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## Organic ultraviolet filters in Irish sediments and biosolids

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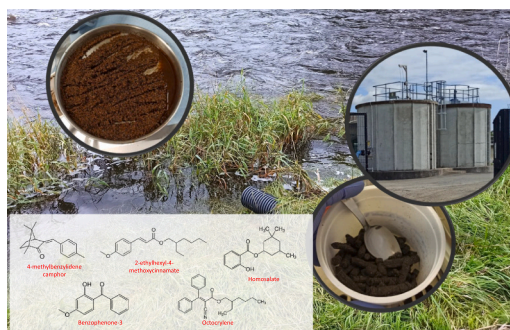
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### HIGHLIGHTS

- Highest mean concentrations of OC and HMS in both sediments and biosolids
- HMS, EHMC, & 4-MBC of moderate ecotox. risk based on concentrations in sediments
- Lower use of SSAs in Ireland indicated herein compared to similar studies worldwide
- Highest concs of HMS in biosolids determined; further international data required
- Ongoing monitoring required to ensure continued compliance with regulations

### GRAPHICAL ABSTRACT



### ARTICLE INFO

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### ABSTRACT

This study provides baseline data on the concentrations of selected ultraviolet filters (UVFs) in 81 inland and transitional sediments as well as in 21 agricultural biosolid samples sourced from seven wastewater treatment plants (WWTP) across Ireland. Concentrations of five prominent UVFs were determined: 4-methylbenzylidene camphor (4-MBC); benzophenone-3 (BP-3); 2-ethylhexyl-4-methoxycinnamate (EHMC); homosalate (HMS); and Octocrylene (OC). Mean concentrations in sediments were as follows: OC (3.66 ng/g); HMS (2.35 ng/g); 4-MBC (1.87 ng/g); BP-3 (0.19 ng/g); and EHMC (0.04 ng/g). Based on 95th percentile concentrations in sediments (MEC<sub>95</sub>), HMS, EHMC, and 4-MBC were found to pose moderate ecotoxicological risk. Concentrations found in Irish sediments were found to be low compared to similar studies internationally. Arithmetic mean concentrations of UVFs in Irish biosolids were: OC (666 ng/g); HMS (453 ng/g); 4-MBC (13.9 ng/g); EHMC (12.7 ng/g); and BP-3 (1.28 ng/g). Fewer international data are available for these UVFs in biosolids, though available results indicate Ireland to have low levels compared to those found internationally for EHMC, 4-MBC, BP-3, and OC. This study is one of the first to report of concentrations of HMS in WWTP-derived biosolids and highlights the need for similar assessments internationally based on levels found in biosolids as well as in sediments. Ongoing

**Acronyms:** 4-MBC, 4-methylbenzylidene camphor; BP-3, benzophenone-3; CPR, Cosmetics Products Regulation; EHMC, 2-ethylhexyl-4-methoxycinnamate; HMS, Homosalate; OC, Octocrylene; PBT, Persistent, Bioaccumulative, Toxic; RQ, Risk Quotient; SSD, Sewage Sludge Directive; SVHC, Substance of Very High Concern; UVF, Ultraviolet Filter; UWWTD, Urban Wastewater Treatment Directive; WFD, Water Framework Directive; WWTP, Wastewater Treatment Plant.

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monitoring in sediment is required for HMS and 4-MBC following their restrictions under the EU's Cosmetic Products Regulation (CPR), to ensure that environmental levels fall to levels concomitant with low ecotoxicological risk. Meanwhile, further assessments are required for OC, EHMC, and BP-3 to facilitate thorough evaluation of their environmental risks.

## 1. Introduction

Ultraviolet filters (UVFs) are a range of compounds used in sunscreens and related cosmetic products in order to protect the skin from harmful UV radiation. Historically used for the purposes of protecting the skin during activities such as sunbathing and snow sports, UVFs are now incorporated into a much larger range of cosmetic products such as moisturisers, hair-care products, aftershave, and makeup (Chisvert and Salvador, 2007). There are two main types of such chemicals: inorganic or physical UVFs, which create a protective layer over the skin to reflect and scatter UV radiation; and organic or chemical UVFs, which penetrate the skin and convert UV radiation to heat. Within the EU, the use of physical UVFs in cosmetics is restricted to Titanium Dioxide (TiO<sub>2</sub>) though Zinc Oxide (ZnO), another physical UVF, is permitted for use in cosmetics as a colourant (EU, 2009). However, a much wider range of organic UVFs are today regularly used in cosmetics (Nitulescu et al., 2023) with quickly evolving research on their toxicological effects (Schlumpf and Lichester, 2009; Schreurs et al., 2002; Kunz and Fent, 2006; Ruszkiewicz et al., 2017) though comparatively less information available on their environmental prevalence.

