

## Indirect electrochemical detection of perfluoro decanoic acid from water

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### Abstract

Electrochemical detection of perfluoro decanoic acid (PFDA) has gained significant interest due to its environmental persistence and potential health risks. This study focuses on the development and utilization of a molecular imprinted polymer (MIP) sensor for the selective and sensitive detection of PFDA in aqueous samples. Commercial boron-doped diamond (BDD) electrode was modified with MIP by electro polymerization in 2:1 (v/v) mixture of 0.1 M sodium acetate buffer solution (pH = 5.8) and methanol containing 10 mM *o*-phenylenediamine (*o*-PD) and 1 mM PFDA concentration. The electro polymerization process was carried out using cyclic voltammetry (CV) by scanning 25 cyclic voltammograms in a potential range of 0.00 → 1.00 V/SCE with a scan rate of 0.05 V·s<sup>-1</sup>. The electrochemical performance of the MIP-BDD sensor using cyclic voltammetry (CV), differential pulse voltammetry (DPV) and square wave voltammetry (SWV) was assessed. Considering both voltametric detections based on oxygen reduction reaction (ORR) and the lack of electrochemical activity of PFDA, investigation of its signal in the presence of increased PFDA concentrations is exploited for development of indirect detection method. Linear dependence between signal decrease vs. increasing PFDA concentrations allowed determining sensitivity and the lowest limit of detection of 0.160 nM (0.08 μg·dm<sup>-3</sup>) PFDA. The integration of MIP technology with electrochemical sensors offers a robust platform for environmental monitoring of PFDA, combining high selectivity, sensitivity and rapid response time. This method holds promise for advancing detection techniques for persistent organic pollutants in environmental and public health contexts.

**Keywords:** electro polymerization, electrochemical detection, molecular imprinted polymer, *o*-phenylenediamine, perfluoro decanoic acid

### INTRODUCTION

Per- and polyfluoroalkyl substances (PFAS), widely used since the 1940s, are known as “forever chemicals” due to the presence of the C-F bond, one of the strongest bonds in their molecular structure, which contributes to their persistence in the environment [1, 2]. Depending on their chemical characteristics, PFAS are widely used in household products such as food packaging [3, 4], the production of aqueous film-forming foams for firefighting [5, 6], metal plating [1], detergents, inks, semiconductors, lubricants [7], coating additives (for walls, furniture, and other surfaces) [8], surfactants [9], agricultural uses (pesticides), textiles, clothing [8÷10], commercial products, and cosmetics [11÷14]. The widespread use of PFAS has made them ubiquitous in the environment,

wildlife, and even in our bodies [15–17]. Released through human activities, these new emerging contaminants have become a serious global issue in recent decades due to their potential to affect the ecosystems and human health. A predominant application for PFAS is aqueous film-forming foam (AFFF) for firefighting, which is also among the most significant sources of PFAS contamination in drinking water (DW) and groundwater (GW) [18]. Epidemiological and toxicity studies have associated perfluoroalkyl compounds with harmful health effects, including liver and testicular cancer, elevated cholesterol levels, preeclampsia, and reduced vaccine responses in children [19].

PFDA belongs to PFAS family, has garnered considerable attention because of its environmental persistence and potential health hazards. As a persistent organic pollutant, PFDA is highly stable and resistant to degradation, posing substantial risks to both ecosystems and human health.

PFDA has been shown to be toxic to various aquatic organisms, including fish, amphibians, and invertebrates. Contaminated water sources can lead to human exposure to PFDA through drinking water, recreational activities, and consumption of contaminated food. Chronic exposure to PFDA is connected to harmful health effects, including liver damage, thyroid disorders, immune system disruptions, and a higher risk of specific cancers. Due to its chemical stability, PFDA is highly persistent in the environment with high mobility in water, leading to widespread contamination. This persistence makes it challenging to remove from water sources once contamination has occurred.

Given these harmful effects, it is crucial to develop and implement effective monitoring and remediation strategies to detect and mitigate PFDA contamination in water bodies, protecting both environmental and human health. As PFDA continues to be detected in various environmental matrices, the development of reliable and sensitive detection methods becomes crucial. Among the analytical methods available for detecting PFDA, liquid chromatography-tandem mass spectrometry (LC-MS/MS) coupled with solid-phase extraction (SPE) is the most commonly employed and dependable technique. This approach offers high selectivity and sensitivity for measuring PFAS in diverse environmental samples.

