

# Electrofluorochromism of Conjugated Polymers Applied to the Development of Chemical Sensors

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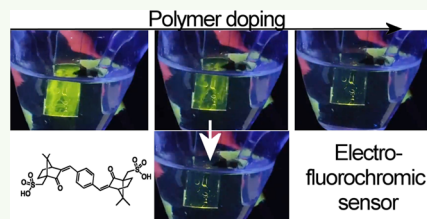
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**ABSTRACT:** Electrofluorochromism (EFC) is a phenomenon widely employed for the development of optoelectronic devices. In this work, we applied EFC as a powerful tool in sensing applications. The ability to tune the photoluminescence emission by controlling the applied potential may help maximize the response to target molecules. We have explored the EFC of the conjugated copolymer poly[9,9-dioctylfluorene-*alt*-benzothiadiazole] (F8BT) to design an electrofluorochromic sensor able to detect emerging pollutants with a significant impact in the marine ecosystems. In particular, a sulfonic acid-based chemical sunscreen (Ecamsule) was used as a model molecule. The photoluminescence emission modulation is affected by the presence of Ecamsule, which is attributed to the enhanced quenching process of F8BT during the p-doping process. The quenching is due to the formation of exciplex-like within the conjugated film upon the insertion of the organic anionic molecule in the polymer chain. The developed F8BT-based electrofluorochromic sensor displays a low limit of detection and good selectivity in the presence of interfering anions that are common in seawater.

**KEYWORDS:** electrofluorochromic sensor, *in situ* fluorescence spectroscopy, F8BT, sulfonic acid-based sunscreen, Ecamsule, Mexoryl SX



## 1. INTRODUCTION

The control of photoluminescence emission (both the intensity and the quality of the light emitted) by applying an electric field or an electrochemical potential is called electrofluorochromism (EFC).<sup>1</sup> This phenomenon is an emerging field for the development of new technologies and devices.<sup>2</sup> Electrofluorochromic devices have found application in optoelectronics applications, such as wearable and portable electronics devices or encrypted information and see-through displays.<sup>3–5</sup>

Several efforts have been made to achieve highly performing electrofluorochromic materials including small organic molecules (such as tetrazine, thienoviologen, or cyanine-based molecules) metal coordination compounds, quantum dots, or molecular dyads.<sup>6</sup> Among the materials garnering great attention within the EFC field, light-emitting conjugated polymers (CP) have a relevant place. These polymers found application in traditional organic optoelectronic devices, such as organic displays, transistors, or photovoltaic devices.<sup>7</sup> The synthetic tunability of conjugated polymers allows for the electronic and physical properties to be extensively optimized. They are low-cost and solution-processed and can be fabricated in lightweight and flexible devices. For the above-mentioned reasons, CPs have been vastly used in electrofluorochromic applications.<sup>8</sup>

The modulation of the photoluminescence of the conjugated polymer is produced upon the injection of charges into the polymer chain. This process can be performed by electrochemical methods in a reversible manner (i.e., electrochemical doping) by the generation of charged polarons in the

conjugated chain. When such electrogenerated polarons encounter the photogenerated excitons residing in the conjugated system, the exciton-polaron annihilation process drives a quenching of the photoluminescence. This phenomenon was explored in some previous works to determine exciton dynamics.<sup>9–11</sup>

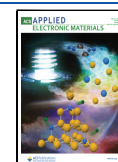
Although electrofluorochromic devices can be used in a wide range of optoelectronics applications, this phenomenon could produce a deep impact on applications focused on chemical sensing since photoluminescence spectroscopies are recognized as one of the most sensitive analytical techniques. However, these applications for EFC are still largely unexplored, as indicated by the excellent critical review performed by Corrente and Beneduci.<sup>2</sup> To the best of our knowledge, only two reports can be found in the bibliography devoted to the use of EFC in sensing applications. Ding et al. detected cyanide ions by EFC in thiophene-carbazole-benzothiadiazole conjugated copolymers.<sup>12</sup> The carbazole and benzothiadiazole anions participated in a significant weakening of the fluorescence inhibition by the polymer oxidation, indicating that the detection was produced by a noncovalent interaction mechanism between electron-deficient benzothiadiazole units and the nucleophilic CN<sup>-</sup> molecule. More recently, Chua et al.

