



Commercial screen-printed carbon electrodes for real-time and in situ monitoring of organic UV filters: Application to study their adsorption onto polyethylene microplastics

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ABSTRACT

This work demonstrates the use of commercial screen-printed carbon electrodes (SPCEs) coupled with differential pulse adsorptive stripping voltammetry (DPAdSV) for the *in-situ* and continuous monitoring, over 24 – 48 h, of the concentration of the organic UV filters benzophenone-3 (BP-3) and octocrylene (OC) as they adsorb onto polyethylene (PE) pellets in stirred aqueous solutions. The approach enables a large set of real-time measurements with minimal disturbance of the solution, providing a valuable complement, with higher temporal resolution, to conventional off-line kinetic studies of UV-filter adsorption onto microplastics, typically performed by HPLC-MS/MS. Moreover, the high reproducibility and durability of SPCE units under stirred conditions highlight their potential for the continuous monitoring of target pollutants in real environmental matrices, especially when integrated into automated systems involving periodic sensor replacement and recalibration.

1. Introduction

In recent years, the impact of emerging organic contaminants and other residues on ecosystems has become an increasingly significant environmental concern, attracting both scientific and regulatory attention. Among the compounds commonly detected in personal care and cosmetic products are organic filters, which, while protecting skin and materials from ultraviolet (UV) radiation, also raise concerns regarding their potential risks to human health and aquatic ecosystems. Benzophenone-3 (BP-3), also known as oxybenzone, and octocrylene (OC) (Fig. 1) are two representative members of this family. Both have been detected in marine organisms and have been associated with toxicological effects in aquatic species such as dolphins [1] and various bird species [2], as well as marine plants such as *Posidonia oceanica* [3]. These findings have led certain countries, such as Palau [4], to ban the use of sunscreens containing specific UV filters, including BP-3 and OC, in order to protect their natural ecosystems.

BP-3 and OC have been widely used in sunscreens due to their

excellent UV-absorption properties. However, several studies demonstrate that these compounds may negatively affect ecosystems by acting as endocrine disruptors, altering hormonal regulation in various species. Additionally, their bioaccumulation and biomagnification across food webs have been reported, impacting organisms ranging from mollusks to humans [5–7]. Furthermore, OC contained in sunscreens can degrade over time through a retro-aldol condensation, generating benzophenone [8], the simplest member of the benzophenone family and classified as a possible human carcinogen by the International Agency for Research on Cancer (IARC). These findings highlight the need for analytical strategies to monitor these contaminants and understanding their environmental behavior, as well as developing solutions or alternatives to the use of these UV filters.

In parallel, numerous publications have documented the widespread presence of microplastics (MPs) in lakes, rivers, oceans, and even indoor environments [9–11]. Polyethylene (PE) (Fig. 1) is among the most abundant polymers found in these systems and is not completely removed by wastewater treatment plants [12,13]. MPs not only exert

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direct negative effects on organisms and ecosystems, but also act as potential adsorption surfaces, thus becoming carriers for chemical contaminants [14,15]. Moreover, plastic materials often contain UV stabilizers and related additives to reduce photodegradation. Once fragmented into MPs, these particles can be ingested by aquatic organisms, causing adverse effects such as oxidative stress in mollusks [16]. Consequently, MPs pose a significant threat to aquatic species and reinforce the need to address this source of pollution.

Recently, exposure experiments involving water fleas (*Daphnia magna*) and PE microplastic fragments with and without BP-3 have shown a significant decrease in population and size, particularly when BP-3 was present. Additionally, leaching of BP-3 adsorbed onto PE has considerably affected reproduction and development, suggesting that BP-3 can alter the digestive system of this aquatic species [17,18]. Although some studies have investigated the adsorption of UV filters onto MPs [16,19], important limitations remain in the analytical methodologies available for continuously monitoring these processes *in situ* and under environmentally relevant conditions. While highly sensitive and selective, conventional approaches provide only discrete time-point measurements, may alter the adsorption system, and are not well suited for automated kinetic studies.

Research on UV filters using electroanalytical techniques began with polarographic analyses in micellar media [20] and later in aqueous solutions using hanging mercury drop electrodes [21,22]. These electrodes offer a wide cathodic range and high reproducibility but are toxic and cannot be modified. Subsequent studies employed solid glassy carbon electrodes (GCE) [23], which provide intermediate detection limits but require very frequent cleaning and polishing and often suffer from reproducibility issues, thus being unsuitable for studies requiring real-time monitoring.

To address these challenges, screen-printed electrodes (SPEs) were proposed for BP-3 and OC analysis [24]. SPEs consist of a three electrode system (working, auxiliary, and reference) printed with conductive inks onto an insulating solid substrate, typically ceramic. Their advantages over conventional electrodes include low cost, disposability, non-toxicity, and no need for pretreatment or polishing nor deaeration, making them ideal as portable sensors in healthcare and environmental applications [25–28]. Alternative electrochemical strategies have also been explored for the determination of BP-3 and OC. For instance, spectroelectrochemical methods based on Pt grid working electrode have also been applied to BP-3 and OC analysis in sunscreens [29]. However, these methods rely on extensive electrochemical oxidation of these substances, i.e. a destructive and time-consuming strategy unable for continuous monitoring.

Regarding *in situ* measurements, a portable potentiostat coupled to a GCE was used to determine OC and BP-3 in cosmetic products with high sensitivity, achieving detection and quantification limits (LODs and LOQs) lower than previously reported [30]. However, this configuration

requires a frequent electrode polishing, which restricts its suitability for continuous monitoring. Carbon paste electrodes modified with a pyridinium composite have also been employed for BP-3 determination in pool water by square-wave voltammetry with good sensitivity [31]. Additionally, molecularly imprinted polymers generated on screen-printed gold electrodes have been successfully applied to OC and BP-3 determination in aquatic ecosystems [32]. However, the requirement for electrode modification reduces commercial availability and reproducibility.

