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Occurrence of twelve UV filters and evidence of cis-octinoxate formation in Adriatic coastal waters: Environmental implications

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ABSTRACT

UV filters used in personal care products are increasingly recognised as emerging contaminants due to their persistence and ecological risks. Detected in aquatic systems at concentrations from ng L^{-1} to μg L^{-1}, their monitoring is essential for environmental risk assessment but challenging because of their diverse physicochemical properties, requiring sensitive analytical methods.

This study optimised a solid-phase extraction gas chromatography–tandem mass spectrometry (SPE-GC–MS/MS) method for the simultaneous determination of twelve UV filters in marine waters. The method showed suitable linearity, precision, and recoveries, with detection limits below 1 ng L^{-1}. Seawater samples collected along the Adriatic coast (Rimini and Riccione, Italy) during peak recreational activity were analysed. All twelve UV filters were detected, with octocrylene (up to 23,100 ng L^{-1}), homosalate (up to 16,400 ng L^{-1}), and ethylhexyl salicylate (up to 10,900 ng L^{-1}) being most abundant. *Trans*-2-Ethylhexyl methoxycinnamate (*E*-EHMC, the trans isomer of octinoxate, 13–1474 ng L^{-1}) occurred consistently with its isomer *Z*-EHMC (cis-octinoxate), contributing 18–64 % of total (*E* + *Z*)-EHMC, suggesting in situ photochemical conversion, since the cis form is negligible in cosmetic products. Benzophenone, ethylhexyl dimethyl PABA, 4-methylbenzylidene camphor, benzyl salicylate, and isoamyl methoxycinnamate were found at lower yet relevant concentrations. UV filters banned under European regulations, such as menthyl anthranilate and etocrylene, were absent or barely detected.

This work provides the first data on sunscreen UV filters in Adriatic coastal waters and the first quantitative evidence of *Z*-EHMC in environmental waters, highlighting its in situ formation and contributing to understanding their environmental fate and risks.

1. Introduction

The UV filters contained in sunscreens and personal care products are widely used to protect the skin from harmful UV radiations, preventing premature skin ageing, sunburn, and skin cancer. However, concerns have been raised about their potential negative effects (Breakell et al., 2024). Their widespread occurrence in aquatic environments accompanied by their potential toxicity have led to recognise UV filters as Contaminants of Emerging Concerns (Gandar et al., 2025). Despite this, environmental quality guidelines are still lacking due to gaps in environmental risk assessments; increasing interest in studying their distribution in water systems.

UV filters are regulated in cosmetics by European Regulation, which limits their concentrations and restricts or prohibits certain compounds (European Union, 2009). Currently, 27 UV filters are authorised for use in the European Union (EU), with concentrations limit ranging from 2 % to 15 % (Cadena-Aizaga et al., 2020). Global production of UV filters (including avobenzone (AVO), benzophenone-3 (BP-3), (*E*)-2-ethylhexyl methoxycinnamate (*E*-EHMC), octocrylene (OCR), and 4-methylbenzylidene camphor (4-MBC)), has increased substantially, from over thousand tons in 2005 to million tons by 2020 (Brausch and Rand, 2011; Sun et al., 2024).

Regarding water policy, UV filters were not specifically addressed until 2015, when EHMC (octinoxate) was added to the EU Watch list for

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monitoring substances in water (Gomez Cortes et al., 2022; European Union, 2015). Subsequently, seven candidates were proposed for the 4th Watch list, three of which were sunscreen agents, underlining the environmental importance of this group of water pollutants (Gomez Cortes et al., 2022). In the 5th Watch list (2025), another UV filter (2-ethylhexyl salicylate) was also included (European Union, 2025).

E-EHMC is of particular concern due to its photostability, undergoing photoisomerisation to (*Z*)-2-ethylhexyl methoxycinnamate (*Z*-EHMC, cis-octinoxate) under sunlight, a transformation product associated with greater toxicity potential than its parent *trans* isomer (Nečasová et al., 2017; Sharma et al., 2017). A recent review by Bordalo et al., 2025, surveyed the levels of EHMC in various marine and coastal compartments and examined its toxicological effects, highlighting the importance of monitoring UV filters to establish appropriate threshold values. In light of recent SCCS evaluations flagging *E*-EHMC for its potential endocrine-disrupting effects (Bernauer et al., 2025), investigating both the parent compound and its transformation product is essential for comprehensive environmental risk assessment. The continuous detection and transformation in the environment highlight the need to assess not only their chemical persistence but also their potential biological and ecological effects. Indeed, the UV filters benzophenone-3 (BP-3) and EHMC have been shown to harm coral reefs and other aquatic ecosystems even at very low concentrations (parts-per-trillion), causing coral bleaching, larval damage, and hormonal disruptions in marine life. These findings prompted Hawaii to ban sunscreens containing these compounds. Few other countries have yet taken this recommendation into account (Celeiro et al., 2019).

The environmental impacts of UV filters have raised growing concerns, leading to more studies in recent years (Brausch and Rand, 2011). These compounds enter aquatic environments (seas, lakes, and rivers) directly through activities like swimming and sunbathing, and indirectly via wastewater (Carve et al., 2021; Mokh et al., 2022; Vila et al., 2017). Several studies found that concentration of UV filters (specifically for OCR and 4MBC) in seawater might be higher than safe levels for marine life (Cadena-Aizaga et al., 2022).

Detecting organic UV filters is challenging due to their low concentrations (in the ng L^{-1} to μg L^{-1} range) and the interference from the sample matrix (Oubahmane et al., 2023). To ensure the reliability and accuracy of results, appropriate sample preparation methods are essential to concentrate the target analytes (Capela et al., 2019; Hejji et al., 2023). The sorbent-bases extraction method SPE is the most widely preferred, as it simultaneously facilitates trace analytes preconcentration and interference removal in a single step (Montesdeoca-Esponda et al., 2012; Pedrouzo et al., 2011; Wang et al., 2018).

Rimini and Riccione, located along Italy's Adriatic coast, are renowned tourist destinations known for their sandy beaches and vibrant summer tourism. Thus, it is expected that during peak summer months, the increased use of personal care products, particularly sunscreens, leads to a higher influx of UV filters into the sea. However, to the best of our knowledge data on the occurrence of UV filters in seawaters of Adriatic Sea is not available in the current literature apart Venice lagoon for EHMC (Pizzini et al., 2024). Concentration data on sediments highlighted the importance of river as the main contributor of many contaminants in the Adriatic basin, but the touristic coastal areas were identified as a direct input of UV filters from recreational activities (Combi et al., 2016).