In recent years, two prominent UVFs have been heavily restricted for use in cosmetics under the Cosmetics Products Regulation (CPR) (EU, 2009). The first, homosalate (HMS), is currently flagged by the European Chemicals Agency (ECHA) as very toxic to aquatic life and suspected of damaging unborn children (ECHA, 2024a); as such, the maximum allowable concentration in cosmetics was recently revised to below 0.5 % by the CPR and only allowed to be used in face products (EU, 2022a). The second, enzacamene or 4-methylbenzylidene camphor (4-MBC), is a listed substance of very high concern (SVHC), a known endocrine disruptor (ECHA, 2024b), and will from the 1st of May 2025 be prohibited from entering the European market and prohibited from sale in the EU market from the 1st of May 2026 (EU, 2024). Under the Water Framework Directive, three further organic UVFs have been identified as needing urgent monitoring and assessment due variously to: no monitoring data being available; extensive use in products; high volumes of discharge to the aquatic environment; and/or suspected toxicity. In 2015, 2-ethylhexyl-4-methocinnamate (EHMC) was placed on the WFD Watch List for monitoring in water and suspended matter or sediment (EU, 2015). It was removed from the Watch List in 2018 pending the acquisition of further data in sediments, to be reintroduced under a later monitoring call to allow for a conclusive analysis on that monitoring matrix (EU, 2018). The 4th Watch List published in 2022 included both Octocrylene (OC) and Oxybenzone/Benzophenone-3 (BP-3) (EU, 2022b) and are furthermore listed on the most recent Watch List as of February 2025 (EU, 2025). The former, OC, is under assessment as persistent, bioaccumulative, and toxic (PBT) (ECHA, 2024c) and was flagged by the WFD as needing further assessment in sediments and biota due to its high hydrophobicity. The latter, BP-3 is currently under assessment as an endocrine disruptor (ECHA, 2024d) and was flagged by the WFD as needing further assessment in either water or sediment.

Numerous studies have reported links between the environmental uptake of sunscreen agents and discharge of wastewaters into the environment (Duis et al., 2022; Ramos et al., 2016; Al Falahi et al., 2022), as wastewater treatment technologies have been shown to be only partially effective in removing UVFs from wastewater with lipophilic UVFs in particular shown to adsorb onto sludges (Ramos et al., 2016). Other likely environmental sources include: uptake from products treated with UVFs such as plastics, textiles, and paints from general wear and tear, along with their disposal in waste streams (Al Falahi

et al., 2022); and the application of biosolids from wastewater treatment operations on land (Mitchellmore et al., 2021; Liu et al., 2012) with the potential for uptake into crops, particularly root crops, grown on treated land (Ramos et al., 2020; Fernandes et al., 2024). The EU's Sewage Sludge Directive (SSD) (EU, 1986) and Urban Wastewater Treatment Directive (UWWTD) (EU, 1991) do not include cosmetics and pharmaceuticals within their remit and the potential environmental contamination from these sources is not currently monitored. However, both the SSD (EU, 2023) and UWWTD (EU, 2022c) are intended to be updated by the European Commission, with the monitoring and control of such chemicals of emerging concern to be considered.

The assessment and monitoring of UVFs in environmental matrices is of vital importance. For those which are already restricted under the CPD, ongoing monitoring is necessary to verify levels in the environment fall and remain below ecotoxicological risk factors. Similarly, assessment and determination of risk is required for those which are potentially of concern, but do not yet have sufficient data available for a consensus. To that end, we report herein the results of a nationwide campaign which investigated levels of these five prominent UVFs (Table 1) in inland and transitional sediments across Ireland, and samples of sewage sludge/biosolids emanating from seven small- to large-scale wastewater treatment plants. We furthermore investigate the correlations between environmental contamination and output from wastewater treatment works, as well as the factors which may impact concentrations of UVFs in WWTP-derived biosolids. To the authors' knowledge, this is the largest ever survey of these UVFs in inland or transitional sediments conducted worldwide to date, and one of the first to determine concentrations of homosalate in WWTP-derived biosolids.

## 2. Materials and methods

### 2.1. Sample collection

A total of 81 samples were collected from inland ( $n = 62$ ) and transitional ( $n = 19$ ) waters across Ireland in 2023 for analysis of the ultraviolet filters outlined above. Inland sediments comprised of freshwater rivers, while transitional sediments comprised of those collected in brackish water in estuaries. Full details of this sampling campaign are reported elsewhere (Sharkey et al., 2024). Briefly, approximately 1 kg aliquots of sediments were collected from inland and transitional sites (Fig. 1) prior to homogenisation, wet sieving to a maximum grain size of

**Table 1**

– Overview of UVFs of relevance to this study. \*Allowable use as specified in the Cosmetics Products Regulation (EU, 2009).

