K_{\text{POM}} (L_{\text{water}} \text{ kg}_{\text{POM}}^{-1})$
BPA	0.11 ± 0.01	2.5 ± 0.2	0.99	2.2 ± 0.3
BPAF	0.08 ± 0.02	3.4 ± 0.1	0.99	3.2 ± 0.7
BPAP	0.09 ± 0.02	3.4 ± 0.1	0.99	4.1 ± 0.7
BPB	0.11 ± 0.01	2.8 ± 0.2	1.00	3.5 ± 0.7
BPF	0.13 ± 0.03	2.3 ± 0.3	1.00	2.3 ± 0.7
BPM	0.09 ± 0.03	4.2 ± 0.4	0.83	5.3 ± 0.7
BPP	0.10 ± 0.01	4.3 ± 0.4	0.90	5.6 ± 0.7
BPS	0.10 ± 0.02	1.3 ± 0.1	1.00	1.5 ± 0.7
BPZ	0.09 ± 0.01	2.6 ± 0.1	0.99	4.2 ± 0.7
BzP-1	0.11 ± 0.00	2.5 ± 0.1	1.00	2.5 ± 0.7
BzP-2	0.09 ± 0.01	2.1 ± 0.2	1.00	2.0 ± 0.7
BzP-3	0.11 ± 0.01	3.1 ± 0.4	0.87	3.8 ± 0.3
BzP-8	0.10 ± 0.02	2.9 ± 0.2	0.99	3.3 ± 0.7
4-OH BzP	0.14 ± 0.00	2.2 ± 0.1	1.00	2.6 ± 0.3

$$C_{\text{POM},t}/C_{\text{water},t} = K_{\text{POM}}(1 - e^{-kt}) \quad (4)$$

3. Results and discussion

3.1. POM uptake kinetics

POM uptake kinetic data is plotted as the $\log(C_{\text{POM}}/C_{\text{water}})$ ratio as a function of the shaking time for the studied bisphenols and benzophenones in Fig. 1A and B, respectively. Obtained results demonstrated that 14 days of shaking is sufficient to reach equilibrium, practically defined as the time when the $C_{\text{POM}}/C_{\text{water}}$ ratio exceeds 80% of the maximum value or the time the value plateaus (Fig. 1) (Arp et al., 2015). A previous study developed by Morin et al. (Morin et al., 2015) reported an equilibrium time of 7 days for BPA. This difference is hypothesized to be related to the use of different shaker systems. In Morin et al.'s study, an

end-over-end shaker with vials filled at 90% working at 13 rpm was used for the experiment, while in the present research, an orbital shaker at 60 rpm was employed. The first-order uptake rate constant, k , for uptake in POM is presented in Table 1, as described in Eq. 4.

As evident, k values ranged from 0.08 ± 0.02 to 0.14 ± 0.00 (day⁻¹), indicating little dependence on the substance. Compared to Morin et al. who measured a k value of 0.52 using more effective shaking, this collectively indicates that the kinetics is controlled by the water phase and water motion on the water-POM interface, rather than the diffusion into the POM itself, unlike the case for larger molecules, e.g., large polychlorinated biphenyls, where POM diffusion can be a limiting factor (Arp et al., 2015). Additional set-ups using a broad range of organic substances could be used to develop a general theory for uptake kinetics in POM under different mixing conditions.

3.2. POM-water partition coefficients

POM partitioning coefficients (K_{POM}) were calculated at four initial concentrations of 1, 10, 20, and 100 ng mL⁻¹. The obtained values of $\log K_{\text{POM}}$ with the standard deviation and the R^2 for the corresponding sorption isotherms are presented in Table 1. The POM-water sorption isotherms for bisphenols and benzophenones UV-filters are presented in Fig. S1A and S1B, respectively. Eleven out of the 14 POM-water isotherm curves showed an excellent correlation providing R^2 values > 0.99. The isotherms for BPM, BPP and BzP-3 presented lower R^2 values, 0.83, 0.90 and 0.87, respectively. The reason is partially attributed to the large sorption (K_{POM}) for BPM and BPP, leading to most of the measured data close to their respective LOQ. In the case of BzP-3, which is transformed in the environment relatively fast (Mao et al., 2019), the lower correlation is likely related to it presenting the lowest mass recovery after 28 days (Table S6).

