

Article

Fabrication of Reusable Platinum Sensing Platform for Green Electrochemical Analysis

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Abstract

This work reports the design and validation of a reusable platinum (Pt) electrode platform (ST-E) for green electrochemical analysis. The device integrates Pt working and counter electrodes with an external Ag/AgCl reference and is engineered for repeated regeneration and refunctionalization. Surface renewal by alumina polishing followed by electrochemical activation in 0.5 M H₂SO₄ restores a clean, reproducible Pt surface, as confirmed by diffusion-controlled, reversible ferricyanide voltammetry over 5–150 mV s^{−1} with near-Nernstian peak separation and $i_{pa}/i_{pc} \approx 1$. Platform versatility is demonstrated in two applications. First, ST-E is functionalized with PFOA-selective molecularly imprinted nanoparticles on an APTES layer, enabling trace determination of perfluorooctanoic acid (1–5 pg mL^{−1}), with a detection limit of 0.50 pg mL^{−1} and sensitivity of 3.16 μA (pg mL^{−1})^{−1}. Responses correlate with an equivalently modified commercial screen-printed electrode ($r = 0.990$, $p < 0.005$), with Bland–Altman analysis confirming concordance. Second, after regeneration, electropolymerization of o-phenylenediamine yields an insulating poly(o-phenylenediamine) film that attenuates redox currents and increases ΔE_p, illustrating compatibility with diverse surface chemistries. Green metrics (AGREE, AGREE_{prep} ≈ 0.80; BAGI = 75.0) highlight reduced waste and solvent use versus single-use transducers and compatibility with portable potentiostats, supporting circular electrochemical sensing.

Keywords: reusable platinum electrode fabrication; electrochemical sensor regeneration; green analytical chemistry; PFOA; molecular imprinted polymers



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1. Introduction

Electrochemical sensing has evolved into a cornerstone of technological progress because of its versatility for rapid and cost-effective chemical analyses, enabling measurements from laboratory settings to on-site screening [1]. The combination of high sensitivity, short response times, minimal sample volumes, and compatibility with portable instrumentation makes electrochemical methods particularly appealing for trace-level detection

of priority pollutants, biomolecules, and food contaminants [2]. Among the many electrode materials explored for electroanalytical applications—including carbon paste, glassy carbon, bismuth, gold, and various composite substrates—platinum (Pt) stands out for its unique physicochemical properties, including a wide potential window in aqueous media, exceptional chemical inertness, and well-defined surface electrochemistry [3].

The electrocatalytic activity and selectivity of Pt electrodes can be substantially modified through surface modification strategies. Self-assembled monolayers (SAMs) of thiol-based compounds, electropolymerized films, and covalently grafted organic layers have all been applied to Pt to modulate electrode surface access, suppress interference, or provide specific recognition sites [4]. In particular, the electropolymerization of aromatic amines such as *o*-phenylenediamine (oPD) on Pt leads to the formation of insulating poly(*o*-phenylenediamine) (PoPD) films that effectively block the diffusion of interfering species while remaining permeable to small analyte molecules, a property exploited in both biosensors and electrosynthetic applications [5]. Nafion coatings and carbon nanotube composites deposited on Pt have been used to improve the selectivity towards ascorbic acid and catecholamines in complex biological matrices, demonstrating that surface engineering dramatically expands the analytical scope of Pt-based platforms [6]. Organosilane monolayers, including (3-aminopropyl)triethoxysilane (APTES), have also been deposited on platinum oxide surfaces, providing amine layers that serve as anchoring sites for nanoparticles [7], enzymes [8], and molecularly imprinted polymers (MIPs) [9].

The advent of screen-printed electrode (SPE) technology brought Pt-based voltammetry into the realm of disposable, mass-producible sensors. Screen-printed platinum electrodes (SPPtE) have been employed in differential pulse voltammetry (DPV) and square-wave voltammetry (SWV) for the determination of heavy metals [10], pharmaceutical compounds [11], and emerging organic contaminants including perfluoroalkyl substances (PFAS) [12]. Crapnell and Banks recently provided a comprehensive overview of screen-printed electrochemical sensing platforms, highlighting that, despite their analytical convenience, the single-use nature of SPEs generates solid waste, consumes platinum-group metals, and introduces variability associated with batch-to-batch differences in the printed ink composition [13]. Ahamed et al. estimated the environmental footprint of voltammetric SPE-based sensors using life-cycle thinking and concluded that disposable transducers impose a measurable ecological burden that can be substantially reduced by adopting reusable configurations [3]. These sustainability concerns have reactivated interest in the fabrication, characterization, and regeneration of robust, reusable Pt electrodes.

The regeneration of platinum electrode surfaces is a prerequisite for reliable reuse. Mechanical polishing with alumina suspensions of fine grades, followed by ultrasonic rinsing, removes adsorbed contaminants and restores a mirror-like finish, while subsequent CV cycling in 0.5 M H₂SO₄ completes the electrochemical activation by removing residual polishing debris and forming a reproducible surface oxide [14]. Cavanillas et al. took this concept further by demonstrating a self-polishing platinum ring voltammetric sensor that integrates an in-line mechanical renewal mechanism, enabling continuous operation in complex matrices such as wastewater and industrial effluents without manual intervention [15].

Advances in microfabrication have made it possible to produce thin-film Pt electrodes with precisely controlled geometry and thickness on silicon, ceramic, and polymeric substrates [16]. Thin-film Pt electrodes fabricated by sputtering or thermal evaporation on silicon wafers exhibit surface roughness and electrochemical activity comparable to bulk Pt disks, provided that appropriate adhesion layers (typically titanium or tantalum) are used to promote bonding between the Pt film and the substrate [17]. Such microfabricated Pt electrodes have found application in implantable glucose sensors, where the

combination of biocompatibility, wide potential window, and compatibility with MEMS fabrication processes is advantageous [18]. Furthermore, silicon-based Pt platforms benefit from the dimensional precision afforded by wafer dicing, allowing the production of large numbers of identical electrode chips from a single substrate with minimal chip-to-chip variation in active area and film thickness [19]. This manufacturability, combined with the possibility of mechanical regeneration and electrochemical reactivation, positions thin-film Pt-on-silicon architectures as attractive candidates for reusable, high-throughput electroanalytical platforms.

Green Analytical Chemistry (GAC) provides a framework for evaluating and minimising the environmental impact of analytical methods, encompassing waste reduction, solvent substitution, energy efficiency, miniaturisation, and the use of renewable or reusable materials [20]. Electrochemical sensors score favourably against many of these criteria: they consume micro- to nanolitre volumes of reagents, operate at room temperature with low energy demand, and are compatible with miniaturised cell configurations. However, the environmental benefit of SPE-based sensing is partially offset by the large number of disposable units consumed in long-term monitoring campaigns. Quantitative sustainability assessment tools such as AGREE (Analytical GREENness calculator), AGREEprep (for sample preparation steps), and BAGI (Blue Applicability Grade Index) have recently been applied to electrochemical sensor workflows to provide numerical benchmarks for comparisons across technologies [21–23]. Reusable platforms based on metal electrodes, when combined with portable multichannel potentiostats and rapid surface regeneration protocols, can score significantly higher on these indices than their single-use counterparts, particularly when the regeneration cycle consumes only dilute acid and deionised water.