The aim of the present study is to propose an analytical methodology and to investigate the presence of twelve UV filters and one transformation product, *Z*-EHMC, in coastal waters in Rimini and Riccione during the summer season. As mentioned, there is not available information about UV filters in the Adriatic Sea, as well as no quantitative information on the occurrence of *Z*-EHMC in natural waters, revealing a critical knowledge gap that require further investigation. This study contributes to advancing environmental risk assessment methodologies by providing essential data on the concentrations of UV filters and the transformation product *Z*-EHMC in coastal waters, thereby supporting regulatory decision-making and helping to mitigate potential ecological risks.

2. Material and methods

2.1. Standards and reagents

The UV filters analysed in this study included (*E*)-2-ethylhexyl methoxycinnamate (*E*-EHMC; 98.5 %, Dr. Ehrenstorfer, Augsburg, Germany), 4-methylbenzylidene camphor (4-MBC; 99.8 %, Alfa Aesar, Karlsruhe, Germany), benzophenone-4 (BP-4; 98.7 %), benzyl salicylate (BS; >99 %), and menthyl anthranilate (MA; 99.9 %, Fluka, St. Louis, MO, USA). Ethylhexyl dimethyl PABA (EHPABA; 98 %), octocrylene (OCR; 99.1 %), homosalate (HMS; 99.9 %), and ethylhexyl salicylate (EHS; >99 %) were purchased from Sigma-Aldrich (St. Louis, MO, USA); benzophenone-3 (BP-3; 99.9 %), isoamyl methoxycinnamate (IAMC; 96 %), and etocrylene (Eto; 99.7 %) were purchased from Tokyo Chemical Industry (Tokyo, Japan). All solvents and reagents were of analytical grade. Ethyl acetate (EtAc, 99.5 %), methanol (MeOH, 99.9 %), ultrapure water, and acetonitrile (ACN, 99.9 %) were provided by Scharlab (Barcelona, Spain), and dichloromethane (DCM, ≥99.9 %) was provided by Avantor (VWR, Randor, PA, USA). Stock solutions of all the compounds were prepared in MeOH at concentration of 1000 mg L^{-1} and further dilutions were made from the stock. Stock and working solutions were stored in glass vials protected from light and kept in a freezer at −20 °C. HLB and C18 CHROMABOND® SPE cartridges (500 mg, 6 mL) were provided by Machery-Nagel (Düren, Germany).

2.2. Sample collection

Seawater samples were collected from three different beaches in Rimini (bathing zone 17, 18 and 20) and Riccione (bathing zones 34, 87, and 110) at different locations, along the Italy's Adriatic coast (Fig. 1). Sampling was carried out from July–September 2024, mostly around the noon. These months were selected due to peak recreational activities during summer.

A total of fourteen seawater samples were collected between 3 and 4 m from the shoreline, within the central area of the bathing zone, and approximately 50 cm below the water surface. Samples collected in close proximity in time and space were considered independent since water movement and mixing rapidly change water masses. Variability can occur even at the same location but at different depths (Labille et al., 2020), and local water dynamics strongly affect UV filter concentrations (Cadena-Aizaga et al., 2022). All samples were stored frozen and protected from light until analysis. Additional sample information and site details (based on publicly accessible data) are given in Table S1.

Cosmetic samples labelled as containing EHMC (lip balm, hair oil and sunscreen) were purchased from local markets (see Table S2).

2.3. Photoisomerisation experiments

Due to the absence of a commercially available *cis* isomer of EHMC standard, *E*-EHMC was used for the quantification of *Z*-EHMC. This approach was already adopted in the literature (Poiger et al., 2004) and was supported by the observation that both isomers shared identical mass spectrometric fragmentation patterns and transitions during GC–MS/MS analysis. This has been proved by laboratory experiments conducted to obtain *Z*-EHMC from *trans* isomer (*E*-EHMC). Aliquots of *E*-EHMC standard solution in ethyl acetate were placed in quartz cuvettes and irradiated in a photoreactor equipped with two UV lamps ($\lambda = 254$ nm, 8 W, and 11 W; Philip, Holland) at a distance of 9 cm for 10 min. After irradiation, samples were analysed by GC–MS to identify and confirm the retention times, mass spectra and transitions of the *E*- (*trans*-) and *Z*- (*cis*-) EHMC isomers (see Fig. S1).

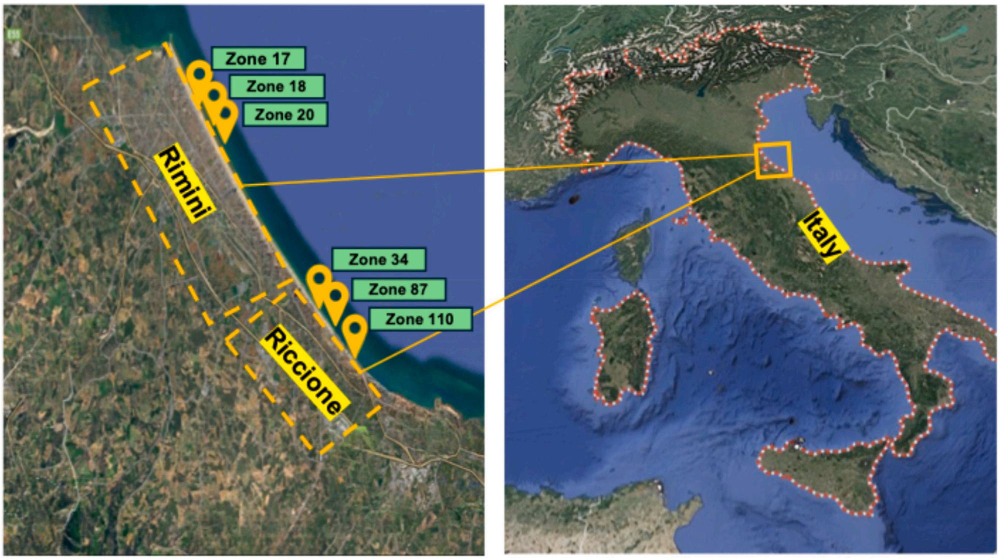

Fig. 1. Sampling area and sites along six beaches on the Adriatic Sea.

