EMERGING CONTAMINANTS IN SEDIMENTS: A REVIEW

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ABSTRACT

Emerging Contaminants (ECs) are chemicals for which new concerns have recently raised in terms of occurrence, fate, adverse effects on human health and the environment. ECs include pharmaceuticals, nanomaterials, compounds used in personal care products, plastics, pesticides and flame retardants, and compounds with a large variety of uses (e.g., phenolic and perfluorinated compounds, chlorinated paraffins, etc.).

The EU Water Framework Directive 2013/39/EU deals with the status of water bodies and reports an updated list of priority substances. Some of these (e.g., nonylphenols, di(2-ethylhexyl)phthalate DEHP, perfluorooctane sulfonate PFOS, hexabromocyclododecanes, polybrominated diphenyl ethers, etc.) are emerging contaminants with high affinity towards suspended solids and sediments, due to their physicochemical properties (e.g., Koc value, etc.). The review focuses on this group of pollutants, reporting about the occurrence and distribution in sediments and biota, bioaccumulation and ecotoxicity, and the target or limit values that have been set in order to protect the aquatic environment and the human health.

Keywords: Emerging Contaminants, Alkylphenols, Phthalic Acid Esters, PerfluoroalkylCompounds, Brominated Flame Retardants, Sediments.

INTRODUCTION

CEs include a wide range of chemicals (e.g. industrial, agricultural and household chemicals, as well as pharmaceuticals and personal care products) that have been produced and released for long, but not commonly monitored in the environment and for which new concerns (occurrence, fate, adverse effects on human health and the environment) have recently raised [1].

Recently, the EU Water Framework Directive [2]set a group of 45 compounds/groups of compounds **(**metals, pesticides, polyaromatic hydrocarbons, chlorinated solvents, …**)**,that have been prioritized and updated for action at European Union level. Before this, 23 chemicals were listedin the globally binding Stockholm Convention, aiming to eliminate or reduce their presence in the environment [3].These international policies established the concentration limits for pollutants in water, sediment or biota that should not be exceeded to protect human health and the environment [3, 4].

Sediments play a significant role in the overall environmental quality of an aquatic system, as important biological habitats, and environmental reservoirs and sources of various contaminants [5, 6]. Many pollutants, originally introduced into the water bodies, have great affinity for sediment particles due to their hydrophobic/lipophilic properties and some of them tend to accumulate in aquatic biota [6].

The determination of pollutants in sediments and aquatic organisms is thus of major importance for the assessment of ecological risks, such as loss of biodiversity, ecosystem change and potential effects to wildlife and human health. Adsorption of ECs to suspended particles or sediments is mainly influenced by the organic carbon (OC) content of the sorbents [6] and the solubility and the octanol-water partition coefficientof the compounds, which modifies according to temperature, salinity, …[6].

However, few studies have analyzed CEs in water, sediment and biota samples collected at the same time and place [7]. Thus, poor information is available about the actual sediment–water partitioning of these compounds, and the organic carbon-water partitioning coefficient (Koc) of the chemicals taken as the reference parameter to calculate the expected concentration in sediments [9]. Moreover current assessment methods of sediment-water distribution predominantly rely on chemical equilibrium partitioning despite several observations reporting an "enrichment" of chemical concentrations in suspended and deposited sediments [6, 8].

Another key factor related to sediment pollution is the biota-sediment accumulation factors (BSAF), that is the ratio between the concentration of a chemical in an aquatic organism (normalized to the lipid fractionofthe organism) and the concentration in surface sediment (normalized to the organic carbon content of the sediment) [9]. BSAF is used to describe bioaccumulation of sediment-associated pollutants into tissues of ecologicalreceptors. However, experimental data processing and interpretation is complicated by contemporary

exposure to interstitial water and suspended particles[10], resulting in huge variability among different sites [11].

Knowledge about toxicity and biological effects is limited or not available for several CEs. Thus, exposure and middle or long-term effects on human health, terrestrial and aquatic ecosystems are largely unknown. Accordingly, regulatory concentration limits or sound standard or trigger values for the different environmental media have not been established yet [12]. Furthermore, currently the majority of environmental standardsare established for surface water only. Monitoring for the long-term trend analysis of ECs that are likely to accumulate in sediment and/or biota, are still required [13, 14].

