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The study of polar emerging contaminants in seawater by passive sampling: A review

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ABSTRACT

Emerging Contaminants (ECs) in marine waters include different classes of compounds, such as pharmaceuticals and personal care products, showing "emerging concern" related to the environment and human health. Their measurement in seawater is challenging mainly due to the low concentration levels and the possible matrix interferences. Mass spectrometry combined with chromatographic techniques represents the method of choice to study seawater ECs, due to its sensitivity and versatility. Nevertheless, these instrumental techniques have to be preceded by suitable sample collection and pre-treatment: passive sampling represents a powerful approach in this regard.

The present review compiles the existing occurrence studies on passive sampling coupled to mass spectrometry for the monitoring of polar ECs in seawater and discusses the availability of calibration data that enabled quantitative estimations. A vast majority of the published studies carried out during the last two decades describe the use of integrative samplers, while applications of equilibrium samplers represent approximately 10%. The polar Chemcatcher was the first applied to marine waters, while the more sensitive Polar Organic Chemical Integrative Sampler rapidly became the most widely employed passive sampler. The organic Diffusive Gradients in Thin film technology is a recently introduced and promising device, due to its more reliable sampling rates. The best passive sampler selection for the monitoring of ECs in the marine environment as well as future research and development needs in this area are further discussed. On the instrumental side, combining passive sampling with high resolution mass spectrometry to better assess polar ECs is strongly advocated, despite the current challenges associated.

1. Introduction

Emerging contaminants (ECs) are a wide group of substances whose presence in the environment has raised the attention of the scientific community in the last two decades. The EC term can refer to "contaminants which have appeared only recently", but also to contaminants of emerging concern, namely "contaminants which have been in the environment for a while but for which concerns have been raised much more recently" (Sauvé and Desrosiers, 2014). Some traditional contaminants can also have "emerging issues" when new information on environmental and human health risks appear (Sauvé and Desrosiers, 2014). Although these contaminants have been detected in the most diverse environmental ecosystems, they are not currently included in routine monitoring programs; nevertheless, most of them have been introduced in some "watchlists", and they may be subjected to future regulation (Jones et al., 2015).

ECs in marine waters include mainly hydrophilic classes such as current-use pesticides, pharmaceuticals and personal care products (PPCPs), veterinary products, stimulants, food additives, corrosion inhibitors and illicit drugs (Brumovský et al., 2017). Measurement of hydrophilic organic contaminants in seawater is challenging mainly due to the high dilutions and the complexity of the matrix (presence of salts, ionic species and biota) (Jones et al., 2015; Mills et al., 2014; Vidal-Dorsch et al., 2012). Furthermore, Environmental Quality Standards (EQS) in coastal waters require for some compounds detection levels at up to an order of magnitude lower than for inland waters (Jones et al., 2015). The remarkable progress in analytical techniques for trace analysis, with the development of new and more sensitive methods for detecting chemicals has permitted to study analytes that were not considered in the past. In this context, mass spectrometry (MS) plays a central role, as demonstrated by the considerable number of publications on environmental studies carried out by means of this technique (Magi

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and Tanwar, 2014). Combined with chromatographic techniques, MS is the most sensitive and versatile technique to meet the requirements of seawater quality monitoring, thus currently the method of choice for such studies. However, these powerful methodologies need suitable sample collection and pre-treatment. Indeed, conventional spot sampling may not allow sufficient sensitivity (Jones et al., 2015), as contaminants are often detected at concentrations close to detection limits (Vidal-Dorsch et al., 2012). Moreover, due to the complex dynamics of the coastal marine environment, discrete samples may not offer a representative picture of contaminant concentrations (Jones et al., 2015). Passive sampling represents a powerful approach to overcome these problems. Passive samplers are generally deployed in water for several days or weeks, allowing an in-situ pre-concentration of the contaminants, which can improve sensitivity (Vrana et al., 2005). Also, in the case of integrative samplers, the relatively long deployments permit an estimation of time-weighted average (TWA) concentrations, rather useful for the assessment of marine water quality.

The operating principle of passive sampling in water has been described in detail previously (Godlewska et al., 2021). It is based on contaminant diffusion from a medium (the water) to a receiving phase (the sampler) resulting from the difference in chemical activity of the contaminant in the two compartments (Allan et al., 2009). The uptake rate of the analytes into a sampler depends on the sampler design, the physicochemical properties of the analytes and environmental variables (Godlewska et al., 2021). Passive samplers can generally be classified as either single- or two-phase passive samplers (a receiving phase and diffusion membranes) (Godlewska et al., 2021). Passive sampling technologies have widely spread in recent years for freshwater monitoring, while, in comparison, the application to marine waters remains a less investigated field.

A recent work (Gong et al., 2018) discussed the use of the most popular integrative samplers for the study of ECs in freshwater, by focusing on the sampler configurations and characteristics. Other reviews regard seawater monitoring of certain EC groups, without giving specific attention to the sampling approach (Branchet et al., 2021; Cole et al., 2015). Only the paper by Schintu et al. described the use of passive samplers for the monitoring of marine contaminants, by focusing on traditional ones, such as polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) (Schintu et al., 2014). The current review fills the gap, giving an update on the different studies using passive sampling techniques (combined with mass spectrometry) for polar ECs in seawater and complements the other reviews present in the literature. Current challenges and future prospects in the field are discussed.

2. Passive samplers for polar emerging contaminants

Marine applications for passive samplers were first mainly for traditional and mostly hydrophobic contaminants, such as PAHs, PCBs and organochlorine pesticides (Huckins et al., 1997; Prest et al., 1995). Some have also been applied for several classes of emerging hydrophobic contaminants (Pintado-Herrera et al., 2020; Sacks and Lohmann, 2011).

In the past two decades, more and more work has been conducted on passive sampling of polar compounds ($1 < \log K_{ow} < 4$) in marine waters, mainly using two-phase passive samplers with a solid sorbent (Branchet et al., 2021). The most popular devices are the Polar Organic Chemical Integrative Samplers (POCIS), the polar version of the Chemcatcher and the organic Diffusive Gradients in Thin film technology (o-DGT). Such samplers typically work in the kinetic sampling mode as time-integrative samplers: contaminant uptake should remain linear over the whole duration of the deployment, thanks to the presence of diffusion limiting membranes. As such, each TWA concentration can be estimated from the mass of compound sampled, using the sampling rate (Rs) and the exposure time (Vanryckeghem et al., 2021). Alongside with the integrative samplers, the use of equilibrium-type devices has been reported, although less frequently.