Therefore, despite the progress achieved to date, a simple, commercially available, non-modified electrochemical platform-based methodology suitable for *in situ* and continuous monitoring of organic UV filters in aquatic environments remains lacking. Thus, in this work, a simple, cost-effective and sustainable methodology based on commercial screen-printed carbon electrodes (SPCE) is proposed for the *in situ* and continuous monitoring of organic UV filters in marine ecosystems. The same SPCE remains immersed throughout each experiment, thereby avoiding signal discontinuities associated with electrode replacement, while a new SPCE is used for each independent experiment to minimize memory effects and eliminate the surface regeneration procedures required for conventional solid electrodes. Furthermore, the integrated three-electrode configuration and standardized geometry of SPCEs facilitate reproducible positioning and make them well suited for automated and long-term monitoring applications. Considering the role of MPs as vectors for UV filters in marine environments, the proposed methodology is applied to monitor the time-dependent concentration of BP-3 and OC during their adsorption onto PE pellets, thus providing insight into the adsorption kinetics.

2. Experimental

2.1. Reagents

Unless otherwise indicated, the water used for the preparation of all solutions was obtained from a Milli-Q Reference A + Water Purification System by Millipore (Burlington, MA, USA).

Benzophenone-3 (BP-3, 98% purity) was purchased from Thermo Scientific (Waltham, MA, USA), and octocrylene (OC, 97% purity) and cetyltrimethylammonium bromide (CTAB, >99% purity) were obtained from Sigma-Aldrich (St. Louis, MO, USA).

Artificial seawater was prepared using HPLC-MS grade LiChrosolv® water (Merck KGaA, Darmstadt, Germany) and the reagent “Sea-Salt ASTM D 1141–98” (Lake Products Company LLC), a mixture of high-purity salts formulated to reproduce the ionic composition of natural seawater. The detailed composition of the salt mixture is provided in Table 1.

Stock solutions of BP-3 and OC (prepared in ethanol), and synthetic seawater, were stored at 4 °C in amber glass bottles protected from light.

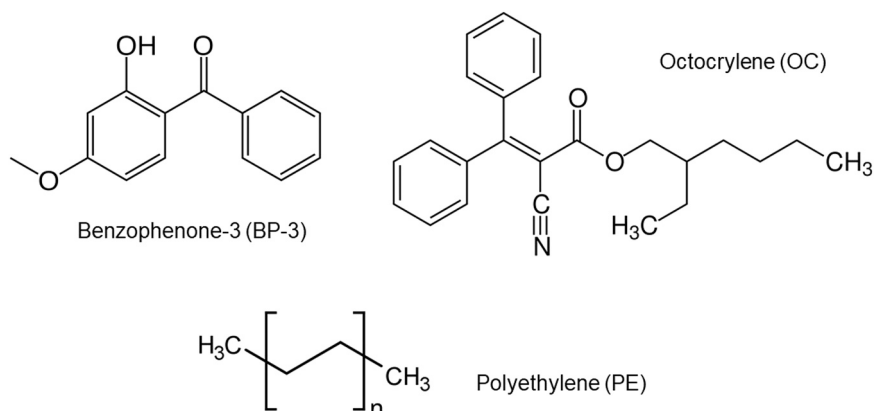


Fig. 1. Chemical structures of the compounds considered.

Table 1

Composition of the synthetic seawater “Sea-Salt ASTM D 1141–98” employed. Its density is 1.025 g/mL at 15 °C. The percentage of each substance is expressed on a mass basis. Data were obtained from the product label.

Substance	Concentration	Substance	Concentration
NaCl	58.490%	NaHCO ₃	0.477%
MgCl ₂ · 6 H ₂ O	26.460%	KBr	0.238%
Na ₂ SO ₄	9.750%	H ₃ BO ₃	0.071%
CaCl ₂	2.765%	SrCl ₂ · 6 H ₂ O	0.095%
KCl	1.645%	NaF	0.007%

All volumetric additions were carried out using Nichiryo micropipettes or class-A volumetric pipettes.

Low-density PE pellets were obtained from Sigma-Aldrich. Previous material-characterization studies [33] determined that the pellets are round and nearly spherical, with a diameter of 4.2 ± 0.3 mm. Scanning electron microscopy (SEM) revealed the presence of both smooth and rough surface regions, as well as cracks and fissures generated during the pellet manufacturing process.

2.2. Voltammetric determination of UV filters

All voltammetric experiments were performed using a μ Autolab Type III potentiostat connected to a 663 VA Stand (Autolab-Metrohm, Herisau, Switzerland). SPCEs (Dropsens-Metrohm, ref. DRP-C110) were connected to the potentiostat by means of a DRP-CAC cable (Dropsens-Metrohm). The system was controlled using the General Purpose Electrochemical System (GPES) software, version 4.9 (Autolab-Metrohm).

Differential pulse adsorptive stripping voltammetry (DPAdSV) measurements were carried out using the instrumental parameters previously optimized [24,33]. The procedure consisted of applying a deposition potential of -0.6 V for 80 s under stirring to promote the adsorption of the UV filter onto the SPCE surface. The accumulated analyte was subsequently electrochemically reduced during a differential pulse scan from -0.5 to -1.6 V, performed without stirring at a sweep rate of 15 mV/s. During the scan, a pulse amplitude of 50 mV and a pulse time of 50 ms were applied. In addition, a conditioning potential of -0.2 V was applied for 60 s between measurements, in order to remove electrochemically reduced species adsorbed onto the electrode surface, and to improve signal recovery before the subsequent measurement. In the present study, DPAdSV was selected not only because of its analytical sensitivity, but also because it provided well-defined signals suitable for repeated and automated measurements throughout the adsorption experiments.

Following a common practice derived from the pioneering works of Wang et al. [34], solutions were analyzed without prior nitrogen deaeration. Unlike mercury electrodes, SPCE are not significantly affected by dissolved oxygen, and previous studies have shown that BP-3 and OC remain stable under the experimental conditions used. Consequently, the removal of dissolved oxygen was considered unnecessary.

pH measurements were carried out using a Crison basic 20 pH meter (Barcelona, Spain).