This review describes current knowledge on the occurrence in sediments of selected classes of ECs based on the EU Water Framework Directive priority list and the Koc values (Table 1).

ALKYLPHENOLS

Alkylphenols (APs) belong to the group of non-ionic surfactants and are widely used for many applications (as formulants in pesticides, as lubricating oil additives, and as catalysts in the curing of epoxy resins). APsare also the degradation products of the non-ionic surfactants alkylphenolpolyethoxylates (APEs) which are largely used in domestic and industrial applications, such as detergents, emulsifiers, dispersants, and plasticizers [15]. Since January 2005, an EU Directive has restricted the use of nonylphenol (NP) and nonylphenoletoxylates (NPEs) by banning their use in cleaning products at concentrations >0.1% [16].

Nonylphenols (NPs) and Octylphenols (OPs) are ubiquitous around cities and factories [17]; they are almost stable in the environment, with a half-life of several decades in sediments [18]. Higher accumulation of OPs and NPs in sediments than expected from their hydrophobicity has also been often reported, possibly due to in situ biotransformation of nonylphenolethoxylates (NPnEO) [19].

Alkylphenols have been considered for many years as endocrine disrupters, leading to several official restrictions [20]. NPs and OPs are more toxic to aquatic organisms and have higher anti-estrogenic effects than parent etoxylates compounds [21, 22]. They alsobioaccumulate in clam tissues at high concentrations [10, 11].

Casatta et al. [10] report the guideline values provided by European and Canadian authorities for nonylphenol (Table 1). NPs predicted no effect concentration (PNEC) for sediments is 39 ng/g dw (dry weight); for OPs a PNEC of 3.4 ng/g dw for saltwater and 34 ng/g dw for freshwater sediments have been proposed [15]. Although NPs and OPs are listed as hazardous, no environmental quality standard is given for biota for secondary poisoning or human health protection. LogBASF in literature for NPs range from 0.05 to 2.6 [11, 23, 24]. Bakke et al. [25] proposed aRfD value of 0.10 mg/day/kg body weight, which should be protective for human health under chronic exposure to NP and NPEs. For OP, as a threshold level for human health, a NOAEL of 0.15 mg/day/kg body weight was assumed. Danish Institute of Safety and Toxicology (DIST) derived preliminary TDI value of 5 µg/day/kg body weight for NP and 10 µg/day/kg body weight for OP [26].

NPs and OPs in sediments range from 3.6 µg/kg dw to 72 mg/kg dwand 0.08-250 µg/kg dw, respectively(Table 2), with the highest values in lakes [27]. In river sediment high NP and OP concentrations are generally related to specific point sources, such as industrial plants, or large amounts of domestic wastewater entering the river in populous regions [28]. In estuarine and marine sediments, a decrease in APs concentrations were often observed with increasing distances from the coast [29]. In Italy, NPs were measured in areas with different soil uses (urban, industrial, agricultural, open country) in the Rieti district, along the Tiber river and in the Venice Lagoon, resulting in similar values, between 44 and 970 µg/kg dw [30, 31, 32]. Alkylphenols represent approximately 50% of the overall amount of organic chemicals detected in six Italian lagoons, with a marked prevalence of nonylphenol and its monoetoxylate [10].

PHTHALIC ACID ESTERS

Phthalate esters (PAEs) have been widely used in plastic manufacturing since the 1930s, and they can also be found as a common additive in paints, lubricants, adhesives, insecticides, packaging, toys, and cosmetics [33, 34, 35]. Di(2-ethylhexyl) phthalate (DEHP) and di-n-butyl phthalate (DBP) are the most abundantly produced and used phthalate esters.

Dumps and sanitary landfills are significant PAEs source, as leaching from the plastic materials into runoff water or leachate is possible [36]. The use of agricultural plastics has exacerbated soil contamination in rural areas [37]. Sediments may act as an ultimate repository of these contaminants entering water bodies, as they are almost insoluble in water [38].