Fig. 1 shows the number of publications for each methodology over the last 21 years, highlighting the trend in the application of the different approaches.

Most published works on freshwater monitoring use the POCIS (Mills et al., 2014), and this is also the case for seawater.

2.1. POCIS

The "standard" POCIS is generally used in marine occurrence studies. It consists of a Hydrophilic-Lipophilic-Balanced (HLB) solid sorbent phase, sandwiched between two microporous polyethersulfone (PES) membranes and held together by stainless steel rings (Alvarez et al., 2004). Table 1 shows a summary of the most relevant information regarding works on seawater quality assessment using standard POCIS.

Some of the first documented uses of standard POCIS in marine waters were to detect alkylphenols (some of which are still considered as ECs), in the open sea. In particular, POCIS played an important role in seawater column monitoring for alkylphenols (APs) and naphthenic acids close to two offshore oil production platforms in Norway (Harman et al., 2009, 2010, 2011b, 2014, 2011b). $\mathrm{Log}K_{ow}$ for APs is in the range 2.3–5.8 (Harman et al., 2008). It follows that for some of them, hydrophobic samplers should be more suitable (Vrana et al., 2005). Nonetheless, POCIS have been shown to be applicable for APs with $\log K_{ow} < 5$. Six APs as well as a large list of pesticides and pharmaceuticals were also monitored in large-scale characterization studies of coastal waters along French coastlines (Mediterranean Sea and oversea territories (Gonzalez-Rey et al., 2009; Gonzalez et al., 2014; Turquet et al., 2010)).

More recent studies showed an extension of the POCIS use to numerous classes of hydrophilic and moderately hydrophobic ECs, such as PPCPs, munition constituents, pesticides and perfluorinated chemicals. In most cases the focus was on the contribution of wastewater in the contamination of coastal areas or on large-scale monitoring.

Compared to deployment in freshwaters, it is rather complex to check the POCIS status during the deployment time in the sea: it is not usually possible to know if biofouling is starting to accumulate on the membranes or if the POCIS have been damaged prior to their final recovery. This problem is compounded by the need for longer deployments in less contaminated remote areas due to higher dilution factors or greater distance from point sources. By contrast, easy-to access deployment sites, such as ports and harbours, may allow POCIS to be regularly checked during their exposure.

Many of the studies do not specify the deployment depth which must be chosen with consideration, so that the sampler always remains submerged throughout the deployment. Some POCIS were reported to have been deployed approximately 1–2 m below the surface. While this

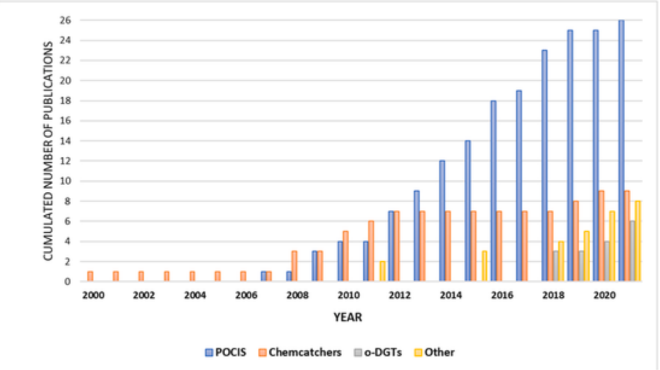

Fig. 1. Cumulative publications on integrative passive samplers (POCIS, Chemcatchers, o-DGTs and others) for polar emerging contaminants since the first application in marine waters.

Table 1
Marine applications of standard-POCIS to monitor polar ECs. (Only classical targeted studies were considered while researches employing untargeted analysis are discussed in paragraph 3.4.)

