ECSA-12 2025 Conference
The 12th International Electronic Conference on Sensors and Applications
12–14 November 2025 | Online
MDPI

VOLTAMMETRIC DETECTION OF THIOCTIC ACID USING INORGANIC-ORGANIC COMPOSITE MATERIAL

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INTRODUCTION & AIM

In this work, we have developed a sensing platform containing a hybrid material composed of an inorganic redox mediator, namely Prussian Blue (PB), and an organic polymer like poly(3,4-ethylenedioxythiophene), PEDOT, for thioctic acid detection. The hybrid material is prepared onto glassy carbon electrode (GCE) using alternate current protocol. This protocol consists in the application of an excitation sinusoidal current having defined frequency and amplitude over a dc constant current [1,2]. The sensing platform was characterized by cyclic voltammetry and electrochemical impedance spectroscopy revealing the electrochemical activity of the PB component in aqueous solutions containing potassium ions. The PEDOT-PB sensing platform was applied successfully in the detection of thioctic acid in synthetic and real samples with good accuracy.

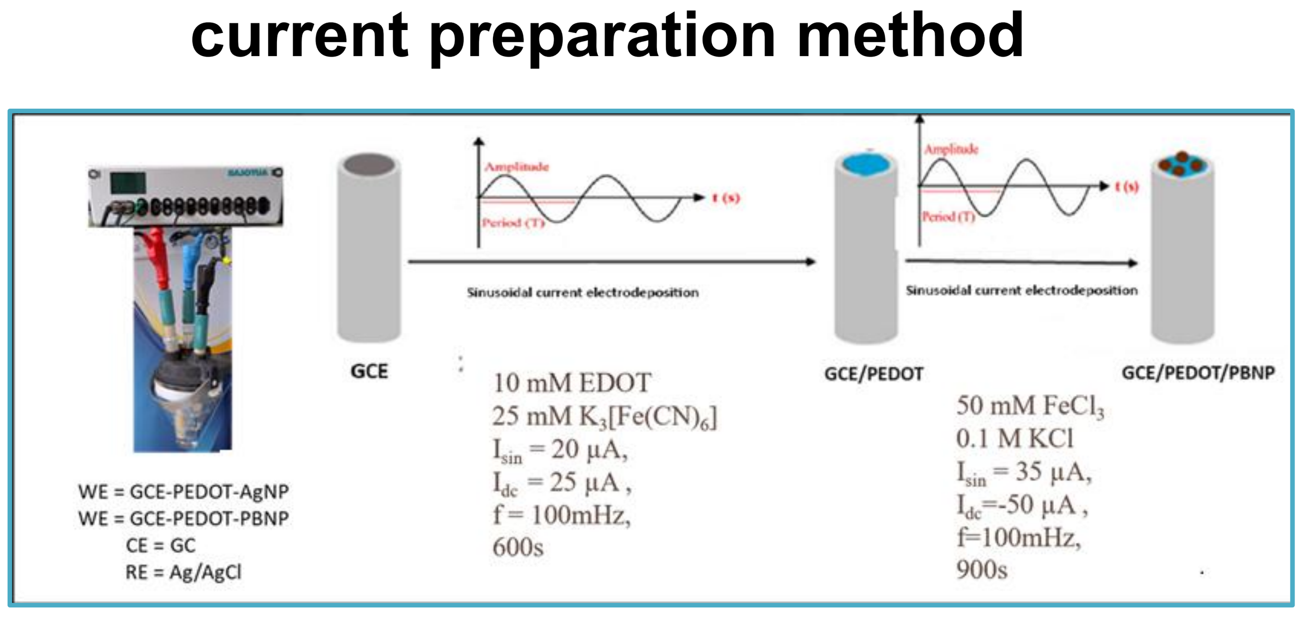
METHOD

Method description: The hybrid sensing material has been electrochemically synthesized in two steps: firstly, the organic polymer is prepared in the presence of ferricyanide ions. In the second step, the ferricyanide-doped polymer layer is subjected to the in situ electrochemical reduction of Fe(III) via the alternate current protocol. This approach ensured the in-situ formation of the PB inorganic redox mediator within the organic matrix. The PEDOT-PB based sensing platform was characterized by cyclic voltammetry and electrochemical impedance spectroscopy techniques.

