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Palacký University Olomouc

Optimizing Flow Injection Systems for Electrochemical Sensing of Pesticides

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Flow Injection Technique

The first definition, given by Ruzicka and Hansen 1975 was "A method based on injection of a liquid sample into a moving un segmented continuous stream of a suitable liquid."

The injected sample forms a zone, which is then transported toward a detector

The analysis of the Flow injection depends on several factors, including

- Injection of samples
- Dispersion
- The time necessary to collect events in each cycle

Continuous Flow Systems

Reverse Flow Injection Analysis (r FIA)

Stopped Flow Injection Analysis (s FIA)

Sequential Injection Analysis (SIA)

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Optimizing Flow Injection Systems for Electrochemical Sensing of Pesticides

Advantage of FIA

- On-Site and Real-Time Monitoring
- Automation
- High Throughput
- Cost-Effective
- Rapid Analysis
- Minimized Sample and Reagent Consumption
- Increased precision compared to batch methodologies
- High Sensitivity and Selectivity

Application of FIA

Typical applications of flow injection analysis include the following fields:

Environmental analysis

- **River/Sea water**
- Waste water
- Sediments

Pharmaceutical application

Food analysis

Clinical assay

Bio analytical chemistry

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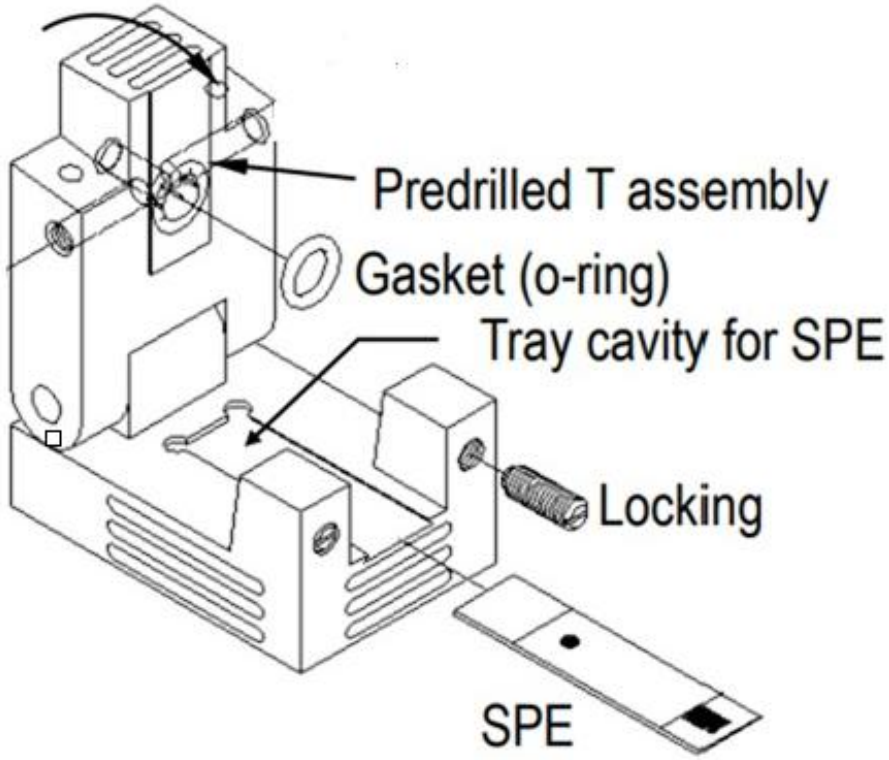
MINISTRY OF EDUCATION, YOUTH AND SPORTS