Chemical	Acronym	CAS #	Allowable Use*
4-Methylbenzylidene Camphor	4-MBC	36,861–47-9 / 38,102–62-4	4 %
Benzophenone-3 (Oxybenzone)	BP-3	131–57-7	6 % in face, hand, and lip products (excluding propellants); 2.2 % in body products; 0.5 % in other products
Ethylhexyl Methoxycinnamate	EHMC	5466-77-3	10 %
Homosalate	HMS	118–56-9	7.34 % in face products (with the exception of propellant sprays)
Octocrylene	OC	6197-30-4	9 % in propellant sprays; 10 % in other products

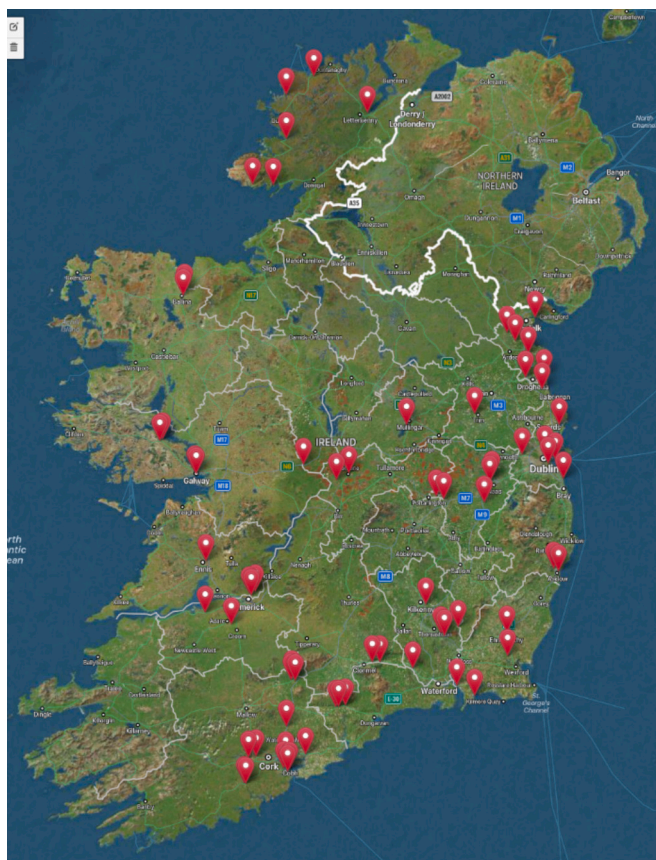


Fig. 1. – Sampling points for 2023 campaign of Inland and transitional sediments [map adapted from irishgridreferencefinder.ie].

2 mm, and freeze-drying under a 0.133 mBar vacuum to a uniform temperature of  $-40^{\circ}\text{C}$  over a period of approximately six days. Samples were then sealed using parafilm and screw-top caps, and stored prior to analysis.

In collaboration with Uisce Éireann ([www.water.ie/](http://www.water.ie/)), seven WWTP sites were selected for the collection of biosolid samples used as agricultural fertilisers. Table 2 shows a summary of the characteristics of the WWTPs sampled for biosolids which are relevant to the analysis and interpretation of data. In total, 21 biosolid samples were collected from the seven WWTPs: three samples were collected from each site at four-month intervals (January, May, and September 2023) to assess any seasonal variations, along with nuances relating to relative size and types of treatment occurring at each site. Collected samples were then homogenised and freeze-dried using the same procedure as outlined above for sediments.

Table 2

- Available metadata for biosolid samples collected from 7 WWTPs in Ireland. AD = Anaerobic Digestion; TD = Thermal Drying; THP = Thermal Hydrolysis Processing; P = Pasteurization; LS = Lime Stabilisation.

Site Reference #	Population Equivalent (PE) Band	Output Flow Band ( $\text{m}^3/\text{day}$ )	Types of Biosolids Treatment
WWTP 1	> 100,000	> 30,000	Advanced AD
WWTP 2	> 100,000	> 30,000	AD, TD
WWTP 3	> 100,000	> 30,000	AD, TD
WWTP 4	>100,000	>30,000	THP, AD
WWTP 5	2000 - 50,000	1000 - 10,000	AD, P
WWTP 6	2000 - 50,000	1000 - 10,000	AD, TD
WWTP 7	50,000-100,000	10,000 - 30,000	LS

## 2.2. Chemicals and standards

All native standards (4-MBC, BP-3, OC, EHMC, and HMS) were purchased from Chiron (Trondheim, Norway), and  $d_4$ -4-MBC was purchased from LGC Standards (Teddington, UK). Sodium perfluoro-1-(1,2,3,4- $^{13}\text{C}_4$ ) octanesulfonate (MPFOS) used as a recovery determination (syringe) standard was purchased from Wellington Laboratories (Guelph, ON, Canada). Acetonitrile (HPLC grade), methanol (Optima LC/MS grade), and water (Optima LC/MS grade) were obtained from Fisher Scientific (Loughborough, UK). Q-Sep QuEChERS extraction salt was purchased from Restek Thames (Buckinghamshire, UK). PRiME HLB SPE cartridges ( $3\text{ cm}^3$ , 60 mg) were purchased from Waters (Wilmslow, UK).