2.4. *Extraction procedure*

The SPE extraction procedure was performed by modifying a method previously reported (Liu et al., 2011). A SPE method coupled with HPLC-DAD was initially optimised for two of the UV filters, *E*-EHMC and OCR. Later, under the optimised conditions (see Section 3.2 discussion), the method was validated and applied to the determination of twelve UV filters and one transformation product using GC–MS/MS for their analysis. Both HLB and C18 SPE cartridges were used for extraction. Each cartridge was preconditioned with 6 mL of DCM, 6 mL of MeOH followed by 6 mL of Milli-Q water. The unfiltered seawater samples (~ 400 mL) were loaded onto the cartridges at a flow rate of 5 mL min^{-1}. A cleaning step (salt or impurity elimination) was carried out with 10 mL of Milli-Q water (5 % MeOH). Elution was done with 2 mL of MeOH (fraction discarded), and 4 mL of DCM. DCM extract was reduced to the final volume of 1 mL under the flow of N_2 gas, using an evaporation system.

2.5. *Instrumental analysis*

2.5.1. *HPLC-DAD analysis*

Chromatographic analyses were carried out using an Agilent Technologies SL 1200 Series HPLC system (Waldbronn, Germany) equipped with a binary pump and micro vacuum degasser. Separation was achieved on a ZORBAX SB-C18 column (150 × 4.6 mm, 3.5 μm). The gradient elution was programmed at a flow rate 2 mL min^{-1}: from 55 % to 45 % H_2O in 1 min, 45–30 % H_2O in next 10 min and, then to 100 % ACN in next 10 min. The injection volume was 20 μL and the column temperature was set at 40 °C. Detection was performed with UV/DAD detector set at 310 nm as the primary wavelength. Data acquisition and processing were managed using Agilent ChemStation software. Optimal sensitivity and peak resolution for two representative UV filters (OCR and *E*-EHMC) were obtained using eluent A (ACN) and eluent B (H_2O). A standard solution containing both compounds was analysed, and the resulting chromatogram is shown in Fig. S2.

2.5.2. *GC–MS/MS analysis*

Gas chromatography coupled with tandem mass spectrometry (GC–MS/MS) was performed using a Thermo Scientific Trace 1310 GC system combined with a triple quadrupole mass spectrometer (TSQ 8000) and an IL 1310 autosampler (Thermo Scientific San Jose, CA, USA).

The chromatographic GC–MS/MS conditions for the determination of the target UV filters were previously developed by the authors (Celeiro et al., 2019; Vila et al., 2015). Separation was achieved on a Zebron ZB-Semivolatiles column (30 m × 0.25 mm i.d., 0.25 μm film thickness, Phenomenex). Helium (purity 99.999 %) was used as the carrier gas at constant flow rate of 1.0 mL min^{-1}. The oven program was set as follows: initial temperature 100 °C (held 1 min), ramped to 290 °C at 25 °C min^{-1} (held 10 min). Samples were introduced in surged splitless mode (200 kPa, 1.20 min), with the injector maintained at 260 °C. The MS was operated in electron impact (EI) positive ionisation mode at +70 eV, with transfer line and ion source temperatures held at 290 °C and 350 °C, respectively. MS/MS acquisition was optimised using the Selected Reaction Monitoring (SRM) tool in the TSQ 8000. Two to three transitions were selected per compound: one for quantification, and the additional transition(s) for identification and confirmation purposes. The SRM transitions and corresponding collision energies are summarised in Table 1. Instrument control and data processing were carried out with Xcalibur 3.0 and Trace Finder™ 3.2 software.

Since BP-3 and BP-4 co-eluted and exhibited identical transitions due to their similar chemical structures, sharing the same benzophenone core, they were analysed and quantified together as a combined mixture. *Z*-EHMC and *E*-EHMC eluted at different retention times but showed the same spectra and transitions. Quantification of the target compounds was performed using external calibration with standard solutions of the compounds in DCM, since the extraction was quantitative, and no matrix effect was observed.

3. Results and discussion

3.1. *SPE optimisation*

The extraction and elution parameters that potentially affect the SPE technique were optimised to achieve the best extraction efficiencies for the target UV filters using synthetic seawater (3.5 % NaCl). Initially, the SPE was optimised for two of the target UV filters, EHMC and OCR, since only an HPLC-DAD instrument was available at that time (see Section 2.5.1). Subsequently, the final optimised conditions were applied to the other studied UV filters, demonstrating good overall suitability. For SPE optimisation, different parameters were studied: cartridge type, sample pH, salting-out effect, and elution solvent.

SPE extraction was carried out using HLB and C18 cartridges, employing identical conditioning and elution protocols. Water samples (50 mL) were fortified with the target UV filters at 100 μg L^{-1} and then passed through the SPE cartridges at a flow rate of 5 mL min^{-1}. Elution

Table 1
GC–MS/MS: retention times and selected SRM transitions.

Acronym	INCI Name[a]	RT (min)	SRM (*m/z*)[b]			CE (eV)[c]
EHS	Ethylhexyl salicylate	8.64	*120*	→	*92*	*10*
			138	→	120	10
			250.1	→	120	15
BS	Benzyl salicylate	9.00	*91*	→	*65*	*15*
			228.1	→	91	10
HMS	Homosalate	9.04	120	→	92	10
			138	→	*120*	*10*
			262.2	→	120	15
IAMC	Isoamyl methoxycinnamate	9.61	*161*	→	*133*	*10*
			178.1	→	161.1	10
			248.1	→	178	10
BP-3	Benzophenone-3	9.68	*151*	→	*95*	*10*
			227.1	→	127.9	35
			227.1	→	184	20
BP-4	Benzophenone-4	9.68	*151*	→	*95*	*10*
			227.1	→	127.9	35
			227.1	→	184	10
4MBC	4-Methylbenzylidene camphor	9.79	127.9	→	102	20
			170.6	→	128.1	15
			254.1	→	*239.2*	*10*
MA	Menthyl anthranilate	9.95	119	→	91.8	10
			137	→	*199*	*10*
			275.2	→	137	10
Z-EHMC	*Cis*-Ethylhexyl methoxycinnamate	10.09	161	→	133.1	10
			177.9	→	133.1	20
			290.2	→	*178.1*	*10*
Eto	Etocrylene	10.12	231.9	→	176.5	20
			248	→	164.9	25
			276.9	→	*248.1*	*10*
EHPABA	Ethylhexyl dimethyl PABA	10.51	148	→	104.2	25
			165.1	→	148.6	25
			277.2	→	*164.9*	*10*
E-EHMC	*Trans*-2-Ethylhexyl methoxycinnamate	10.68	161	→	133.1	10
			177.9	→	133.1	20
			290.2	→	*178.1*	*10*
OCR	Octocrylene	12.27	232	→	203	20
			248	→	*165*	*30*
			360.2	→	276.1	20

[a] International nomenclature of cosmetic ingredients.