This work introduces the fabrication of a new platinum platform (ST-E) engineered for electrochemical sensing and designed to facilitate setup reuse. The platform comprises Pt electrodes (working and counter electrodes) and an Ag/AgCl electrode as a reference, connected to a potentiostat. The resulting system was activated, characterized, and tested using voltammetric techniques. The results were then statistically validated. To demonstrate the application and potential of the developed system, the ST-E was modified with imprinted nanoparticles for the detection of perfluorooctanoic acid (PFOA). The results obtained were compared with those of a similarly modified commercial SPE. To further investigate the regeneration, multiple electropolymerization cycles of o-PD were performed as a case study. oPD was chosen because it features electropolymerization with controllable film growth, forming insulating films that attenuate redox currents and increase peak separation (ΔE_p). Beyond performance, the environmental impact of this reusable Pt platform is significant. Regeneration avoids the use of single-use materials, reducing solid waste and the demand for consumables. Fabrication based on a long-lasting metal platform minimizes reliance on other substrates, while integration with portable instrumentation can reduce sample transport and laboratory processing, decreasing the overall environmental impact of monitoring campaigns.

2. Materials and Methods

2.1. Chemicals

Potassium Ferricyanide(III) ($\geq 99.0\%$), used as an electrochemical probe, aminopropyltriethoxysilane (APTES, 99%), and o-Phenylenediamine (99.5%) were purchased from Sigma–Aldrich, Milan, Italy. Potassium chloride ($\geq 99.5\%$, Puriss. p.a.), used as a supporting electrolyte, was purchased from Fluka, Milan, Italy. Sodium Phosphate Dibasic Heptahydrate and Sodium Phosphate Monobasic Monohydrate (analytical grade) were purchased from Fisher BioReagent™, Milan, Italy. Sulfuric acid (95.0–98.0%) was purchased from VWR Italia, Milan, Italy. For the proof of application of fabricated ST-E, imprinted

nanoparticles (nanoMIPs) already synthesized for a previous work [24] were used. The results were compared with those obtained with commercial screen-printed platinum electrodes (DRP-550-U75, Metrohm DropSens, Apeldoorn, The Netherlands). The DRP-550 integrates a platinum working electrode and a platinum counter electrode, while the on-board reference is a printed silver (Ag) electrode. Milli-Q water (resistivity 18.2 MΩ·cm, conductivity 0.055 μS/cm), obtained using an ADRONA system, model CT-5100, Riga, Latvia, was used throughout the experiments.

2.2. Fabrication of ST-Electrode

The ST-Electrode (ST-E) platform was fabricated by STMicroelectronics in Agrate Brianza, Italy, starting from commercially available silicon substrates. Single-side polished silicon wafers (p-type, <100> orientation) were purchased from GlobalWafers Co., Ltd., Bristol, UK. and served as the mechanical support for the electrode structure. A metallic bilayer was deposited on the polished face of each wafer by thermal evaporation under high vacuum (base pressure $< 5 \times 10^{-6}$ mbar): first, a 10 nm-thick titanium (Ti) adhesion layer was evaporated to promote bonding between the native silicon oxide and the noble metal film; subsequently, a 100 nm-thick platinum (Pt) film was deposited on top of the Ti layer without breaking the vacuum, ensuring a continuous and contaminant-free metallic stack. Deposition rates and film thicknesses were monitored in situ using a quartz-crystal microbalance. Finally, the metallized wafers were diced into individual electrode chips, with dimensions of 0.8 cm × 2.3 cm, using a precision wafer dicing saw. Each chip thus constitutes a flat, non-patterned Pt electrode.

2.3. Analytical Performance Test

Platinum platforms were used as working (WE) and counter (CE) electrodes, linked with the PalmSens4 potentiostat (Metrohm, Amsterdam, Netherlands) controlled by the PStTrace v. 5.13 software, through 4 mm banana terminal wire with crocodile clips, along with the traditional Ag/AgCl as the reference (RE) electrode. During electrochemical measurements, the crocodile-clip contact area was kept outside the electrolyte. Each Pt chip was immersed for 1.0 cm only, resulting in a geometric exposed area of 0.80 cm² on the Pt surface. No insulating mask was used; the exposed area was defined by maintaining a constant immersion depth in all experiments. Platinum was selected as WE and CE due to its chemical inertness, wide potential window in aqueous environments, and ease of cleaning. The importance of using Ag/AgCl in three-electrode measurements lies in the accuracy and reproducibility of the potential applied to the working electrode, which directly depends on the stability of the reference electrode. Drift or instability in the reference potential can introduce qualitative and quantitative errors. Single-use SPEs exploit a printed Ag/AgCl element that can function as a quasi-reference, whose potential can vary with chloride activity and matrix composition. ST-E, however, uses an external Ag/AgCl reference electrode, designed to maintain a virtually invariant potential by keeping a controlled chloride environment separated from the sample via a liquid junction, improving protocol robustness.

Prior to each measurement session, the ST-E was electrochemically activated to improve its electrochemical response and surface cleanliness. Activation was performed by immersing the electrode in a 0.5 M H₂SO₄ aqueous solution and subjecting it to cyclic voltammetry by applying multiple consecutive potential scans within a potential window (−0.5 V–1.5 V, −0.3 V–1.5 V, −0.25 V–1.5 V vs. Ag/AgCl). Different potential windows were tested. After activation, the electrode was thoroughly rinsed with milliQ water and dried before subsequent electrochemical measurements. The electrochemical characterization of the ST-Electrode was performed using potassium ferricyanide(III) as the redox probe.

Cyclic voltammetry measurements were carried out in a 5 mM solution of $K_3[Fe(CN)_6]$ prepared in 0.1 M KCl as the supporting electrolyte. Scan-rate studies were conducted by recording CVs at 5, 10, 25, 50, and 100 $mV\ s^{-1}$. For each scan rate, the anodic (I_{pa}) and cathodic (I_{pc}) peak currents were extracted and plotted as a function of both the scan rate and the square root of the scan rate, obtaining the linear relationship between I_{pa} and the square root of the scan rate over the investigated range (5–100 $mV\ s^{-1}$) (Figure 1). Furthermore, the diffusion and electron exchange of the developed system were investigated, according to the Randles–Ševčík Equation (1), which states that the peak current varies with the square root of the voltage scan rate and is an important analytical result, indicating that a redox couple is dissolved in solution rather than adsorbed on the electrode surface, the latter identified by a linear relationship between peak current and scan rate [25,26].

$$i_p = 2.69 \times 10^{-5} n^{3/2} AC \sqrt{Dv} \quad (1)$$

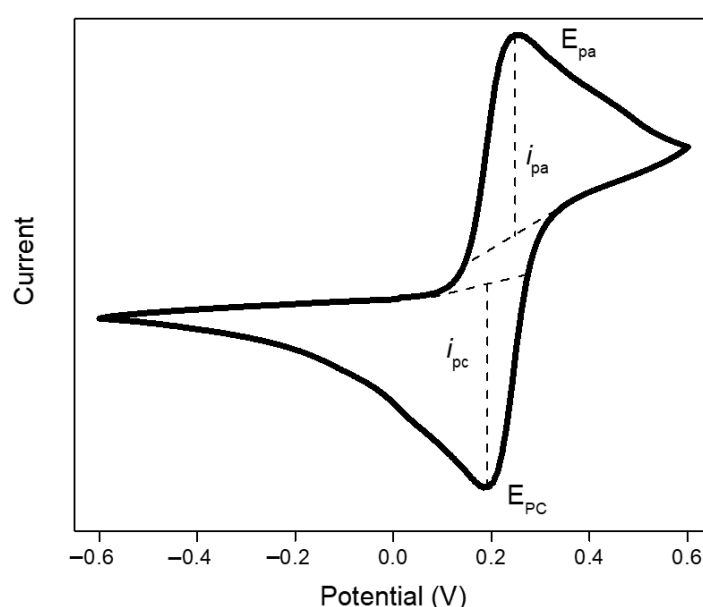


Figure 1. Example of cyclic voltammograms of a redox probe in KCl as a support electrolyte. Parameters: E_i : $-0.6\ V$; E_f : $0.6\ V$; step potential: $5\ mV$; scan rate: $50\ mV/s$.

