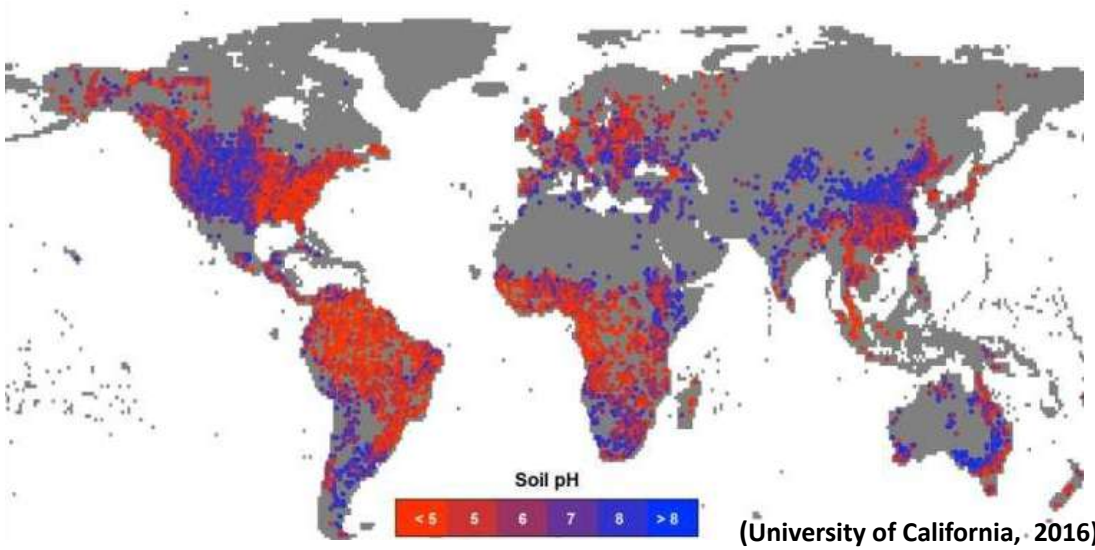
Laser-Induced Graphene- and Bismuth-based Sensor for *in situ* Detection and Quantification of Al^{3+} in soils

Vanessa Reyes-Loaiza , Jhonattan De La Roche, Orlando Idárraga, Drochss Pettry Valencia, Thaura Ghneim-Herrera and Andrés Jaramillo-Botero

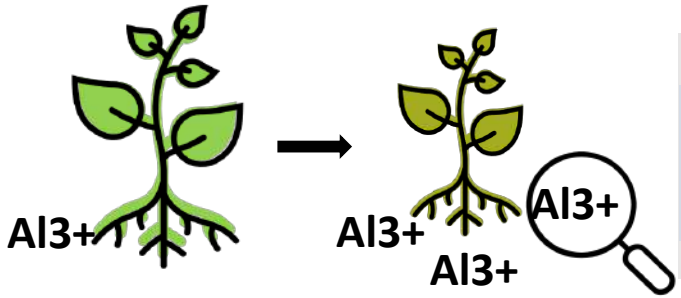
Rationale

Global map of soil pH



(University of California, 2016)

- **40%** Worldwide
- **80%** Colombia

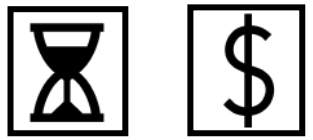


- **Cation exchange sites**
- **Monitor**
- **Enhance amendment / remediation strategies**
- **2- 3 ppm Phytotoxicity**

Detection methods

Atomic Absorption

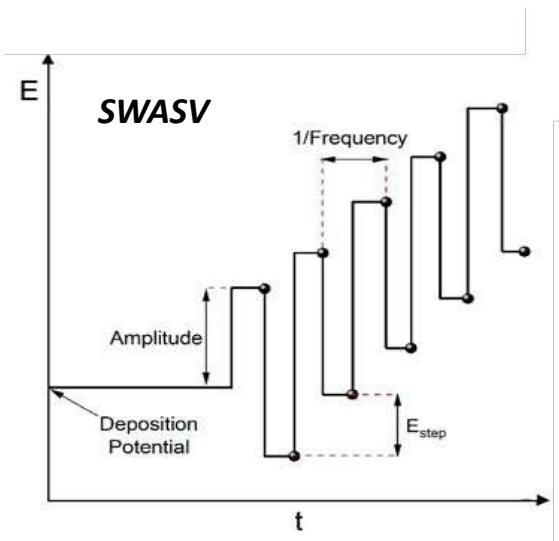
ICP-MS/OES

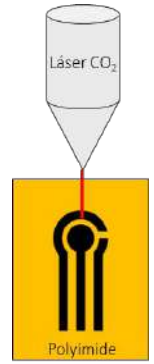


- **Unsuitable for real time *on-site* characterization**

Alternative

Electrochemical systems





LIG

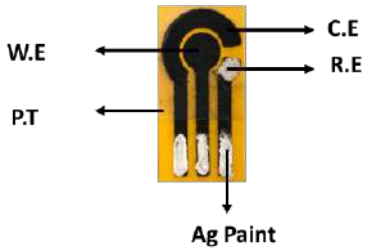
In situ Bi^{3+} deposition

Ayala et al. J. Electroanal. Chem., vol. 919, no. April, p. 116552, 2022.