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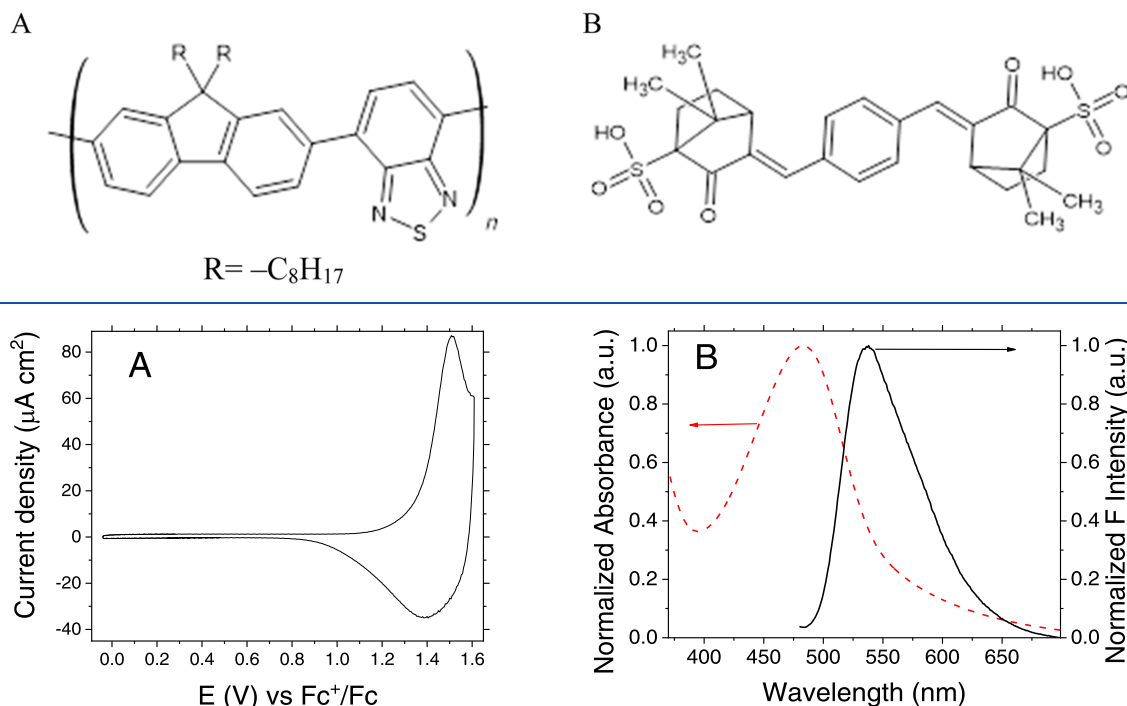
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Scheme 1. (A) Molecular Structure of F8BT Conjugated Polymer and (B) Ecamsule



**Figure 1.** (A) Stabilized cyclic voltammogram of F8BT on the ITO electrode in solution 0.1 M TBAHFP in ACN. Scan rate: 100 mV/s. (B) Normalized UV-vis absorption (dashed red line) and fluorescence emission spectra (solid black line) of the F8BT/ITO electrode ( $\lambda_{\text{exc}} = 470 \text{ nm}$ ).

detected nitroaromatic compounds in an electrofluorochromic system of polyacrylates containing triphenylamine units.<sup>13</sup> EFC-based sensing was based on a turn-on fluorescence at high voltages in the presence of the target molecule. The authors suggested a mechanism of electron transfer between the nitrocompounds and oxidized polymer radical that regenerates the neutral emissive polymer. Therefore, even though there are very few studies reporting on the EFC in sensing applications, the mechanism is the focus of controversy.