[b] Italicised values show the quantification transitions.

[c] CE: collision energy (eV).

was carried out with a total of 6 mL: 2 mL of MeOH and, 4 mL of DCM, recovering the analytes. As depicted in Fig. 2a, HLB cartridges provided better extraction efficiency, offering a higher chromatographic response for both analytes compared to C18 cartridges. This performance is consistent with literature reports demonstrating the broad retention capabilities of HLB sorbent for compounds with mixed hydrophobic and polar characteristics (Liu et al., 2011).

The influence of pH on analyte extraction was assessed at pH 3 (0.2 M HCl) and neutral seawater pH (~ 8). As can be seen in the Fig. 2b, chromatographic responses at pH 3 were comparable to those at neutral pH. Since the analytes are non-ionisable under this pH range hydrophobic interactions should enhance; on the other hand, acid addition risks co-exiting humic substances that can foul cartridges (Tayeh et al., 2025). Therefore, maintaining the native seawater pH simplifies sample preparation, avoiding hazardous acid handling, and it aligns with greener analytical practices without compromising extraction efficiency.

To probe the salting-out effect NaCl was added to seawater at 10 % and 20 % (*w*/*v*). A slight decrease in peak areas was observed compared to natural salinity (~3.5 % NaCl), but overall signals remained similar (Fig. 2c). Since seawater already contains sufficient salt to induce portioning effects, further salting-out was unnecessary, reducing reagent consumption and streamlines the workflow.

The elution solvent, as well as the elution volume, are also a significant parameter to optimise, as they can significantly affect the overall performance of analytes extraction. The initial selection of solvents was based on previous studies (Liu et al., 2011; Poiger et al., 2004). Three different elution conditions and solvent mixtures were tested: 1) MeOH (2 mL, first fraction), DCM/MeOH (50:50; *v*/*v*) (4 mL, second fraction); 2) MeOH (2 mL first fraction), isopropanol (4 mL, second fraction), DCM (4 mL, third fraction); 3) MeOH (2 mL, first fraction), DCM (4 mL, second fraction). For all the experiments, the MeOH extract (first fraction) did not show any detectable peaks, resulting inefficient for the extraction of the target analytes. Regarding the second fraction, DCM/MeOH (50/50, *v*/*v*) yielded low recoveries (between 10 and 30 %), as did isopropanol, which also showed a poor performance. Only DCM provided satisfactory results (data not shown). Hence, the last elution condition (3) 2 mL of MeOH as first fraction followed by 4 mL of DCM as second fraction) was selected. The first MeOH fraction was kept as a clean-up step and subsequently discarded, while the DCM fraction was analysed.

Therefore, the selected SPE conditions for the extraction of UV filters from water samples are described as follows: raw water samples, without pH adjustment or salt addition, were passed through an HLB cartridge (previously conditioned with 6 mL of DCM, 6 mL of MeOH followed by 6 mL of Milli-Q water). The target compounds were then recovered with 4 mL of DCM after a clean-up step of MeOH. This optimised method was further applied to recovery studies and method validation of the target compounds.

3.2. Method validation

To confirm method suitability for the quantitative determination of the twelve target UV filters in water, method performance parameters were assessed. They are summarised in Table 2. Analyses were carried out by GC–MS/MS (see conditions in Section 2). The method was evaluated in terms of linearity, limits of detection (LOD), limits of quantification (LOQ), precision, and recoveries. External calibration standards

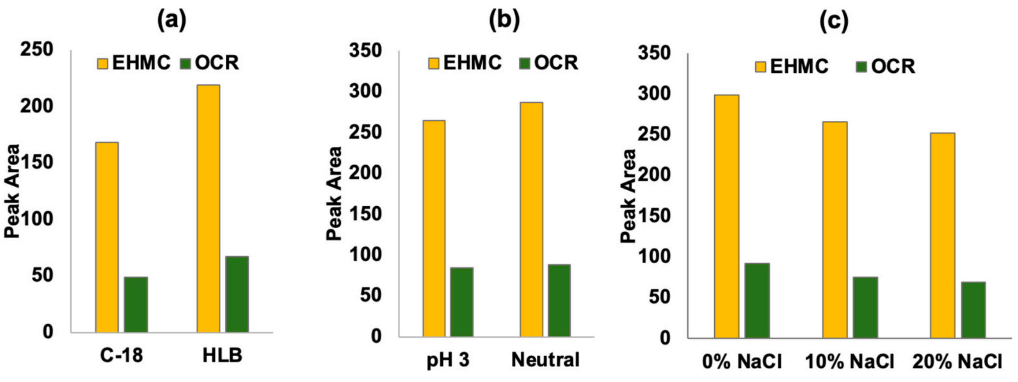

Fig. 2. Effect of different parameters on the extraction of EHMC and OCR: (a) SPE cartridge type, (b) sample pH, and (c) salting-out effect.

Table 2
Performance of the SPE-GC–MS/MS method for the target compounds.