Flow Injection Cell



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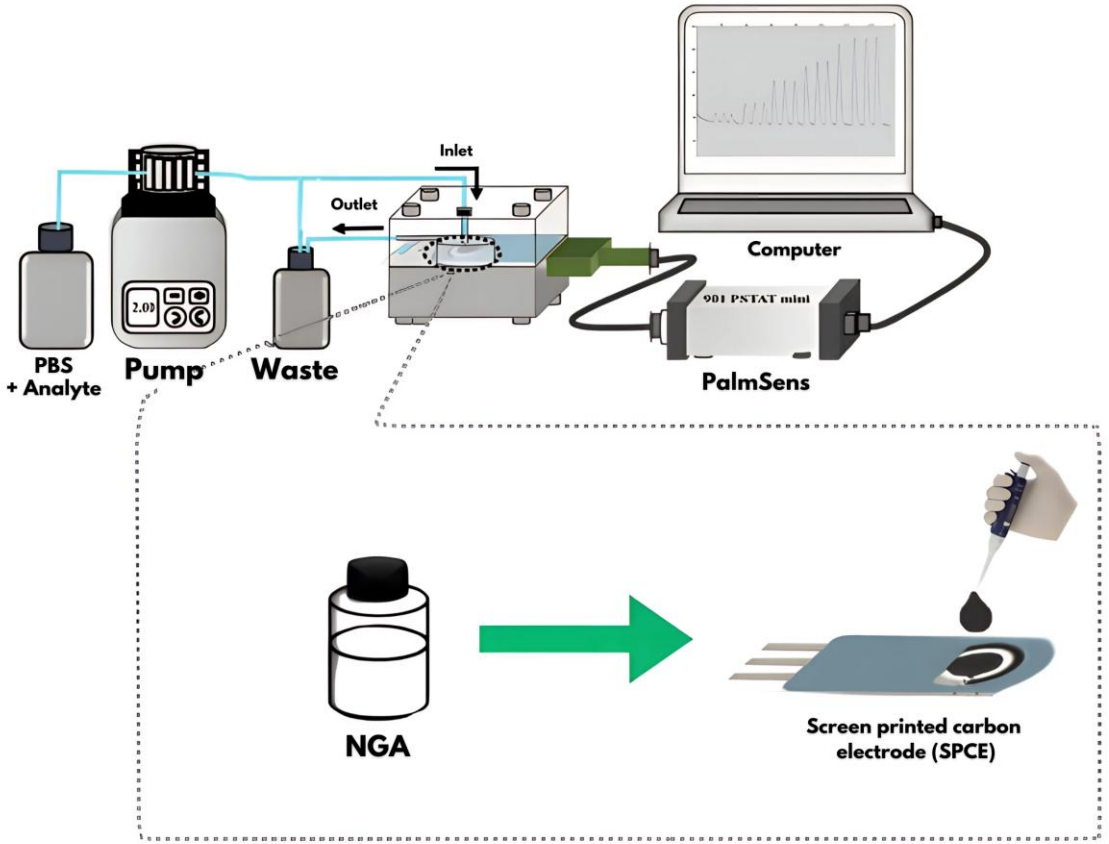
Flow Injection System