For the detection of 29 different PFAS including PFDA, LC-MS/MS has been officially approved by the United States Environmental Protection Agency (EPA) [20, 21]. However, this method is expensive, relies on sophisticated instruments, requires highly skilled users, and is unsuitable for applications outside of the laboratory. To overcome these drawbacks, rapid, simple, and sensitive methods suitable for on-site analyses are of increasing interest worldwide.

For these reasons, electrochemical sensors have been developed to address the disadvantages of combined chromatography and mass spectrometry approaches [22, 23]. Electrochemical detection has emerged as a promising approach for identifying and quantifying PFDA. This method leverages the principles of electrochemistry to offer high sensitivity, selectivity, and rapid response times. Techniques such as voltammetry and amperometry have been extensively studied and optimized to enhance detection capabilities. The beneficial characteristics of electroanalytical methods and sensors have motivated many researchers to explore and design new electrochemical approaches for the detection of PFAS. Modified electrodes, incorporating nanomaterials or conductive polymers, have shown significant improvements in detection limits and accuracy. MIPs are polymers that have been synthesized around template molecules, so as to create a cavity for the selective recognition of that template or a similar structure. They are used to modify the electrodes to detect the particular analyte for the development of advanced electrochemical sensors. MIP is designed to provide the recognition capability of the sensor, having within the polymer matrix specific cavities that bind to the target analyte (PFDA).

In recent years, considerable efforts have been invested in developing electrochemical sensors for the indirect detection of non-electroactive target pollutants, such as organic environmental contaminants. Electrochemical detection of PFAS through direct oxidation or reduction of PFAS is generally not feasible due to their stability under oxidative or reductive conditions. Although such direct electrochemistry has been studied primarily for the destruction of PFAS [24, 25], the electrochemical behavior is not sufficiently selective or sensitive to warrant its use in electroanalytical methods. However, strategies such as specific adaptation of electrode surfaces (MIP) combined with the use of redox-active indicators e.g. ferrocene carboxylic acid (FcCOOH) that serves as signal generation

through its oxidation from the surface of the electrode., as well as exploiting the ionizable and interfacial properties of PFAS, have been reported as indirect detection [26]. Thus, it can be stated that the use of electrochemical sensors based on carbon modified with MIP is of particular interest in electroanalytical applications due to the flexibility in functional design, high sensitivity, mechanical resistance, low cost and ease of operation.

This work aim was to develop MIP-based strategy for indirect detection of PFDA using BDD electrode as substrate electrode material. Oxygen-reduction reaction (ORR) that is commonly for aqueous solution based on dissolved oxygen, is considered as electroactive process. The effect of PFDA on ORR at MIP-modified BDD electrode is studied using CV, DPV and SWV.

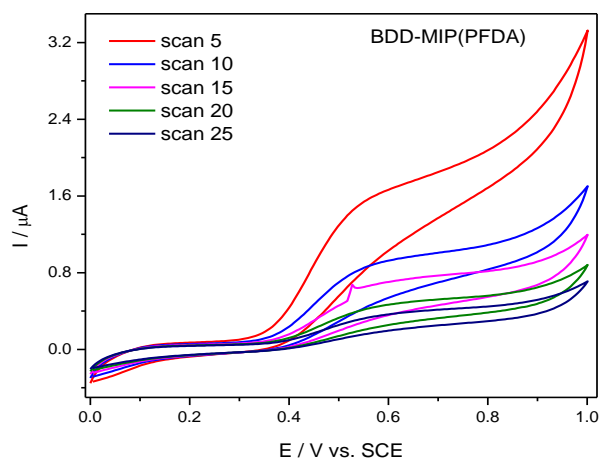
## EXPERIMENTAL PART

### *Materials and reagents*

Electrochemical experiments were carried out using an Autolab PGSTAT 302 potentiostat / galvanostat, operated via GPES 4.9 software and a three-electrodes based cell. The cell configuration included a BDD working electrode, a platinum (Pt) counter electrode, and a saturated calomel electrode (SCE) as the reference. Reagents, such as: sodium acetate ( $\text{CH}_3\text{COONa}$ , >99.5%), o-phenylenediamine (o-PD,  $\geq 98\%$ ), methanol ( $\text{CH}_3\text{OH}$ ,  $\geq 99\%$ ), glacial acetic acid, and PFDA were used as received from obtained from Sigma-Aldrich. Solutions were prepared using ultrapure water. The BDD electrode (2.5 mm diameter) and the SCE electrode (stored in 1 M KCl) were sourced from Metrohm Romania.