Biodegradation can be the most important process for the removal of PAEs in the environment. Field and laboratory studies have shown that aerobic or anaerobic microorganisms from various habitats (water, sediment, soil) are able to degrade PAEs [39]. However, the half-lives of PAEs depend strongly on the condition of each habitat such as oxidant,

microbial density, and temperature, with half-lives in the wide ranges 0.5-37.4 d under aerobic conditions and 9.2-392 d in anaerobic environments [40, 41].

PAEs are considered to be endocrine disrupting chemicals and are suspected to interfere with biological processes in human and wildlife, in particular aquatic organisms, even at very low concentrations [38]. U.S.EPA [42] has recently classified DEHP under probable human carcinogen (Class B2) and DBP under possible human carcinogen (Class C). Consequently, six of them (including DEHP and DBP) have been placed on the priority pollutant list of the U.S.EPA, the European Union and on the list of priority pollutants in Chinese waters [43]. Therefore, the use of PAEs is now subject to stricter control and some phthalates restriction/banning or their reduction in numerous products has been recommended.

The NOEC values of DEHP and DBP in sediments are 470 and 2200 µg/kg dw, respectively [44] (Table 1). The screening benchmark value (SCB) of phthalates given by U.S.EPA [45] is 180 µg/kg dw. The environmental risk limits (ERLs) for phthalates in soil are 700 µg/kg for DBP and 1 mg/kg for DEHP based on no observed effect concentrations (NOECs) for Rana arvalis [46, in 47].

In sediments, DBP and DEHP were found in abundance all over the world (Table 2), at concentrations significantly varying between urban and rural samples because of higher discharge of PAEs from plastic materials in urbanized areas [48]. The investigation of phthalate ester occurrence in sediments from the Qiantang River, China, demonstrated that on average the concentrations in urban regions were almost 3 times than those in rural locations [49]. DEHP is the most found PAEs in sediments [36, 37] with the highest values registered in Iraq, Shatt al-Arab River [50] and in Taiwan, Kaohsiung Harbor [47]. In Chen et al. [47], in 70 out of 80 sediment samples (88%) AEPs concentrations are 1 to 35 times higher the ERLs. In Selvaraj et al. [38], among the quantified phthalates, only DEHP exceeded the screening levels, with higher levels, 8 times the SCB, in river sediments and 1.5 times the SCB in estuarine environments. DEHP levels in fish were often below the LOD, as reported by Peijnenburg and Struijs [51].

PERFLUOROALKYL COMPOUNDS

Perfluorinated compounds (PFCs) contain a fully fluorinated hydrophobic alkyl chain with variable number of carbon atoms (typically C4 to C16) [52]. Due to their thermal and chemical stability, PFCs found application in industrial and household products, like surfactants, pesticides, surface protective layers, firefighting foams. Perfluorooctane sulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) were found as the dominant PFCs in sediments [53, 54].

Concerns over persistence and potential for toxicological effects led to the phase-out of PFOS, and related compounds beginning in 2000 by the major global manufacturer in United States [55]. Since then, studies on environmental, toxicological and health effects have grown rapidly, and production, usage and emission of PFCs have been strictly controlled [56].

To date there are not available screening values or suggested limits for sediments (Table 1). Oral reference dose (RfD) values for most PFCs have not been yet established by any government or regulatory agency. However, provisional RfDs for PFOS and PFOA of 25 and 333 ng/day/kg body weight, respectively, have been derived, on the basis of a rat chronic carcinogenicity study and a rat multigenerational study [57].

Sediments are one of the most important environmental sinks and reservoirs for PFCs [58]. PFCs have been found in sediments from several countries and regions, with concentrations ranging from non-detectable to several hundred nanograms per gram dry weight [10, 11, 13]. The distribution pattern of PFCs are closely related to the land-use, and high concentrations generally occur in the industrialized and urbanized areas [14]. PFOS and PFOA in sediments range from 0.05 µg/kg dw to 600 µg/kg d.w, being PFOS the most abundant with an average concentration of 74.4 ng/g dw.; some recent case studies are reported in Table 2. In the review by Becanova et al. [52], in many European rivers PFCs concentrations do not exceed 50 µg/kg dw (up to 20 µg/kg for PFOS and PFOA); the highest levels are reported for Tangxun Lake, in China, located near a fluorochemical industry [53].