- ❑ NGA/SPCE
- ❑ rGO@Ni

- ❖ $[Fe(CN)_6]^{3-/4}$ solution
- ❖ Pesticide solution

- ❖ Chronoamperometry
- ❖ Differential pulse voltammetry



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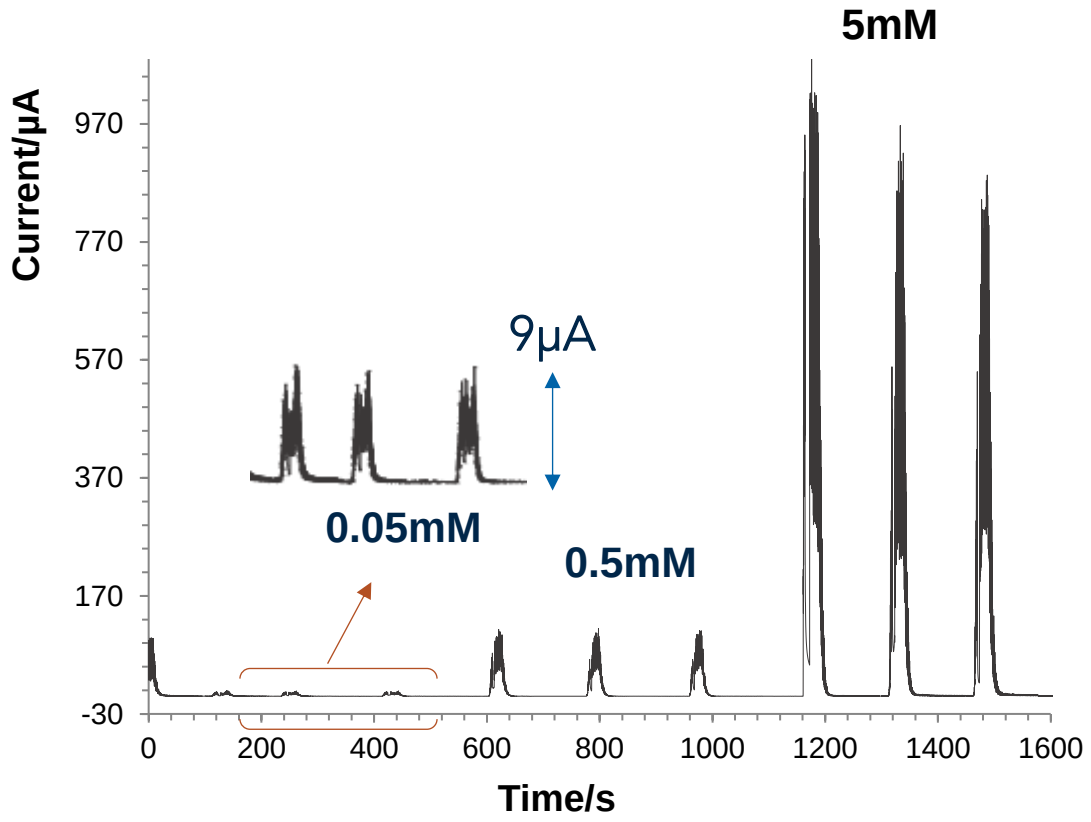
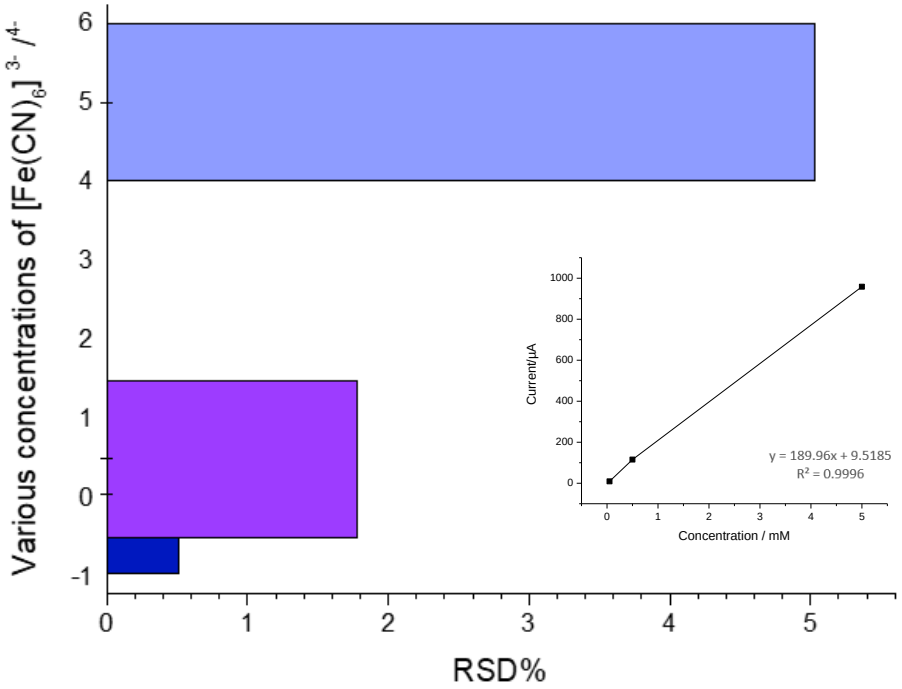
Flow Injection System Performance Testing



FIA responses of NGA/SPCE in various concentrations of $[Fe(CN)_6]^{3-/4-}$



Test Setup

- ❑ *Strong Linearity*
- ❑ *High Reliability and Reproducibility*
- ❑ *Fast Operation*