Compounds	Linearity	R^2	Precision (RSD, %)		Recoveries (% ± RSD)		LOD	LOQ
	μg L^{-1}		Intra-day (n = 3)	Inter-day (n = 5)	Seawater	Spring water	ng L^{-1}	ng L^{-1}
EHS	0.2–500	0.9982	2.3	5.5	77.3 ± 5.5	107.0 ± 4.7	0.73	2.4
BS	1–500	0.9989	11	12	87.3 ± 7.3	91 ± 14	0.40	1.3
HMS	1–500	0.9993	2.0	2.5	74.4 ± 2.2	69 ± 13	0.49	1.6
IAMC	1–500	0.9996	1.8	3.2	98.0 ± 6.3	93 ± 11	0.30	1.0
BP-3,4	5–500	0.9942	2.4	11	74.4 ± 6.3	96.0 ± 6.3	3.4	11
4MBC	5–500	0.994	6.5	9.3	87.4 ± 3.3	102 ± 11	1.9	6.2
MA	0.1–500	0.999	1.6	5.9	90.1 ± 2.9	94.1 ± 6.0	0.17	0.56
Eto	0.2–500	0.9998	1.6	4.8	107 ± 12	88 ± 11	0.10	0.33
EHPABA	0.1–500	0.9997	4.5	12	80 ± 13	78.4 ± 5.4	0.14	0.46
E-EHMC	1–500	0.9996	4.7	2.1	95.3 ± 8.8	72.4 ± 8.8	0.94	3.1
OCR	1–500	0.9994	7.0	8.0	104 ± 12	89 ± 12	0.56	1.9

were prepared in DCM at twelve levels, covering from 0.1 μg L^{-1} to 500 μg L^{-1} (see Table 2 for compound specific ranges). Linear relationships between analyte concentration and chromatographic response with determination coefficients $R^2 \geq 0.994$ were confirmed. Instrumental precision, expressed as relative standard deviation (RSD, %), was satisfactory, with intraday ($n = 3$) and interday ($n = 5$) values ranging from 1.8 to 7.0 % and 2.1–12 %, respectively.

Recovery studies were performed at two concentration levels using real water samples (400 mL): spring water and seawater, spiked at 0.02 μg L^{-1} and 0.1 μg L^{-1}, respectively. Recoveries were determined as the ratio of the response obtained from spiked samples to that of standards at the corresponding concentration level and expressed as percentages. For the seawater matrix, a sample collected during the winter season was selected to minimize the likelihood of the background contamination by UV filters. Procedural blanks confirmed the absence of the target compounds, ensuring accurate recovery assessment. Fig. 3 shows satisfactory recovery values for both seawater and spring water, generally exceeding 80 %, with the standard deviation values (± SD) between 2 and 14 %, indicating the absence of significant matrix effect in real samples using the validated methodology. The LODs and LOQs were calculated as the concentration in real sample giving a signal-to-noise ratio of three (S/N = 3) and ten ($S/N = 10$), respectively for each compound. The method demonstrated high sensitivity with LODs below 1 ng L^{-1} for most compounds (see Table 2).

3.3. Application to seawater samples

The validated SPE-GC–MS/MS method was applied to fourteen seawater samples collected during the recreational activities from six beaches along the Adriatic coast. As previously mentioned in Section 2.2, each of the collected fourteen seawater samples was considered independent, even when collected in close proximity in time and space, due to marine currents, particle interactions, and wave motion mixing.

All twelve target UV filters and the transformation product *Z*-EHMC were detected across the sampling sites, confirming their ubiquitous presence in coastal waters. Fig. 4 displays SRM reconstructed SPE-GC–MS/MS chromatograms of a representative seawater sample (Sa4). Table 3 summarises the quantification results for each analyte and the summed concentrations of UV filters for each sample.

Compounds consistently detected in all samples were EHS, BS, HMS, BP-3,4, *Z*-EHMC, *E*-EHMC and OCR. Eto and IAMC were detected in seven and six of the fourteen samples, respectively. This trend reflects the widespread use of the UV filters in personal care products. On the other hand, Eto and MA which are prohibited in EU cosmetics formulations (European Union, 2009), were detected in few samples at low levels (< 3 ng L^{-1}). In fact, MA was detected in one sample (Sa4).

Total UV filter concentrations of all detected UV filters spanned from 0.2 to 10 μg L^{-1}, with the exception of S11 (Table 3). Sample S11 exhibited the highest overall UV filter load (53 μg L^{-1}), with extreme levels of OCR (23,100 ng L^{-1}), HMS (16,400 ng L^{-1}), and EHS (10,900 ng L^{-1}). These extreme values could represent an anecdotal scenario due

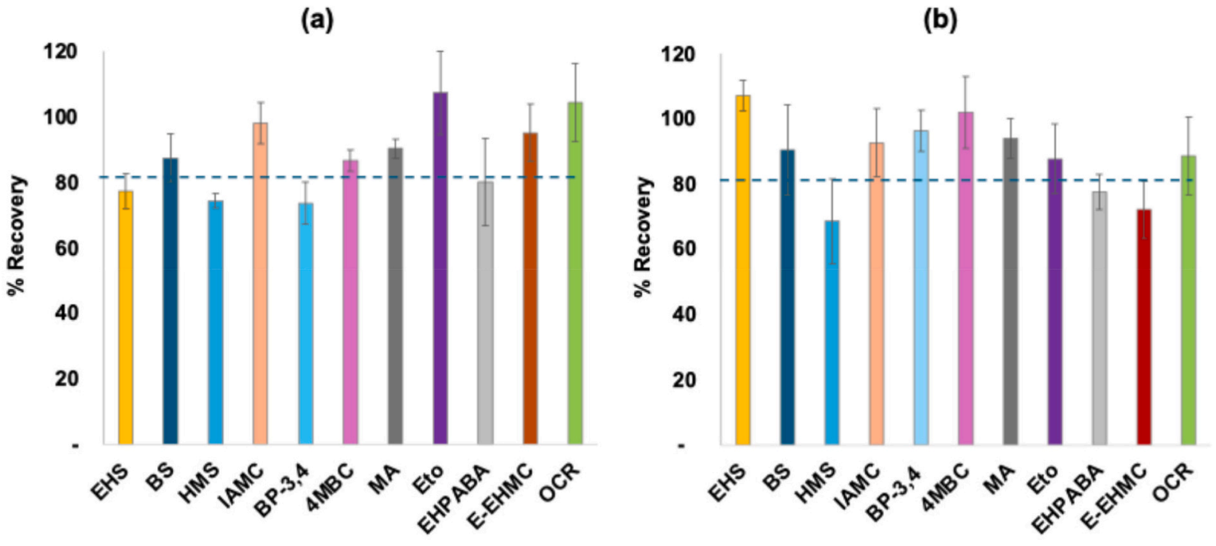

Fig. 3. Recoveries of UV filters in real water samples: (a) seawater, and (b) spring water.