### *Fabrication of the MIP electrode*

BDD substrate was modified with MIP (PFDA) through the electro polymerization process. In the first stage, the surface of the electrode with the role of electrochemical sensor was modified by the molecular printing process using the target analyte (PFDA). The electro polymerization process was carried out using the electrochemical technique of CV by recording 25 cyclic voltammograms in a potential range of  $0 \rightarrow 1$  V with a scan rate of  $0.05 \text{ V s}^{-1}$  in a 2:1 (v/v) mixture of 0.1 M  $\text{CH}_3\text{-COONa}^+$  buffer solution (pH = 5.8) and  $\text{CH}_3\text{-OH}$  with 10 mM o-PD and 1 mM PFDA (target analyte) (fig. 1).



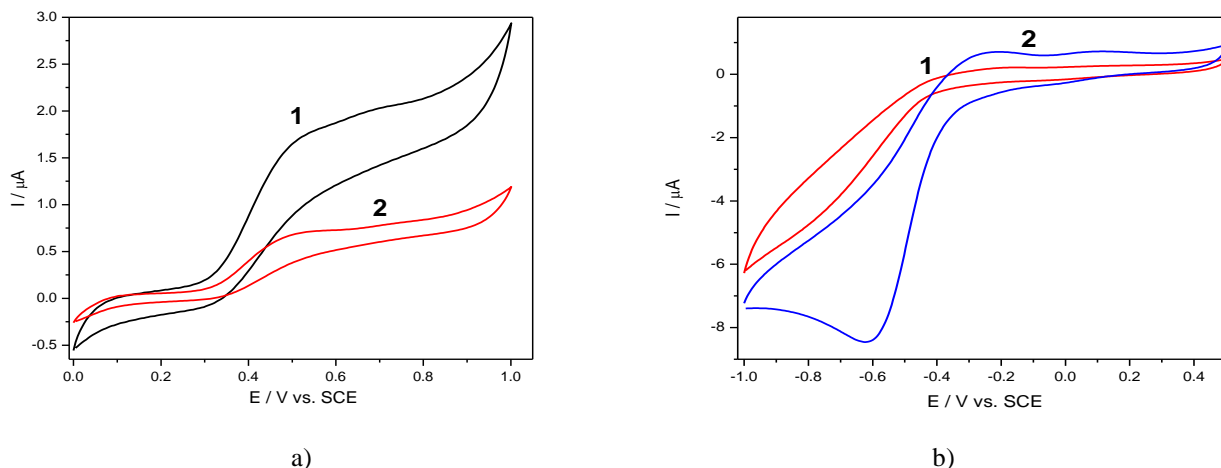
**Fig. 1.** Cyclic voltammograms recorded in 2:1 (v/v) mixture of 0.1 M  $\text{CH}_3\text{-COONa}^+$  and  $\text{CH}_3\text{-OH}$  containing 10 mM o-PD and 1 mM PFDA using the BDD electrode during electro polymerization process of BDD-MIP (PFDA), within the potential range of  $0 \rightarrow 1$  V/SCE, at the scan rate  $0.05 \text{ V} \cdot \text{s}^{-1}$

The modified BDD-MIP (PFDA) electrode was then immersed in a water/ methanol solution, in a ratio 1:1, under gentle mixing for 10 minutes, then rinsed with distilled water to remove PFDA from polymer template and dried at room temperature resulting as namely MIP(PFDA)-BDD electrode. In the second stage of voltametric detection, the supporting electrolyte solution used for the electrochemical detection of PFDA was 0.1 M  $\text{CH}_3\text{-COONa}^+$  and  $\text{CH}_3\text{-OH}$  in a ratio of 2:1. The electrochemical techniques used in the development of detection protocols for PFDA were CV, DPV, and SWV.

## RESULTS AND DISCUSSION

### *BDD modification with MIP by CV*

The electrochemical behavior of the unmodified BDD electrode and modified by the electro polymerization process at a scan rate of  $0.2 \text{ V}\cdot\text{s}^{-1}$  [26] and  $0.05 \text{ V}\cdot\text{s}^{-1}$  was studied by CV in the potential range between  $-1 \text{ V/ESC} \rightarrow +0.5 \text{ V/ESC}$ . The results are illustrated in figure 2 (a, b).