2.3. Voltammetric sensing in adsorption experiments

Continuous DPAdSV measurements of BP-3 or OC concentrations in the presence of the PE pellets were carried out in a 100 mL beaker, thus reproducing the same experimental conditions as in our previous study [33]. In each experiment, 25 mL of the saline medium containing a known concentration of BP-3 or OC and 100 mg/L of CTAB (to prevent precipitation of BP-3 and, especially, OC) were placed in the beaker. CTAB concentration was kept constant in both calibration and adsorption experiments to ensure comparable voltammetric responses and adequate UV filter solubility during the study. Subsequently, the SPCE device and the stirring bar of the electrode stand were immersed in the

solutions. The beaker was covered with aluminum foil to minimize light-induced degradation of the UV filters and PE pellets. A magnetic stirrer placed beneath the beaker provided continuous agitation by means of a magnetic core. (Fig. 2).

Before adsorption experiments, a new SPCE was calibrated in the same beaker and medium through five successive additions of BP-3 or OC to the saline medium containing CTAB. The fifth addition corresponded to the initial concentration selected for the adsorption study. Three replicate measurements of the initial solution were then performed in the absence of PE pellets, to accurately determine the initial concentration with the SPCE. Finally, seven PE pellets (0.19–0.21 g total mass) were introduced into the beaker. Assuming a spherical geometry (without accounting for the surface roughness, cracks and fissures) and a mean pellet diameter of 4.2 mm, the seven PE pellets provided an estimated geometric surface area of approximately 3.9 cm^2 , corresponding to a surface-area-to-solution-volume ratio of approximately $0.155 \text{ cm}^2 \text{ mL}^{-1}$. The adsorption experiment was considered to start after the pellets addition, and periodic measurements were subsequently initiated, with the electrode permanently immersed in the solution. During most of the experiment, stirring was maintained at 350 rpm using the magnetic horizontal stirring system moved by the magnetic stirrer beneath the beaker to homogenize the solution and maximize contact between the UV filters and the PE pellets. This continuous agitation was briefly stopped during each DPAdSV measurement cycle, which was performed in triplicate. During these measurements, only the vertical rotating stirring bar integrated into the stand operated, providing agitation during the deposition step of DPAdSV and stopping during the stripping step. At the end of the third measurement, stirring was resumed at 350 rpm with the magnetic stirrer below. Of the three measurements carried out, the third one was selected for quantification, considered as optimal, since the first two scans contributed to equilibrating the electrode after many minutes in contact with the solution (likely with adsorption of the analyte and other substances onto the electrode surface). As the adsorption experiment progressed, the height (or the area) of the DPAdSV peak indicates the evolution of the BP-3 or OC concentration over time, which decreased as adsorption onto the PE pellets occurred. It is important to note that the presence of the SPCE, PE pellets and stirring elements may modify the local hydrodynamic conditions. However, their position and the stirring rate were kept constant throughout all the experiments. Each voltammetric measurement cycle also required approximately 11 min, during which the horizontal stirring was temporarily interrupted. Although adsorption continued during this period, the same measurement sequence was consistently applied to all experiments, minimizing systematic differences among kinetic profiles.

As explained above, two complementary agitation systems were combined in these experiments. On the one hand, the long-term magnetic stirrer homogenized the solution and maximized UV filters contact with the PE. On the other hand, the rotating stirring bar incorporated into the electrode stand provided agitation during the 80 s in which the deposition potential was applied, thus improving the analyte transport from the solution towards the working electrode surface. Given the long duration of adsorption experiments (usually extended overnight), the measurement sequence was automated. For this purpose, the magnetic stirrer was connected to a Ketotek KTTS01 programmable socket, allowing magnetic stirring to be switched on and off in coordination with the voltammetric sequence (Fig. 3). Furthermore, a series of executable commands was developed in the GPES Project tool to perform the DPAdSV measurements automatically.

2.4. Data processing

The results obtained from voltammetric measurements can be fitted to different kinetic models that describe the rate of the adsorption process of UV filters onto PE. These models have previously shown excellent performance in adsorption studies involving other contaminants and

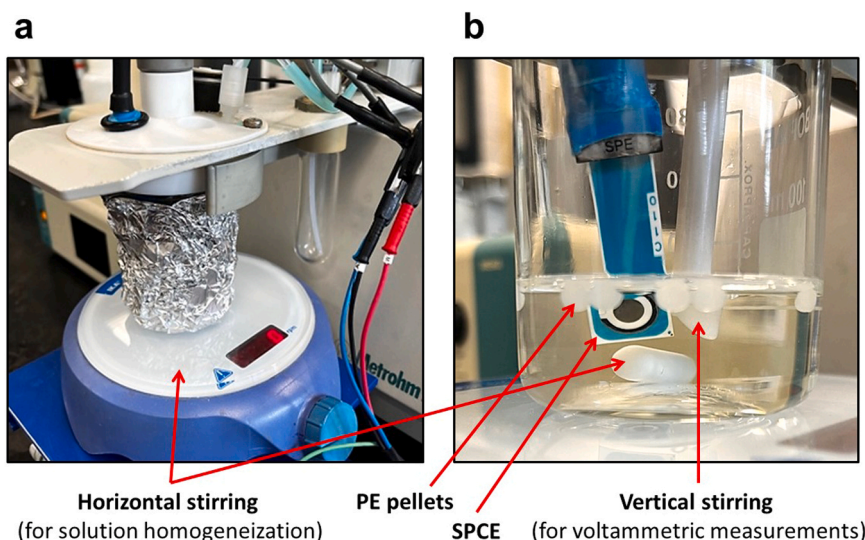


Fig. 2. Setup for experiments of BP-3 or OC adsorption onto PE pellets with voltammetric monitoring using SPCE. The setup is shown with (a) and without (b) the aluminum foil.



Fig. 3. Time-sequence of the automated adsorption experiments.

MPs of different compositions and polymeric nature [35–40]. The equations for the pseudo-first order and pseudo-second order kinetics are those described below.

Pseudo-first order kinetics:

$$Q_t = Q_e (1 - e^{-k_1 t}) \quad (1)$$

where Q_t ($\text{mg} \cdot \text{g}^{-1}$) is the concentration of BP-3 or OC adsorbed onto PE at time t (h), Q_e ($\text{mg} \cdot \text{g}^{-1}$) is the concentration adsorbed once the equilibrium is achieved, and k_1 (h^{-1}) is the rate constant of the adsorption process.