## 2.3. Sample extraction

The sample extraction and clean-up protocol was modified from a previous publication (He et al., 2019). Sediment (0.5 g) and biosolid (0.1 g) samples were spiked with 10 ng of  $d_4$ -4-MBC as the internal standard (surrogate standard), and then equilibrated for 30 min. Each sample was vortexed for 3 min with 5 mL of acetonitrile and 5 mL of deionised water, after which QuEChERS salt (2 g of  $\text{MgSO}_4$  and 0.5 g of NaCl) was carefully added. Samples were then vortexed for 1 min to ensure complete mixture before being centrifuged at 3000 RPM for 10 min. The acetonitrile (upper) layer was loaded into a PRiME HLB SPE cartridge ( $3\text{ cm}^3$ , 60 mg) preconditioned with 6 mL of acetonitrile followed by 6 mL of acidified water ( $\text{pH} = 3$  – adding 83  $\mu\text{L}$  of 37 % HCl solution into 100 mL deionised water). Each cartridge was then eluted with 4 mL of acetonitrile, with the eluent dried under a gentle flow of nitrogen and then reconstituted into 0.5 mL of water and 0.5 mL of methanol (0.1 %  $\text{NH}_4\text{OH}$ ) containing 10 ng of MPFOS as the recovery determination standard (syringe standard).

## 2.4. Instrumental analysis

Concentrated extracts were analysed on a Sciex Exion UPLC coupled to a Sciex 5600+ triple TOF MS. An XBridge BEH C18 ( $2.5\text{ }\mu\text{m}$ ,  $2.1\text{ mm} \times 150\text{ mm}$ ) column was employed for the analysis of target compounds. LC and MS conditions are summarised in SI 1.1 and SI 1.2, respectively.

## 2.5. QA/QC

Good linearity was obtained from 5-point calibrations (concentration range: 10–200 ng/mL;  $R^2 > 0.99$ ). Results from five replicate analyses of matrix (0.5 g of sediment) and five replicate analyses of matrix spikes (10 ng of native standards were spiked into 0.5 g of sediment) showed excellent precision and consistency (SI 1.3). Recoveries of the I.S. ranged 69–78 % in the replicate analyses. Recoveries of 4-MBC, BP-3, OC, EHMC, and HMS ranged 54–59 %, 60–65 %, 32–40 %, 50–54 %, and 76–93 %, respectively, in the matrix spikes. A method blank was conducted along with every 5 samples, and recoveries of the internal standard were  $69 \pm 28\%$ . Limits of quantification (LOQ) values for the target analytes were calculated from a signal to noise ratio of 10 (SI 1.4). For those analytes detected in blanks (see SI 1.5), blank correction was performed by subtracting the absolute mass detected in the blank (in ng) from the mass measured in the corresponding sediment or biosolid sample. The blank-corrected concentration was then calculated based on the net mass and the dry weight of the sample..

## 2.6. Data analysis and risk assessment

For comparisons of mean concentrations determined with various metadata recorded (inland vs transitional waters and proximity to UWW emission points for sediments; population equivalent, treatment type, and month of collection for biosolids), non-parametric statistical tests are used as data cannot be assumed or is not observed to follow a specific

distribution. For two sample means, Mann-Whitney *U* tests are used while, for greater number, Kruskal Wallis H tests are used (confidence interval of 95 %, Significance Level (*p*) of 0.05). Where data are recorded to be below the limits of quantification (LoQ), for the purposes of statistical analysis calculations, proxy values were determined using {LOD x Detection Frequency}.

Risk Quotients (RQs) were employed to evaluate ecotoxicological risks for targeted compounds in sediment samples collected in inland and transitional sediments in 2023. RQs were calculated per the following equation:

$$RQ = \frac{MEC_{95}}{\text{Lowest PNEC}}$$

where  $MEC_{95}$  is the 95th percentile of the maximum measured environmental concentration from collected samples herein, and lowest predicated no-effect concentration (PNEC) in sediments are taken from the Norman Ecotoxicology Database ([www.norman-network.com/normans/ecotox](http://www.norman-network.com/normans/ecotox)). Degree of risk is assessed based on the resulting risk quotient, i.e.:  $RQ > 1$  – high risk;  $1 > RQ > 0.1$  – moderate risk;  $RQ < 0.1$  – low risk.

### 3. Results and discussion

#### 3.1. Concentrations of ultraviolet filters in sediments

Table 3 shows concentrations of the five UVF chemicals in inland and transitional sediment samples collected from sites across Ireland. A comparison of means was conducted between inland samples and transitional samples. While mean concentrations of 4-MBC, OC, and HMS in transitional sediments were slightly higher in inland sediments, this difference was not statistically significant ( $p < 0.05$ ). Therefore, for the purposes of reporting and statistical analysis, sediment data will be considered as a single dataset of  $n = 81$  samples.

Of our target UVFs, the highest concentrations detected in sediments were of: OC, followed by HMS, and 4-MBC, with 4-MBC additionally being the most frequently detected above LOQ (92.6 %). While no formal data on the market share of these UVFs in Ireland is available, the CPR lists the allowable concentrations of these chemicals in various cosmetics products. The high concentrations OC are consistent with the allowed use of 9–10 % in cosmetics products. HMS and 4-MBC were also until recently permitted at concentrations of 4 % and 10 % respectively, consistent with the relatively high concentrations detected in sediment samples. Several studies report that of these five targeted UVFs, EHMC and BP-3 are the most widely used organic UVFs in the cosmetics industry (Chayada et al., 2021; Menzie et al., 2022; Pniewska and Kalinowska-Lis, 2024). EHMC is currently permitted to be used in most products at concentrations up to 10 %, with BP-3 permitted between 0.5 and 6 % depending on the product. Data reported here however shows BP-3 and EHMC are not very prevalent in either Irish sediment or biosolid samples (Section 3.2.1). This trend is similarly reflected in

international data: both BP-3 and EHMC (along with OC) are found at quite low concentrations, while those of HMS, and 4-MBC are in the moderate range (Table 4). Investigation of the regional prevalence of UVF-containing products in various markets would be highly informative and aid targeted screening measures based on regional needs.