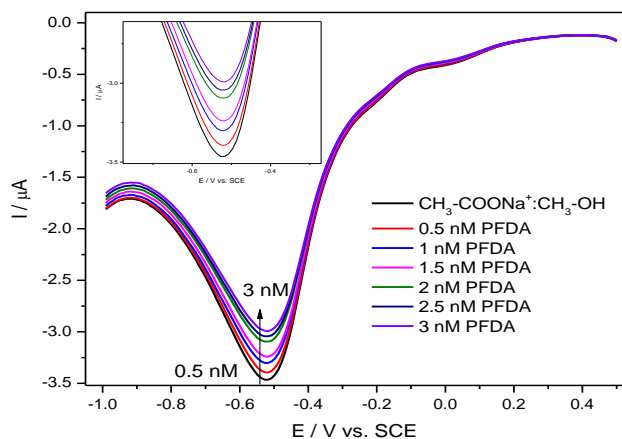


**Fig. 2.** 25 th cycle of the CVs recorded: a) in the electro polymerization process of BDD at scan rates  $0.2 \text{ V}\cdot\text{s}^{-1}$  (curve 1) and  $0.05 \text{ V}\cdot\text{s}^{-1}$  (curve 2); b) in  $\text{CH}_3\text{-COONa}^+ : \text{CH}_3\text{-OH}$  (2:1) at the scan rates  $0.05 \text{ V}\cdot\text{s}^{-1}$  with BDD-MIP electrodes electropolymerized at: at scan rates  $0.2 \text{ V}\cdot\text{s}^{-1}$  (1) and  $0.05 \text{ V}\cdot\text{s}^{-1}$  (2)

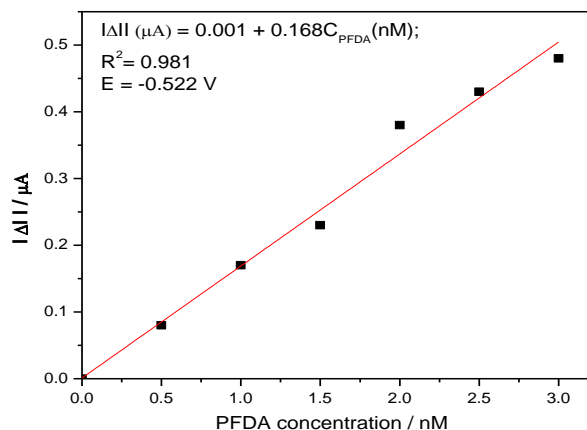
The results presented in figure 2 b) showed clearer CV shapes and a depolarization effect of oxygen reduction reaction (ORR) for BDD-MIP electropolymerized at  $0.05 \text{ V}\cdot\text{s}^{-1}$  compared to  $0.2 \text{ V}\cdot\text{s}^{-1}$ . The results of electropolymerization process after 25 scans expressed by CV recorded in supporting electrolyte for detection are presented in Figure 2. a) for  $0.05 \text{ V}\cdot\text{s}^{-1}$  and  $0.2 \text{ V}\cdot\text{s}^{-1}$  that was selected based on the literature data [26].

### *Indirect voltametric detection of PFDA with MIP (PFDA)-BDD electrode. Differential pulse voltammetry (DPV)/ Square wave voltammetry (SWV) studies*