Q_t is computed from the initial concentration C_0 ($\text{mg} \cdot \text{L}^{-1}$) of the UV filter at $t = 0$, the concentration C_t at time t ($\text{mg} \cdot \text{L}^{-1}$), the volume V (L) of the measuring solution, and the mass m (g) of the seven PE pellets, according to:

$$Q_t = V (C_0 - C_t) / m \quad (2)$$

Pseudo-second order kinetics:

$$Q_t = k_2 Q_e^2 t / (1 + k_2 Q_e t) \quad (3)$$

with Q_t , Q_e , t as before, and k_2 ($\text{g} / (\text{mg} \cdot \text{h})$) as the rate constant of the adsorption process.

These equations can be linearized to fit the sets of Q_t vs. t data in the pseudo-first order and pseudo-second order cases, respectively:

$$\ln(Q_e - Q_t) = \ln(Q_e) - k_1 t \quad (4)$$

$$t/Q_t = t/Q_e + 1/k_2 Q_e^2 \quad (5)$$

In Eq. (4), the Q_e value on the left side can be initially approached from the limiting value of Q_t as a function of t and later iteratively computed using the Solver function in Excel.

3. Results and discussion

3.1. Analytical performance of SPCEs for BP-3 and OC determination in synthetic seawater

Synthetic seawater was employed to simulate *in situ* and real-time measurement of UV filters in marine environments. In this medium, the presence of bicarbonate ensured a stable pH around 8.2, suitable for BP-3 and OC determination. In addition, CTAB prevented precipitation of the UV filters, while the high ionic strength provided the adequate conductivity for voltammetric measurements. Preliminary experiments in this medium also demonstrated the suitability of the built-in silver pseudo-reference electrode integrated into the SPCEs, as no improvement in signal quality was observed when using an external Ag/AgCl reference electrode.

Under these conditions, the analytical performance of the voltammetric determination of BP-3 and OC was evaluated. Figs. 4a and 4b show representative voltammograms obtained for OC and BP-3, respectively, over the concentration range $0.3 - 33 \text{ mg L}^{-1}$. The corresponding peak heights and areas increased linearly with concentration up to approximately 15 mg L^{-1} . At higher concentrations, signal

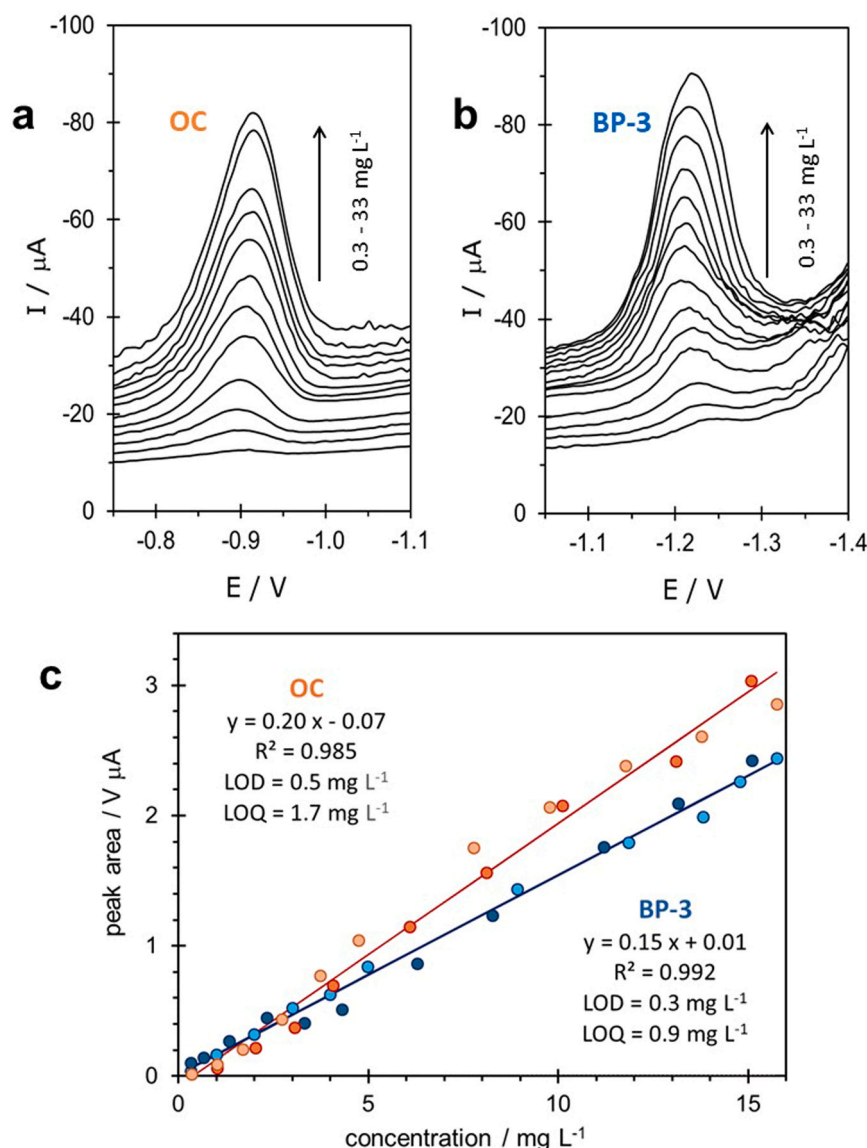


Fig. 4. DPAdSV study with SPCEs for both UV filters in synthetic seawater and in the absence of PE pellets, showing the evolution of voltammetric signals of OC (a) and BP-3 (b) at increasing concentrations from 0.3 to 33 mg L^{-1} . Fig. (c) shows the corresponding calibration lines computed from peak areas for each substance in two replicate experiments within the linear range.

increase becomes less pronounced, suggesting partial saturation of the electrode surface. Within the linear range, peak areas provided better analytical performance than peak heights. Then, such areas were used for quantification in further experiments. Fig. 4c shows calibration lines obtained from two replicate experiments for both BP-3 and OC, as well as the LODs and LOQs, computed as 3 and 10-fold, respectively, the standard deviation of the intercept over the slope of the calibration line. The resulting LOD and LOQ values were 0.3 and 0.9 mg L^{-1} for BP-3, and 0.5 and 1.7 mg L^{-1} for OC, respectively. Peaks remained visible at concentrations corresponding to the LOD. Based on these figures of merit, further adsorption experiments were restricted to the 2 – 15 mg L^{-1} concentration range (i.e., between quantification and linearity limits). The analytical performance of the method is summarized in Table 2.