For solutions at room temperature, where i_p is the maximum current, n is the number of electrons transferred in the redox reaction (typically 1), A is the electrode area in cm^2 , D is the diffusion coefficient in cm^2/s , C is the concentration in mol/cm^3 , and v is the scan rate in V/s . The constant with a value of 2.69×10^{-5} is expressed in $C\ mol^{-1}\ V^{-1/2}$.

Furthermore, the evaluation of a redox system involves a cyclic voltammetric (CV) study at multiple scan rates to establish the kinetic nature of the couple (reversible, quasi-reversible, irreversible). In CV, a reversible system typically shows $\Delta E_p \approx 59\ mV$, $i_{pa}/i_{pc} \approx 1$, and ΔE_p independent of scan rate. Systematic deviations and increasing with scan rate, as broadened or asymmetric peaks, indicate quasi-reversibility ($\Delta E_p \leq 200\ mV$) or irreversibility (>200) [27].

2.4. PFOA-nanoMIP Test and Proof of Concept for Reusability

The three-electrode system developed for this work was tested with PFOA-selective MIP nanoparticles developed for a previous work. [24] and where a commercial screen-printed electrode was previously used. To obtain a nanoMIP-modified system for the detection of perfluorooctanoic acid, APTES was used as a silanizing agent on which the imprinted nanoparticles could be immobilized by activating the carboxyl groups via

EDC/NHS. The sensor developed with ST-E was tested with PFOA solutions at known concentrations using the same parameters already studied in the previous work by DPV, in the potential range of -0.15 V to $+0.4$ V vs. Ag/AgCl reference electrode with the potential step of 4.95 mV; modulation amplitude of 0.2 V; pulse duration of 20 ms; and scan rate of 50 mV/s.

To assess the concordance between the responses of the two sensor systems, the Pearson correlation coefficient (r) [28] and Bland–Altman analysis [29] were applied. The sensor response was calculated for each point as the difference (Δ) between the measurements and their mean (m), graphically representing Δ as a function of m . Bias was defined as the mean of the differences, while variability was expressed as the standard deviation (SD) of the differences. The Limits of Agreement (LoA) were calculated as $\text{bias} \pm 1.96 \times \text{SD}$, which represents the interval within which 95% of the expected differences between the two methods fall.

After testing with the imprinted nanoparticles, the working electrode was mechanically cleaned with alumina and then activated with H_2SO_4 . To confirm the successful cleaning of the WE, it was characterized with an electrochemical probe. Subsequently, electropolymerization of ortho-phenylenediamine was performed. The surface of the WE was again characterized to demonstrate the formation of a polymer layer.

3. Results

3.1. Electrochemical Activation and Characterization of ST-E

The electrochemical activation of the ST-E was performed using cyclic voltammetry in sulfuric acid, as shown in Figure 2a, as an electrochemical preanodization of the unmodified electrode increases the heterogeneous transfer constant for the redox couple, improving the electrochemical reversibility and analytical performance [30] thanks to an increase in surface functional groups, surface roughness, and the removal of surface contaminants [31]. Specifically, three different CVs were performed with different initial applied potentials (-0.50 , -0.30 , -0.25) and a fixed final potential of 1.2 V. All curves show a pronounced cathodic current at the beginning of the scan, corresponding to the reduction of surface oxides and adsorbed species, typical of the H_2SO_4 voltammogram. As the scan progresses toward positive potentials, a broad anodic feature becomes visible, particularly evident for activation starting at -0.50 V, indicating the formation of oxygenated functional groups. Among the tested potential windows, the activation protocol starting from -0.5 V was selected for subsequent experiments, as it provided the most pronounced hydrogen adsorption/desorption features and improved Pt surface activation. The hydrogen oxidation peak is strongly influenced by the potential window. The hydrogen adsorption/desorption region is strongly influenced by the start-of-scan potential. Extending the potential window toward more negative values generally promotes hydrogen-related surface processes, making this region more prominent, as reported for platinum group metal oxide electrodes [32]. However, the intensity of the corresponding anodic feature cannot be interpreted solely as a characterizing function of the initial potential, since it is also influenced by the previous electrochemical history of the Pt surface, the reduction of residual oxide, capacitive currents, and possible hydrogen evolution during the cathodic excursion. Therefore, the cyclic voltammetric activation curves in Figure 2a were used only as a qualitative comparison of the activation windows. The initial potential of -0.50 V was chosen because it provided the most complete activation profile and, more importantly, led to a reproducible ferricyanide response after activation. For better visualization, Figure 2a shows the potential window from -0.3 V to 1.2 V. The complete plot is shown in Figure A1.