Considering this, in the present work, we aim to study the EFC of the conjugated copolymer poly[9,9-dioctylfluorene-*alt*-benzothiadiazole] (F8BT, see Scheme 1A). This yellow emitter containing electron-acceptor benzothiadiazole units was originally designed for use in OLED devices because of its high electron mobility, making this a model polymer for laser and photovoltaic applications.<sup>14,15</sup>

This polymer presents electroactivity and can suffer reversible electrochemical doping due to oxidation (*p*-doping) or reduction (*n*-doping). The electrogenerated holes (in *p*-doping) are compensated for by the insertion of anions present in the electrolyte solution. Based on this anion insertion and its possible interaction with the light-emitting polymer, we intended to develop an electrofluorochromic sensor for chemical sunscreens. A great family of chemical sunscreens are anionic aromatic molecules that absorb UV light. These sunscreens, commonly used in cosmetics, have widespread use in coastal zones and are threatening the ecological environment<sup>16</sup> and for that reason are considered as emerging pollutants.<sup>17</sup>

The present work focuses on the electrofluorochromic detection of a model sunscreen, Ecamsule (also Mexoryl SX or terephthalylidene dicamphor sulfonic acid, see Scheme 1B), that belongs to the family of sulfonic acid-based screens and is present in major cosmetic creams showing a broad-spectrum

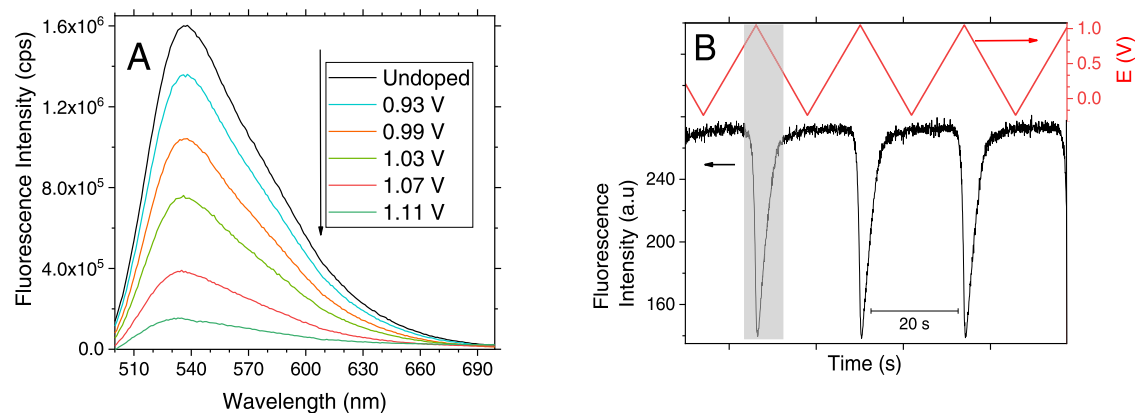
UVA light absorber.<sup>18</sup> Given the characteristics of F8BT that contains benzothiadiazole electron-acceptor functionalities, a strong interaction with aromatic substances is expected.

## 2. EXPERIMENTAL SECTION

Poly[9,9-dioctylfluorene-*alt*-benzothiadiazole] (ADS 133YE, F8BT, was purchased from American Dye Source,  $M_n = 21,000$ ). Tetrahydrofuran (THF, inhibitor-free, HPLC,  $\geq 99.9\%$ ), acetonitrile (ACN, anhydrous,  $\geq 99.8\%$ ), tetrabutylammonium hexafluorophosphate (TBAHFP, for electrochemical analysis,  $\geq 99\%$ ), ferrocenium hexafluorophosphate (Fc,  $\geq 97\%$ ), and Ecamsule sunscreen analytical standard (terephthalylidene-3,3'-dicamphor-10,10'-disulfonic acid) were supplied by Sigma-Aldrich.