3.2. Long-term stability and automated monitoring

A key requirement for adsorption-kinetic studies is the long-term stability of the SPCE response. Therefore, a control experiment was performed to evaluate electrode repeatability and durability by

Table 2

Analytical performance of the DPAdSV method for BP-3 and OC determination using commercial SPCEs in synthetic seawater. Standard deviations are denoted by parenthesis.

Analyte	Linear range (mg L^{-1})	Sensitivity ($\mu\text{A V mg}^{-1} \text{L}$)	R^2	LOD (mg L^{-1})	LOQ (mg L^{-1})	Inter-calibration RSD (%)
BP-3	0.9 – 15.7	0.153 (0.003)	0.992	0.3	0.9	3.4
OC	1.7 – 15.7	0.202 (0.006)	0.985	0.5	1.7	5.1

performing continuous voltammetric measurements of a fixed UV filter concentration over time in synthetic seawater and under the stirring conditions required for adsorption-kinetic studies. Initially, an increase in the signal over time was observed, suggesting that keeping the electrode immersed between measurements under hydrodynamic conditions enhanced the transport of UV-filter toward the electrode surface, leading

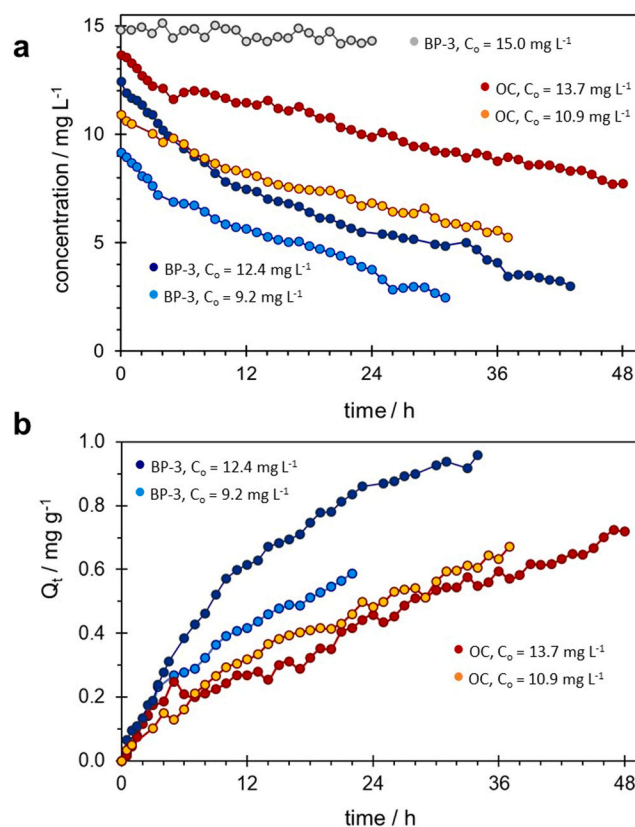


Fig. 5. Adsorption experiments of BP-3 and OC onto PE pellets carried out by DPAdSV in synthetic seawater at pH 8.2. (a) Concentration remaining in solution as a function of time for BP-3 at initial concentrations of 12.4 (●) and 9.2 (●) mg L^{-1} , and OC at initial concentrations of 13.7 (●) and 10.9 (●) mg L^{-1} . The control experiment performed with 15 mg L^{-1} BP-3 in the absence of PE pellets is also included (●). (b) Corresponding Q_t values computed using Eq. 2. Each experiment was carried out with a single SPCE immersed into the stirred solution (Fig. 2).

to increased accumulation at the electrode interface. To obtain a stable and reproducible response, a conditioning protocol was introduced before data acquisition. This protocol consisted of the application of -0.2 V for 60 s immediately before each voltammetric scan, followed by the recording of three consecutive measurements. The first two measurements were systematically discarded, as both were related to a transient stabilization regime, and only the third measurement was used for analytical purposes. Following this protocol, good stability was observed over 25 analytical measurements carried out during a 24 h period in stirred synthetic seawater solution containing ca. 15 mg L^{-1} of the UV filter, with the signal remaining practically constant throughout the experiment and retaining 97% of its initial value after 24 h. This high stability suggests that SPCE units are suitable for continuous monitoring of organic UV filters over extended periods and under hydrodynamic conditions, and are thus suitable for adsorption-kinetic studies within the investigated time window. Furthermore, although this protocol increases the time required for each analytical determination, it still allows a high number of measurements to be obtained during an adsorption kinetic study, considerably higher than that typically achieved in off-line approaches.

3.3. Adsorption profiles of BP-3 and OC onto PE pellets

Fig. 5 shows the results of several adsorption experiments performed with a single SPCE at different initial concentrations of BP-3 or OC in the presence of PE pellets, and compares them with a series of repeated measurements of BP-3 over 24 h in the absence of pellets. Under these conditions the system showed stable voltammetric responses over the operational time window, as discussed in Section 3.2. In all cases, and in contrast with the stable measurements in the absence of PE, a

progressive decrease in the analyte signal was observed during the operational lifetime of the SPCE, which remained operative for at least 24 h and, in some experiments, up to 48 h (Fig. 5a). This decrease is consistent with analyte depletion from solution as a result of adsorption onto PE. Considering the 25 mL solution volume and 0.19–0.21 g of PE, an adsorbed amount of 1 mg g^{-1} corresponds to a concentration decrease of approximately $7.6\text{--}8.4 \text{ mg L}^{-1}$. Therefore, the adsorption levels determined in this work are expected to produce concentration changes of several mg L^{-1} , well within the quantifiable range of the DPAdSV method.