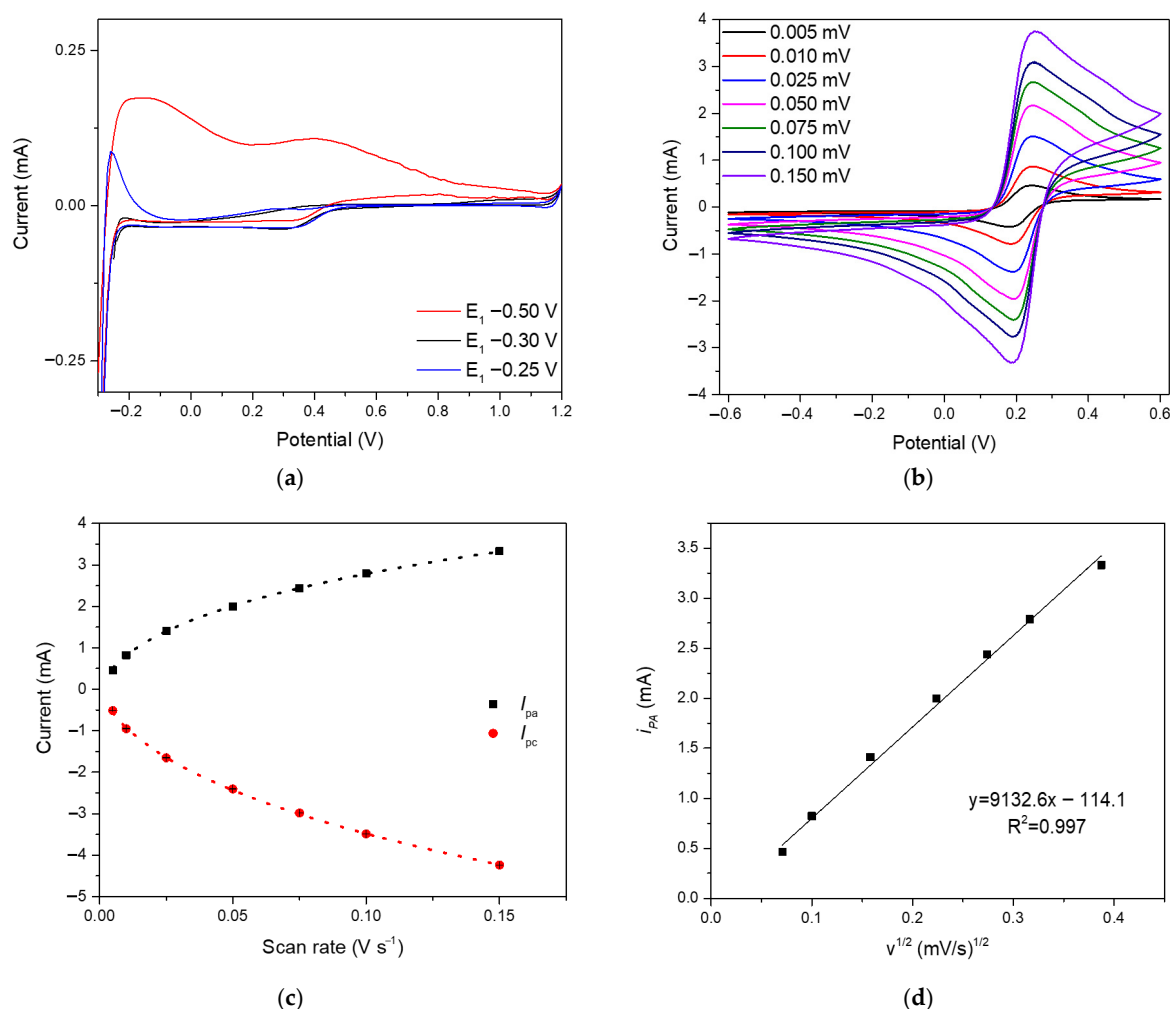


Figure 2. (a) Cyclic voltammograms recorded during electrochemical activation of the ST-E in H₂SO₄ at different initial potentials ($E_1 = -0.50$, -0.30 , and -0.25 V). (b) Cyclic voltammograms of a redox probe recorded at different scan rates (5, 10, 25, 50, 75, 100 and 150 mV·s⁻¹). (c) Dependence of the anodic peak current (i_{pa}) and cathodic peak current (i_{pc}) on the scan rate for the redox probe. (d) Dependence of the anodic peak current (i_{pa}) on the square root of the scan rate for the redox probe.

Figure 2b shows the cyclic voltammograms obtained for the redox probe at different scan rates (5, 10, 25, 50, 75, 100 and 150 mV s⁻¹). As the scan rate increases, an increase in the anodic and cathodic peak currents is observed, consistent with diffusive behavior (Figure 2c). The anodic currents (i_{pa}), shown in Figure 2d, indicate a linearity of the peak current with respect to the square root of the scan rate, confirming the Randles–Ševčík relationship, typical of diffusive processes in cyclic voltammetry, with a correlation coefficient of 0.992.

Table A1 summarizes the CV study conducted at $v = 5$ – 150 mV s⁻¹, reporting, for each scan rate, the potential peaks and the corresponding peak-to-peak separation ΔE . The measured ΔE_p spans 0.038–0.059 V, varies slightly with scan, and averages 0.048 V over the full range. Therefore, these results indicate the kinetics of a reversible, diffusion-controlled process. Furthermore, the i_{pa}/i_{pc} ratio in all cases shown is very close to 1, which is the ideal condition.

3.2. PFOA-nanoMIP Test

The developed system was modified with MIP nanoparticles for PFOA detection. To enable nanoparticle immobilization, the electrode surface was first functionalized with

APTES, prepared in a 5% *v/v* ethanolic solution, and deposited on the ST-E electrode surface through three consecutive applications of 5 μL each, allowing the solvent to evaporate completely between depositions. The electrode was then placed in an oven at 150 $^{\circ}\text{C}$ for 1 h to promote silane crosslinking. After placing an APTES layer on the work surface, 100 μL of EDC/NHS and nanoMIPs mixture was deposited for 45 min. The baseline of the functionalized electrode was initially recorded after incubation with a phosphate buffer solution at pH 7.4 without analyte. Subsequently, the sensor was tested against different PFOA concentrations of 1, 2, 3, 4, and 5 pg mL^{-1} . Each solution at a known concentration of PFOA was first incubated for 10 min to allow interaction with the immobilized MIP nanoparticles, and then the electrochemical responses were recorded using differential pulse voltammetry (DPV). Figure 3a reports the calibration curve obtained from the response of the sensor developed on ST-E within the linear range 1–5 pg mL^{-1} compared with the responses obtained with the commercial SPE, achieving a limit of detection of 0.50 pg mL^{-1} . Figure 3b instead shows the voltammograms obtained from the sensor.