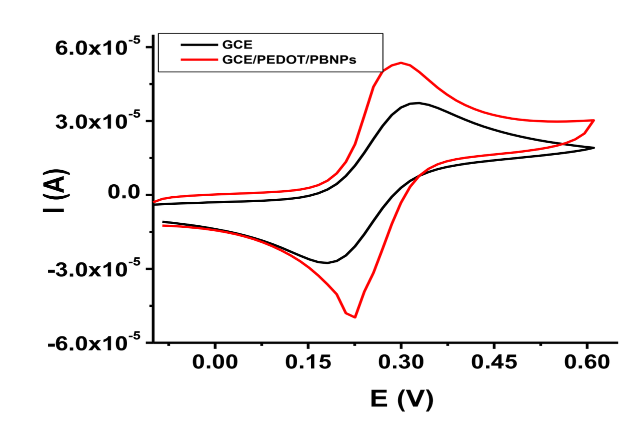
Apparatus and materials:

- potentiostat/galvanostat Autolab 302N (Ecochemie, Utrecht);
- working electrode: glassy carbon disk (Metrohm);
- reference electrode: Ag/AgCl/KCl 3M (Metrohm);
- counter electrode: glassy carbon rod (Metrohm)
- undivided glass electrochemical cell (Metrohm).

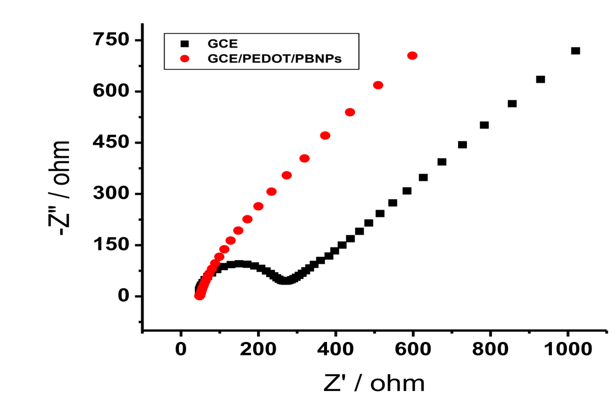
The apparatus and the principle of the alternate current preparation method



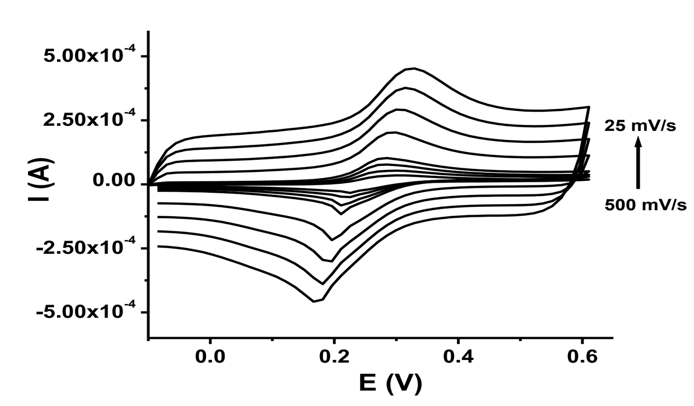
RESULTS & DISCUSSION



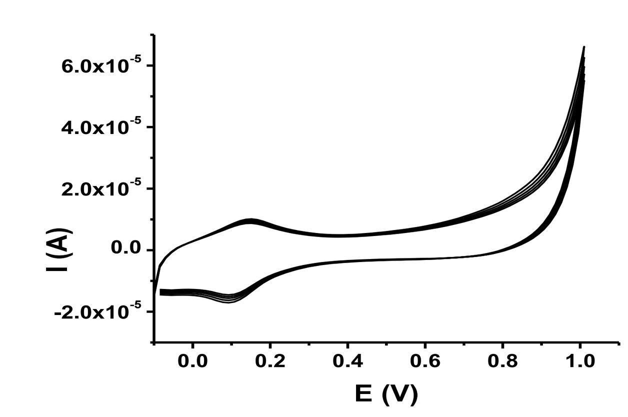
CVs recorded at GCE and GCE/PEDOT-PB electrodes in 0.5 M KNO_3 containing 5 mM $Na_4[Fe(CN)_6]$; v = 50 mV/s.