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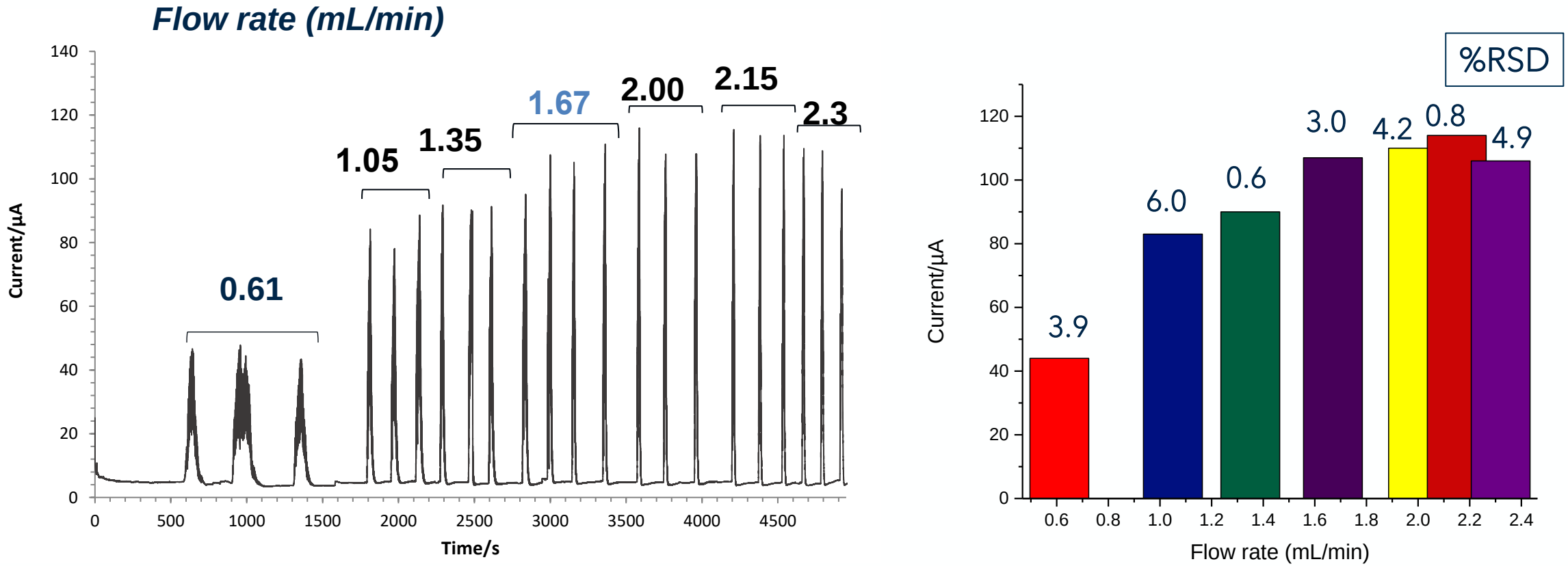
Optimizing the Flow Injection Setup