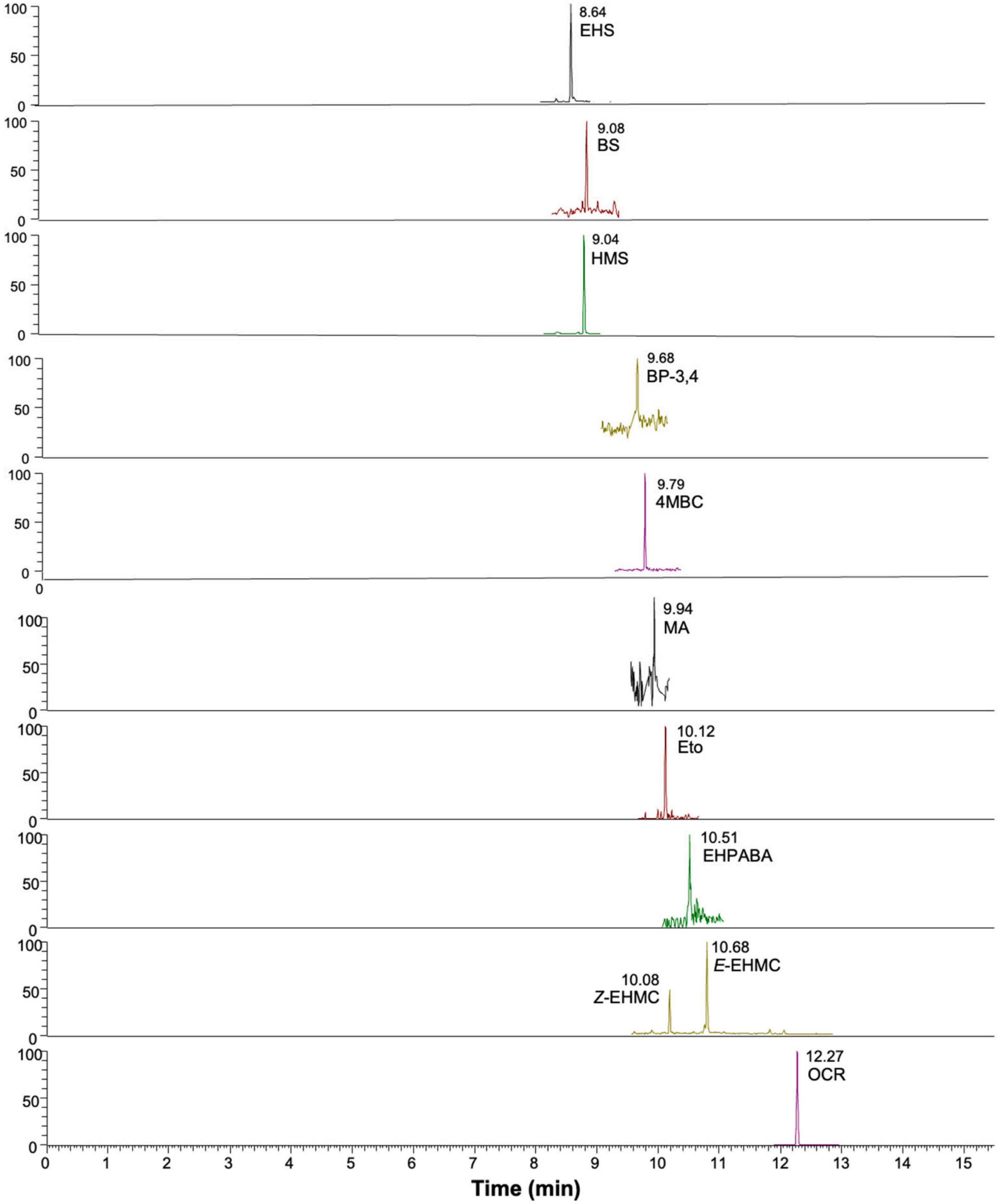

Fig. 4. SRM reconstructed SP*E*-GC–MS/MS chromatograms of a real seawater sample (Sa4, see Table 3).

to concomitant factors such as a relatively high number of bathers (10 people were counted nearby sampling). Samples Sd9 and Sd10 also showed elevated levels of several UV filters, including OCR (7317 ng $L^{-1}$and 6830 ng $L^{-1,}$ respectively) in agreement with other studies on locations nearby important cities (Tsui et al., 2014). In the other 11 samples, the concentrations of all the investigated UV filters fall well below 1000 ng L^{-1} concordantly with literature (Grant et al., 2025). Replicate sampling showed a rather high variability for each single analyte, with relative standard deviation (RSD) lowest for OCR and highest for *E*-EHMC.

The UV filter *E*-EHMC was found in all the analysed samples at significant levels (1–22 % of the total mass of target UV filters) and was the second most abundant UV filter in the samples. Notably, its *cis* isomer (*Z*-EHMC) occurred at similar if not higher (samples Sd9 and Sd10) concentrations in comparison to the *trans* isomer. The comparison with other water systems was challenging as the *cis* isomer was rarely

Table 3

Concentrations (ng L^{-1}) of single UV filters and total concentrations (μg L^{-1}) in seawater samples.

Sample name	EHS	BS	HMS	IAMC	BP-3,4	4MBC	MA	*Z*-EHMC	Eto	EHPABA	*E*-EHMC	OCR	Total
Sa1	159	4.8	150		90	51		47	1.6	0.55	219	727	1.4
Sa2	224	10	187		62	90		50	2.0	0.49	112	556	1.3
Sa3	223	10	204		62	122		43	2.6	*0.29*	45	661	1.4
Sa4	78	5.4	133		66	97	<LOQ	20	2.2	*0.37*	43	712	1.2
Sb5	31	6.1	26		52	12		7.5	0.40		64	101	0.30
Sb6	20	4.0	17		120			4.8		*0.26*	18	96	0.28
Sc7	27	5.6	24		154	54		5.7			19	126	0.42
Sc8	19	4.8	17		172	37		3.7			13	123	0.39
Sd9	319	6.7	2020	4.8	60	26		195	1.2	4.1	157	7317	10
Sd10	228	9.3	1560	2.5	50	25		149		3.2	84	6830	8.9
S11	10,900	16	16,400	223	44			1008		5.4	1474	23,100	53
S12	206	8.2	163	29	44	17		73	1.3	3.8	74	406	1.0
S13	103	7.1	81	15	54			52			224	491	1.0
S14	16	3.0	18	2.5	66			21			39	74	0.24

*In sample name, the same lower-case letter indicates similar sampling location and time.