Analytes	Number of sites	Water type	Study area	Duration (days)	TWA range (ng L^{-1})	Analysis	Reference
14 pharmaceuticals	1	Transitional (Estuarine)	Seine estuary, on the dam of Poses (France)	3 and 34	ng to hundreds of ng L^{-1} (over 3 days)	GC-MS	Togola and Budzinski (2007)
46 APs	8	Open sea (reference site/contaminated site)	Nearby the Ekofisk oil production platforms (Norway)	42	∑APs = 20–57 and 11 at the ref site	GC-MS after derivatization	Harman et al. (2009)
23 pharmaceuticals and pesticides	1	Coastal (fish farm cages)	Southeast of Spain	14–15	ND–75.6	LC–QLIT-MS/MS	(Martínez Bueno et al., 2009)
APs	6	Open sea (reference site/contaminated site)	Nearby the Statfjord B platform (Norway)	42	∑APs = 40–70	GC-MS after derivatization	Harman et al. (2010)
21 pharmaceuticals, 6 APs and 27 pesticides	13	Transitional/Coast	Bays, lagoons and coast (France)	14–28	ND–41	GC-MS (herbicides), LC-MS/MS (pharmaceuticals and herbicides) LC-MS (APs)	Munaron et al. (2012)
7 pesticides, 2 TPs	1	Transitional (Lagoon)	Thau Lagoon, Hérault (France)	14	ND–10	Not specified which between LC-MS, LC-MS/MS and GC-MS	Miège et al. (2012)
68 compounds including ECs (fragrances, pesticides …)	4	Coastal	Coastal reefs, St. John (US)	28	ND–820	GC-MS/ECD	Bargar et al. (2013)
166 wastewater compounds, pharmaceuticals, and pesticides	11	Coastal	San Francisco Bay and Southern California Bight (US)	28–30	ND–8900	GC-MS (pesticides), HPLC-MS/MS (pharmaceuticals)	Alvarez et al. (2014)
12 pharmaceuticals and EDCs	1	Transitional (estuarine)	Mangrove estuary (Singapore)	28	ND–265	LC–MS/MS	Bayen et al. (2014)
19 pharmaceuticals and 6 TPs	3	Coastal water (reference site/contaminated site)	Nearby a WWTP effluent discharge Helsinki, (Finland)	31	concentrations expressed in ng/POCIS	LC–MS/MS	Turja et al. (2015)
19 pharmaceuticals and 47 pesticides	2	Estuarine (impacted)	Arade estuary (Portugal)	30	ND–804	LC–MS/MS	Gonzalez-Rey et al. (2015)
7 pharmaceuticals and TPs	4	Coastal (impacted)	Nearby a submarine pipeline outfall + 2 beaches (France)	30	ND–1.17	LC-HRMS (LC-Orbitrap-MS)	(Martínez Bueno et al., 2016)
17 pharmaceuticals and EDCs	9	Mangroves (urban pressure)	Singapore	28–29	ND–1918	LC-MS/MS	Bayen et al. (2016)
28 wastewater micropollutants	3	Coastal (wastewater outfall)	Cambridge Bay, Nunavut (Canada)	9 and 14	ND–2.7	UHPLC-MS/MS	Chaves-Barquero et al. (2016)
15 PPCP wastewater markers	3	Estuarine and coastal	Auckland marine environment (New Zealand)	20–21	ND–5.87	LC-MS/MS	Stewart et al. (2016)
7 alkylphenols, BPA and 6 steroids	2	Coastal	La Maddalena Archipelago (Italy)	49	ND	GC-MS/MS	Moschino et al. (2017)
Organic wastewater contaminants	3	Transitional (Lagoon)	Biscayne Bay (US)	NS	ND-710	GC-MS	Bargar et al. (2017)
1 pesticide	5	Transitional (Lagoon)	Arcachon Bay (France)	28	ND–40	LC-MS/MS	Fauvelle et al. (2018)
1 pharmaceutical	3 (x 3 depths)	Open Sea	Baltic Sea	21	concentrations expressed in ng/g POCIS	LC-MS/MS	Björlenius et al. (2018)
2 industrial additives	3	Transitional (Lagoon)	Coastal area of Portofino Promontory (Italy)	22, 45, 72	29–547	GC-MS	Di Carro et al. (2018)
25 pesticides and 7 TPs	1	Transitional (Lagoon)	Bizerte lagoon (Tunisia)	21	ND–13.5	HPLC-MS/MS	Mhadhbi et al. (2019)
1 pharmaceutical, 3 EDCs, 8 pesticides	3	Transitional (Estuarine)	Dublin and Cork catchments (Ireland)	30	ND–2.36 for estrogens. Pesticides and diclofenac results in ng/POCIS	HPLC-MS/MS	Jones et al. (2019)
3 microcystins + 4 TPs	3	Transitional (Estuarine)	Geum River Estuary (Korea)	7	2.1–28	HPLC-MS/MS	Kim et al. (2021)

APs: alkylphenols, EDCs: Endocrine Disrupting Compounds, ND: not detected, NS: not specified, PPCP: Pharmaceuticals and Personal Care Product, TPs: metabolites/transformation products, WWTP: wastewater treatment plant.

may work in semi-enclosed sites (harbours, ports, lagoons), large waves may destroy the POCIS membranes in more demanding marine areas, such as open seas. For example, in a campaign in 2008, 2 samplers out of 9 were lost due to bad weather (Gonzalez-Rey et al., 2009) and in a large-scale study of the Mediterranean coast, the loss regarded 30% of the POCIS (7/20 sites, 42/180 POCIS) (Gonzalez-Rey et al., 2009). Deploying the POCIS deep enough may protect the sampler from damage as well as biofouling. However, in deeper deployments, turbulence is generally lower, thus increasing the thickness of the water boundary layer (i.e. the stagnant diffusion layer) and decreasing sampling rates. In addition, contaminant concentrations may be lower, especially those of more hydrophobic compounds (Aminot et al., 2017), thus making it advisable to place the POCIS closer to the surface. A compromise should be selected considering both the physico-chemical properties of the ECs and the characteristics of the site. In one study, no great difference was found for the carbamazepine accumulation in POCIS deployed at different depths, within the first 40 m from the surface (Björlenius et al., 2018). Still, further studies need to be conducted for more hydrophobic and/or more degradable ECs (Björlenius et al., 2018).

As already mentioned, the most used sorbent material is HLB. Another sorbent, which consists of Isolute ENV + polystyrene divinylbenzene resin and Ambersorb 1500 carbon dispersed on S-X3 Biobeads, was also tested for seawater. In two comparative calibration studies, the standard HLB-POCIS gave better results for the targeted analytes (munition constituents) in terms of compound loss from the sorbent (Belden et al., 2015; Hernando et al., 2005). In recent years, POCIS-like samplers have been developed using innovative sorbents such as: mixed-mode ion exchange polymeric sorbents, ionic liquids, molecularly imprinted polymers and carbon nanotubes, to increase the range of POCIS applicability (Godlewska et al., 2021). Some of these have been successfully applied for seawater and are summarized in Table S1 (Supplementary Material).

Regarding the form in which the data is provided, some of the studies only reported the mass of analytes accumulated in the POCIS. On the other hand, among those which measure TWAs, lab-derived Rs are mainly used, some from freshwater evaluations. Still, Rs can be affected by marine conditions, such as the salinity, with a compound-specific behaviour (Bayen et al., 2014; Lis et al., 2019; Shi et al., 2014; Togola and Budzinski, 2007; Zhang et al., 2008). Thus, the application of freshwater-derived Rs to the marine environment should be carried out with caution (Harman et al., 2012), while the determination of more accurate Rs, under appropriate salinity conditions, should be desirable (Bayen et al., 2014). Shi et al. focused on the use of POCIS in the estuarine environment and used a range of field Rs, calculated at different salinities (Shi et al., 2014).

An alternative to Rs measurement under conditions matching the deployment site, is the use of Performance Reference Compounds (PRCs). Nevertheless, only Gonzalez-Rey et al. corrected sampling rates by using this strategy (Gonzalez-Rey et al., 2015). Harman et al. preferred to adjust POCIS sampling rates by studying the dissipation of the PRCs from co-deployed hydrophobic passive samplers (Harman et al., 2009, 2011b). The questions on the reliability of the PRC approach for POCIS (Harman et al., 2011a) may have limited its application in seawater studies.