FIA responses of NGA/SPCE at various flow rates in 5mM $[Fe(CN)_6]^{3-/4-}$

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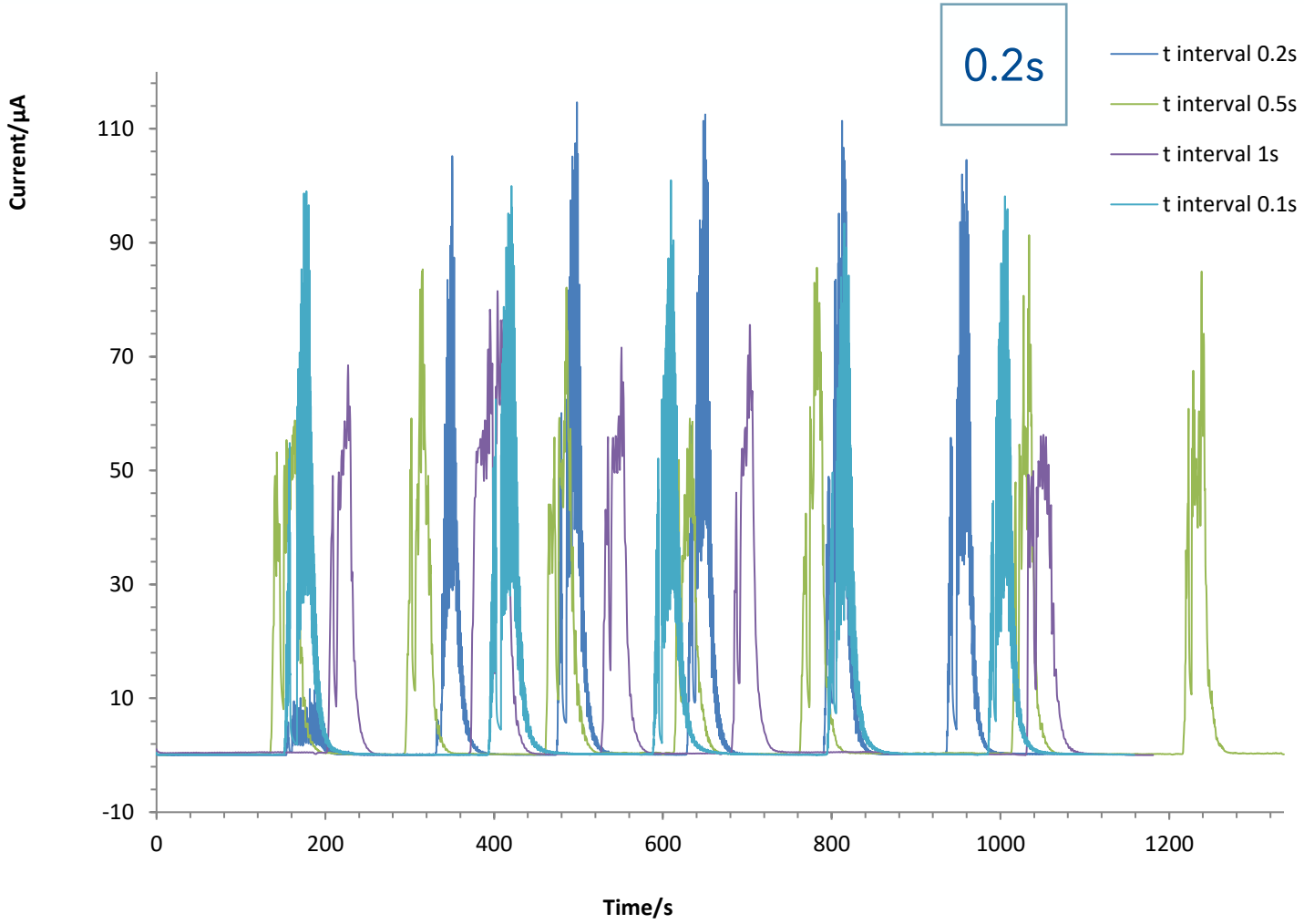
Optimizing Flow Injection Systems for Electrochemical Sensing of Pesticides

Optimizing the Flow Injection Setup

- Signal Stability
- Reaction Kinetics
- Sample Resolution
- Minimizing Carry-Over
- System Throughput