Electrochemical measurements were carried out in an EDAQ EA163 Potentiostat connected to a waveform generator (EG&G Princeton Applied Research model 175) and an e-Corder 410 signal acquisition. Conventional three-electrode glass cells were used for the electrochemical measurements. The cell consists of a silver wire as a pseudoreference electrode, a platinum wire as the counter electrode, and a transparent glass/ITO electrode (SOLEMS, model SOL-30) covered by the conjugated polymer. All potentials were referred to the ferrocenium/ferrocene redox couple (Fc/Fc<sup>+</sup>) that was added to the electrochemical cell at the end of the experiments for the calibration of the reference electrode. The preparation of working electrodes was as follows: (i) ITO electrodes were cleaned by sonication in an acetone bath for 10 min; (ii) on the conductive side of the ITO electrode, an area of 1 cm<sup>2</sup> was coated by dropping 20  $\mu\text{L}$  of F8BT dissolved in THF (1 mg mL<sup>-1</sup>), leading to a thin layer; (iii) this layer was dried for 30 min under an IR lamp.

Fluorescence spectroscopy experiments were acquired using a PTI QuantaMaster spectrofluorometer (model QM-62003SE). *In situ* spectroelectrochemical measurements were performed in a modified 1 cm long fused silica cell capped with a Teflon plate, which also served as the electrode support. Details on the experimental setup were given elsewhere<sup>19</sup> and in Scheme S1 in the Supporting Information.



**Figure 2.** (A) Evolution of the fluorescence emission spectrum during p-doping of F8BT ( $\lambda_{\text{exc}} = 470$  nm). (B) Evolution of the fluorescence intensity ( $\lambda_{\text{em}} = 540$  nm) for an F8BT as a function of applied potential (electrofluorochromism, EFC) during successive cyclic potential scans between  $-0.23$  and  $+1.06$  V at  $100$   $\text{mV s}^{-1}$ . The zone marked in gray corresponds to potential values where the polymer is in a p-doped state.

### 3. RESULTS AND DISCUSSION

**3.1. Spectroelectrochemical Characterization of F8BT.** The electrochemical response of polymer layers in blank solutions has been characterized to determine the potentials that induce the injection of charge into the conjugated chain.

Figure 1A shows the stabilized cyclic voltammogram of the F8BT thin layer on an ITO electrode. In the positive-going scan, a rise in the current density is observed from  $+0.9$  V to a maximum peak current density at  $+1.50$  V related to the injection of positively charged species in the conjugated chain (p-doping). During the p-doping, the positive charges are compensated by the diffusion of anions coming from the solution to keep the electroneutrality in the polymer structure, which produces the polymer swelling.<sup>22</sup>

Figure 1B shows the UV–vis absorbance and fluorescence spectra of the pristine F8BT on the ITO electrode. The absorbance spectrum presents a main absorption band at  $480$  nm caused by  $\pi\pi^*$  electron transitions of  $\pi$ -delocalized electrons along the conjugated polymer chain.<sup>20</sup> The fluorescence emission spectrum shows a unique band of maximum emission at  $538$  nm corresponding to the main electronic  $S_1 \rightarrow S_0$  transition.

In the following experiments, we determine how doping processes affect the emissive properties of the polymer using fluorescence spectroelectrochemical characterization. Figure 2A shows the evolution of the fluorescence spectrum of F8BT as a function of the applied potential. The emitting properties of the polymer remain unchanged for potentials below the p-doping onset, but for potentials more positive than  $+0.9$  V, the electrochemical doping results in a progressive inhibition of the emission (electrochemical quenching).

Figure 2B shows the evolution of the fluorescence intensity emitted by F8BT at a fixed wavelength of  $540$  nm measured during the continued potential sweeping from  $-0.23$  to  $1.06$  V at  $100$   $\text{mV s}^{-1}$ . The figure's upper part (red line) represents the applied potential sequence, while the lower part (black line) shows the fluorescence intensity as a function of time. Initially, in the range of potentials where the polymer is undoped state, the PL intensity of the polymer stays at its maximum value. Beyond  $+0.9$  V, the photoluminescence (PL) intensity sharply decays at the potential corresponding to the onset of the p-doping process observed previously in the electrochemical response. In the reverse potential sweep, the PL intensity