Materials and Methods

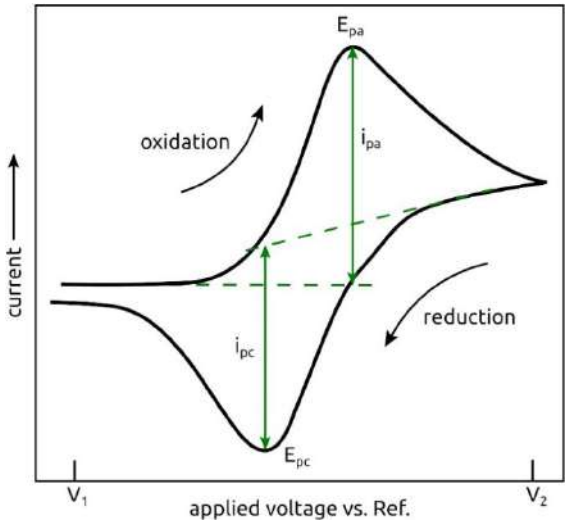
❑ Optimization ❑ Calibration curve ❑ Interference analysis

Commercial potensiostat

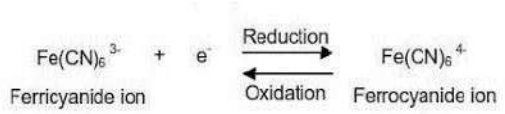


Ag/AgCl KCl 3M

Cyclic Voltammetry

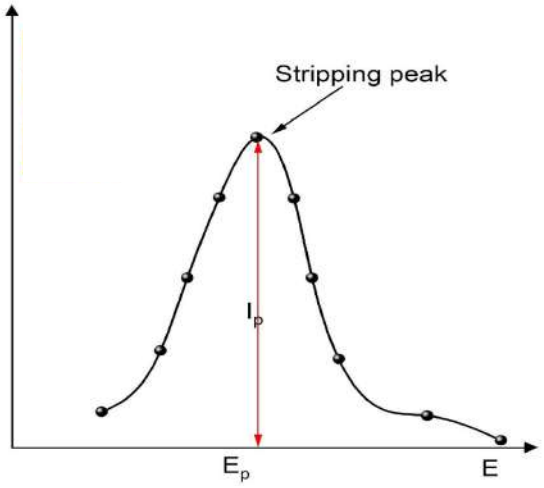


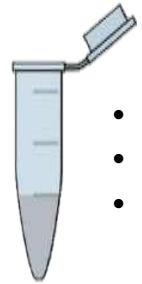
P. Kissinger and W. Heineman, J. Chem. Educ., 1983, 60, 702-706.



-0.4 to 0.6 V; ΔEp 75mV; (Ipa/Ipc) near to 1

SWASV and Working Solution





- Bismuth
- Acetate Buffer 0.1M
- Analyte of interest

Deposition time: 60s Deposition potential: -1.4 V

Other parameters from:

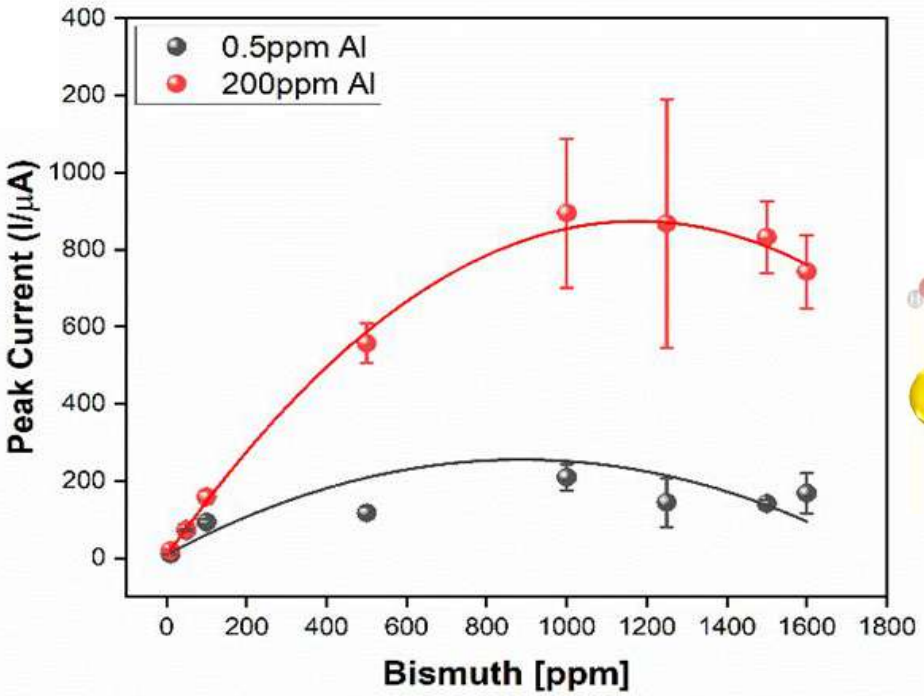
Ayala et al., (2022)
Jeong et al., (2022)