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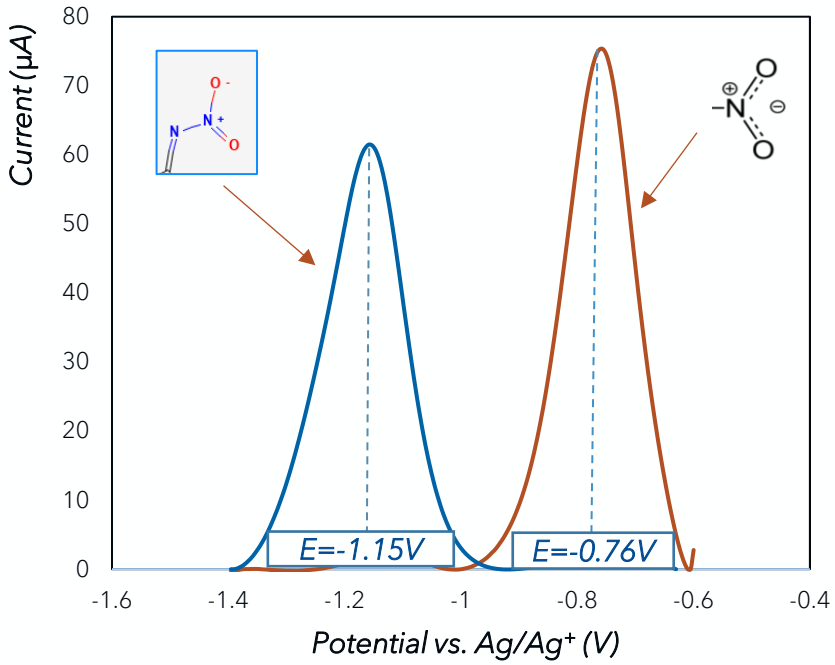
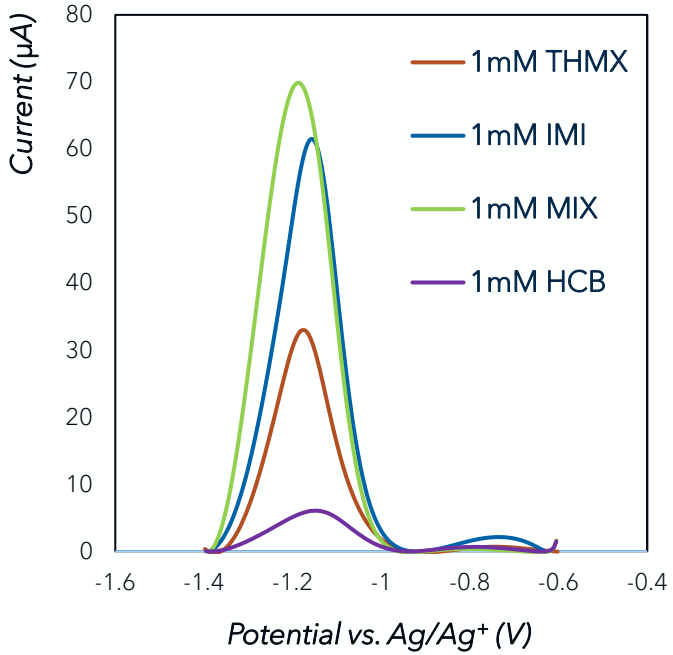
NGA/SPCE in Pesticide Detection



AdSWVs of NGA/SPCE, in 1mM Imidacloprid (blue curve) and 1mM nitrobenzene (orange curve)



AdSWVs voltammograms in 0.1M PBS pH=7: Mix solution of THMX and IMI (green curve), THMX (orange curve), IMI (blue curve) and HCB (purple curve, at) NGA/SPCE

Pesticides	Spiked Concentration	Founded Concentration	Recovery	RSD%
Imidacloprid IMI	0.1mM	0.10142mM	101.42 %	5.33 %
Imidacloprid IMI	1mM	0.8561 mM	85.61 %	1.18 %
Thiamethoxam THMX	0.1mM	N. D	-	-
Thiamethoxam THMX	1mM	0.9576 mM	95.76 %	2.62 %