The $\log K_{\text{POM}}$ results were compared with literature values for BPA and estimated values for the other substances, using the POM-specific poly-parameter linear free energy relationship by Endo et al. (2011) and the substance descriptors obtained from the UFZ-LSER database (Ulrich et al., 2017). From this database, experimental descriptors were only available for BPA, BzP-3 and 4-OH-BzP, indicating that these estimates are the most reliable. The rest of the estimates used were

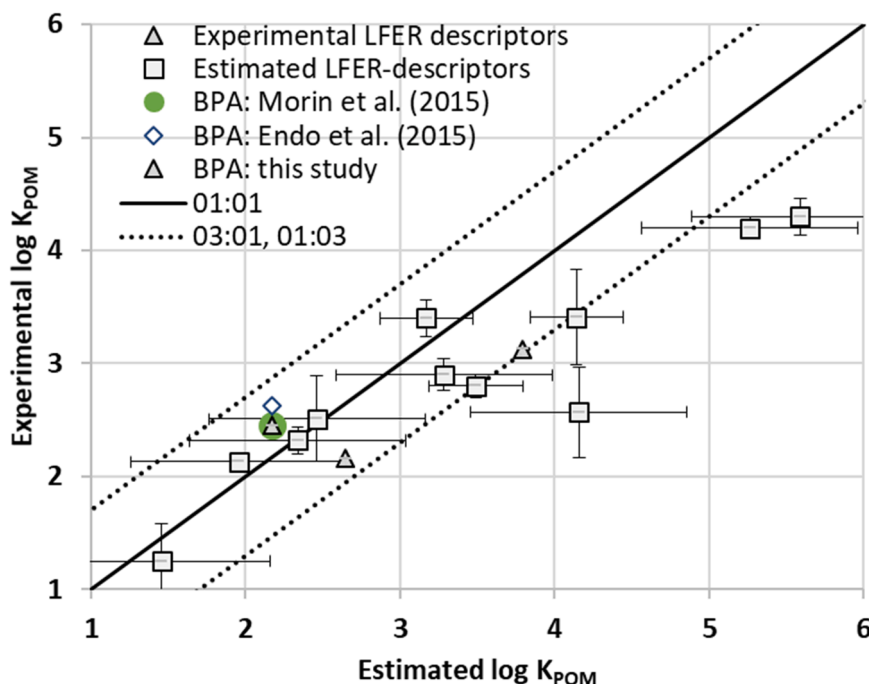


Fig. 2. Comparison between experimental and estimated $\log K_{\text{POM}}$ values for bisphenols and benzophenones.