Ayala et al. J. Electroanal. Chem., vol. 919, no. April, p. 116552, 2022

Jeong et al. Microchemical Journal 172 (2022) 106950

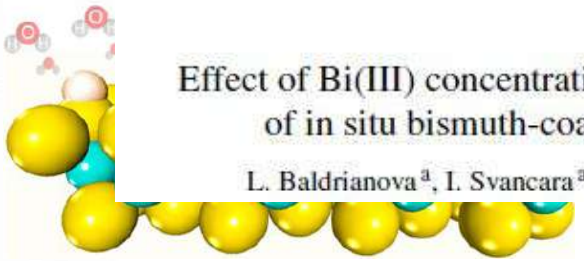
Results

Optimization of chemical parameters



- 10 to 1600ppm Bi^{3+}
- $[Al^{3+}]$ constant

Hydrogen evolution



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Electrochimica Acta 52 (2006) 481–490

Effect of Bi(III) concentration on the stripping voltammetric response of in situ bismuth-coated carbon paste and gold electrodes

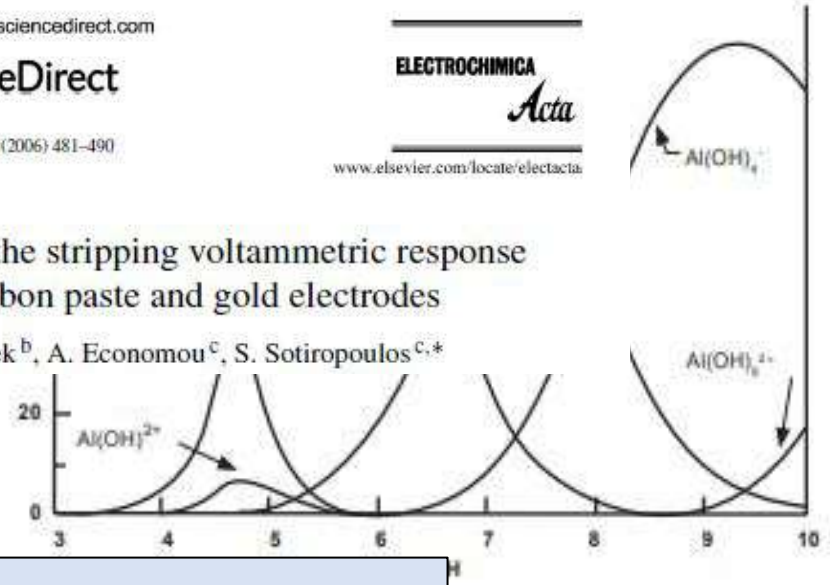
L. Baldrianova [a], I. Svancara [a], M. Vlcek [b], A. Economou [c], S. Sotiropoulos [c,*]

Tang & Jiang . (2016)

Speciation diagram

ELECTROCHIMICA Acta

www.elsevier.com/locate/electacta



al. (2003)

- W.E. Saturation
- Thick Bi film limit mass transfer for Al3+ diffusion in the electrochemical system

Zhao et al. Int. J. Electrochem. Sci., 11 (2016) 1840 - 1851

Porta, J et al (2003). Edafología para la agricultura y el medio ambiente. Madrid: Mundi-Prensa