A further parameter to consider is the deployment time. As highlighted in Table 1, a rather wide range of deployment durations (3–72 days) has been used in POCIS studies. This may be justified by the fact that the duration choice strictly depends on the expected concentrations in water, on the Rs and the site conditions. Still, if the purpose is monitoring the water quality of several geographical areas, a standardization of exposure times should be applied. If not, a reliable comparison among the different studies is hindered. On the one hand, the longer the deployment, the higher the probability that the site conditions would change, thus increasing the uncertainty connected with the Rs. On the other hand, longer deployments do not guarantee that the sampling is still in its linear uptake regime. A strategy to verify this assumption is to perform overlapped deployments, in order to check that an integrative sampling is occurring. However, this approach is rarely followed, or linear accumulation is actually not observed, especially due to high biofouling generated during long exposures (Togola and Budzinski, 2007).

2.2. Chemcatchers

The Chemcatcher is composed of a sorbent disk and a membrane sealed into a support of polytetrafluoroethylene or polycarbonate (Charriau et al., 2016). Polar Chemcatchers were first developed by Kingston et al. who also deployed this novel sampler in a harbour to study two polar pesticides, no longer considered as ECs (https://www.norman-network.net/) (Kingston et al., 2000). Since then, several configurations have been successfully developed for use of Polar Chemcatchers in aquatic environments (Charriau et al., 2016). The main receiving phase, in disk form, are silica bonded with octadecyl groups (C18), styrenedivinylbenzene-exchange (SDB-XC) and styrenedivinylbenzene-reverse phase sulfonated (SDB-RPS) while the most used membranes are PES, polysulfone (PS) and cellulose acetate (CA). Naked versions of the Chemcatcher, namely samplers without membrane, have also been applied. However, relatively few studies have been conducted on ECs in the marine environment and are shown in Table 2.

The papers mainly focused on two classes of compounds in coastal waters: pesticides and organotin compounds. A short list of semi-polar pesticides ($1.8 < \log K_{ow} < 4.0$) have been sampled by Chemcatchers. Notably, in one study, diuron and atrazine, which can be considered as

Table 2
Marine applications of Chemcatchers to monitor ECs. (Only classical targeted studies were considered while researches employing untargeted analysis are discussed in paragraph 3.4.)

Analytes	Receiving phase Empore™ disk	Membrane type (pore size)	Study area	Duration (days)	Depth (m)	TWA range (ng L^{-1})	Analysis method	Reference
2 pesticides	C18	PS (0.2 μm)	Portsmouth Harbour (UK)	7 and 14	0.1–0.2	8.3–1382	GC-MS	Kingston et al. (2000)
4 pesticides	C18	LDPE (0.2 μm)	Langstone and Portsmouth Harbours (UK)	7 and 14	NS	concentrations in ng/sampler	GC-MS (SIM)	El-Shenawy et al. (2010)
8 pesticides, 2 TPs	SDB-RPS	–	Great Barrier Reef (Australia)	4–14	NS	ND–6.4	HPLC-MS/MS	Shaw et al. (2010)
13 pesticides and TPs	SDB-RPS	PES (0.45 μm)	Great Barrier Reef (Australia)	30 or 60	NS	ND–430	HPLC-MS/MS	Kennedy et al. (2012a)
78 herbicides and PPCPs	SDB-RPS	–	Great Barrier Reef (Australia)	7	NS	ND–0-68 for 14 compounds with available Rs	LC-MS/MS	Gallen et al. (2019)
4 organotins	C18	CA (0.45 μm)	Alicante Harbour (Spain)	14	0.3–0.4	concentrations in ng/sampler	GC-ICP-MS or GC-FPD after derivatization	Aguilar-Martínez et al. (2008a)
4 organotins	C18	CA (0.45 μm)	Alicante Harbour (Spain)	14	1	ND–8.7	GC-ICP-MS or GC-FPD after derivatization	Aguilar-Martínez et al. (2008b)
3 organotins	C18	CA (0.45 μm)	Alicante Harbour (Spain), Portsmouth harbour and Ribble Estuary (UK)	14	0.8–1	ND–8.7	GC-ICP-MS after derivatization	Aguilar-Martínez et al. (2011)
3 organotins	C18	CA (0.45 μm)	Port Camargue (France)	14	NS	0.5–14.5	GC-ICP-MS after derivatization	Garnier et al. (2020)

SDB-RPS: styrenedivinylbenzene-reverse phase sulfonated; PS: polysulfone; PES: polyethersulfone; CA: cellulose acetate; ND: not detected; NS: not specified.

"semi-polar", were sampled by exploiting low density polyethylene (LDPE) diffusion-limiting membranes (El-Shenawy et al., 2010), typically used for more hydrophobic compounds, with $3.5 < \log K_{ow} < 7$ (Charriau et al., 2016). The other pesticide studies, which took place along the Great Barrier Reef Marine Park in Australia, employed SDB-RPS disks with or without PES membranes, more suitable for polar compounds (Charriau et al., 2016). Large annual monitoring campaigns were conducted over 12 sites, between 2005 and 2011 using Chemcatchers with 0.45 μm PES membranes (Kennedy et al., 2012a). The long-term deployments (1–2 months) performed during both wet and dry seasons, permitted a study of the influence of a period of extreme weather conditions on the passive sampling (Kennedy et al., 2012a). However, the linear uptake behaviour (integrative sampling) during these long deployments was not verified, generating uncertainty on the final results. Simultaneous shorter deployments would have been useful to guarantee that the samplers were operating in the kinetic linear range throughout the two month-exposures.

Alongside the classical Chemcatcher setup, a "naked" configuration was employed in several studies: a monitoring campaign on numerous polar pesticides took place in 2004–2005, deploying Chemcatchers for short durations (4–14 days) (Shaw et al., 2010). During the 2010–2011 wet season, passive sampling was complemented with grab sampling over an extended period to better characterise variability in exposure over a transect. Naked samplers were used for shorter deployments, while the complete configuration was chosen in the other cases, due to a better control over sampling rates as well as protective functions (Kennedy et al., 2012a).

Regardless of the configuration, the probability of sampler loss in the sea seems to be comparable to that of POCIS. For example, approximately 18% of the samplers (5/27) were lost during different monitoring campaigns due to extreme weather conditions (Kennedy et al., 2012a). However, Chemcatchers are less employed than POCIS, thus more studies are required to strengthen the comparison significance.