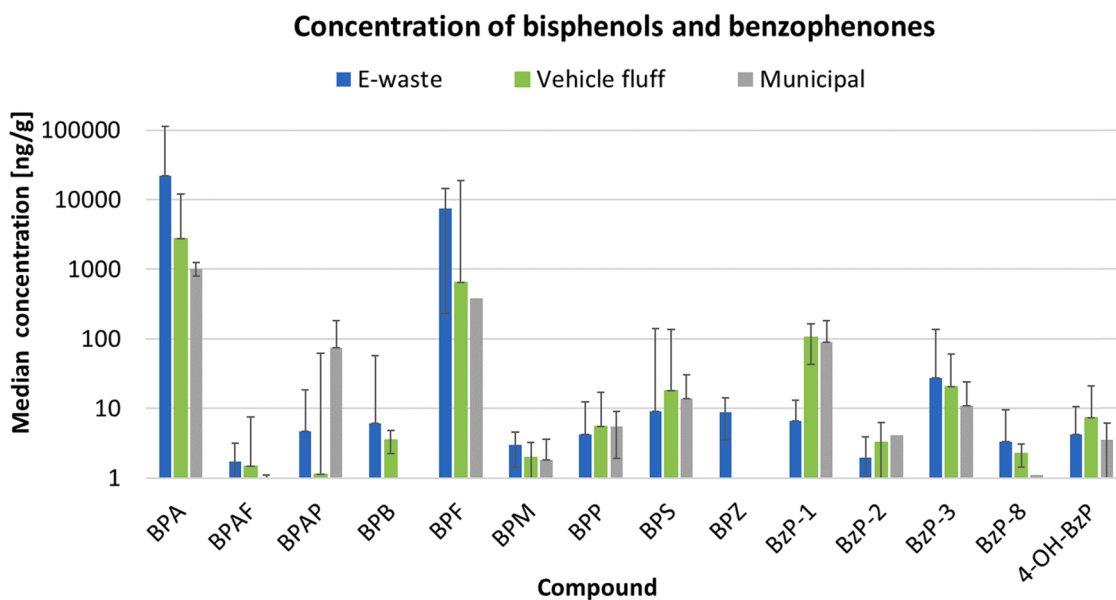


Fig. 3. Median concentrations of bisphenols and benzophenones measured in homogenized e-waste plastic (n = 6), vehicle fluff (n = 4) and municipal waste (n = 2).

predicted descriptors that were considered outside the chemical applicability domain, and therefore should be used with caution. Fig. 2 shows the comparison between the experimental and estimated log K_{POM} values for bisphenols and benzophenone UV-filters, providing a correlation coefficient (R^2) of 0.83, including the outliers.

The measured log K_{POM} in this study (2.5 ± 0.2) matched those reported previously of log K_{POM} 2.45 ± 1.2 (Morin et al., 2015) and 2.63 (Endo et al., 2011). For comparisons with estimated K_{POM} values, all were within a factor 3 (Fig. 2), except for BPM, BPP and BPZ, which were overestimated by 1.6, 1.1, and 1.3 orders of magnitude, respectively. Possible reasons include the uncertainty in the K_{POM} estimations (Table S2), high standard deviations for BPM and BPP (0.4 orders of magnitude) and the lower recoveries of BPZ (52%).

3.3. Bisphenols and benzophenone UV-filters in e-waste and vehicle fluff samples

The concentrations of bisphenols and benzophenone UV-filters in the collected e-waste plastic, vehicle fluff and municipal waste samples are presented in Fig. 3. The mean, median, maximum and minimum concentrations, as well as their standard deviation (SD) and detection frequency (%) can be found in Table S7. To the best of our knowledge, concentrations of bisphenol analogues (other than BPA) and benzophenones were reported in these types of samples for the first time.

The highest median concentrations were for BPA and BPF, which both had a detection rate of 100%. Regarding the type of samples, median concentrations of BPA in e-waste was higher than in vehicle fluff, $22,022$ and 6719 ng g^{-1} , respectively, while in case of BPF the tendency was the opposite, with median concentrations of 7426 ng g^{-1} in vehicle fluff versus 4637 ng g^{-1} in e-waste. Similar concentrations were reported for BPA in e-waste and vehicle fluff by Arp et al., $71,188$ and 6492 ng g^{-1} , respectively (Arp et al., 2017). The higher concentration of BPF than BPA in vehicle fluff, despite BPA being historically the most utilized bisphenol, may be related to BPF being more commonly used in applications with increased thickness and durability requirements (İyigünođdu et al., 2020). In the case of individual benzophenone UV-filters, median concentrations between 2 and 104 ng g^{-1} were measured. Even though the concentrations of this class of chemicals is not as large as those found for bisphenols, it is noteworthy to report that the detection frequency of BzP-1 and BzP-3 was 100%.

A comparison of median concentrations measured for bisphenols and

Table 2

Measured K_{waste} values and percentage of substance leached to the water phase in the batch tests (L/S = 31).

Substance	Vehicle fluff (n = 1) log K_{waste} (L kg^{-1})	E-waste (n = 3) log K_{waste} (L kg^{-1})	Fluff and E-waste (n = 4) Average percentage leached (%)
BPA	2.62	2.55 ± 0.14	7.6
BPAF	2.25	3.58 ± 0.35	3.5
BPAP	nd	3.46	0.5
BPB	nd	nd	–
BPF	3.65	3.18 ± 0.36	1.1
BPM	nd	4.03 ± 0.63	0.4
BPP	nd	4.24 ± 0.48	0.1
BPS	1.36	1.72 ± 0.32	83
BPZ	nd	2.39 ± 0.17	18
BzP-1	2.58	2.54 ± 0.61	7.8
BzP-2	1.79	1.68 ± 0.18	62
BzP-3	2.50	3.21 ± 0.47	2.2
BzP-8	nd	nd	–
4-OH-BzP	2.04	2.37 ± 0.15	14
Morin et al. (2015)	(n = 5)	(n = 7)	
BPA	2.9 ± 0.7	2.5 ± 1.1	–