**Empty cells indicate UV filters not detected.

Values in italics are estimated, below LOQ.

analysed in environmental samples (Poiger et al., 2004), or it was analysed as a sum of isomers (Langford and Thomas, 2008; Tashiro and Kameda, 2013). Plagellat et al. (Plagellat et al., 2006) analysed both *E*- and *Z*-EHMC in sewage sludge from different wastewater treatment plants in Switzerland, but the concentrations of the single isomers were not reported.

OCR exhibited the highest concentrations in all the investigated samples (74–23,100 ng L^{-1}) is coherent with the study by Combi et al. found, that OCR was the most abundant UV filter in the sediments of Adriatic Sea with highest concentrations in the northern sector (16.3 ± 9.6 ng g^{-1}) (Combi et al., 2016). Among the other two UV filters investigated by the authors, EHMC was more abundant than BP-3, similarly to what observed in samples where (*E* + *Z*)-EHMC > BP-3,4. The concentrations of *E*-EHMC in seawater from various locations exhibited large variability, generally in the <1–100 ng L^{-1} range in accordance with our data (13–224 ng L^{-1}) with the exception of sample 11, up to 1474 ng L^{-1}. When considering the Adriatic basin, the concentrations of EHMC in the Venice lagoon were reported to fall in the 0.23–22.4 ng L^{-1} range with the highest concentrations observed in summer and attributed to effluents from wastewater treatment plants (Pizzini et al., 2024). This range was considered among the lowest in natural waters worldwide where the upper limit exceeded 1000 ng L^{-1} (Pizzini et al., 2024) underlining the importance of direct introduction from recreational activities (e.g. swimming, bathing). Benzophenones (BP-3,4) were detected at concentrations between 44 and 172 ng L^{-1} in all the samples, on average similar to the levels observed in bathing areas of the Western Mediterranean Sea (Gandar et al., 2025) and lower than the levels reported in the tourist resorts in the Black Sea (Chiriac et al., 2021). The detection of *Z*-EHMC in notable concentration is of particular concern, emphasizing the ecological relevance of monitoring these compounds in marine ecosystems.

3.4. *Environmental implications*

3.4.1. *Potential risk*

The above findings supported the widespread presence and varied distribution of UV filters in coastal environments, likely influenced by intense seasonal recreational activities and coherent with the fact that in cosmetic formulations different UV filter are mixed to obtain a broad-spectrum coverage against the diverse UV radiation. Although specific environmental risk assessment is not compulsory for cosmetic products, some UV filters used in sunscreens are being evaluated as important environmental stressors, for example BP3, IAMC, 4MBC as endocrine disrupters and OCR as persistent, bioaccumulative and toxic substance (Kroll et al., 2025). Thus, the impact associated with the levels observed in this study is difficult to assess given the absence of official values for hazard characterisation, such as predicted no-effect concentration (PNEC), or environmental quality standards (EQS). Estimated values of PNEC from a limited set of data proposed by Fent et al., 2010 (BP3 6000 ng L^{-1}, 4MBC and EHMC 290–9900 ng L^{-1}) and Cadena-Aizaga et al., 2020 (OCR 400–800 ng L^{-1}, 4MBC 10–87,800 ng L^{-1}, BP3 600–285,000 ng L^{-1}), 4MBC and EHMC 290–9900 ng L^{-1}) were well above the concentrations observed in this study with the possible exception of OCR. Most recent values of PNEC for marine waters based on publicly available data reported by Pawlowski et al., 2025 (BP3 67 ng L^{-1}, EHMC 300 ng L^{-1}, EHS 82 ng L^{-1}, HMS 49 ng L^{-1}, OCR 27 ng L^{-1}) would indicate potentials risk for EHS, HMS and OCR. A detailed study on freshwaters organisms highlighted the lack of data for reliable environmental risk assessments for UV filters, nevertheless some EQS values were proposed based on EU technical guide (Kroll et al., 2025). A conservative comparison with our concentrations could be attempted by considering that saltwater organisms should be less sensitive than freshwater ones. Our measured concentrations resulted lower than EQS for all the samples in case of BP3 and most of the samples for 4MBC, apparently not critical for EHMC, while all the samples presented values higher than EQS for OCR (EQS chronic: BP3 500 ng L^{-1}, EHMC <900 ng L^{-1}, 4MBC 85 ng L^{-1}, OCR 53 ng L^{-1}; Kroll et al., 2025). In addition OCR and 4MBC are poorly biodegradable, when EHS, EHMC and IAMC are readily and HMS is inherently biodegradable (Kroll et al., 2025, Pawlowsky), it comes that OCR. In conclusion, OCR appears to be the most critical UV filter in the investigated area.

However, the levels of stressors emerged from this study should belong to a worst-case scenario for a number of reasons. First of all, the concentrations of UV filters were expected to be the highest as samples were collected during high frequency of bathers in terms of daytime and season (see the tourist presence in Table S1). Several studies showed that higher concentrations of UV filters were associated with intense recreational activities (touristic season, number of beachgoers) confirming the dominance of direct release from leave-in cosmetics (Picot-Groz et al., 2018; Labille et al., 2020; Cadena-Aizaga et al., 2022). Actually, concentrations should be averaged from a temporal sequence of data monitoring for a more adequate risk assessment to take into account diurnal and seasonal variability. Concentrations of UV filters exhibited low values at night or early morning (Sankoda et al., 2015; Picot-Groz et al., 2018).

Besides, our study measured total concentrations of UV filters (dissolved and particulate similarly with Picot-Groz et al., 2018) as water samples were not filtered prior to analysis, thus they did not reflect the fraction available to living organisms (Pawlowski et al., 2025). Thus, any conclusion about risk should be considered premature. Moreover, stereoisomerism, as in the case of EHMC, has not been taken into account even though it may affect environmental and ecotoxicological properties (Kroll et al., 2025).