The remaining publications focused on 3 or 4 organotin compounds ($-0.14 < \log K_{ow} < 3.2$) in coastal waters (Table 3). In these applications, a combination of C_{18} Empore™ disks and CA membranes were used (Aguilar-Martínez et al., 2008b, 2011, 2008a; Garnier et al., 2020). Unlike the long-term pesticide monitoring described previously, the deployment lasted only for 14 days in all 4 studies, perhaps to limit fouling or to remain in the integrative phase. Due to better detection limits than for spot water sampling, most of the organotins could be detected in the studied harbours and estuaries (Aguilar-Martínez et al., 2011).

As far as TWAs are concerned, it was estimated in most of the cited works, regarding both pesticide and organotin classes. The studies on pesticides in the Barrier Reef used different sampling rate estimation methods, such as a reference diuron or atrazine sampling rate (Kennedy et al., 2012b, 2012a; Shaw et al., 2010), which leads to poor TWA accuracy. Gallen et al. estimated the water concentrations of several ECs detected in their naked Chemcatchers, but without specifying which Rs were used (Gallen et al., 2019).

Finally, the influence of ionic strength or salinity on Chemcatcher performances has rarely been evaluated, except for organotins (Lissalde et al., 2016). For these compounds, no significant differences were observed comparing tap water and artificial seawater (Aguilar-Martínez et al., 2008a). However, when using real seawater calibration, some of the Rs were found to be higher than in tap water (Garnier et al., 2020), thus suggesting the need for a more thorough study of the calibration in marine water.

2.3. o-DGT

O-DGTs are small samplers, usually composed of two hydrogels, a binding gel covered by a diffusive gel (DG), and a microporous membrane to protect it. Generally, the thickness of the DG is greater than that of the diffusive boundary layer (DBL). Thus, compound mass transfer during sampling mostly derives from the mass transfer rate through the DG layer, reducing the influence of the hydrodynamic conditions in the exposure medium (Guibal et al., 2019).

The introduction of o-DGTs in marine passive sampling is rather recent, since the first development of o-DGT samplers dates to 2012 (Chen et al., 2012). Table 3 summarizes the six studies regarding marine applications, four of which took place on the same coastline (Dalian coast, China). In three works, o-DGTs containing the non-polar XDA-1 resin were used for antibiotics, EDCs and pesticides during short deployment times (8 h– 3 days) (Xie et al., 2018a, 2018b, 2021, 2018a). Three other sorbent materials were employed to develop innovative applications, with longer deployments (from 6 to 28 days) (Amato et al., 2021; Ren et al., 2018; Stroski et al., 2020).

The Ø 0.45 μm PES filter membrane is the most popular for sampling polar organic compounds using o-DGTs (Guibal et al., 2019; Ji et al., 2022). However, in several studies, this membrane significantly adsorbed (from 10 to 100%) several ECs (Guibal et al., 2019). To avoid this issue, in the study on EDCs, Xie et al. decided not to use the PES membranes, considering the short deployments in relatively clear seawaters (Xie et al., 2018b). No biofouling was observed on either the Ø 0.45 μm PES filter membrane or the naked diffusive agarose gel of the o-DGT devices during brief exposures (Xie et al., 2018a, 2018b). Fouling was not observed either on o-DGTs deployed for up to 14 days in a harbour, using analogous agarose gels and protective membranes (Guo et al., 2019).

Although naked configurations seem suitable for short deployments, in a recent study in Oostende Harbour (Belgium), extensive damage and loss of diffusive gels was observed after 15 days when employing o-

Table 3
Marine applications of standard o-DGTs to monitor ECs.

Analytes	Binding phase	Diffusive phase	Study area	Duration	TWA range (ng L^{-1})	Analysis method	Reference
20 antibiotics	XDA-1 in (1.5% m/v) agarose	agarose	Dalian coast (China)	8 h	2.6–45.4	UPLC-MS/MS	Xie et al. (2018a)
4 estrogens, 2 pesticides	XDA-1 in (1.5% m/v) agarose	agarose	Dalian coast (China)	3 days	ND–19.4	UPLC-MS/MS	Xie et al. (2018b)
20 antibiotics	Metal-organic framework derived porous carbon in acrylamide	agarose	Xinghai Bay, Dalian (China)	7 days	ND–43.3	HPLC-MS/MS	Ren et al. (2018)
34 pharmaceuticals, 10 PFASs	25 mg Sepra-ZT (pyrrolidone modified styrene divinylbenzene) in 1.5% agarose	agarose	Iqalut Bay, Cambridge Bay and Kugluktuk, Nunavut (Canada)	7–28 days	ND–514	LC-MS/MS	Stroski et al. (2020)
1 pharmaceutical, 2 pesticides	HLB in (1.5% m/v) agarose	agarose	Port of Zeebrugge (Belgium)	6, 8 and 15 days	<LOQ–19.1	LC-MS/MS	Amato et al. (2021)
5 pesticides, 5 pharmaceuticals	XDA-1 in (1.5% m/v) agarose	ceramic	Dalian coast (China)	3 days	ND–13.1	UPLC-MS/MS	Xie et al. (2021)

PFASs: per- and polyfluorinated alkyl substances; LOQ: limit of quantitation; ND: not detected.

DGTs without protective membranes (Amato et al., 2021). Due to the fragility of the agarose gel, Xie et al. decided to use a ceramic diffusive gel instead (Xie et al., 2021). The new design was found to be less impacted than the one with agarose by the diffusive boundary layer when applied in seawaters with low flow velocity.

Overall, the use of o-DGTs in seawater quality monitoring is limited. Generally, the deployment and sample analysis are performed to verify the field applicability of novel configurations. In five of the six studies, TWAs in coastal waters are given, but poor information is provided on the deployment sites. Stroski et al. were the first to apply o-DGTs to study the impact of wastewater treatment in marine water (Stroski et al., 2020), albeit in extreme environments (Arctic Sea).

The effect of ionic strength on sampling rates in o-DGTs was discussed in a recent review (Guibal et al., 2019). The influence of salinity on sampling rate, investigated in synthetic seawater, was found to be compound-dependent. Some studies on tetracyclines, macrolides and personal care products indicated an increase in sampling rate due to the "salting out" effect. On the other hand, a decreased sampling was observed at low ionic strength for other antibiotics and PFOS. This phenomenon can be explained by modification of electrostatic repulsions among the chemicals and the diffusive gel.

Overlapped deployments have been carried out by Amato et al. for 6 and 8 d (those left for 15 d were damaged) and showed integrative sampling, but only one polar EC could be assessed (Amato et al., 2021).