In the literature PFOS is reported as the predominant PFCs also in biota. PFOS was accounted for 94% and 93% of the total PFCs for crucian carp and sharpbelly, respectively [53]. In estuarine and coastal areas of Korea [59], concentrations in biota ranged from 0.26 to 612 ng/g. Prosser et al. [60] investigated also the accumulation of PFCs in three laboratory-cultured species exposed to field-collected sediments; PFOS resulted in concentrations greater than all the other PFCs analyzed, but the study demonstrated, also, these contaminants are potentially readily available for accumulation in aquatic organisms.

BROMINATED FLAME RETARDANTS

Brominated flame retardants (BFRs) include diverse chemical classes of compounds widely incorporated as additives in consumer products such

as electronic devices, textiles, polystyrenefoams, epoxy resins and polycarbonate polyesters, to reduce the likelihood of ignition of the flammable materials [61, 62]. BFRs account for about 21% of the total consumption of flame retardants. Polybrominated diphenyl ethers (PBDEs), a class of substances with 209 theoretical congeners depending on number of bromine atoms and the substitution pattern, with1,2,5,6,9,10-hexabromocyclododecane (HBCDs) are among the most used BFRs in the world [63, 64].

BFRs have low vapor pressures, very low solubility and are very lipophilic, thus they are expected to sorb strongly on sediments [65]. Half-lives for pentaBDE are 600 days in aerobic sediment [66].Many BFRs have been identified as bioaccumulative, LogBSAFs ranged from 0.59 ng/g dw to 1.2 ng/g dw [66, 67].

Increasing concern due to their persistence and potential toxic effects on human health [69] has promoted some international organizations and countries (including European Union, US, Canada) to ban some of them following the precautionary principle. In fact, there is evidence of the endocrine-disrupting properties of several congeners and of their induction of neurobehavioral and reproductive disorders and thyroid hormone-level alterations in both humans and wildlife [68].

US companies phased out the production and use of penta- and octaBDE at the end of 2004 and had decided to voluntarily cease the production and use of decaBDE by the end of 2012 [70]. Finally under the Stockholm Convention, tetra- to heptaBDEs have been listed for elimination in 2009 and HBCD in 2013 and the proposed listing of decaBDE is presently discussed [71]. Overall, these restrictions led to a shift in the market with an increased use of alternative BFRs to substitute for the discontinued PBDEs.

The threshold concentration in sediment was set to 19 ng/g dwforBDE-209 and 0.4 for penta-BDE ng/g dw [66]. In the EU, HBCDs have been identified as Substances of Very High Concern [72]; the environmental quality standard proposed by Baltic Commission [73] for sediment is 170 µg/kg dw, derived on eco toxicological effects on predators and 167 µg/kg fish wet weight (Table 2).

In Wang et al., [74] the concentrations (ranged from 1.46 to 6.76 ng/g) of eight PBDEs presented a decreasing gradient with increasing distance off the shore indicating that anthropogenic activities and river inputs may be the major sources of PBDEs in the sediments. In Sühring et al., [71] 53 brominated retardants were investigated in sediment samples from the German rivers (Table 2); concentrations in general were low DL (<1 ng/g dw) with exception for BDE-209 with up to 7 ng/g dw and tetrabromobisphenol with 2.7±1.5 ng/g dw. Recently in Italy, it was shown that among the many flame retardants reaching the Po delta, only PBDEs were identified as potentially of (eco)toxicological concern [10]. HBCDs have sometimes been found at substantially higher concentrations than PBDEs in sediments [76]; the highest concentrations were measured downstream of HBCD production sites and of industrial users. HBCDs have been widely detected in sediments from Europe, Asia and North America [63]. For instance, sediments from the Detroit River and Lake Erie showed similar concentrations (0.1-5 µg/kg) with other Great Lakes, San Francisco Bay, English Lakes and Taihu Lake.