#### 3.2. Ecotoxicological risk assessment of UVFs in Irish sediments

Based on the sediment data collected and the corresponding  $MEC_{95}$  concentrations (Table 5), calculated risk quotients (RQs) show that 4-MBC, EHMC, and HMS are all of moderate ecotoxicological risk, based on current PNECs as promulgated by the NORMAN Network (Norman, 2024). The RQ determined herein for EHMC in sediments exceeds those reported in sediments by the Review of the 1st Watch List for the WFD (Loos et al., 2018). However, a relatively small number of studies have been conducted in sediments at an EU-level with the review highlighting the likelihood of EHMC partitioning into the solid phase due to high lipophilicity. Results herein indicate a moderate risk of EHMC in sediment and thus a potential risk to biota due to these lipophilic properties, thus further studies are recommended to determine regional ecotoxicological implications. 4-MBC was not hitherto on the WFD watch list, though is currently listed by ECHA as an Endocrine Disruptor (ECHA, 2024b) and will be restricted in cosmetics in the EU as of May 2026 (EU, 2024). Levels detected in this current study suggest a moderate ecotoxicological risk; therefore, ongoing surveillance monitoring would be recommended post-restriction to ensure concentrations fall to Low Risk levels after restriction. Homosalate would similarly require a degree of ongoing monitoring to verify whether environmental levels continue to fall. Meanwhile, concentrations of both OC and BP-3 suggest low ecotoxicological risk in Irish sediments, though this low risk is not necessarily indicative of low risk in other jurisdictions based on levels found internationally. Both these chemicals are currently on the WFD's Watch List for further environmental and toxicological assessment. As these chemicals are more recent additions to the WFD Watch List, comparatively less assessment has been conducted in terms of both environmental contamination and toxicology, thus the relevant PNECs may be liable to change following the accrual of further data. Further assessments may therefore be necessary should PNECs be significantly lowered based on emerging toxicological information.

#### 3.3. Concentrations of ultraviolet filters in biosolids

Table 6 shows the summary statistics for UVFs in biosolid samples. As expected, higher concentrations are present in WWTP-derived biosolids compared to sediment samples as wastewater is a known sink of UVFs, which are likely to partition to the solid fraction due to their lipophilic properties (Ramos et al., 2020). By a wide margin, the highest average concentrations were of OC (666 ng/g) and HMS (456 ng/g), matching the pattern seen in Irish sediment samples. Contrastingly, 4-MBC, while present at concentrations a similar order of magnitude to

**Table 3**  
– Summary statistics for UVFs in 81 inland and transitional sediment samples collected in 2023.

	ng/g	EHMC	4-MBC	BP-3	HMS	OC
<i>Inland and Transitional Sediments (n = 81)</i>	Mean	0.04 (<LOQ)	1.87	0.19 (<LOQ)	2.35	3.66
	Median	<LOQ	1.09	<LOQ	<LOQ	1.82
	Range	<LOQ-0.99	<LOQ-10.7	<LOQ-3.76	<LOQ-32.0	<LOQ-31.8
	DF (%)	9.9	92.6	60.5	21.0	61.7
<i>Inland Sediments (n = 62)</i>	Mean	0.02 (<LOQ)	1.67	0.18 (<LOQ)	1.97	3.35
	Median	<LOQ	0.98	0.05	<LOQ	1.82
	Range	<LOQ-0.24	<LOQ –10.7	<LOQ –3.76	<LOQ –16.4	<LOQ –29.4
	DF (%)	8.1	90.3	59.7	19.4	61.3
<i>Transitional Sediments (n = 19)</i>	Mean	0.07 (<LOQ)	2.54	0.21	3.57	4.69
	Median	<LOQ	2.10	0.08	<LOQ	3.04
	Range	<LOQ-0.99	0.20–7.12	<LOQ-1.08	<LOQ-32.0	<LOQ-31.8
	DF (%)	15.8	100	63.2	26.3	63.2
	LoQ	0.13	0.14	0.02	1.42	0.13

**Table 4**

– Concentrations of organic ultraviolet filters in sediments in Ireland (this study) compared to similar studies worldwide (Costa et al., 2024; Tovar-Salvador et al., 2023; Li et al., 2022; Peng et al., 2017; Pintado-Herreram et al., 2017; Langford et al., 2015; Gago-Ferrero et al., 2011a). <sup>a</sup>4-MBC was only quantified in 48 of 66 samples.