As a final remark, it should be noted that calibration experiments are less performed for o-DGTs, in comparison with POCIS or Chemcatchers, since sampling rates can be predicted from known aqueous diffusivity relations. Thus, the calculation of concentration can be performed by using the diffusivity coefficients, correcting them for the site temperature (Zheng et al., 2015). However, some marine sites with low flow velocity, such as tidal channels or inner bays, may still require DBL corrections which entails the cumbersome deployment of o-DGTs with various thicknesses of diffusive gels, or the development of less-affected o-DGT gels (Xie et al., 2021).

2.4. Other samplers

Alongside with the integrative passive samplers, some methodologies are described in the literature for monitoring ECs in equilibrium conditions. Single-phase passive samplers are generally used for quantification of mostly hydrophobic pollutants, by using nonpolar polymeric sorbent materials, such as polydimethylsiloxane (PDMS) and LDPE (Taylor et al., 2021). PDMS sheets, often called silicon rubbers, have also been used as equilibrium passive samplers for more hydrophilic pharmaceuticals and pesticides in the marine environment (Belgian coastline) (Wille et al., 2011). Tributyltin and dibutyltin were detected in coastal waters at the Zanzibar Port, although the silicon rubbers were shown to sample much less efficiently monobutyltin and dibutyltin, the more polar metabolites of tributyltin (Sheikh et al., 2020). In a recent article, silicon rubbers were applied to perform a combined targeted/non targeted screening in Australian seawater (Great Barrier Reef) (Gallen et al., 2019). While the selected target analytes were hydrophobic (PAH, pesticides, PCBs), the non-target screening allowed the detection of 13 semi-polar ECs including PPCPs and pesticides (Gallen et al., 2019). Still, PDMS is a relatively apolar polymer and was found to have limited enrichment capacity for polar organic compounds with $\log K_{ow} < 2$ (Magnér et al., 2009).

According to Magnér et al., other materials may be more appropriate such as poly (ethylene-co-vinyl acetate-co-carbon monoxide) films (Magnér et al., 2009). Ethylene vinyl acetate, in the forms of sheets or titanium plate coating, was employed in two estuaries in Long Island Sound (US) and was found to be effective for pesticides of mid-hydrophilicity (Raub et al., 2015; St. George et al., 2011). Several polar munition compounds were also successfully detected after a 4-day deployment in marine field sites of the Baltic Sea containing unexploded ordnances (Warren et al., 2018). Novel hydrophilic-lipophilic balance sorbent-embedded cellulose acetate membranes (HECAM) were deployed to monitor 4 PFRs in coastal marine waters of the Arctic Sea, during a 7-day exposure (Gao et al., 2020).

A novel device, called Simple Teabag Equilibrium Passive Sampler (STEPS), was recently introduced (Vanryckeghem et al., 2021). It consists of 20 mg of hydrophilic divinylbenzene enclosed by a nylon mesh and is designed for combined sampling of hydrophilic and hydrophobic compounds ($-0.1 < \log K_{ow} < 6.8$). Deployment of STEPS in two Belgian harbours of the North Sea allowed detection of up to 40 ECs (hormones, APs, phthalates, PPCPs and pesticides), with concentrations ranging from 0.003 ng L^{-1} to 1.9 μg L^{-1}. The simplicity of these devices facilitates their use in the marine environment and should lead to a lower occurrence of damage or loss. Only one out of 48 deployed STEPS was lost and extensive sorbent loss (>50%) was noticed in just four STEPS. However, the deployment was in closed sites, not subjected to extreme weather conditions. More studies are definitely necessary to evaluate the performance of these devices, especially in the open sea.

3. Future perspectives

3.1. Evaluation of method performances

An important issue regarding the main passive sampling applications (POCIS, Chemcatchers and o-DGTs) is that several studies lack in information about the overall method performances. Valuable parameters such as method quantitation limits and analysis accuracy are often disregarded, independently from the chosen sampler. Fig. 2 shows the subdivision of the reviewed works, depending on the analytical figures of merit which were or were not specified, while Table S2 provides details on the single studies.

Regarding sensitivity, approximately half of the researches do not report any quantitation limit or just indicate the instrumental ones. While this is quite common for classical analytical methodologies, when dealing with passive sampling, limits of quantitation (LOQs) in the water body should be stated, by considering Rs and deployment time. Indeed, if these values are not specified, comparing the different passive sampling approaches may become difficult. The determination of the actual LOQs in water is performed in 40% of the POCIS studies and in 60% of the Chemcatcher studies. On the other hand, almost all papers regarding o-DGT applications report these values. This may be due to the relative ease in calculating Rs for those samplers and the consequent simpler evaluation of the TWA quantitation limits.

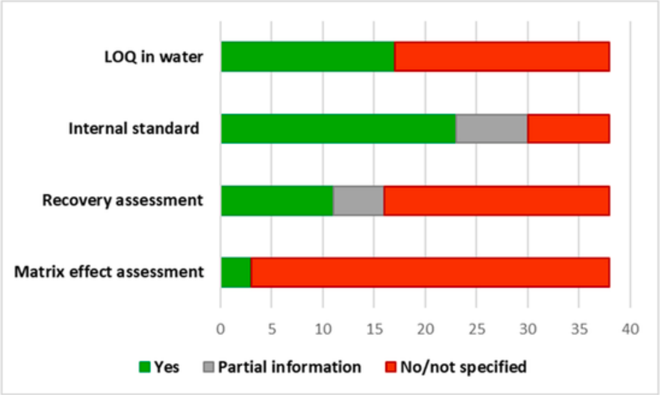

Fig. 2. Evaluation of the analytical figures of merit in the main reviewed studies. The bars indicate the number of papers which respectively evaluated the considered parameters (green bars), did not evaluate them (red bars) and only gave partial information about them (grey bars). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

As for analytical accuracy, three aspects must be considered: the use of internal standards (IS), the recovery of analytes from the sampler and the matrix effect in the final extract, namely the potential signal suppression or enhancement, correlated with the co-elution of interferent species from the sorbent phase. IS are employed in 80% of the cases, even though in seven studies out of 38 they are just used as volumetric or instrumental references. Usually, there are only a few labelled IS (even a single one) for many analytes which may be significantly different from the corresponding internal standard. This may not be the most suitable approach, since losses or compound-related signal modifications may not be correctly accounted for. The evaluation of the elution recovery as well as the matrix effects (ME) represents a valid approach to assess the method accuracy and complements the use of IS. Still, only 30% of the investigations perform recovery tests and 8% of them verify ME. A better and broader assessment of those metrics is desirable in all passive sampling applications, in order to provide more reliable monitoring data.