Although the pellet-free control experiments demonstrated a stable SPCE response under the same hydrodynamic conditions, a minor contribution from PE-induced electrode fouling cannot be completely excluded. Surface-associated additives or low-molecular-weight compounds released from the pellets could progressively alter or partially passivate the electrode surface. Consequently, the signal decrease observed in the presence of PE should be interpreted primarily as analyte depletion due to adsorption, while acknowledging the possibility of a small contribution from pellet-induced changes in electrode response.

Nevertheless, the adsorption of BP-3 and OC onto the same PE pellets was independently confirmed in our previous study by HPLC-MS/MS. In that work, the concentrations obtained by DPAdSV were also in good agreement with the chromatographic results [33], providing additional support for the interpretation of the signal decrease as a consequence of the adsorption process.

Nevertheless, although the observed signal decrease can be mainly attributed to analyte adsorption onto PE, the long-term operation of the SPCE under continuous stirring and in the presence of pellets also revealed some limitations in sensor durability. Under the most demanding conditions, a sudden loss of electrode function was noticed

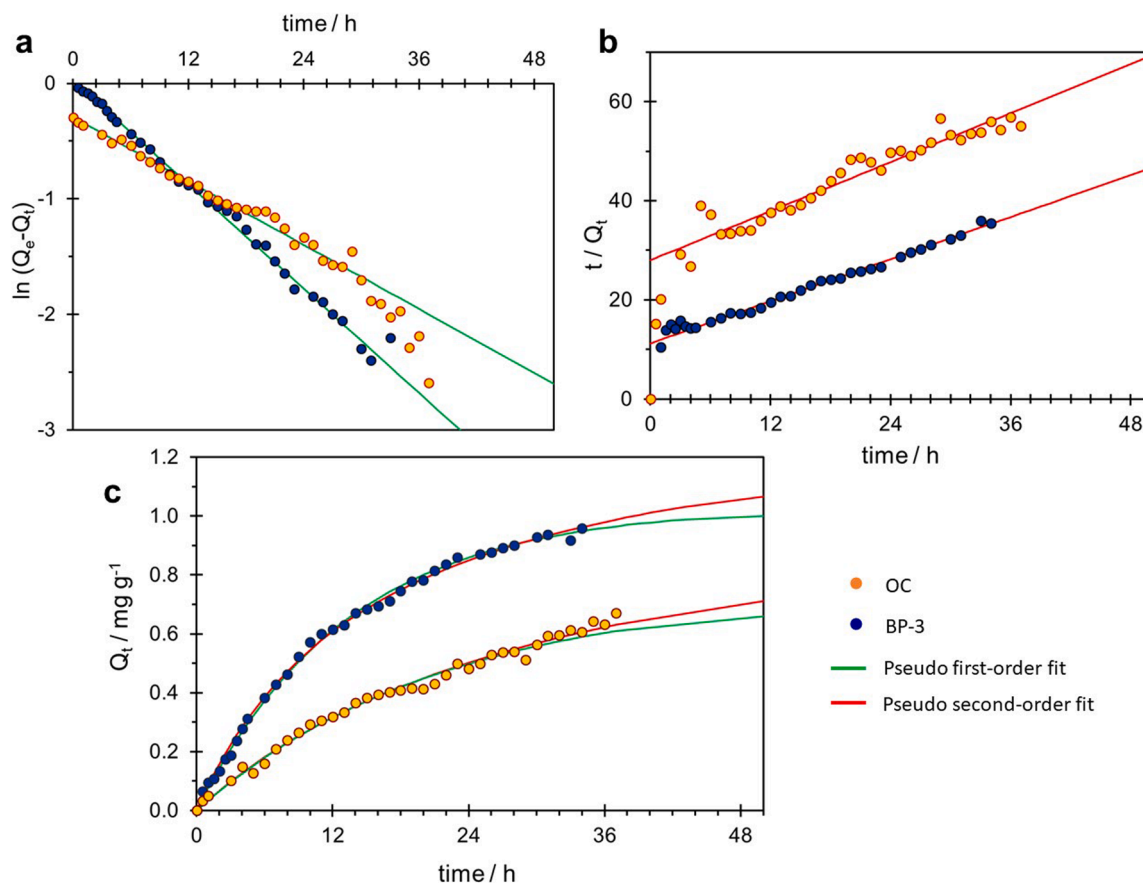


Fig. 6. Linear fitting of Eq. 4 (a) and 5 (b) to the data measured in the adsorption experiments with BP-3 at 12.5 mg L⁻¹ (●) and OC at 9.0 mgL⁻¹ (●). In (c) the experimental Q_t values are compared with the predictions obtained with the parameters fitted in (a) and (b) using Eq. 2 and 3 1 and 3. In all figures, the fitted curves are shown in green (—) for the pseudo first-order kinetics and in red (—) for the pseudo second-order kinetics.

by a measured current dropping abruptly to zero, which is consistent with complete loss of electrochemical activity or electrical connectivity. More commonly, however, the electrode exhibited a gradual decline in expected current response (as illustrated by the last points of BP-3 curves in Fig. 5a), suggesting signal depletion during extended operation in the presence of pellets. This progressive decay introduces significant deviations in the Q_t values (Fig. 5b) and, especially, in the fitting of kinetic models (4 and 5) since the assumption of a strictly time-invariant sensor response is not fully fulfilled under these experimental conditions.

3.4. Kinetic modelling

Fig. 6 compares the fitting of the two kinetic models considered to selected adsorption experiments for BP-3 and OC. As shown in Figs. 6a and 6b, both the pseudo-first-order (PFO) and pseudo-second-order (PSO) models yield linear plots over a wide time interval, indicating that both approaches can adequately describe the adsorption profiles under the tested conditions. When the values of the kinetic parameters

deduced from the slopes and the intercepts of the adjusted lines (Eq. 4 and 5) are applied to Eq. 2 and 3, the predicted plots of Q_t vs t shown in Fig. 6c are obtained. Comparison with the experimental values shows a good fitting in both cases within the investigated experimental time window. However, when the models are extrapolated to longer times, differences emerge. The PFO model tends to provide lower limiting values of Q (i.e., Q_e) than the PSO model, which predicts higher adsorption capacity. Since the adsorption experiments did not reach equilibrium conditions, and the end of the titrations appears to be still far from Q_e , it is not possible to conclusively determine which model provides the most representative description of the adsorption process. In addition, as previously discussed (Section 3.3), progressive changes in the SPCE response during long-term operation may also contribute to deviations at extended times and therefore introduce additional uncertainty into the fitted kinetic parameters.