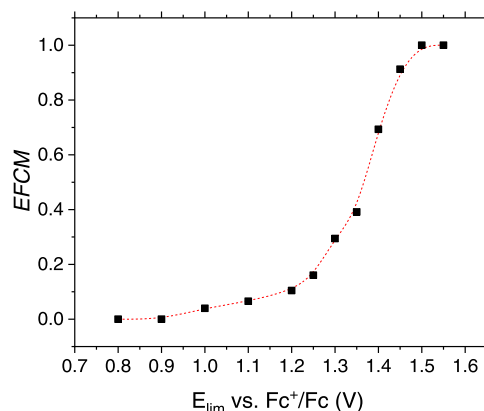
undergoes full recovery upon electrochemical reduction (recovery of the semiconducting state of the polymer upon the undoping process). This response remains consistent over multiple potential cycles, sustaining stability through at least 20 voltammetric cycles.

For the experimental conditions depicted in Figure 2B, the electrofluorochromism modulation (EFCM) can be defined as the variation of the normalized fluorescence during the voltammetric experiment (eq 1)

$$\text{EFCM} = \frac{F_0 - F(E_{\text{lim}})}{F_0} \quad (1)$$

being  $F_0$  the fluorescence intensity of the undoped polymer and  $F(E_{\text{lim}})$  the fluorescence of the polymer at the potential limit established in the voltammetric experiment. The EFCM depends on the anodic limit reached in the voltammetric experiment since higher potential limits drive higher doping levels.

Figure 3 shows the EFCM values registered in voltammetric experiments for a potential scan between  $-0.23$  V and different anodic limits. For anodic limits below  $+0.7$  V, no modulation of the fluorescence is observed since the polymer preserves its semiconducting state in this range of potentials, but for



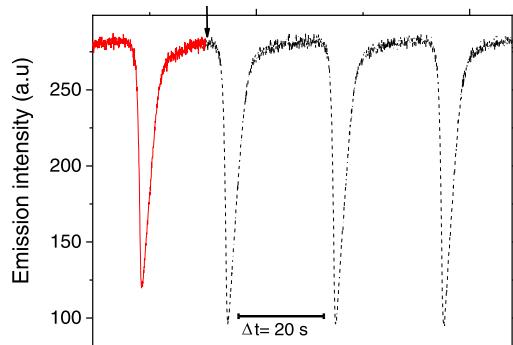
**Figure 3.** Stabilized electrofluorochromic modulation (EFCM) as a function of the potential of the anodic limit ( $E_{\text{lim}}$ ) of F8BT ( $\lambda_{\text{exc}} = 470$  nm,  $\lambda_{\text{em}} = 540$  nm). Cyclic voltammetric scans between  $-0.23$  V and the anodic limit are indicated in the abscissa at  $100$   $\text{mV s}^{-1}$ .

potentials above +0.7 V, an EFCM is observed due to the doping onset.

Two regimes can be observed in this plot; at low doping levels (up to +1.2 V), the EFCM increases linearly with the applied potential. The major mechanism of quenching at low doping levels is the collisional polaron-exciton annihilation dominated by diffusional processes of both excitations.<sup>10</sup> From +1.2 V, a sigmoidal increase is observed related to enhanced quenching due to static contributions, i.e., long-range Förster resonance energy transfer (FRET) between excitons and polarons.<sup>11</sup> From this potential, the EFCM increases sharply, and the complete quenching (EFCM = 1) is attained at potential limits above +1.5 V.

### 3.2. Spectroelectrochemical Response of Ecamsule.

To evaluate the viability of the F8BT/ITO electrodes as electrofluorochromic sensors, fluorescence modulation at 540 nm was followed under potentiodynamic conditions in the absence and presence of the analyte of interest. Figure 4 shows



**Figure 4.** Electrofluorochromism of F8BT in the absence of Ecamsule (red solid line) and after the addition of 15  $\mu\text{M}$  Ecamsule to the solution (black dashed line). The arrow marks the moment of the injection of Ecamsule. Scan rate of the voltammetric cycle 100  $\text{mV s}^{-1}$ . Anodic limit: + 1.37 V.