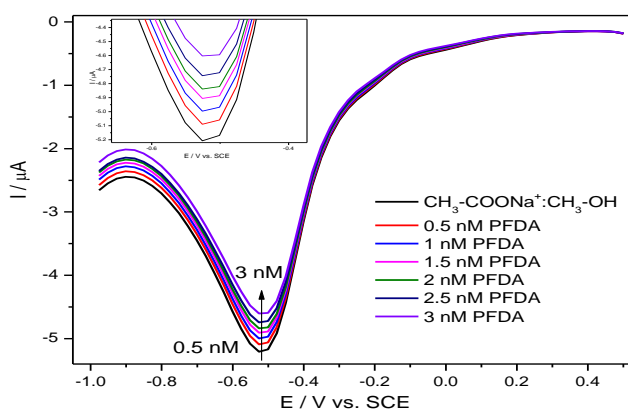
Considering the inert characteristics related to the electrochemical conductivity of PFDA, its effect of mitigating the oxygen reduction reaction ORR current was considered using PFDA incorporated within molecular imprinted polymer modified BDD electrode to develop indirect detection of PFDA by reduction of electrochemical signal corresponded to ORR reaction. Regarding the advantages of DPV and SWV techniques in minimizing the capacitive component of the background current, their applicability for detecting PFDA was evaluated under various operating conditions. The goal was to achieve the best sensitivity and the lowest detection limit using a supporting electrolyte consisted of 2:1 (v/v) mixture of  $0.1 \text{ M CH}_3\text{-COONa}^+$  and  $\text{CH}_3\text{-OH}$  with  $10 \text{ mM o-PD}$  and  $1 \text{ mM PFDA}$ . For the DPV technique, the selection of modulation amplitude (MA), step potential (SP), and scan rate (v) was guided by the need for both electrode stability and reproducibility, alongside superior electroanalytical performance. MA values of  $100 \text{ mV}$  and  $200 \text{ mV}$  were tested, while SP varied between  $10 \text{ mV}$  and  $50 \text{ mV}$ . The results obtained are illustrated in figures 3÷6.



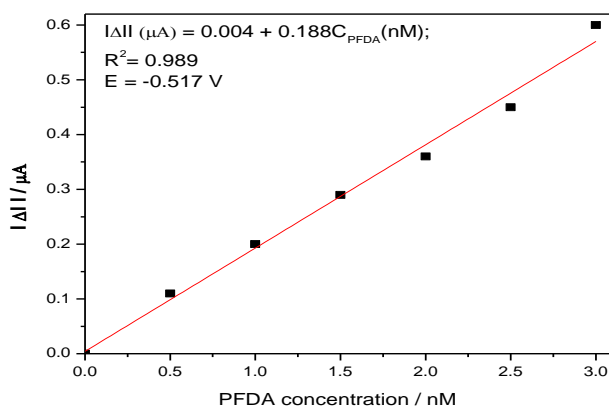
**Fig. 3. a)** The differential pulse voltammograms recorded under the conditions of AM 100 mV, SP 10 mV,  $\nu$  0.02 V·s<sup>-1</sup> with the BDD-MIP electrode in 0.1 M CH<sub>3</sub>-COONa<sup>+</sup>:CH<sub>3</sub>-OH supporting electrolyte and in the presence of 0.5-3 nM PFDA concentrations; potential range: -1V → +0.5V / SCE.



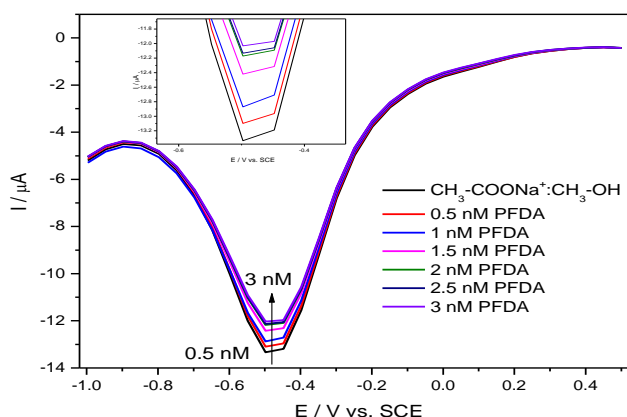
**Fig. 3. b)** The calibration curve of the current intensity corresponding to the detection peak at E = -0.522V/SCE vs. the concentration of PFDA.



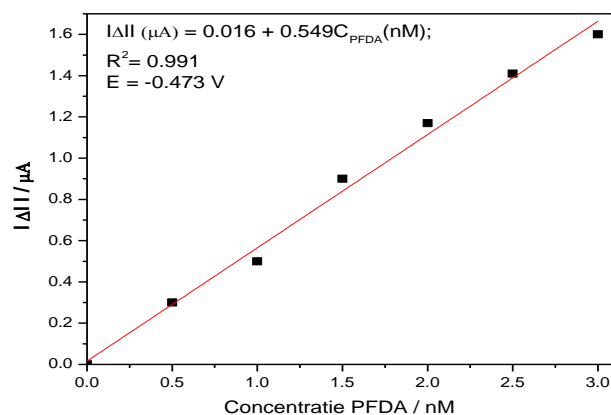
**Fig. 4. a)** The differential pulse voltammograms recorded under the conditions of AM 100 mV, SP 25 mV,  $\nu$  0.05 V·s<sup>-1</sup> with the BDD-MIP electrode in 0.1 M CH<sub>3</sub>-COONa<sup>+</sup>:CH<sub>3</sub>-OH supporting electrolyte and in the presence of 0.5-3 nM PFDA concentrations; potential range: -1V → +0.5V / SCE.