		EHMC	4-MBC	BP-3	HMS	OC
This Study	Mean	0.04	1.87	0.19	2.35	3.66
	Median	<LOQ	1.09	0.06	<LOQ	1.82
	Range	<LOQ – 0.99	<LOQ – 10.7	<LOQ – 3.76	<LOQ – 32.0	<LOQ – 31.8
	DF (%)	9.9	92.6	60.5	21.0	61.7
Costa et al., 2024, Admiralty Bay, Antarctica (n = 17)	Mean	5.23	<LOQ	9.70	19.7	20.2
	Median	3.60	<LOQ	<LOQ	5.00	11.5
	Range	<LOQ-26.2	<LOQ	<LOQ-86.0	<LOQ-251	<LOQ-93.8
	DF (%)	76.5	0	35.3	88.2	93.8
Tovar-Salvador et al., 2023, Atacama, Chile (n = 36)	Mean	0.36	0.05	0.08	0.28	4.79
	Median	–	–	–	–	–
	Range	nd-2.52	nd-1.61	nd-0.5	nd-1.06	nd-33.4
	DF (%)	>71	>11	>71	>71	>70
Li et al., 2022, Tianging, China (n = 59)	Mean	–	–	–	–	–
	Median	1.56	1.47	–	2.04	–
	Range	0.625–10.3	0.359–8.91	–	<1.45–25.8	1.03–175
	DF (%)	–	–	–	–	–
Peng et al., 2017, Pearl River Delta, China (n = 27)	Mean	3.36	–	0.44	–	6.93
	Median	1.30	–	0.32	–	1.04
	Range	<LOQ-22.4	–	0.14–1.07	–	0.05–91.7
	DF (%)	81.5	–	100	–	100
Pintado-Herreram et al., 2017, Andalusia, Spain (n = 66 <sup>a</sup> )	Mean	7.49	1.70	0.75	3.24	19.3
	Median	–	–	–	–	–
	Range	–	–	–	–	–
	DF (%)	100	75	86.9	89.8	100
Langford et al., 2015, Norway (n = 5)	Mean	–	–	–	–	–
	Median	–	–	<LOD	–	–
	Range	8.5–19.8	–	<LOD	–	<LOQ-82.1
	DF (%)	–	–	–	–	–
Gago-Ferrero et al., 2011a, 2011b, Ebro, Spain (n = 20)	Mean	4.85	<LOQ	4.60	–	240
	Median	<LOD	<LOQ	<LOQ	–	62.0
	Range	<LOQ-42.0	<LOQ	<LOQ	–	<LOQ-2400
	DF (%)	20.0	0	35.0	–	95

**Table 5**

– Risk Quotients determined from lowest predicted no-effect concentrations (PNECs) taken from the NORMAN Toxicological Database (as of 5th November 2024) using 95th percentile concentrations (MEC<sub>95</sub>). QSAR (Quantitative Structural Activity Relationship) utilised for determining PNEC where experimental PNECs not yet determined.

Chemical	PNEC (ng/g)	PNEC Determination	MEC <sub>95</sub> Concentration	RQ (95th percentile)
EHMC	1.28	QSAR	0.165	0.129
4-MBC	14.4	Experimental	5.82	0.404
BP-3	122	Experimental	0.649	0.005
HMS	127	QSAR	13.9	0.110
OC	606	Experimental	12.4	0.021

OC and HMS in sediment samples, is much less abundant in biosolids at a mean concentration ~ 30–50 times lower than OC and HMS. While data on the half-lives of these chemicals is limited – particularly for OC and HMS (PubChem, 2024a; PubChem, 2024b; PubChem, 2024c; PubChem, 2024d; PubChem, 2024e) – a recent study suggests that the half-life of 4-MBC is substantially shorter than that of OC under controlled conditions (170 vs 7000 h) (O'Malley et al., 2021) which may provide insight to the disparity between sediment and biosolid results.

Despite the elevated levels of OC in Irish biosolids, levels of almost all UVFs determined herein are low compared to data reported

**Table 6**

– Summary statistics for UVFs in 21 biosolid samples collected in 2023.

	ng/g	EHMC	4-MBC	BP-3	HMS	OC
Biosolids (n = 21)	Mean	12.65	13.91	1.28	456	666
	Median	<LOQ	14.30	<LOQ	413	393
	Range	<LOQ-63.87	4.596–30.04	<LOQ-7.77	122–914	90.1–1880
	DF (%)	42.9	100	47.62	100	100
	LoQ	0.71	0.18	0.10	7.09	0.64