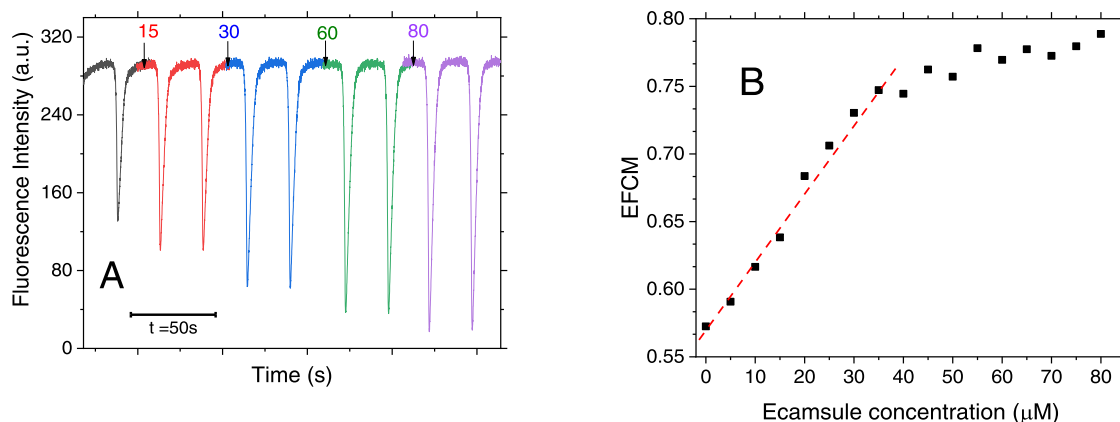
the EFC of F8BT in a similar experiment as that presented in Figure 2B. In this case, the anodic limit was fixed to obtain an EFCM of 0.45 ( $E_{\text{lim}} = +1.37$  V). After stabilization of the electrofluorochromic response (marked with the arrow in Figure 4), Ecamsule (15  $\mu\text{M}$ ) was spiked to the solution in the spectroelectrochemical cell while the fluorescence was followed.

The addition of Ecamsule does not modify the electrochemical response of the polymer, but an enhancement of EFCM is observed from the first scan. This response is stable along successive cycles that reached an EFCM of about 0.63, showing the responsiveness of the doped polymer toward the Ecamsule in solution. Since the fluorescence of the polymer in an undoped state remains unaffected by the presence of the sunscreen (see Figure S2 in the Supporting Information), the EFCM enhancement can be related to the incorporation of this molecule into the polymeric network upon electrochemical quenching. The doping process produces the injection of positive charges into the polymer chain, and this charge is compensated with anions from the solution, causing the polymer swelling. In the presence of the Ecamsule anion, the fluorescence of the polymer is quenched more intensely.

The quenching mechanism caused by the sunscreen is difficult to determine, although we can discard charge-transfer processes between the polymer and the incorporated molecule since the presence of this molecule does not affect the electrochemical response of the polymer. Förster-type energy transfer mechanisms can also be ruled out given the absorbance spectrum (see Figure S1 in the Supporting Information) since there is no spectral overlap between the emission spectrum of the polymer (donor) and the absorption spectrum of the potential acceptor, Ecamsule. The lack of optical activity at 470 nm for Ecamsule also discards the possibility of “inner filter” effects that would produce static quenching in the polymer.

Therefore, the most plausible quenching mechanism is the formation of an Ecamsule-F8BT charge-transfer exciton complex (or exciplexes). This phenomenon may be favored by the aromatic nature of the sunscreen because of  $\pi\pi$  interactions between the aromatic rings of fluorene and the sunscreen.

We obtained the spectrum of the doped polymer in the presence of Ecamsule, obtained at the potential of the anodic limit presented in Figure 4. The spectrum (see Figure S3 in the Supporting Information) presents a similar shape to that obtained under similar conditions in the absence of Ecamsule, with no additional emission features. In this case, after exciplex formation, the photoinduced electron transfer complex by the polar surrounding drives to ion pairs separation and concomitant photoluminescence quenching.<sup>21–24</sup>



**Figure 5.** (A) EFC of F8BT in the presence of different concentrations of Ecamsule indicated in  $\mu\text{M}$  units. Conditions are as in Figure 4. (B) EFCM as a function of Ecamsule concentration. Fitting parameters: slope ( $k$ ) =  $5.32 \times 10^3 \text{ M}^{-1}$ ,  $R^2 = 0.990$ .