Nevertheless, PSO kinetic model has frequently been reported in the literature for the adsorption of organic contaminants onto MPs often providing slightly improved fitting under comparable experimental

Table 3

Parameters obtained in the fitting of the data from adsorption experiments to kinetics of pseudo first-order (PFO, Eq. 4) and pseudo-second-order (PSO, Eq. 5), being n the number of points fitted.

Substance	C_0 (mg·L ⁻¹)	Pseudo first-order kinetics (PFO)				Pseudo second-order kinetics (PSO)			
		R^2	n	k_1 (h ⁻¹)	Q_e (mg·g ⁻¹)	R^2	n	k_2 (g mg ⁻¹ h ⁻¹)	Q_e (mg·g ⁻¹)
BP-3	6.5	0.991	15	0.070	0.30	0.956	29	0.073	0.57
	7.5	0.973	19	0.099	0.36	0.929	20	0.121	0.56
	9.2	0.990	21	0.103	0.61	0.914	11	0.078	0.91
	12.4	0.997	32	0.075	1.03	0.995	27	0.044	1.41
	10.0	0.985	32	0.039	0.69	0.973	36	0.024	1.14
OC	10.9	0.980	31	0.046	0.75	0.944	29	0.024	1.21
	13.7	0.983	37	0.025	0.99	0.927	23	0.013	1.43

Table 4

Kinetic parameters obtained for the PSO kinetic model in different publications. (LDPE low density polyethylene, HDPE high density polyethylene, PET polyethylene terephthalate).

Plastic	Substance	Technique	k_2 (g · mg ⁻¹ h ⁻¹)	Q_e (mg · g ⁻¹)	Reference
LDPE	BP-3	HPLC/MS-MS	0.32	0.89	Contreras-Llin et al. (2025) [33]
LDPE ^a	BP-3	DPAdSV			
LDPE ^a	BP-3	HPLC/MS-MS	1260–1600	0.020–0.035	Cui et al. (2022) [40]
HDPE ^{**}	BP-3	GC-MS	54	0.040	Kapelewska et al. (2025) [41]
PET ^{***}	BP-3	UV Spectrophotometry	0.057–0.081	6.62–9.10	Hanun et al. (2023) [42]
LDPE	OC	HPLC/MS-MS	0.20	2.03	Contreras-Llin et al. (2025) [33]
		DPAdSV			
PE	Tylosin	HPLC-UV	0.94	0.67	Guo et al. (2018) [37]
LDPE	Sulfamethoxazole	HPLC-UV	7.90	0.11	Xu et al. (2018) [38]
LDPE	BP-3	DPAdSV	0.04 – 0.12	0.56 – 1.41	This work
LDPE	OC	DPAdSV	0.013 – 0.024	1.14–1.43	This work

^{**} Particle diameter 40–48 µm. Biodegraded by microalgae *Acutodesmus obliquus*.

^{***} Particle diameter 125 µm. Subjected to a natural process of aging during 3 months.

^a Particle diameter 75–550 µm.

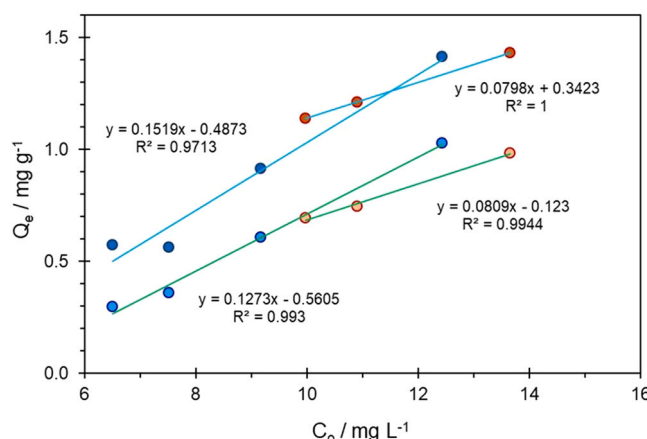


Fig. 7. Fitted values of Q_e according to a pseudo first-order (–) and a pseudo second-order kinetics (–) as a function of the initial concentration C_0 of BP-3 (●) and OC (●). The equations and the corresponding R^2 values of the fitted lines are shown in the graph.

conditions. It should be noted, however, that most of these studies are based on adsorption experiments with ten points or fewer, obtained through off-line sampling. In contrast, the experiments presented here include 20–40 data points measured *in situ* and in real-time, providing a much more detailed description of the adsorption profiles and their temporal evolution.

Table 3 summarizes the kinetic parameters obtained from fitting the PFO and PSO models to the adsorption experiments carried out for BP-3 and OC at different concentrations. Although the literature reports a large diversity of experimental conditions (especially regarding MPs size, nature and aging) and analytical methodology applied, the kinetic constants and Q_e values obtained here are within the same order of magnitude as those previously published (Table 4). Under the particular conditions of this work, the values in Table 3 suggest that the kinetic constants of BP-3 (0.07–0.10 h⁻¹ in PFO and 0.04–0.12 g mg⁻¹ h⁻¹ in PSO) are slightly higher than those obtained for OC (0.03–0.05 h⁻¹ in PFO and 0.01–0.02 g mg⁻¹ h⁻¹ in PSO). This trend may be related to the greater hydrophobic character of OC as compared to BP-3, which could reduce its mass transport in the aqueous medium and adsorption kinetics, despite favoring overall affinity for the PE surface. In both cases, however, no clear dependence of the fitted kinetic constants on the initial concentration of the UV filter was observed. This variability suggests that each compound may be characterized by a single intrinsic kinetic constant, independent of concentration but subject to experimental dispersion. Assuming this approximation, the mean values and standard deviations provide representative kinetic parameters for each model for comparative purposes: 0.09 (0.01) h⁻¹ for BP-3 and 0.04 (0.01) h⁻¹ for OC using the PFO model, and 0.08 (0.03) g mg⁻¹ h⁻¹ for

BP-3 and 0.020 (0.006) g mg⁻¹ h⁻¹ for OC using the PSO model. These averaged values consistently suggest that BP-3 adsorbs onto PE pellets at a faster rate than OC under the experimental conditions tested.