The recovery of spiked IMI and THMX with Langarica River matrix

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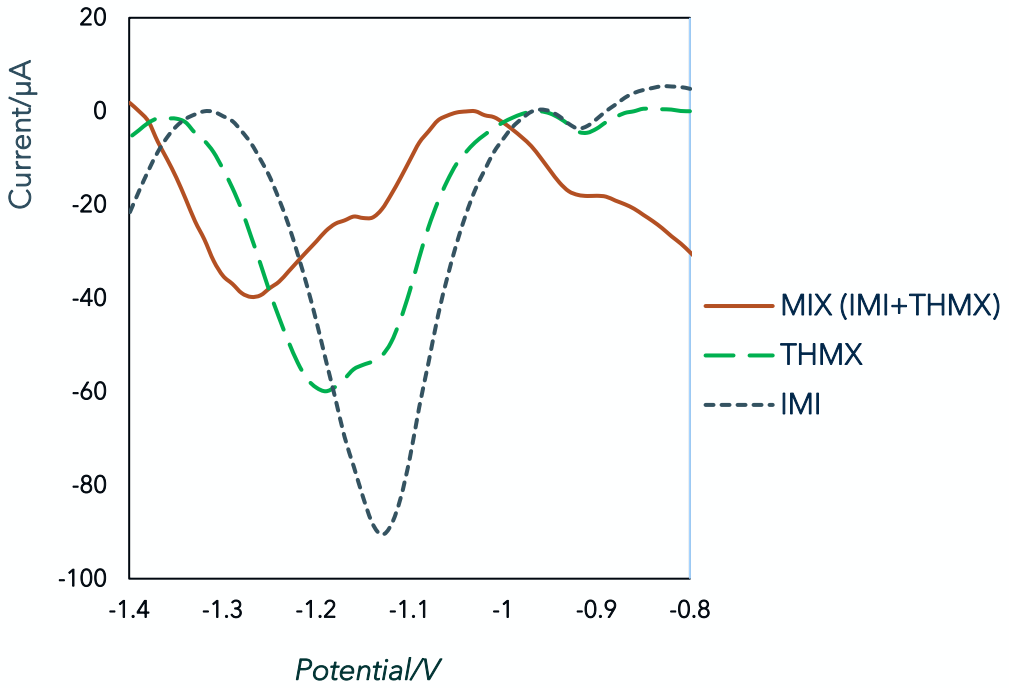
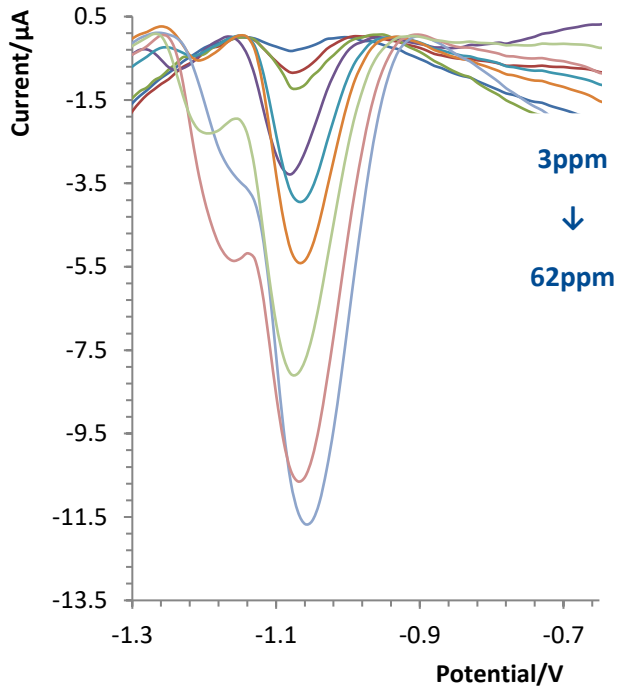
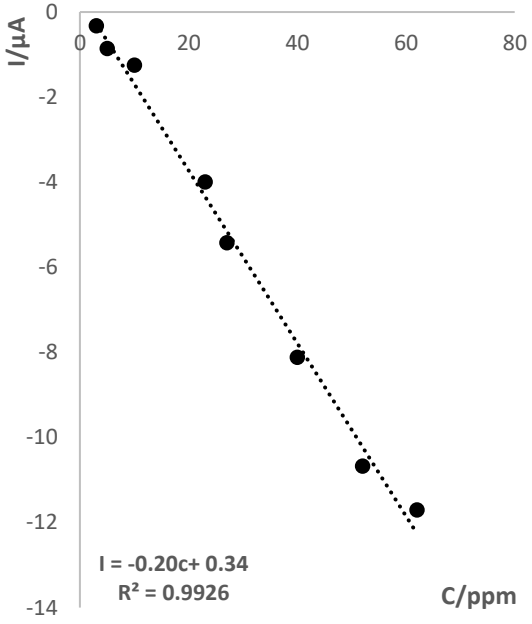
Pesticide Sensing with Ni@rGO



DPV voltammograms in 0.1M PBS pH=6.7: Mix solution of THMX and IMI (orange curve), THMX (green curve) and IMI (grey curve) Ni@rGO/CPE





Differential pulse voltammetry responses to increasing concentration of imidacloprid (left) with corresponding linear regression (right), in phosphate buffer saline (PBS), pH=7 with rGO@Ni laser scribed electrodes