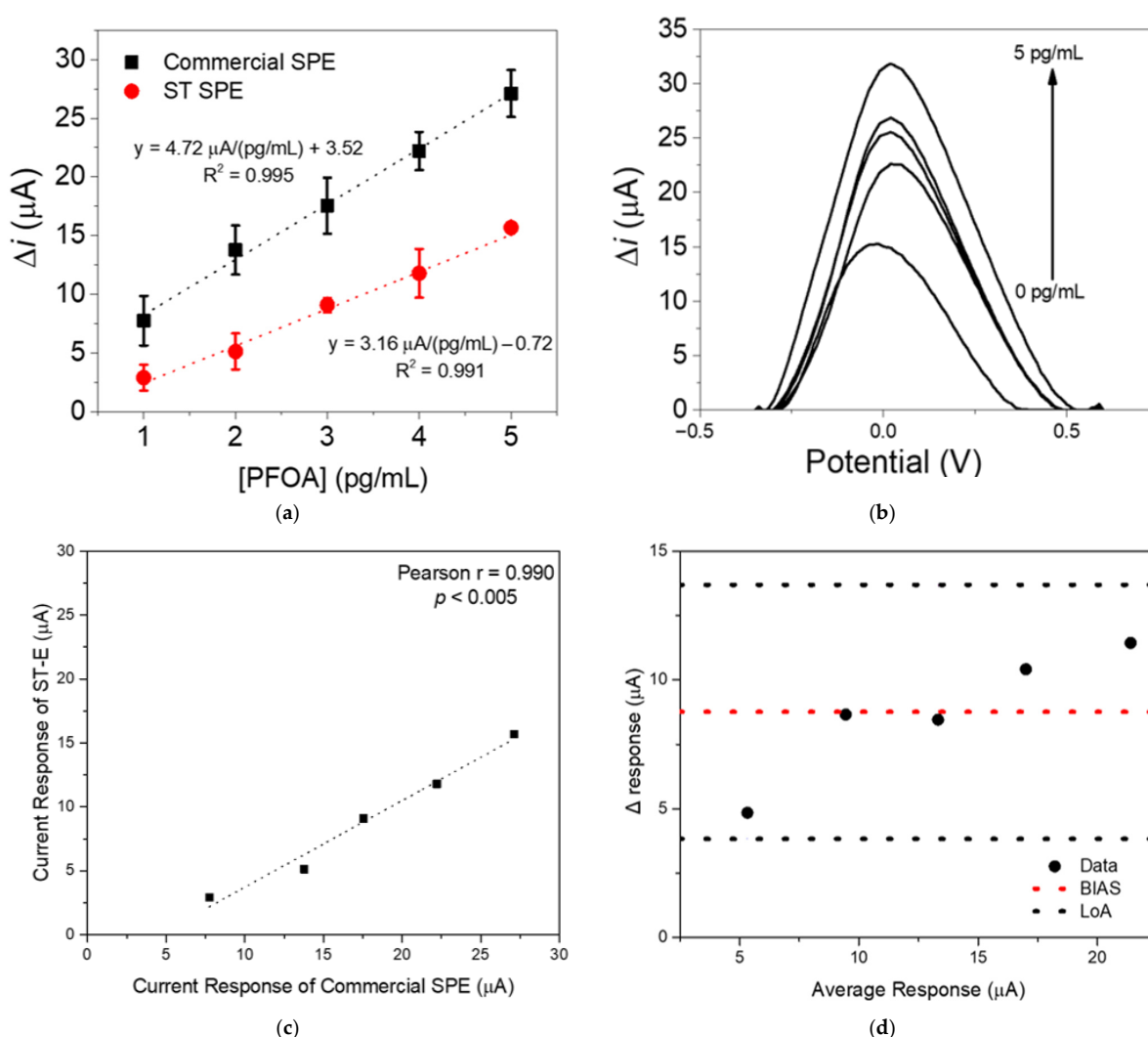


Figure 3. (a) Calibration curve of the modified nanoMIP/APTES/ST-E response compared with the responses of commercial SPE modified with the same imprinted nanoparticles. (b) Voltammograms of the responses obtained via DPV. (c) Relationship between the responses obtained with the modified commercial SPE (*x*-axis) and the modified ST-E method (*y*-axis). The dashed line indicates the linear fit from which the Pearson R coefficient was derived. (d) Bland–Altman plot comparing current responses obtained from modified commercial SPE and modified ST-E.

Figure 3c shows the relationship between the responses obtained from the modified commercial SPE (x -axis) and the responses from the modified ST-E (y -axis), from which Pearson's r was derived and found to be equal to 0.990, indicating a good positive linear correlation between the two methods and a p value of 0.0011. The high correlation confirms that both systems respond proportionally to PFOA concentration changes. Furthermore, the comparability between the two systems was also assessed using the Bland–Altman method, which represents the difference between measurements as a function of their mean. This approach allows us to identify both the systematic error (bias) and the variability of the differences. The bias, defined as the mean of the differences, was found to be $+8.76 \mu\text{A}$, confirming that the commercial sensor has higher performance than the ST-E. The 95% Limits of Agreement (LoA) were also calculated, obtained as $\text{bias} \pm 1.96 \times \text{SD}$, which was found to be equal to 3.83 and $13.69 \mu\text{A}$. Figure 3d shows that all differences are within the LoA, confirming an acceptable agreement between the systems.

As summarized in Table 1, the proposed reusable Pt platform provides analytical performance in the low pg mL^{-1} range, comparable to state-of-the-art electrochemical PFOA sensors based on molecular imprinting, while adding the distinctive advantage of a regenerable metallic transducer. Although some recently reported devices achieve lower detection limits, these are often based on disposable or non-reusable architectures, whereas the present platform was specifically designed for regeneration and refunctionalization.

Table 1. Comparison of analytical performance of electrochemical/electroanalytical devices reported for PFOA determination.

Reference	Type of Device	Linear Range	LOD	Reusability
This work	Reusable Pt platform	$1\text{--}5 \text{ pg mL}^{-1}$	0.50 pg mL^{-1}	platform regenerable
[24]	Electroactive nanoMIP/APTES/SPPtE	Up to 7.5 pg mL^{-1}	0.40 pg mL^{-1}	Yes (at least 3 work cycles)
[12]	MIP-modified screen-printed electrode	$0.1 \text{ nM--}10 \mu\text{M}$	19 pM	Not reported
[33]	MIP-modified Au interdigitated microelectrode chip	$0.4\text{--}40 \text{ fg L}^{-1}$	$4.5 \times 10^{-4} \text{ fg L}^{-1}$	Not reported
[34]	Electropolymerized MIP on $\text{Au/Cu}_2\text{O@C@NiCo}_2\text{O}_4$ microchip sensor	$207\text{--}4140 \text{ ng L}^{-1}$	19.46 ng L^{-1}	No (disposable microchip sensor)
[35]	PFDT self-assembled monolayer on AuNP/GCE	$100\text{--}5000 \text{ ppt}$	24 ppt	Yes (stable for at least 200 cycles)

3.3. Regeneration Study

A key advantage of the ST-E platform is the ability to regenerate the working electrode surface. Mechanical regeneration of the working electrode was performed manually by polishing the exposed Pt surface using a $0.05 \mu\text{m}$ alumina slurry. The chip was held parallel to the polishing surface and moved under gentle pressure in a figure-eight motion, then thoroughly rinsed with Milli-Q water and briefly sonicated to remove residual alumina before electrochemical activation in $0.5 \text{ M H}_2\text{SO}_4$. First, ST-E was characterized by a redox probe to determine the current of the non-modified electrode. Subsequently, electropolymerization of *o*-phenylenediamine (5 mM in $0.5 \text{ M H}_2\text{SO}_4$) was performed to reform a thin polymer layer on the WE. The surface was again characterized to demonstrate the formation of the polymer film, as evidenced by the obtained voltammogram. WE surface was mechanically cleaned with alumina, thoroughly rinsed, and electrochemically activated

in H_2SO_4 to restore a clean, electroactive surface. The cleaning and activation of the WE were verified by characterizing the electrode with an electrochemical probe (Figure 4a). Then, electropolymerization of o-PD was performed to reform a thin polymer layer on the WE, and the cleaning and regeneration operation was reproduced two more times.