Figure 5A shows the electrofluorochromism of F8BT in a solution containing different concentrations of Ecamsule. It is observed that EFCM becomes more intense as the concentration increases. A maximum EFCM of 0.78 was obtained for an Ecamsule concentration of 80  $\mu\text{M}$ . Further increase of Ecamsule concentrations does not modify this value, indicating a saturation of the response as a result of the swelling capacity of F8BT at the generated doping levels.

Figure 5B shows the EFCM as a function of the concentration of Ecamsule where a linear variation of EFCM, with a dynamic range from 0 to 40  $\mu\text{mol L}^{-1}$  is observed. For concentrations higher than 40  $\mu\text{mol L}^{-1}$ , the response is still linear but with a lower value of sensitivity. We have determined the limit of detection (LOD), considering the calibration curve using eq 2

$$\text{LOD} = 3.3 \times \frac{s_y}{k} \quad (2)$$

where  $k$  is the slope of the calibration curve and  $s_y$  is the standard deviation of the blank.<sup>25</sup> The LOD was 2.64  $\mu\text{mol L}^{-1}$  (1.48 mg  $\text{L}^{-1}$ ).

We checked whether Ecamsule is selectively detected in the presence of different anionic species (fluoride, iodide, nitrate, and phosphate). The electrochemical doping is not affected by the presence of those anions and the EFCM remains constant, with no influence coming from the addition of the different anions. The EFCM upon oxidative quenching in the absence of Ecamsule and after the addition of the mentioned anions is shown in Figure S4 in the Supporting Information. Only a sharp modification in the EFCM is observed after the Ecamsule addition.

#### 4. CONCLUSIONS

The photoluminescence emission of conjugated polymer F8BT can be modulated by the applied potential. The inhibition of emission is a reversible process, and a variable quenching was reached upon electrochemical doping, but the PL emission was recovered in the reverse process (undoping). The modulation of the electrofluorochromism (EFCM) depends on the anodic limit of the voltammetric experiment. The monitoring of the EFCM has been used to develop a sensor to detect an anionic emerging pollutant (Ecamsule) that is present in cosmetic sunscreens. In the presence of Ecamsule, the EFCM is enhanced since the insertion of such an aromatic molecule in the polymer chain induces a quenching process of F8BT. The proposed mechanism of quenching is the formation of a charge-transfer exciton (exciplex), which might be favored by the aromatic nature of Ecamsule that would facilitate  $\pi\pi$  interactions between the aromatic rings of the fluorene-benzothiadiazole units and the sunscreen (see Scheme S2 in the Supporting Information). The EFC of F8BT at different concentrations of Ecamsule was assessed, observing that modulation of fluorescence is linear at concentrations of Ecamsule lower than 40  $\mu\text{M}$ . Notably, the F8BT-based electrofluorochromic sensor exhibited a limit of detection around 3  $\mu\text{M}$ , and no interference in the presence of typical anions in seawater was observed. This methodology would allow the detection of this molecule in water. Preliminary measurements in water:acetonitrile mixtures show a modulation of the photoluminescence of F8BT upon electrochemical doping of the polymer (see Figure S5 in the Supporting Information).

#### ■ ASSOCIATED CONTENT

##### Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsaelm.3c01384>.

Experimental setup for EFC measurements (Scheme S1); UV and fluorescence spectra of Ecamsule and F8BT (Figure S1); fluorescence spectra of F8BT films in the absence and in the presence of Ecamsule in solution (Figure S2); spectra of the doped polymer in the absence and in the presence of Ecamsule (Figure S3); EFC of F8BT after the addition of different anions in solution (Figure S4); and tentative structure of doped F8BT-Ecamsule exciplex (Scheme S2) (PDF)  
EFC of F8BT upon electrochemical doping (MP4)

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##### Notes

The authors declare no competing financial interest.

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