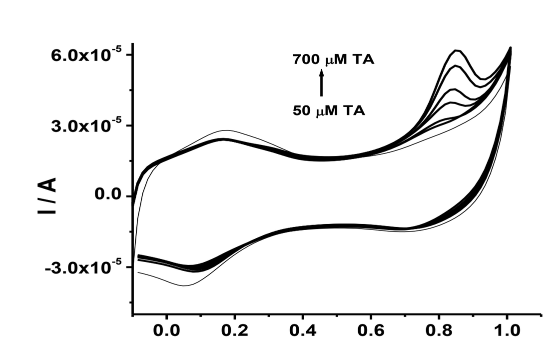
EIS spectra recorded at GCE and GCE/PEDOT-PB in 0.5 M KNO_3 containing 5 mM $K_3[Fe(CN)_6]$/$Na_4[Fe(CN)_6]$



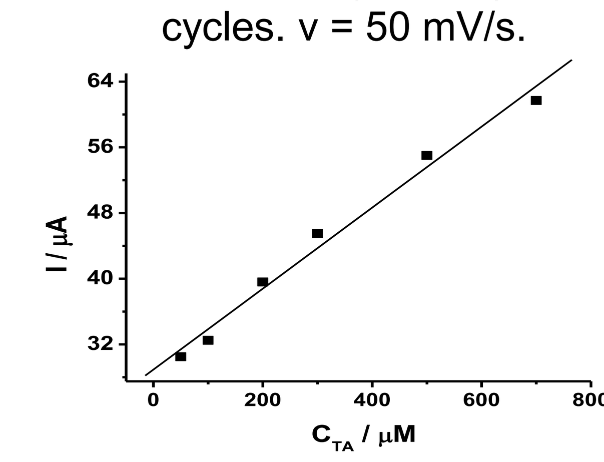
CVs recorded at GCE/PEDOT-PB sensor in 0.5 M KNO_3 containing 5 mM $Na_4[Fe(CN)_6]$; Potential scan rates from 25 to 500 mV/s.



CVs recorded at GCE/PEDOT-PB sensor in 0.1 M acetate buffer (pH=5.0), 5 potential cycles. v = 50 mV/s.



CVs recorded at GCE/PEDOT-PB sensor in 0.1 M acetate buffer (pH=5.0) containing various amounts of thioctic acid (TA) from 50 to 700 μM. v= 50 mV/s.



The calibration plot of GCE/PEDOT-PB sensing platform in the concentration range 50 - 500 μM thioctic acid (TA).

CONCLUSIONS

- A wide linear response range of 50 - 700 μM was obtained.
- The detection limit was determined to be 7.5 μM thioctic acid.
- The sensing platform was successfully applied to the analysis of broccoli extract.
- The recovery values were in the range of 99.38 % to 103%.
- The sensing platform displayed good accuracy.

FUTURE WORK / REFERENCES

1. S.A. Leau, M. Marin, A.M. Toader, M. Anastasescu, C. Matei, C. Lete, S. Lupu, Biosensors, 14(7), **2024**, 320.
2. S.A. Leau, C. Lete, M. Marin, F.J. del Campo, I. Diaconu, S. Lupu, J. Solid State Electrochem., 27, **2023**, pp. 1755-1766.

Aknowledgment: Authors thank the Ministry of Research, Innovation and Digitization, CCCDI–UEFISCDI, for the grant PN-III-P2-2.1-PED-2021-3693