benzophenones in different waste types is presented in Fig. 3. The waste category with the largest median concentrations of BPA, BPB, BPM, BPZ, BzP-3 and BzP-8 was e-waste. The vehicle fluff presented the highest median concentrations of BPAF, BPAP, BPF, BPP, BzP-1, BzP-2 and 4-OH-BzP. BPS was present within the same order of magnitude in the two waste types. Mixed municipal waste, by comparison, demonstrated lower concentrations of bisphenols than e-waste and vehicle fluff, but higher concentrations of benzophenones, particularly for BzP-1 (median concentration 90.3 ng g^{-1}). Evidentially, there is a large variety of BPA alternatives and benzophenone UV-filters in materials ending up in e-waste and vehicle fluff.

3.4. Waste-water partition coefficients

Obtained K_{waste} values, as well as the percentage of bisphenols and benzophenones that entered the water phase during the partitioning experiments, are presented in Table 2. BPS is the bisphenol that is most readily leached into water (83% leached in the experiment), and BzP-2 is

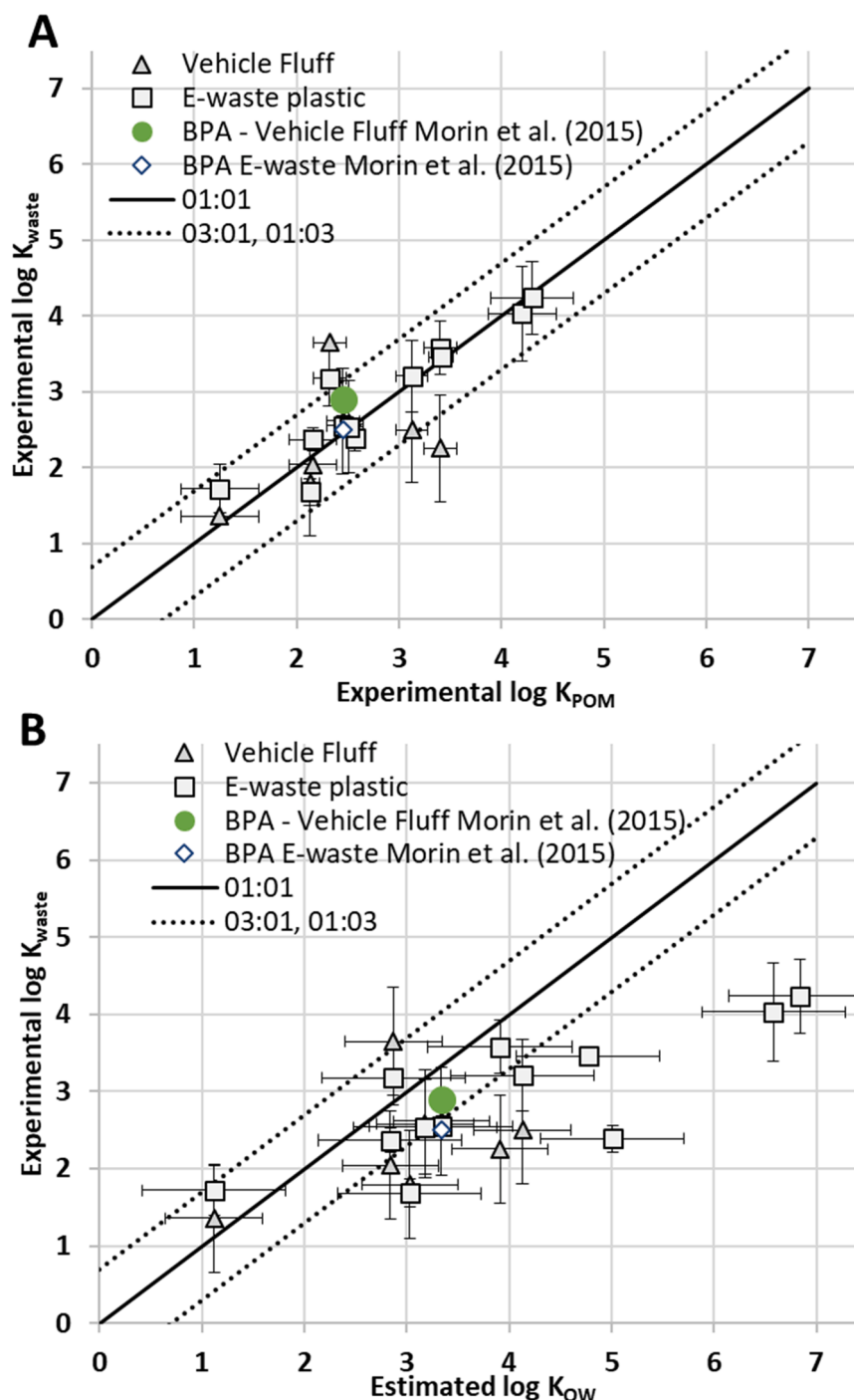


Fig. 4. Comparison between experimental $\log K_{\text{waste}}$ values for bisphenols and benzophenones compared to a) experimental $\log K_{\text{POM}}$ ($r^2 = 0.82$) and b) estimated $\log K_{\text{OW}}$ ($r^2 = 0.67$).

the most leachable of the benzophenones (62% leached). The strongest sorbing bisphenols are BPP (0.1% leached) and BPM (0.4% leached), and the strongest sorbing benzophenone is BzP-3 (2.2% leached). All the $\log K_{\text{waste}}$ values in Table 2 are reported for first time in the literature, except for BPA, where there was a good agreement with a previous study using a different method (Morin et al., 2015). Here we measured $\log K_{\text{waste}}$ for BPA in vehicle fluff and e-waste at 2.62 and 2.55 ± 0.14 , respectively, whereas the previous study measured 2.9 ± 0.7 and 2.5 ± 1.1 , respectively.

The $\log K_{\text{waste}}$ data are compared with $\log K_{\text{OW}}$ data (SI Table S2) in Fig. 4. $\log K_{\text{OW}}$ is often used as a screening parameter for the sorption of