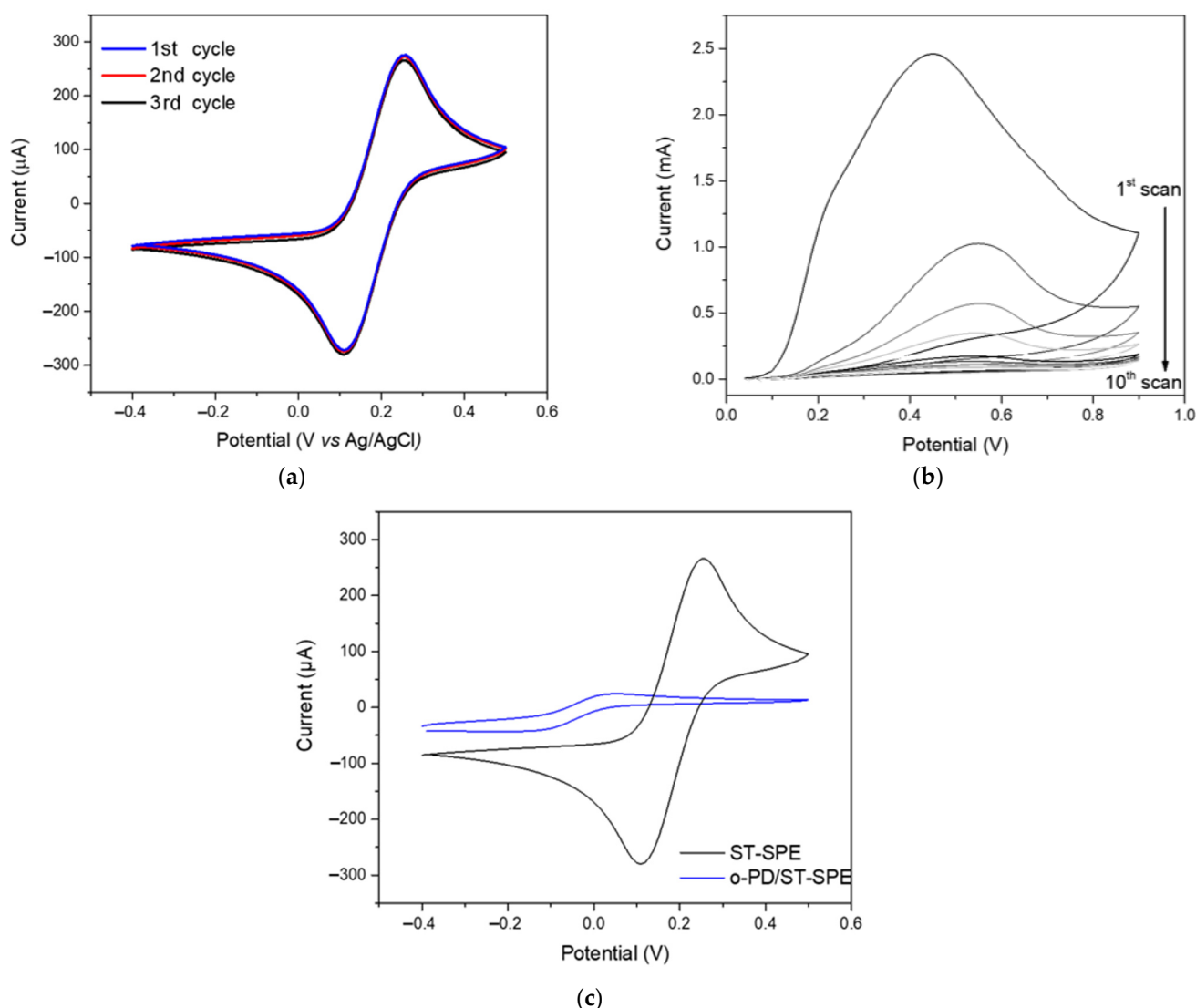


Figure 4. (a) Cyclic voltammetry of the redox probe (vs. Ag/AgCl) recorded before the first electropolymerization and after the first and second regeneration. (b) o-phenylenediamine electropolymerization (5 mM in 0.5 M H_2SO_4) by 10 scans of CV. (c) Comparison of CVs in the redox probe for the bare ST-E and the o-PD/ST-E.

Regeneration and subsequent re-functionalization: representative cyclic voltammograms are shown in Figure 4. Specifically, in Figure 4a, the redox probe response (vs. Ag/AgCl) recorded after regeneration overlaps with the voltammogram before regeneration, with comparable peak positions and currents, indicating that mechanical cleaning with alumina followed by electrochemical activation in 0.5 M H_2SO_4 restored the working surface. Electropolymerization of o-phenylenediamine (5 mM o-PD in 0.5 M H_2SO_4) was then performed using 10 CV scans in the -0.04 – 0.9 V potential window at 0.05 V s^{-1} (Figure 4b). In subsequent scans, a decrease in the oxidation peak and overall current is observed due to the growth of the polymer film, which then becomes poly(o-phenylenediamine) (PoPD). Figure 4c compares the voltammograms obtained by redox probing on the bare ST-E surface and the modified surface (PoPD/ST-E) after electropolymerization. The o-PD-modified

electrode shows a significant decrease in peak currents and increased peak separation (ΔE_p) compared to ST-E, due to an insulating film that prevents electron transfer.

Platinum is known to undergo transient dissolution during repeated oxide formation/reduction cycles. ICP-MS studies in acidic media report dissolved Pt amounts on the order of $\sim 1\text{--}5.9\text{ ng cm}^{-2}$ per potential cycle of fifty scans, depending on the scan rate [36]. Converting the worst-case dissolution (5.9 ng cm^{-2} per cycle) to an equivalent thickness yields $\sim 0.028\text{ \AA}$ per cycle, equivalent to $\sim 1.4\text{ \AA}$ (0.14 nm) for a 50-cycle regeneration step. For a 100 nm ($\approx 1000\text{ \AA}$) Pt film, this corresponds to an upper limit of estimation, approximately 714 regeneration steps, assuming only uniform dissolution. However, thin-film Pt can exhibit non-uniform, edge-localized, defect-assisted material loss during cyclic oxidation/reduction, and performance may degrade before full thickness depletion. Therefore, applying a conservative margin to account for additional wear mechanisms beyond uniform electrochemical dissolution, 150–200 working regeneration cycles have been estimated as the practical upper limit for reuse.

4. Green Chemistry Assessment: Sustainability Evaluation

The platform thus developed and used in this work presents numerous green advantages thanks to its versatility. The ST-E platform promotes Green Analytical Chemistry (GAC) by enabling systematic reuse and rapid surface regeneration of the working electrode, reducing material consumption, solvent use, and solid waste associated with single-use transducers [37]. To quantify the environmental and operational impact, the method was evaluated with two complementary tools: AGREE (Analytical GREEness calculator), developed by Pena et al. [21], consolidates the 12 GAC principles into a unified pictogram and a score from 0 to 1, weighting criteria such as waste generation, hazardous reagents, energy requirements, number of procedural steps, miniaturization, and automation. Using the AGREE software, the workflow achieved an AGREE = 0.80 (Figure 5a), indicating a high level of eco-sustainability. The software can be downloaded from <https://mostwiedzy.pl/AGREE> (accessed on 12 January 2026). Furthermore, since sample preparation can dominate the environmental impact, the green approach was also validated with AGREEprep, developed by Wojnowski et al. [22], which provides an ecological assessment for the preparation steps and offers clear pictogram-based output, as does AGREE. The index was found to be 0.8, in line with other chemical and biomimetic sensors (Figure 5b). The software can be downloaded from mostwiedzy.pl/AGREEprep (accessed on 12 January 2026). BAGI (Blue Applicability Grade Index), developed by Manousi et al. [23], assesses the practicality/applicability of analytical methods, considering attributes such as analysis type, concurrent analyses, samples per hour, reagents/materials, required instrumentation, parallel sample handling, preconcentration, automation, sample preparation, and sample quantity. This approach achieved a BAGI score of 77.5 (Figure 5c). To obtain the BAGI index, simply fill out the form at <https://bagi-index.anvil.app/> (accessed on 12 January 2026). A recurring limitation of MIP-based detection is simultaneous multi-analyte analysis [38]. The ST-E concept mitigates this issue by featuring multichannel devices with independent, regenerable Pt work surfaces functionalized with different receptors. Such architectures improve throughput, parallelization, and applicability, strengthening the practicality dimension captured by BAGI and contributing to the broader vision of sustainability in analytical chemistry. However, in the case of the previously developed PFOA detection device, AGREE and AGREEprep do not differ, but the BAGI index may vary by a few points due to the improvements expected from the simultaneous analysis and the semi-automated device.

To further contextualize the greenness and applicability metrics, the proposed ST-E platform was compared with a recently reported electrochemical PFOA sensor based on SnW-CNT nanocomposites deposited on a glassy carbon electrode (GCE) [39]. The comparison was performed using the AGREE (0.65), AGREEprep (0.78) and BAGI (67.5) index

based on the experimental and analytical protocol reported in the original study. The estimated AGREE score for the SnW-CNT/GCE method was lower than that obtained for ST-E, mainly due to the energy-demanding preparation of the sensing material, which includes hydrothermal synthesis, calcination and nanocomposite fabrication steps. In contrast, the reusable Pt platform relies on a regenerable transducer and avoids the repeated fabrication of complex sensing substrates. Similarly, the estimated BAGI score of the SnW-CNT/GCE method was lower than that of ST-E because the procedure is entirely manual, and the sensing material requires laboratory-scale synthesis before use. These results indicate that, beyond analytical performance, the reusability and operational simplicity of the ST-E platform contribute positively to both greenness and practical applicability assessments.