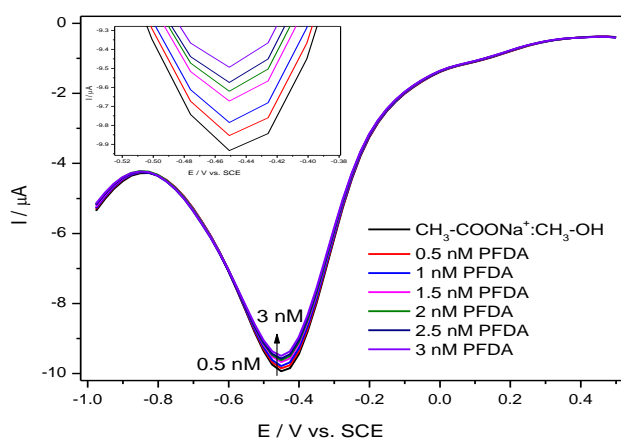
**Fig. 4. b)** The calibration curve of the current intensity corresponding to the detection peak at E = -0.517V/SCE vs. the concentration of PFDA.



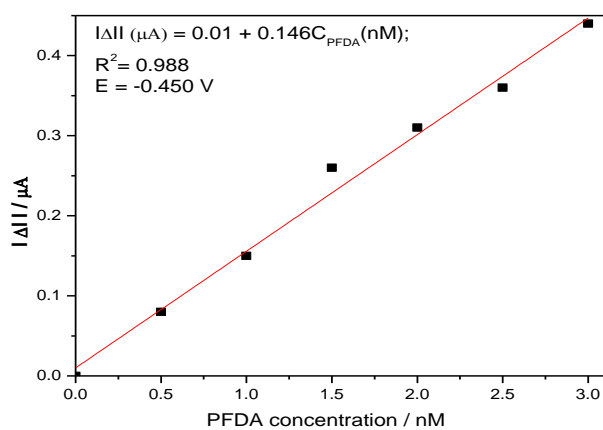
**Fig. 5. a)** The differential pulse voltammograms recorded under the conditions of AM 200 mV, SP 50 mV,  $\nu$  0.1 V·s<sup>-1</sup> with the BDD-MIP electrode in 0.1 M CH<sub>3</sub>-COONa<sup>+</sup>:CH<sub>3</sub>-OH supporting electrolyte and in the presence of 0.5-3 nM PFDA concentrations; potential range: -1V → +0.5V / SCE.



**Fig. 5. b)** The calibration curve of the current intensity corresponding to the detection peak at E = -0.473 V/SCE vs. the concentration of PFDA.

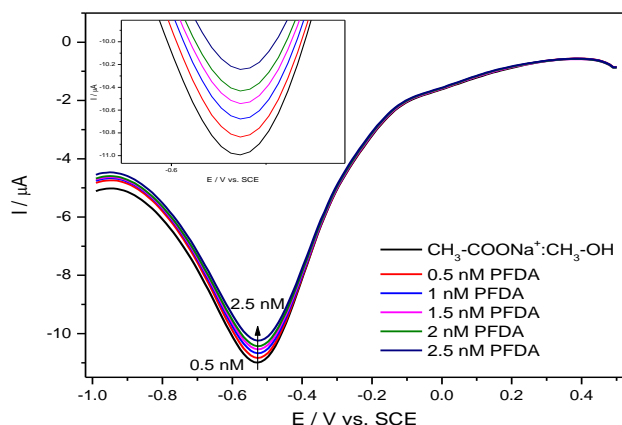


**Fig. 6. a)** The differential pulse voltammograms recorded under the conditions of AM 200 mV, SP 25 mV,  $\nu$  0.05 V·s<sup>-1</sup> with the BDD-MIP electrode in 0.1 M CH<sub>3</sub>-COONa<sup>+</sup>:CH<sub>3</sub>-OH supporting electrolyte and in the presence of 0.5-3 nM PFDA concentrations; potential range: -1V → +0.5V / SCE.