neutral organic compounds to soil, lipids, and other environmental matrices. As evident from Fig. 4, $\log K_{\text{OW}}$ values and $\log K_{\text{waste}}$ were somewhat correlated ($R^2 = 0.67$); though, for most substances $\log K_{\text{OW}}$ tend to be much larger than $\log K_{\text{waste}}$, sometimes by over a factor 100, such as for BPP ($\log K_{\text{waste}} 4.24 \pm 0.48$ vs $\log K_{\text{OW}} 6.84$) and BPM ($\log K_{\text{waste}} 4.03 \pm 0.63$ vs $\log K_{\text{OW}} 6.58$). This may be because the waste materials contain crystalline domains, where solutes cannot enter, unlike in octanol. However, because the e-plastic and vehicle fluff waste fractions assessed here were either explicitly plastic or rich in plastic, and the POM passive sampler used in this study is also a plastic, and a semi-crystalline one at that, the $\log K_{\text{POM}}$ values derived in this study

could be considered an even more appropriate surrogate K_{waste} than $\log K_{\text{POM}}$. A comparison between $\log K_{\text{waste}}$ with K_{POM} and K_{ow} is presented in Fig. 4. As evident from Fig. 4, most bisphenols and benzophenones have K_{waste} values that agree with the corresponding K_{POM} within a factor of 3 (only 4 substances fall slightly out of this range), and the two parameters exhibit a good correlation (Fig. 4A, $R^2 = 0.82$ for all waste and substances, while 0.84 just for e-waste plastic). On the other hand, the correlation between K_{waste} and K_{ow} (Fig. 4B) presented a lower R^2 value ($R^2 = 0.67$), as mentioned. The reason for this can be attributed to two causes. The first cause is that the K_{ow} were estimated, as experimental values were not available, and by-default there is some uncertainty with such estimations. The second cause is due to octanol being a completely liquid phase, whereas most waste plastics and POM are semi-crystalline solids, as mentioned above. It would be of interest to compare these K_{waste} values with K values for other plastic materials; however, for the substances considered in this study, experimental sorption coefficients could only be found for BPA (Liu et al., 2019), and not the other bisphenol analogues nor the benzophenone UV-filters.