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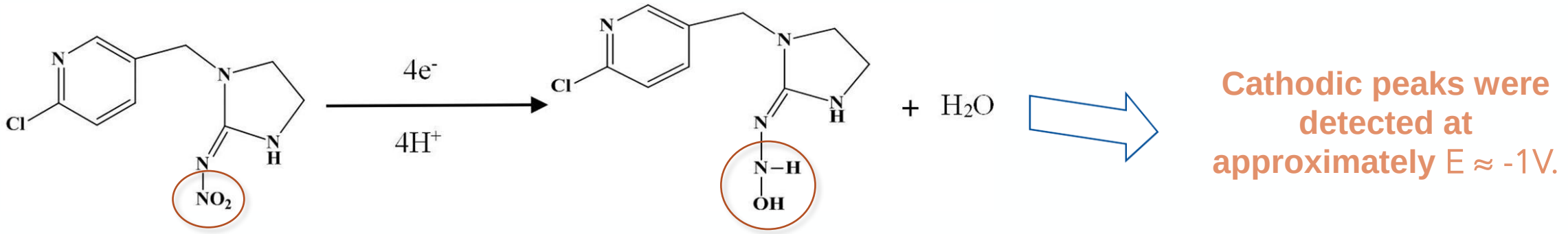
Optimizing Flow Injection Systems for Electrochemical Sensing of Pesticides

Pesticide Electrochemical Sensing



Cathodic peaks were detected at approximately E ≈ -1V.

Electrochemical Reduction Pathway of Imidacloprid: Suggested Mechanism

FIA and chronoamperometry in pesticide electrochemical detection

I. *Impact of Negative Potential on the Flow System*
- ❖ Hydrogen Evolution: Form of hydrogen gas
- ❖ Interaction with Flow: Gas bubbles form
- ❖ Turbulence and Flow Instabilities

II. *Impact of Continuous Flow on Negative Potential*
- ❖ Mass Transport Effects
- ❖ Concentration Gradients

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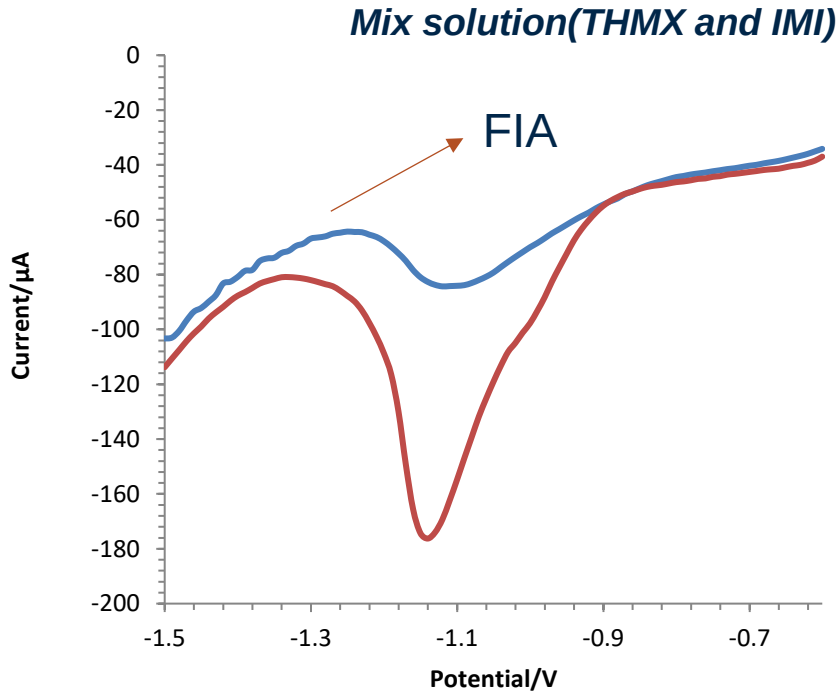
Pesticide Electrochemical Sensing



DPV voltammograms in 0.1M PBS pH=6.7 and mix solution of THMX and IMI: FIA (blue curve) and drop casting (red curve)

Solutions to Minimize Noise & Improve Signal

- **Optimize Flow Rate**
- **Handle Gas Evolution (Hydrogen bubbles)**
- **Hydrolysis of Imidacloprid (Imidacloprid degradation)**
- **Electrolyte and Buffering**

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