CONCLUSIONS

Several CEs, some of them considered priority pollutants by environmental agencies, were found in sediments worldwide. The presence of CEs in sediments and biota, even though at low levels, might be harmful and should not be neglected due to their persistence and toxic effects. The impact on human health after long-time exposure cannot be ignored, but information still is lacking (Table 1). Thus, their presence should be monitored in the future studies. Even compounds already banned are still found in sediments and biota. Studies concerning sediments and biota should be encouraged to provide valuable information for assessing the fate of these pollutants in the environment. Studies concerning bioaccumulation and toxicological effects as an integrative measure of a water body quality should be also stimulated..

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Table 1. Koc values and proposed standards or limits in sediments (a) and biota (b). (c) NOEC; (d) Screening benchmark values; (e) Environmental risk limits; (f) Environmental Quality Guidelines; (b) environmental quality standards in biota from UE 2013.
(-) not found

Group/ Compound	logKoc	St. SED. (ng/g) (a)	St. B. (b) (µg/kg)	RfD (µg/kg/d)
ALKYLPHENOLS				
4-Nonylphenol isomers	3.4-5.5 [10]	39-180(c) [10];	-	100 [76]
4-Octylphenol	3.4-5.6 [77,10]	3.4 (f) [109]	-	
PAE				
Di(2-ethylhexyl)phthalate (DEHP)	5.2 [78]	470(c) [44]; 180(d) [38]; 1000(e) [46]	-	20 [79]
Di-n-butylphthalate (DBP)	3.14-4.17 [45]	220(f) [108]	-	100 [105]
PFC				
Perfluorooctanesulfonic acid (PFOS)	2.4-4.7 [80]	-	9.1	0.02 [26, 106]
Perfluoroctanoic acid (PFOA)	1.3-4.5 [80]	-		0.02 [107]
PBDEs (209 congeners)				
Penta-BDEs	5.0-6.8 [82]	0.4(f) [66]	0.0085	0.1 [81, 82]
Deca-BDEs	4.8-9.1 [82]	19(f) [66]		
HBCDs				
1,2,5,6,9,10-Hexabromocyclododecane (HBCD)	4.77-5.07 [83]	170(f) [73]	167	0.2 [84]

Table 2. Occurrence of selected CEs in sediments and biota in recent international case studies

Area	Sediment (µg/kg)			Biota (µg/kg)
	APs	NPs, NP, 4-NP	OP	
Costallagoons, Po River Delta - Italy	24.2-247.9 [10]	19.4-202.3 (NP), <DL-1.24 (4-NP) [10]; 47-192, Mean: 89 (NPs) [32]	0.4-4 [10]	APs: 4500-6635 NP: 3046-5857, 4-NP: <DL, OP:105-204 (clam tissues) [10]; NPs: 115-240 (Mussel, *Mytilus gallo provincialis*) [32]
Sea and river sediments - Spain	<DL-1693 (6 APs) [85]	<0.24-1693 (4-NP, branched) [85]; 79-521, mean 229 (4-NP, branched) [86]; <DL-4460 (NP), <DL-1.7 (4-NP) [11]	<0.24-76 [85]; 38-39 [86]	NPs 306-2258 (*C.Fluminea*) [11]; 4-NP: 16-58, mean 35, OP: 7 (Mussel) [86]
San Francisco Bay and South California - USA	<DL-3900 (10 APs) [87]	122-3200 (NP) [87]; 21.5-86, Mean: 34.7 (4-NP) [88]	1.9-8.2 [87]	4-NP: <DL-94.5 (mussel) [88]
Sea, Lake and River - China		10.3–338 (4-NP) [27]; 3540-32430, Mean: 10490 (4-NP) [89]; 349.5 -1643, Mean: 890 (NPs) [18];	0.8 -9.3, Mean: 4.7 [18]; 1-27.4 [27]; 58-1245 [89]	
Estuarine sediments in Auckland - New Zealand		<100 (4-NP), <100-32000, Median: 153 (NPs) [90]		
Masan Bay - Korea		130-2810 (NPs) [91]		NPs: 51-290 (mussel) [91]
	PAEs	DEHP	DBP	
Rivers in industrialized and urbanized zone - France–Belgium	1090-11890 [43]; 66-28782 (6 PAEs) [48]	500-6300 [43]; 62-16546 [48]	<400-1200 [43]; 3-1104 [48]	DBP: 92-1110; DEHP:376-1461, PAEs: 718-3156 (fishes) [48]
Sea and river sediments - Spain	1.9-4317 (4 PAEs) [86]	1.9-107, mean 39 [86]		4 PAEs: 0,5-34; DEHP: 2-21 (Mussel): [86]
River, Lake, Marine, Estuary, Harbor sediments - China	400-450000 [92]; 381–2044, mean 789 [94]; 2270-74940, mean 21000 (16 PAEs) [95]	320-760 [93]; 227–567, mean 343 [94]; 210-14160, mean 3640 [95]	82-1260, mean 370 [95]	PAEs:1570-7100 (20 fish species) [92]
Kaveri River - India	2-1438 [38]	<DL-1400, mean 278 [38]	<DL-664, mean 35.5 [38]	
Ogun river catchments, Ketu, Lagos, Nigeria		20-820 [96]	190-1420 [96]	DEHP; 30–300, DBP: 380-3970 (3 fish species) [96]
Kaohsiung Harbor and rivers -Taiwan	400-34820 (6 PAE), mean 5020 [47]	400-34800, mean 4900 [47]; 500-23900 [41]	<DL-1130, mean 290 [47]	