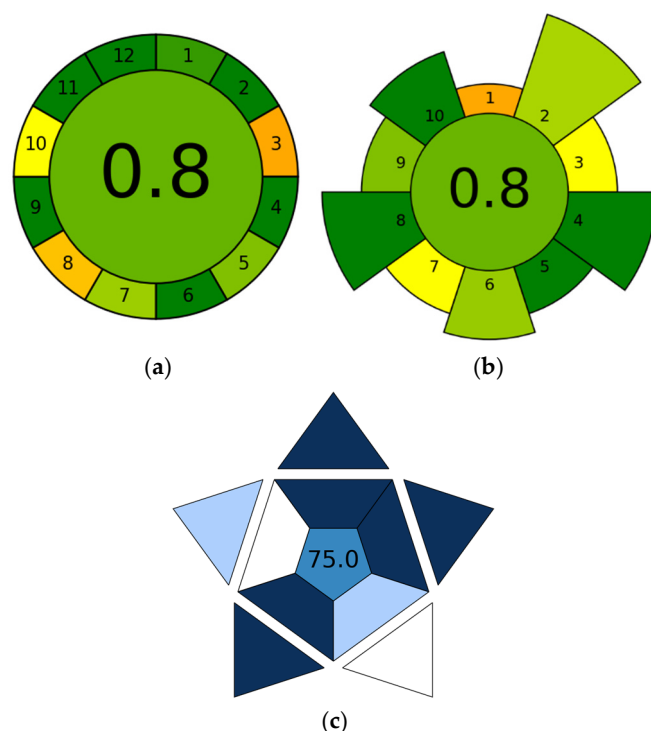


Figure 5. Graphical representation of AGREE (a), AGREEprep (b) and BAGI (c) indices for ST-E for PFOA detection by nanoMIP. For (a,b), the numbers displayed within each sector identify the criterion assessed by the tool. Each sector is colored based on the score (0–1) assigned to that criterion: red = slightly green approach; orange/yellow = intermediate; green = green approach. For (c), each sector is colored based on the score (0–100) assigned to the relative criterion according to the BAGI scale: dark blue = high compliance/practicality; light blue = intermediate compliance; white = low or no compliance. The overall BAGI score (0–100), which is placed in the center, summarizes the applicability/practicality of the method.

5. Future Outlook and Conclusions

In recent years, multiplexed electrochemical sensors have made significant progress using electrode arrays and parallel readout. Biomimetic receptor-based multiplexing strategies enable simultaneous detection of multiple targets on voltammetric/impedimetric platforms by leveraging differentiated labels and nanostructured surfaces to discriminate signals, with compelling demonstrations in environmental, food, and clinical applications, but remain challenging to translate into routine applications due to cross-interactions and signal management. Therefore, field-ready portable multichannel potentiostats compatible with SPE or metal electrodes are available. Multiplexing on a single WE (multiple receptors/labels on the same surface) often suffers from site competition, fouling, and redox crosstalk; in MIPs, the “multi-template” pathway can enable multi-target recognition

but introduces trade-offs in site selectivity and polymerization optimization, with risks of cross-reactivity and validation complexity [40]. Instead, a multichannel architecture (as in Figure 6) overcomes the “one contaminant at a time” constraint. A module with multiple physically separate and independently addressed WEs allows each WE to be functionalized with a dedicated receptor and to read signals in parallel with potential windows, protocols, and chemical conditions tailored to each target. This brings advantages such as (i) accelerated throughput (simultaneous measurements on N channels), (ii) increased metrological robustness, (iii) facilitates regeneration/reuse, (iv) makes replacing an electrode easy, and (v) enables cross-confirmation strategies.

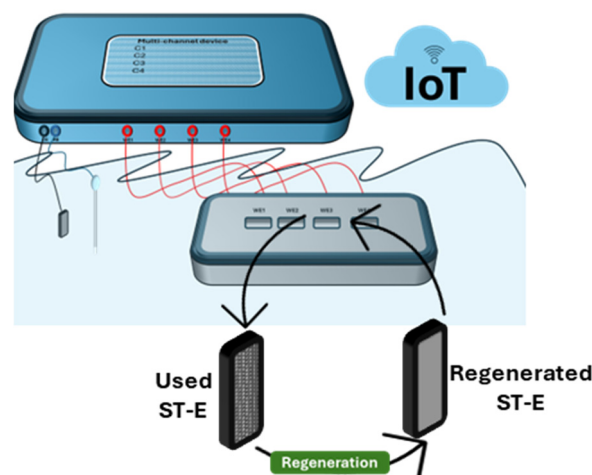


Figure 6. Example of ultimate front-end device.

Furthermore, endurance directly impacts consumables and measurement waste, thus impacting the overall sustainability of the implementation. A regeneration endurance test will be designed to quantify the long-term robustness of the ST-E reusable Pt platform under repeated use/clean/reuse cycles, in view of reducing the environmental impact in large-scale monitoring campaigns. The test will likely imply subjecting the electrode to N consecutive cycles, each including (i) a characterization phase, (ii) a representative functionalization and measurement phase, and (iii) a standardized regeneration sequence based on mechanical cleaning. Surface recovery will then be assessed using a redox probe via cyclic voltammetry, monitoring the i_{PA} . The operational “end of life” could be defined as the first cycle in which the regenerated electrode reduces its performance, in this case, the i_{PA} by 60%, thus developing a practical measurement of its lifecycle. Reusable electrochemical biosensing platforms can withstand multiple regeneration/reuse cycles before significant performance loss, with reported examples reaching up to 50 cycles depending on the regeneration strategy and device design [41]. Notably, however, this result is highly dependent on the emphasis on regeneration put on the design stage. Reusable biosensors designed with a dedicated refresh mechanism have demonstrated regeneration endurance exceeding 200 cycles [42]. High-cycle reuse is then feasible when regeneration is integrated at the device level, supporting the idea that a Pt platform, optimized for rapid and standardized mechanical regeneration, can offer high end-of-life cycles compared to conventional reuse attempts.

This work introduces a reusable Pt-on-silicon thin-film electrochemical platform specifically designed for environmentally friendly regeneration, refunctionalization, and electroanalysis. Unlike conventional single-use screen-printed transducers, the proposed ST-E platform combines the dimensional accuracy and manufacturability of Pt-on-wafer fabrication with a rapid surface renewal protocol, enabling the repeated recovery of a clean and electroactive Pt surface. The platform demonstrated stable diffusion-controlled voltam-

metry with reversible ferricyanide behavior and proved to be a versatile substrate for two different surface chemistries. First, after functionalization with APTES, it supported the immobilization of PFOA-selective nanoMIPs, enabling the determination of PFOA at trace levels in the range of 1–5 pg mL^{−1} with a limit of detection (LOD) of 0.50 pg mL^{−1} and good agreement with equivalently modified commercial SPE. Second, after regeneration, the same Pt surface was successfully reconfigured by electropolymerization with o-phenylenediamine, demonstrating repeatable refunctionalization beyond a single sensing chemistry. Compared to many electrochemical PFOA sensors in the literature, often based on disposable or non-regenerable architectures, the distinctive advantage of this strategy lies in the combination of pg mL^{−1} level analytical performance with a regenerable metal transducer. This makes the platform attractive not only for PFOA detection but also as a general reusable basis for electrochemical sensor development.