3.2. Choice of passive sampler

The choice of the most suitable passive sampler depends on several aspects, such as the applicability and knowledge of the technology, the reliability in TWA evaluations and the required sensitivity.

Despite being developed slightly ahead of POCIS, Chemcatcher studies have been limited both geographically (mainly to the Great Barrier Reef in Australia and the Alicante Harbour in Spain) and in the range of ECs studied (Table 2). On the other hand, POCIS have been deployed for all sorts of monitoring studies: arctic/temperate/tropical waters, transitional/coastal/open sea, shallow and deeper waters and for a large list of compounds (Table 1). Compared to POCIS and Chemcatchers, the o-DGT is rather new in marine monitoring. Moreover, marine application of o-DGTs is still in a phase of development of novel configurations and feasibility studies to fully understand the optimal o-DGT characteristics, with few field deployments (16 sites in total). POCIS marine studies were conducted using either commercial POCIS, or, like Chemcatchers, by assembling commercial sorbents and membranes. On the other hand, binding and diffusive gels for o-DGTs are prepared in the lab, leading to lower reproducibility in the final devices, which may affect the performances. Considering the applicability, POCIS and Chemcatchers are fairly interchangeable (Vermeirssen et al., 2012). Still, the Chemcatcher is easier to handle, as the sorbent is in the form of a disk instead of powder (Petrie et al., 2016).

As far as reliability is concerned, o-DGTs seem promising for future marine applications. In fact, they can be applied without calibration (Xie et al., 2018b), unlike polar Chemcatcher and POCIS samplers. For POCIS and Chemcatchers, great uncertainties affect lab-calculated Rs, due to site-specific environmental conditions, and methods to adjust Rs are still limited. PRC corrections were applied in some cases, but this method has limitations for both types of samplers. In situ calibration is a costly and time consuming approach. It has been used in some surface water field studies, especially wastewater treatment plants, using parallel grab or composite water samples (Iparraguirre et al., 2017; Petrie et al., 2016; Škodová et al., 2016). This approach was only used in one Chemcatcher study and does not seem viable in most marine sites, due to changing conditions and/or too low concentrations for robust grab sampling results and/or marine sites that are too remote for frequent grab sample collection.

In terms of sensitivity, an important parameter is the sampler dimension, making POCIS the best option, followed by Chemcatchers and finally o-DGTs. In fact, due to higher sorbent mass and exposed membrane surface, POCIS generally gives better Rs (Ahrens et al., 2015; Gravell et al., 2020). In addition, the amount of sorbent can be easily increased to enhance the capacity and allow longer deployments (Vermeirssen et al., 2012). This versatility may explain the dominance in the use of POCIS and the limited use of Chemcatchers for polar ECs in the marine environment, both in terms of marine sites and EC diversity. Overall, o-DGTs show lower Rs than POCIS and Chemcatchers, giving higher quantification limits for a given deployment time. The naked HLB-DGT has been found to be competitive with POCIS and Chemcatchers in terms of "surface-area corrected" sampling ability. Still, due to the small size, lower Rs than those of larger samplers are obtained (Guibal et al., 2017). In addition, the naked o-DGT configuration can only be deployed for few days to avoid damage to the gels, further decreasing the reachable sensitivity. POCIS samplers, on the other hand, are often deployed for up to a month, which further increases the mass of analytes accumulated in this device. Hence, POCIS may be more desirable for screening marine waters with low contaminant concentrations or for medium-long deployment studies, despite less precision. Considering o-DGTs protected by membranes, extending the deployment time may be an option to improve the overall sensitivity. The lower sampling rates could provide a longer linear kinetic regime, allowing long exposures in less contaminated areas. Still, the overall capacity of the sampler and the integrative behaviour should be verified. Moreover, higher biofouling would be expected, which usually affects the analysis accuracy. The current studies report short deployments, except for one regarding arctic seawater (Stroski et al., 2020). No information about biofouling was presented in this study, since biofilm formation is generally lower at extreme temperatures. Based on the studies currently present in the literature, o-DGTs seem to be more attractive for shorter deployment studies, in marine waters where higher anthropic pollution is expected.

3.3. Future trends in passive sampler configurations

In the future, more marine trials with HLB-DGTs may help in achieving a better comparison of o-DGTs and POCIS. Only 3 compounds have yet been studied in marine applications of o-DGTs with the HLB binding phase (Table 3), despite being one of the two most commonly used sorbents in other o-DGT applications (Liu et al., 2021).

Whereas in the past a general trend was the development of miniaturised passive sampling devices (Vrana et al., 2005), the situation may be inverted in the future, since increasing the passive sampler size may lead to lower detection limits in seawater. Indeed, if the o-DGT sampling rates are too low for their application, scaling up the size of o-DGTs could offer a potential solution (Challis et al., 2016). A two-sided design with a larger sampling surface area of 22.7 cm^2 and without any membranes has already been tested in river water (Urík and Vrana, 2019). A further example is represented by a "naked" o-DGT-like sampler with a surface area of 78 cm^2 proposed by Belles et al. and deployed in several water bodies, including one marine site (Belles et al., 2017). Such configurations may be promising for short-term marine applications. However, it has been estimated that a 160 cm^2 o-DGT surface area is required for sampling rates comparable to those offered by POCIS, considering a constant deployment time (Guibal et al., 2019).

Regarding POCIS, a further increase in size may be limited by the physical constraints of the fragile membrane, leading to the risk of losing the sampler. Yet, increasing its size to 95 cm^2 while changing its shape has already been successfully conducted in other applications (Berho et al., 2013). As an alternative, the use of new more resistant membranes could be a valuable option.

Single-phase passive samplers tend to be cheaper than dual-phase ones. Nonetheless, sensitivity is limited due to the equilibrium behaviour. An increase in surface area and/or thickness of these devices as well as the combination of extracts deriving from a larger number of deployed samplers may improve quantification limits. More investigation is surely required to compare equilibrium and integrative samplers in different marine waters applications.