	PFCs	PFOS	PFOA	
comparable river basins in parts of Europe	mean 8.4 (15 PFC) [97]; max 37.5 [52]	mean 4.3 [97]; max 14 [52]	<0.1 [97]; max 16.8 [52]	
Jucar River -Spain	14.3–75.9, mean 34.7 (21 PFC) [98]	0.06-9.8, mean 2.6 [98]	0.15-6.7, mean 2.5 [98]	PFOS: 0.56–8.13, mean 2.2;21 PFC: 83.7–1140, mean 369 [98]
Sediment from a creek in Southern Ontario - Canada	<0.1-4.8 (11 PFC) [60]	0.2-4.8 [60]	<0.1-0.15 [60]	PFOS: 11-105 (*Hexagenia)*, 5.6-87 (*Lumbriculus variegates)*, 1.4-58 (*Fathead minnow*) [60]
Charleston - SC USA	0.22-19.14, mean 3.68 (11 PFC) [99]	0.09-7.37, mean 1.52 [99]	0.02-2.52, mean 0.42 [99]	
Nansi, Tangxun, TaihuLakes - China	0.47–1.81 [54]; 41.8-800, mean 151 [53]; 5.8-35 [100]	0.17–0.83 [54]; 10.9-623, mean 74,4 [53]; 4.8-21.7 [100]	0.11–0.44 [54]; 0.48-6.4, mean 2,4 [53]	PFOS: mean 263, PFOA: <DL (Crucian carp). PFOS: mean 384, PFOA: <DL and (Sharpbelly) [53]
Estuarine and coastal areas - Korea		<DL-2 [59]		PFOS: 0.34-0.77, PFOA: <DL-0.94 (Mussel) [59]
	PBDEs	HBDCs		
Costal lagoons, Po River Delta - Italy	1.64-42.4 [10]			8 PBDEs: 0,9-162,5 (Clam tissues) [10]
German part of the rivers Elbe and Weser, the German Bight, East Frisian Coast and UK East coast	<0.01-7.2 [71]	<0.01-1.75 [71]		
50 various locations throughout Flanders – Belgium	10-5811, mean 242 [101]	16-4397, mean 394 [101]		
Southern California Bight, Yadkin River, USA	mean 20, max. 560 [102]; < 1 - 3287 [104]	< 1 - 3718 [104]		10 PBDE max. 68, mean 1.2 (Mussels, Mytilus) [103]; 20 PBDE: <1 - 1777, 3 HBDC: <1 - 11428 (Mollusks *Corbicula fluminea, Elimia proxima*) [104]
Bohai Sea, Surface sediments of Shanghai - China	1.46-6.76 [74]	0.01-13.7, mean 3.4 [64]		
Estuarine sediments in Auckland - New Zealand	0.55-573 [90]			