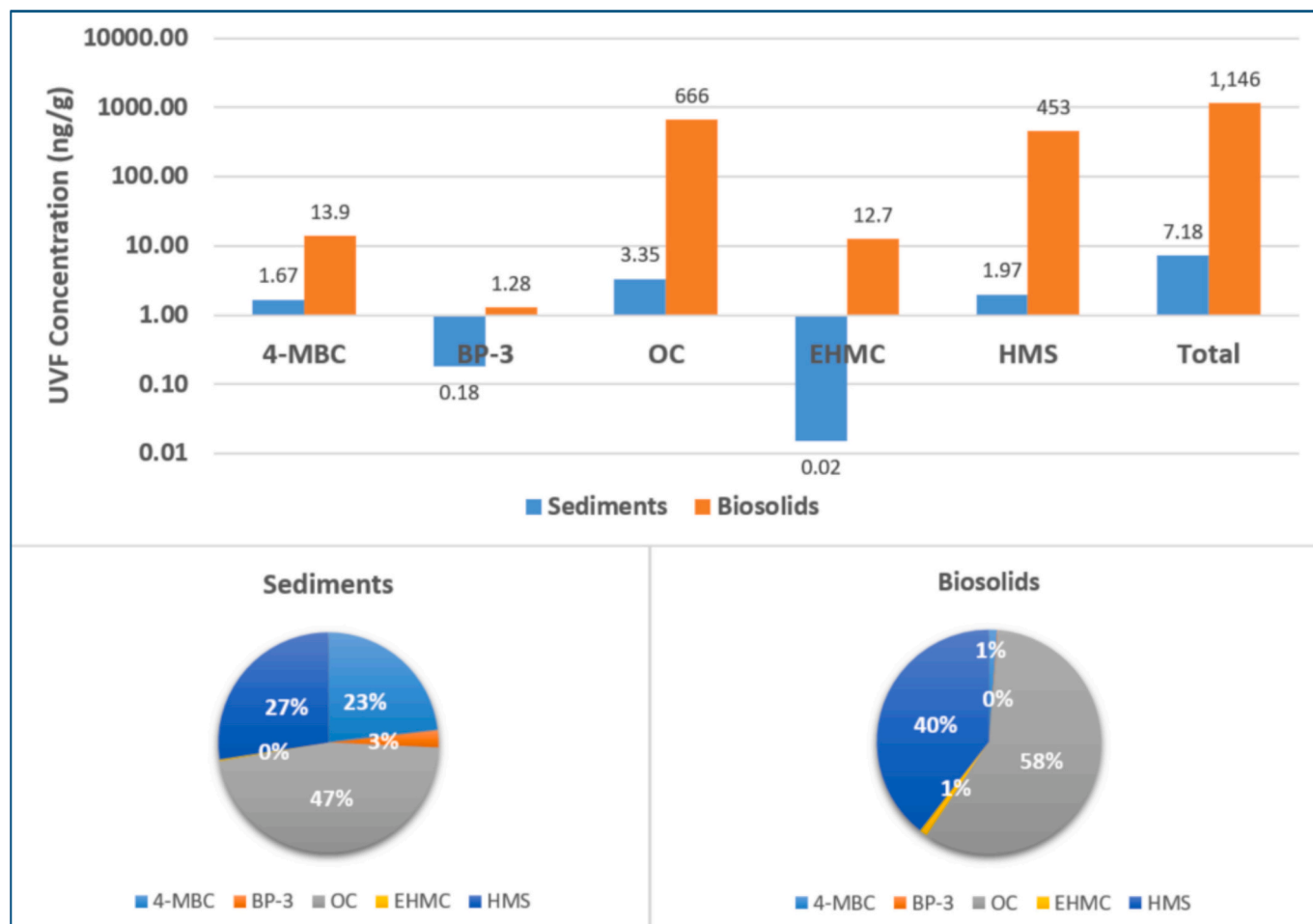
internationally (Table 7). This is with the exception of homosalate (which has been quantified in a single other study to the authors' knowledge) which is slightly higher than levels detected in a single WWTP in Spain. Coupled with the generally lower levels observed in sediments (Fig. 2), points to lower use of UVF-based products in Ireland, with the potential exception of homosalate. The market share of sun protection products in Ireland had an estimated revenue of approximately \$42 m in 2024, compared to the EU-average of approximately €80 m (Statista, 2024). Despite this, levels determined in sediments are still indicative of a moderate ecotoxicological risk, risks which are likely higher in other jurisdictions based on the comparable data. As an additional outlier, few other data is available on the levels of HMS in WWTP-derived biosolids or their presence in inland and transitional sediments. Given this, along with the recent restriction at EU-level due to aquatic toxicity, HMS should be much more thoroughly monitored in the environment as well as in potential sources to monitor the efficacy of restrictions, as well as internationally to assess the environmental risk where restrictions may not yet be implemented.

Statistical tests were conducted to identify trends with available metadata (SI 2.1). While variations in mean concentrations and statistically significant differences were observed for some UVFs between both treatment technologies and population equivalent of the WWTPs (SI 2.3 & 2.4), no significant trends are discernible. Variations in concentrations as a function of collection month (SI 2.4) also show no clear

**Table 7**

– Concentrations of ultraviolet filters in biosolids in Ireland (this study) compared to similar studies worldwide [, (Mao et al., 2020; Biel-Maeso et al., 2019; Liu et al., 2011; Gago-Ferrero et al., 2011b; Nieto et al., 2009; Plagellat et al., 2006)].