Regarding the limiting adsorbed amount (Q_e), Table 3 shows a clear increase with the initial concentration (C_0) of BP-3 or OC. Fig. 7 confirms this trend, displaying a linear relationship for both compounds with R^2 values between 0.97 and 1.00. This behavior is consistent with a linear Henry-type isotherm, that may correspond to the initial part of a Langmuir-type isotherm, still far from the saturation. Nevertheless, the number of experimental points is limited, and the adsorption experiments did not reach equilibrium conditions, resulting in considerable uncertainty in the estimated Q_e values. A more rigorous analysis should be based on equilibrium concentrations rather than initial concentrations C_0 . Achieving this would require long-term experiments allowing the system to reach equilibrium, which falls outside the scope of the present real-time electroanalytical methodology. These observations suggest that the proposed electroanalytical approach is particularly well suited for studying adsorption kinetics, thanks to its high temporal resolution and real-time monitoring capability, whereas the determination of adsorption isotherms would benefit from complementary equilibrium-based measurements performed after extended contact times.

The observed increase in Q_e values for BP-3 at higher initial concentrations (12.4 mg L⁻¹) may be interpreted in terms of the influence of its acid–base behavior and the complexity of the medium (synthetic seawater, pH ≈ 8.2), which also contains CTAB, that is a cationic surfactant. BP-3 is a very weak acid, with a pK_a of 7.6 close to the medium pH, and thus both neutral and deprotonated species coexist under the

conditions of the experiments [33]. This speciation is relevant since the neutral form is considerably more hydrophobic and, therefore, has a stronger tendency to adsorb onto non-polar phases such as PE pellets, whereas the ionic form remains more soluble in the aqueous medium. Additionally, CTAB could further influence how BP-3 behaves in solution. For instance, BP-3 may be present as surfactant–analyte associations or may form hydrophobic microenvironments that facilitate the transport of the neutral species towards the pellets surface. Moreover, adsorption onto PE is not a surface process exclusively, since partitioning mechanisms into amorphous regions of plastic polymers have also been reported, which becomes more relevant as the chemical potential gradient increases with concentration [35]. All these factors taken together may explain why BP-3 appears to show higher adsorption capacities at higher initial concentrations. Finally, it is important to note that as equilibrium was not fully reached in these experiments and that the calculated Q_e values were obtained from kinetic model extrapolations, part of the observed increase should also partially be the consequence of model fitting under non-equilibrium conditions.

4. Conclusions

This work demonstrates that DPAdSV measurements using commercial SPCE provide a simple, robust, cost-effective and sustainable electroanalytical platform for the *in situ* and continuous monitoring of the organic UV filters BP-3 and OC in simulated aquatic (marine) media at mg L^{-1} concentrations. The main novelty of this approach lies in its ability to deliver real-time datasets of concentrations as a function of time with an improved temporal resolution (more than 30 measurements along 24–48 h) compared with conventional off-line sampling strategies, mostly based on chromatographic-mass spectrometry techniques. In the case of the adsorption of organic UV filters on PE pellets studied in this work, this continuous monitoring capability offers a more precise and direct assessment of adsorption kinetics, revealing transient states that are not accessible through conventional sampling approaches based on collecting, storing and analyzing a limited number of off-line samples along the experiment.

The richer datasets obtained in this study reveal that the commonly reported preference for the PSO models in the literature is not directly applicable under real-time monitoring conditions. In contrast, PFO models provided slightly more consistent descriptions of the experimental data within the investigated time window, highlighting the importance of measurement density and temporal resolution in kinetic model selection. The kinetic parameters obtained for BP-3 and OC are consistent with values previously reported in the literature for plastic-organic contaminants systems, and indicate that BP-3 adsorbs onto PE at a faster rate than OC under the tested conditions. The results also suggest a concentration-dependent increase in the adsorption capacity, although this should be interpreted within the context of non-equilibrium conditions and model extrapolation. Longer experiments approaching true equilibrium would help to clarify differences between the two kinetic models and refine the estimation of limiting adsorption capacities. Despite the experimental limitations, the results suggested a linear Henry-type behavior for both compounds. Future studies should evaluate cross-interference between BP-3 and OC and the influence of additional matrix components before applying the method to multi-contaminant systems.

Overall, this study highlights the strong potential of SPCE-based electrochemical monitoring as a powerful tool for continuously analyzing trace concentrations of organic UV filters with high temporal resolution. This may be the case of kinetic adsorption studies such as this above described, but also the case of kinetic studies of degradation of such substances by UV radiation or the monitoring of samples with relatively high concentrations of organic UV filters such as wastewaters in water treatment plants.

Finally, further developments aimed at SPCE regeneration or replacement as well as recalibration strategies and integration into

autonomous monitoring systems could significantly expand the applicability of SPCE devices to long-term analysis of dynamic systems.

CRediT authorship contribution statement

Albert Contreras-Llin: Writing – review & editing, Methodology, Conceptualization. **Joel González-Molero:** Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Clara Pérez-Ràfols:** Writing – review & editing, Methodology, Conceptualization. **Núria Serrano:** Writing – review & editing, Methodology, Funding acquisition, Conceptualization. **M. Silvia Díaz-Cruz:** Writing – review & editing, Resources, Funding acquisition, Conceptualization. **José Manuel Díaz-Cruz:** Writing – original draft, Supervision, Funding acquisition, Data curation, Conceptualization.

Declaration of Competing Interest

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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Data availability

Data will be made available on request.

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