From a sustainability perspective, ST-E reduces solid waste, minimizes the consumption of disposable transducers, and is compatible with portable electrochemical instrumentation, achieving high scores in terms of environmental sustainability and practicality (AGREE = 0.80, AGREEprep = 0.80, BAGI = 75.0). Overall, ST-E should not be considered simply a disposable sensor but a circular, reconfigurable electroanalytical platform with the potential for extension to multichannel, multireceptor, and field applications. The AGREE, AGREEprep, and BAGI assessments were applied to the preparation and use of the final analytical sensor, the nanoMIP/APTES-modified ST-E sensor used for PFOA determination, and not to the industrial fabrication of the ST-E platform itself. Therefore, the preparation of the silicon wafer, the deposition of the titanium adhesion layer and the platinum film, as well as the associated energy and material requirements, were not included in the calculation of these indices. These aspects will be addressed in future studies. Future work will focus on comprehensive endurance testing over repeated sensing/regeneration cycles, extension to additional target analyses, and integration into portable multichannel architectures for simultaneous analysis.

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Abbreviations

The following abbreviations are used in this manuscript:

AGREE	Analytical GREENness Calculator
AGREEprep	Greenness Metric for Sample Preparation
APTES	(3-Aminopropyl)triethoxysilane
BAGI	Blue Applicability Grade Index

CE	Counter Electrode
CV	Cyclic Voltammetry
DPV	Differential Pulse Voltammetry
EDC	1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide
GAC	Green Analytical Chemistry
I_{pa}	Anodic Peak Current
I_{pc}	Cathodic Peak Current
LoA	Limits of Agreement
LOD	Limit of Detection
MIP	Molecularly Imprinted Polymer
nanoMIP	Molecularly Imprinted nanoparticles
NHS	N-Hydroxysuccinimide
oPD	o-Phenylenediamine
PBS	Phosphate-Buffered Saline
PFOA	Perfluorooctanoic Acid
PoPD	Poly(o-phenylenediamine)
Pt	Platinum
RE	Reference Electrode
SPE	Screen-Printed Electrode
ST-E	ST-Electrode
WE	Working Electrode
ΔE_p	Peak Potential Separation

Appendix A

Table A1. Summary of scan rate study. E_{pa} and E_{pc} values refer to the average potential at which the peaks fall.

v (mV s^{-1})	E_{pa} (V) *	E_{pc} (V) *	ΔE_p (V)	i_{pa}/i_{pc}
5	0.237	0.179	0.059	0.90
10	0.240	0.186	0.054	0.86
25	0.237	0.192	0.045	0.85
50	0.235	0.197	0.038	0.83
75	0.237	0.193	0.044	0.82
100	0.239	0.193	0.045	0.80
150	0.240	0.192	0.048	0.79

* SD < 0.005 ($n = 5$).

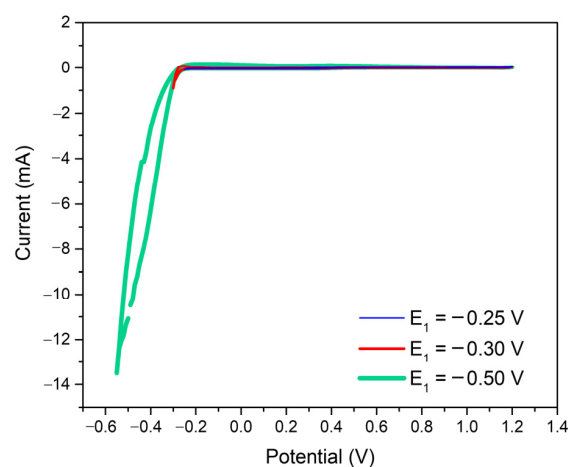
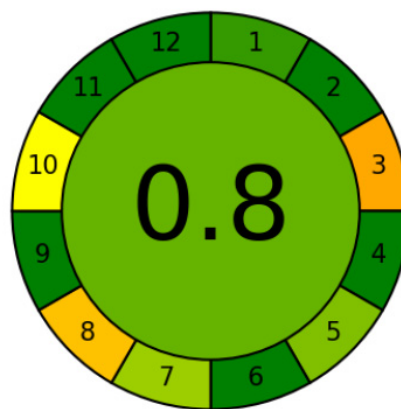


Figure A1. Complete CVs recorded during electrochemical activation of the ST-E platform in 0.5 M H_2SO_4 using different starting potentials. The scans were performed from $E_i = -0.50$, -0.30 , or -0.25 V to $E_f = +1.20$ V vs. Ag/AgCl at the same scan rate used for Figure 2a.

Analytical Greenness report sheet

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Criteria	Score	Weight
	0.9	2
2. Minimal sample size and minimal number of samples are goals.	1.0	2
3. If possible, measurements should be performed in situ.	0.33	2
4. Integration of analytical processes and operations saves energy and	1.0	2
5. Automated and miniaturized methods should be selected.	0.75	2
6. Derivatization should be avoided.	1.0	2
7. Generation of a large volume of analytical waste should be avoided, and	0.69	2
8. Multi-analyte or multi-parameter methods are preferred versus methods	0.38	2
9. The use of energy should be minimized.	1.0	2
10. Reagents obtained from renewable sources should be preferred.	0.5	2
11. Toxic reagents should be eliminated or replaced.	1.0	2
12. Operator's safety should be increased.	1.0	2

(a)

Figure A2. Cont.

AGREEprep

Analytical Greenness Metric
for Sample Preparation

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#	Criterion	Score Weight	
1.	Sample preparation placement Sample preparation placement: On site	0.33	1
2.	Hazardous materials Mass [g] or volume [mL] of problematic materials: 0.1	0.67	5
3.	Sustainability, renewability, and reusability of materials Materials are not sustainable or renewable, but are used SEVERAL TIMES	0.5	2
4.	Waste Mass [g] or volume [mL] of waste: 0.1	1.0	4
5.	Size economy of the sample Mass [g] or volume [mL] of the sample: 0.1	1.0	2
6.	Sample throughput Hourly sample throughput: 20	0.71	3
7.	Integration and automation No. of sample prep. steps: 2 steps or fewer; degree of automation: Semi-automated systems	0.5	2
8.	Energy consumption Approximate energy consumption per analysis [W]: 0	1.0	4
9.	Post-sample preparation configuration for analysis Spectrophotometry, surface analysis techniques, voltammetry, potentiometry, etc.	0.75	2
10.	Operator's safety No. of distinct hazards: No hazards or no exposure	1.0	3

(b)

Figure A2. Cont.

1. Type of analysis	Quantitative and confirmatory
2. Multi- or single-element analysis	Single Element
3. Analytical technique	Simple in operation portable instrumentation (smart-phone based detectors, portable GC, etc.)
4. Simultaneous sample preparation	2-12
5. Sample preparation	Not required or on-site sample preparation if required
6. Samples per h	>10
7. Reagents and materials	Need to be synthesized in the lab with common instrumentation and in a simple way
8. Preconcentration	No preconcentration required. Required sensitivity and /or legislation criteria are met directly.
9. Degree of automation	Manual treatment and analysis
10. Amount of sample	<100 µL (or mg) bioanalytical samples; <10 mL (or g) food/environmental

(c)

Figure A2. Report extracted from (a) AGREE, (b) AGREEprep and (c) BAGI software for ST-E.

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