4. Conclusions

The bisphenols and benzophenone UV-filters displaying the highest concentrations in e-waste and vehicle fluff samples were BPA, BPF and BPS with maximum concentrations of 246,000, 42,400 and 332 ng g^{-1} , respectively, and detection frequencies above 75%. Other substances detected in all samples in considerable amounts were BzP-3, BzP-1 and BPAP, whose maximum concentrations were 285, 155 and 150 ng g^{-1} , respectively.

The POM-water sorption isotherms exhibited strong linearity (from $R^2 = 0.83$ –1.00) over the studied concentration range, with equilibrium being achieved in 14 days using an orbital shaker. A very good correlation between $\log K_{\text{waste}}$ and $\log K_{\text{POM}}$ was obtained ($R^2 = 0.82$). POM can be considered a suitable passive sampler for measuring the partitioning behaviour of bisphenols and benzophenone UV-filters in e-waste and vehicle samples. BPP and BPM displayed the highest K_{POM} and K_{waste} values, as expected by their larger K_{ow} . Similarly, K_{POM} and K_{waste} were smaller for the compounds having lower K_{ow} such as BPS, BzP-2, 4-OH-BzP and BPA. This tendency was also supported by the latter compounds having the highest percentages of leaching from the samples: 83%, 62%, 14% and 7.6%, respectively.

Regarding the recycling of these products, whether a high or low sorption coefficient of a given pollutant is favourable depends mainly on the desired recycling process. If the approach is to keep the substance within the material, an additive such as BPP, BPM, BPAP and BPAF could be appropriate, whereas an additive such as BPS, BzP-2 or 4-OH-BzP could be more suitable if the aim is to extract it. However, further risk assessment regarding the toxicity and environmental hazard of the compounds should be considered within a holistic study of relevant recycling and reuse processes. The methodology in this study could be further expanded towards more types of substances and waste fractions, in order to better assess the presence, leachability and recyclability of waste containing emerging, hazardous contaminants.

Implementing a circular economy means to maintain the value of products and materials for as long as possible, through increasing the reuse and recycling of these products and materials, thereby minimizing the generation of their waste and production from virgin materials. When considering emerging contaminants, e.g., bisphenols and benzophenone UV-filters, the ideal situation would be that these substances were not present. However, whether they have a high or low K_{waste} value has interesting implications for their fate within a circular economy context. Substances with high K_{waste} values (like BPP, BPM, BPAP and BPAF) are more likely to stay within the product during reuse and mechanical recycling processes and would leach out to effluent water at a slower rate during waste treatment. On the other hand, substances with lower K_{waste} values (such as BPS, BzP-2 or 4-OH-BzP) could be easier to extract during a recycling process, such as with aqueous-based solvents;

however, the risk of exposure to the surrounding aqueous environment would be larger. The human health and environmental risks of these and other hazardous substances should be considered within a holistic assessment of recycling, reuse, and waste management processes to ensure a safe and sustainable transition to a circular economy.

CRedit authorship contribution statement

Kristin Runde: Writing – original draft, Investigation, Methodology, Formal analysis. **Gabriela Castro:** Writing – original draft, Writing – review & editing, Formal analysis. **Kristine Vike-Jonas:** Investigation. **Susana Vila González:** Investigation. **Alexandros G. Asimakopoulos:** Writing – review & editing, Conceptualization, Methodology, Formal analysis, Project administration, Supervision, Validation, Funding acquisition. **Hans Peter H. Arp:** Writing – reviewing & editing, Conceptualization, Methodology, Formal analysis, Project administration, Supervision, Validation, Funding acquisition.

Novelty statement

Bisphenols and benzophenones are two groups of high production, hazardous chemicals used in electronics and vehicles. The presence of many of them in e-waste and vehicle "fluff" residues, and their emissions during waste management, are unknown. Herein, a new passive sampler methodology using polyoxymethylene (POM) was calibrated for the first time to understand the leachability of these diverse bisphenols and benzophenones from e-waste and vehicle fluff. This method, and the dataset of concentrations and waste-sorption coefficients for e-waste and vehicle fluff herein, can be used in risk, safety, and sustainability assessments for various waste management strategies.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.jhazmat.2021.127814](https://doi.org/10.1016/j.jhazmat.2021.127814).

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