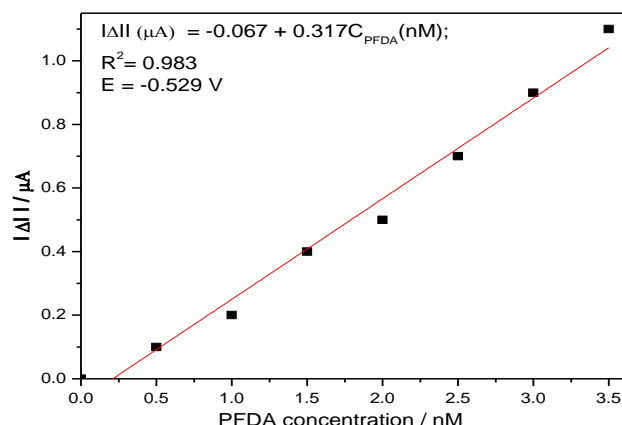


**Fig. 6. b)** The calibration curve of the current intensity corresponding to the detection peak at E = -0.450 V/SCE vs. the concentration of PFDA.

In addition, SWV technique is proposed for testing considering its main advantage of high effective scan rate thus reducing the analysing time. Figure 7 a) shows the square wave voltammograms recorded under conditions of MA 100 mV, SP 10 mV, f 10 Hz,  $\nu$  0.02 V·s<sup>-1</sup>, and calibration curve of the current vs. PFDA concentration is given in figure 7 b).



**Fig. 7. a)** The square-wave voltammograms recorded under the conditions of MA 100 mV, SP 10 mV, f 10 Hz,  $\nu$  0.02 V·s<sup>-1</sup> with the BDD-MIP electrode in supporting electrolyte 0.1 M CH<sub>3</sub>-COONa<sup>+</sup>:CH<sub>3</sub>-OH and in the presence of 0.5-3 nM PFDA concentrations; potential range: -1V → +0.5V / SCE.



**Fig. 7. b)** The calibration curve of the current intensity corresponding to the detection peak at E = -0.529 V/SCE vs. the concentration of PFDA.

The electroanalytical parameters determined for each technique and the operating conditions applied are presented in table 1.

The relative standard deviation (RSD), detection limit (LOD), and quantification limit (LOQ) were calculated for three replicates using the following equations [27]:"

$$\text{RSD} = S / \bar{x} \quad (1)$$

$$\text{LOD} = 3 \times S/m \quad (2)$$

$$\text{LOQ} = 10 \times S/m \quad (3)$$

where:  $\bar{x}$  represents the mean value of the replicates, S is the standard deviation of the replicates, and m is the sensitivity coefficient obtained from the analysis.

**Table 1.** Electroanalytical parameters obtained by DPV and SWV

Technique	Operating parameters			Electroanalytical parameters				
	SP / mV	MA / mV	$\nu$ / V·s <sup>-1</sup>	E (V/SCE)	Sensitivity (μA·nM <sup>-1</sup> )	LOD (nM)	LQ (nM)	RSD (%)
DPV	10	100	0.02	-0.522	0.168	0.273	0.909	0.439
	<b>25</b>	<b>100</b>	<b>0.05</b>	<b>-0.517</b>	<b>0.188</b>	<b>0.160</b>	<b>0.532</b>	<b>0.192</b>
	25	200	0.05	-0.450	0.146	0.616	2.05	<b>0.302</b>
SWV	50	200	0.1	-0.473	0.549	0.546	1.82	0.746
	10	100	0.02 (10Hz)	-0.529	0.317	0.473	1.58	0.453

It was observed that the lowest LOD (0.160 nM) was obtained by DPV at SP 25 mV, MA 100 mV and  $\nu$  0.05 V·s<sup>-1</sup>. This LOD is much better in comparison with one reported in literature data [28].

## CONCLUSIONS

The present research study allowed the development of voltametric detection procedures of PFDA from the PFAS class using the BDD electrode modified with MIP through the electro polymerization process. The ORR on the MIP (PFDA)-BDD electrode was considered for the development of the indirect voltametric detection process of PFDA using the two DPV and SWV techniques. In the optimum conditions of obtaining the MIP (PFDA)-BDD electrode for PFDA and the optimization of the operating conditions for detection using DPV and SWV in tandem with the LOD of 0.160 nM (0.08 μg·dm<sup>-3</sup>), which is bellow MAC, conferring a practical applicability of the method.

## ACKNOWLEDGEMENTS

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