3.4.2. On the origin of the Z-EHMC in seawater samples

To determine whether the *Z*-EHMC detected in environmental seawater samples originates directly from cosmetic formulations during the manufacture or storage, or is instead formed through environmental transformation, a targeted analysis was conducted on commercial products. Cosmetic products explicitly labelled as containing EHMC were selected for this investigation. As mentioned, the isomer used on the cosmetic formulations is the *trans* (*E*-EHMC). The selected products included quite diverse formulations as hair oil, lip balm and sunscreen, widely used by the general population and in recreational coastal settings.

Given the typically high concentrations of EHMC in such formulations required to act as a UV filter sometimes reaching the percentage, a small amount of sample (5–8 mg) was weighted and homogenised in 10 mL of solvent using vortex and ultrasound agitation. Then an aliquot was diluted and finally analysed by GC–MS. Importantly, in all cases, only the *E*-EHMC was detected, with no observable presence of the *Z*-EHMC, indicating that *Z*-EHMC is not inherently present in the original cosmetic formulations.

The *trans* (*E*-) to *cis* (*Z*-) photoisomerisation of EHMC is a well-known process that can occur very rapidly under light exposure (Huong et al., 2007; Pattanaargson et al., 2004). In fact, as commented in Section 2, a solution containing *E*-EHMC was irradiated with the intention of obtaining the *cis* isomer. After 10 min the solvent was analysed and both isomers were present in the solution at quite similar quantity (see Fig. S1 showing the chromatogram and the two mass spectra).

The percentage of *Z*-EHMC to ($E+Z$)-EHMC in the investigated real seawater samples from the Adriatic Sea (see Table 3 and Fig. S3) occurred in a range between 18 and 64 % (34 % on average) underpinning the importance of this isomer. At the best of our knowledge, the percentage of the *cis* isomer in samples of environmental interest was reported only for sewage sludge where on average accounted for 40 % of total EHMC (Plagellat et al., 2006).

These findings suggest that the *Z*-EHMC detected in seawater samples is not released from the original cosmetics, but it is formed probably due to environmental conditions following exposure of *E*-EHMC. As photoinduced *E*-EHMC isomerisation occurs readily in different media (Huong et al., 2007), several sources of *Z*-EHMC in seawater can be considered: direct photoconversion of *E*-EHMC in situ in the water, wash-off from sun exposed skin after sunscreen application, and contributions from wastewater already containing the *cis* isomer.

Table S1 summarizes the environmental conditions during sampling, which did not exhibit clear trends with *E*/*Z*-EHMC ratios, except for radiance. As shown in Fig. S3, samples 9d and 10d showed the highest Z abundance (56 and 60 %) under the highest radiance (sunny), whereas samples 5–8 displayed lower Z proportions (10–23 %) under lower radiance (cloudy). However, the opposite trend was also observed (e.g. samples 1a and 12). Previous studies suggest that Z/E ratios are more influenced by the polarity of the environment and EHMC concentration than by sunlight power (Pattanaargson et al., 2004; Hanson et al., 2015; Herzog et al., 2019). Therefore, the polarity of the microenvironment (sunscreen, skin, water, natural particles), EHMC concentration, and the kinetics of photoisomerization and reverse (dark) reactions should be further investigated to elucidate factors driving EHMC stereoisomerism under real world conditions. Elucidating these formation pathways is critical for accurately assessing the environmental impact of EHMC on seawater quality.

4. Conclusions

A single SPE based sample preparation method coupled with GC–MS/MS was developed and successfully applied for the analysis of twelve UV filters and the transformation product *Z*-EHMC in seawater samples. The method demonstrated excellent analytical performance with the detection limits in the ng L^{-1} range and recovery efficiencies exceeding 80 %, making it suitable for environmental monitoring of these emerging contaminants in marine ecosystems.

The widespread occurrence of UV filters was confirmed across fourteen seawater samples from popular Adriatic beaches, with the target compounds detected at varying concentrations. High concentrations of certain UV filters were observed, with OCR exhibiting critical values, however most of the samples exhibited concentrations lower than 1 μg L^{-1} and probably within a safe zone even though the absence of official quality standard did not allow to estimate the potential environmental risk. A remarkable finding was the ubiquitous presence of *Z*-EHMC in all samples, despite its absence in commercial formulations, confirming significant in-situ photoisomerisation of the parent *E*-EHMC under natural solar radiation. The environmental transformation process generates a potentially more toxic isomer that current regulatory frameworks fail to address.

These findings provide critical insight into the environmental fate of UV filters and highlight the need for further monitoring and risk assessment, especially considering the increasing use of these compounds in personal care products. This research also contributes to the growing body of knowledge regarding contaminants of emerging concern in aquatic ecosystems, calling for improved regulatory measures and more stringent monitoring to mitigate the potential ecological risks associated with UV filters.

Glossary

ACN	Acetonitrile
AVO	Avobenzone
BP-3	Benzophenone-3
BP-4	Benzophenone-4
BS	Benzyl salicylate
DCM	Dichloromethane
EHMC	2-ethylhexyl methoxycinnamate
EHPABA	Ethylhexyl dimethyl paba
EHS	Ethylhexyl salicylate
EI	Electron impact
EtAc	Ethyl acetate
Eto	Etocrylene
GC	Gas chromatography
HMS	Homosalate
HPLC-DAD	High-performance liquid chromatography-diode array detector
IAMC	Isoamyl methoxycinnamate
LOD	Limit of detection
LOQ	Limit of quantification
MA	Menthyl anthranilate
4-MBC	4-methylbenzylidene
MeOH	Methanol
MS	Mass spectrometry
MS/MS	Tandem mass spectrometry
OCR	Octocrylene
SCCS	Scientific committee on consumer safety
SPE	Solid-phase extraction
SRM	Selected reaction monitoring
UV	Ultraviolet

CRediT authorship contribution statement

Hira Zulfiqar: Writing – review & editing, Writing – original draft, Visualization, Validation, Investigation, Formal analysis, Data curation. **Maria Llompart:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Ana Castiñeira-Landeira:** Writing – review & editing, Visualization, Investigation. **Irene Coralli:** Writing – review & editing, Investigation. **Ivano Vassura:** Writing – review & editing, Investigation.

Daniele Fabbri: Writing – review & editing, Supervision, Resources, Methodology, Conceptualization.

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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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

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