		EHMC	4-MBC	BP3	HMS	OC
<i>This Study</i>	Mean	12.7	13.9	1.28	453	666
	Median	<LOQ	14.3	<LOQ	413	393
	Range	LOQ – 63.9	4.60–30.0	LOQ – 7.77	122–914	90.1–1890
	DF (%)	42.9	100	47.6	100	100
Mao et al., 2020, Changsha China (n = 5)	Mean	–	–	1.94	–	–
	Median	–	–	1.43	–	–
	Range	–	–	Nd-4.33	–	–
	DF (%)	–	–	60.0	–	–
Biel-Maeso et al., 2019, Cadiz Spain (n = 10)	Mean	633	622	19.8	322	3200
	Median	473	352	19.5	154	2240
	Range	352–1830	n.d – 1240	11–32	57–1220	654–7200
	DF (%)	100	80	100	100	100
Liu et al., 2011, Adelaide Australia (n = 3)	Mean	31.9	250	74.0	–	138.4
	Median	–	–	–	–	–
	Range	–	–	–	–	–
	DF (%)	–	–	–	–	–
Gago-Ferrero et al., 2011a, 2011b, Catalonia Spain (n = 15)	Mean	1489	1896	52.7	–	–
	Median	1220	1630	Nd	–	–
	Range	610–3350	730–3830	Nd-790	–	–
	DF (%)	100	100	6.7	–	–
Nieto et al., 2009, Tarragona Spain (n = 3)	Mean	–	–	17.7	–	1450
	Median	–	–	20	–	1800
	Range	–	–	10–20	–	700–1840
	DF (%)	–	–	100	–	100
Plagellat et al., 2006, Switzerland (nationwide), (n = 28)	Mean	1780	110	–	–	5510
	Median	1580	100	–	–	3450
	Range	150–4980	10	–	–	700–27,700
	DF (%)	100	390	–	–	100



**Fig. 2.** – Graphical overview of UVF concentrations in biosolid and sediment compartments.

differences based on seasonal sampling. These results are likely due to the inability in this instance to control for extraneous factors such as influent type, seasonal/temporal variations in input, and operational factors, and it is noteworthy that low sample numbers limit the power of these tests. Other studies have noted potential trends in the effects of WWTP operations on UVFs in biosolids (Ramos et al., 2016; Mitchelmore et al., 2021; Liu et al., 2012) though these are also limited by low sample numbers and difficulty controlling for extraneous factors. As these studies and ours suggest, there is also a clear need for dedicated, controlled, and statistically powerful studies on the effects of WWTP operations on emerging contaminants such as UVFs.

### 3.4. Impact of wastewater emission points on UVF concentrations in sediments

Significantly higher concentrations of  $\Sigma$ UVFs ( $p < 0.01$ ) were detected in sediments downstream from known UWW emission points, with highest concentrations of each individual compound also detected in areas directly adjacent to or downstream from (within 0.5 km) UWW or STP emission points. This aligns with previous studies highlighting the correlation between these emission points and environmental prevalence (see Section 1), indicating that environmental emission sources of UVFs in Ireland are likely similar. However, elevated concentrations are also seen in areas isolated from these sources, indicating that other diffuse sources may additionally be resulting in environmental uptake. As outlined in Section 3.3, UVFs are also present in WWTP-derived biosolids, approximately 98 % of which are used as agricultural fertilisers in Ireland (Uisce Éireann, 2024). An extensive review by Ramos et al (Ramos et al., 2020) highlights the ready transfer of UVFs through water as well as their accumulation in soil. The higher lipophilicities of these chemicals potentially indicate a propensity for long-range environmental transport leading to their presence in more rural sediments.

## 4. Conclusions

Environmental assessment of UVFs is of key importance due to their ubiquitous use and the demonstrable ecotoxicological effects of some of the more prominently used chemicals in this class. While restrictions on use of HMS and 4-MBC in light of their hazardous effects is a promising development, the moderate ecotoxicological risk based on the levels found in sediments in this study mean that continued monitoring is recommended to determine whether their restriction will reduce environmental contamination or whether additional measures are required. Meanwhile, though results reported herein for OC, EHMC, and BP-3 add to the international database of sunscreen agents in sediments, further data is needed EU-wide to facilitate more thorough risk assessment. This is most notably required for EHMC as findings here suggest that, despite its low detection frequency, concentrations may indicate moderate ecotoxicological risk in sediments. With regards to biosolids, the elevated levels of homosalate found in samples herein highlight the need for further data internationally to determine whether this is a substance of concern for this matrix. More broadly, in light of the increasing environmental awareness and emerging toxicological effects of UVFs, continued investigation of these and other priority and emerging UVFs would be highly beneficial, in anticipation of revisions to existing directives (EU, 2023) which will allow for the implementation of swifter and more targeted measures to deal with these emerging contaminants.

### CRedit authorship contribution statement

**Martin Sharkey:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Yulong Ma:** Writing – original draft, Visualization, Validation, Supervision, Resources, Project

administration, Methodology, Investigation, Formal analysis. **Liu Yang:** Validation, Methodology, Investigation, Formal analysis. **Stuart Har-rad:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Mark G. Healy:** Writing – review & editing, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **William A. Stub-bings:** Writing – review & editing, Validation, Supervision, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Ann Marie Coggins:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

### 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. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2025.180382>.

### Data availability

Data will be made available on request.

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