3.4. High resolution mass spectrometry and the added value of untargeted screening

Generally, the extracts derived from sampler processing are analysed by LC-MS/MS or GC-MS, depending on the characteristics of the chemicals (Tables 1–3). The typical approach is targeted analysis, which involves the selection of a certain number of compounds and a selective and sensitive measurement. By doing so, the quantitation is limited to the target ECs, and therefore the evaluation of the real sample contamination is incomplete (Magi and Di Carro, 2018). In particular, targeted analysis mostly focuses on the analysis of the parent compounds, while potentially more toxic transformation products may be present at higher concentrations than the initial compounds themselves (Meng et al., 2021). However, recently, two more comprehensive approaches based on MS were found to be suitable for environmental monitoring: suspect screening and untargeted analysis (Menger et al., 2020). These strategies, first introduced in metabolomics studies, are based on high resolution mass spectrometry (HRMS), which allows the detection, discrimination and tentative identification of a large number of compounds (Menger et al., 2020). Nonetheless, the major issue in the application of these approaches to environmental monitoring is the detection of trace contaminants in samples containing other species at high concentration. Passive sampling combined to these analysis modes could represent a winning strategy, thanks to the in-situ pre-concentration usually obtained during the deployment (Castro et al., 2021a).

To the best of our knowledge, only seven studies have been reported on the combination of passive sampling with HRMS for the assessment of polar ECs in seawater: five of these using POCIS (Castro et al., 2021b, 2021a; Martínez Bueno et al., 2016; Mills et al., 2014; Viana et al., 2021), three using PDMS sheets (Castro et al., 2021b; Gallen et al., 2019; Wille et al., 2011) and one using Chemcatchers (Gallen et al., 2019). The scarce literature found on the topic highlights that several difficulties are probably encountered. Besides the issues regarding trace compound detection, the use of polar passive samplers combined with untargeted HRMS approaches is probably limited because previously measured Rs are required to estimate the concentrations of the identified compounds (Challis et al., 2020; Pinasseau et al., 2019). This issue, along with the need for reference standards to confirm identity, explains why most non-targeted studies only gave qualitative data. For POCIS, the first HRMS study barely gave any information on the mass spectrometric method and workflow (Harman et al., 2014). On the other hand, Martínez Bueno et al. used a small database of 82 ECs which permitted the identification of 20 compounds (Martínez Bueno et al., 2016). Among those, an antidepressant drug and 5 of its metabolites were then re-analysed in positive samples using available reference standards. However, the authors had not calculated Rs in the lab for these compounds – as had been done for the targeted ECs – thus concentrations could only be given in ng/POCIS (Martínez Bueno et al., 2016). Viana et al. applied POCIS in combination with LC-HRMS to perform suspect screening of antibiotics and metabolites in Portuguese seawater (Viana et al., 2021). Five and seven antibiotics were tentatively identified in two coastal sites, respectively, but no reference standard confirmation nor TWA evaluations were performed. Castro et al. evaluated four different chromatographic retention methods in combination with suspect screening HRMS with a library of 3227 chemicals to attain the most information on POCIS extracts of four estuary samples (Castro et al., 2021a). The same authors showed an example of the coupling between POCIS and PDMS sheets with GC-HRMS untargeted analysis. (Castro et al., 2021b). Seventy-five compounds in a wide polarity range were detected and tentatively identified in the passive sampler extracts. In the application of naked Chemcatchers coupled to HRMS, little information could be derived from non-target analysis of the extracts, due to high levels of background interference.

Future applications of HRMS may be best suited for extracts of sorbents protected by membranes. Indeed, as already mentioned, a protective layer generally helps in reducing interferences, hindering biofouling on the sorbent phase and allowing to obtain cleaner extracts. O-DGTs associated to suspect and non-targeted analysis may offer semi-quantitative TWA seawater concentrations by predicting sampling rates of suspect compounds, based only on their chemical structure (Challis et al., 2020). For qualitative purposes, POCIS will detect more analytes and with a better reproducibility due to their higher capacity than o-DGTs (Renaud et al., 2021).

A lot more effort is necessary to obtain suitable sampler processing as well as to choose the right sampler(s), based on the matrix, the site conditions and the final purpose (screening of one or more chemical classes or comprehensive characterization).

4. Conclusion

Passive sampling of ECs in marine waters has focused on polar compounds, with POCIS representing approximately two-thirds of the publications in the literature.

A wide range of chemical classes has been investigated by passive samplers, with a prevalence of pharmaceuticals and pesticides. As for the deployment sites, coastal areas are usually chosen, but several offshore sites have been studied as well either as reference sites or close to sources of pollution. Compared to the other integrative passive samplers for polar compounds, o-DGT sampling rates are less influenced by flow variations. Therefore, the o-DGT has a great potential for monitoring a large class of organic pollutants in the marine environment. However, this sampler cannot currently reach sensitivity offered by POCIS and further developments, such as the assessment of bigger o-DGTs, are necessary. Employing POCIS may be the most reliable screening option to detect a larger range of compounds at low marine concentrations.

Several improvements are required to obtain more reliable and also comparable data. First, a validation of the quantitative analysis by testing accuracy is mandatory, to limit the uncertainty of the final results. In fact, they are affected by errors due to the Rs choice, possible exit from the linear uptake range and sampler reproducibility. To improve these aspects, more accurate calibrations, replicates and overlapped deployments of different durations should be performed. Finally, when possible, a sampling protocol with standardization of exposure times may be desirable to allow the comparison of various monitoring studies.

Coupling with HRMS is a promising though challenging approach for marine applications enabling non targeted analysis. New passive sampling devices could be introduced with improved characteristics, such as the ability to sample ECs with a larger range of polarity, to meet the requirements for a comprehensive and informative untargeted screening. Untargeted studies should be pushed forward, through the acquisition of reference standards and Rs evaluations after the first screening. This should be the direction to follow to gain the most from the coupling of passive sampling and HRMS. Despite the challenging problems to be faced, the development in this field could bring to the comprehensive assessment of the environmental contaminants to which humans are exposed during their lives.

Credit author statement

Henry MacKeown: Conceptualization, Writing – original draft, **Barbara Benedetti**: Writing – original draft, **Marina Di Carro**: Writing – review & editing, **Emanuele Magi**: Conceptualization, Writing – review & editing.

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.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2022.134448.

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