Tang & Jiang. ACS Catal. 2016, 6, 8, 4953–4961

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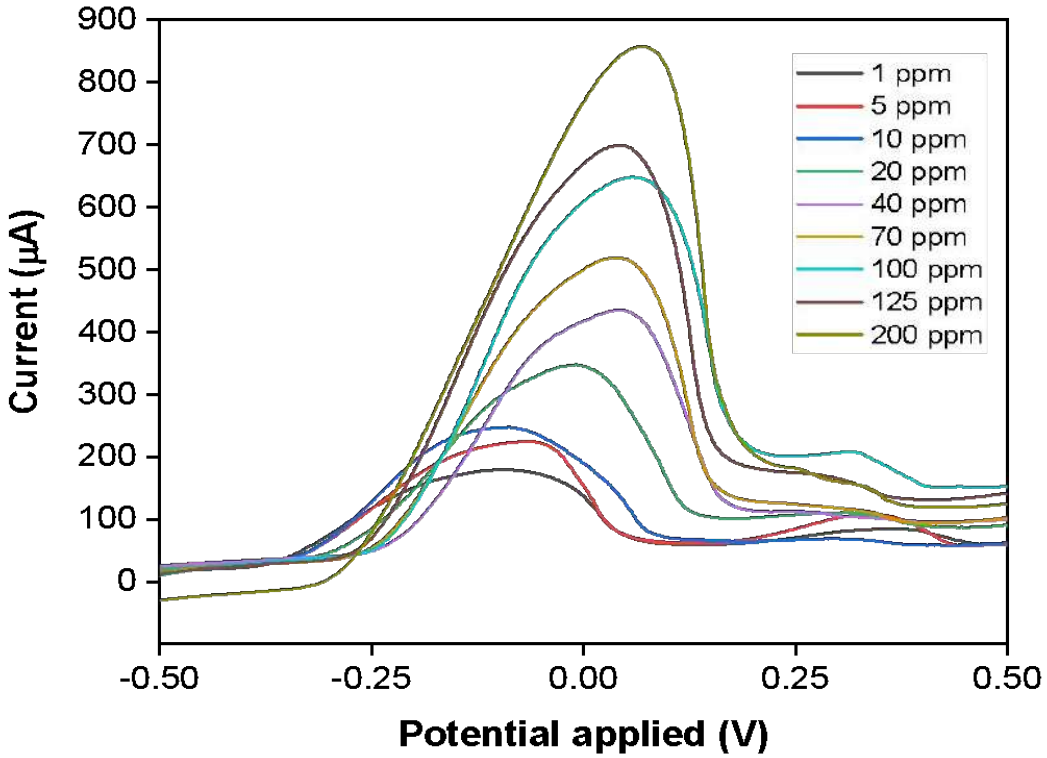
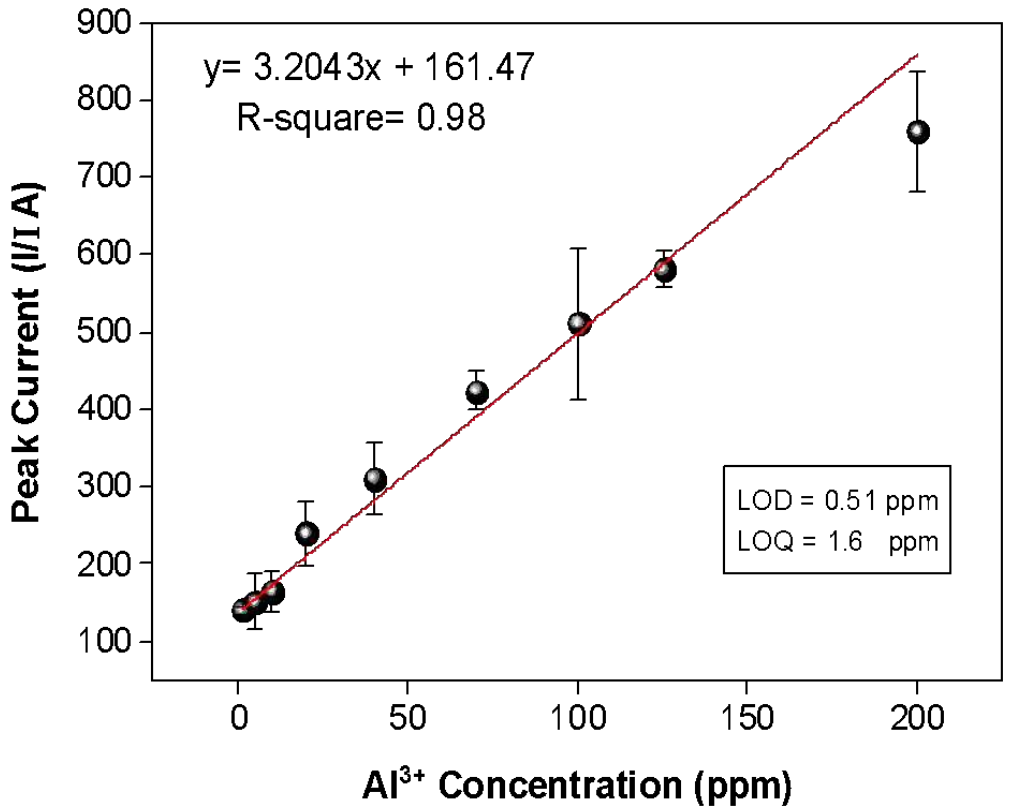
Calibration curve: Individual measurements





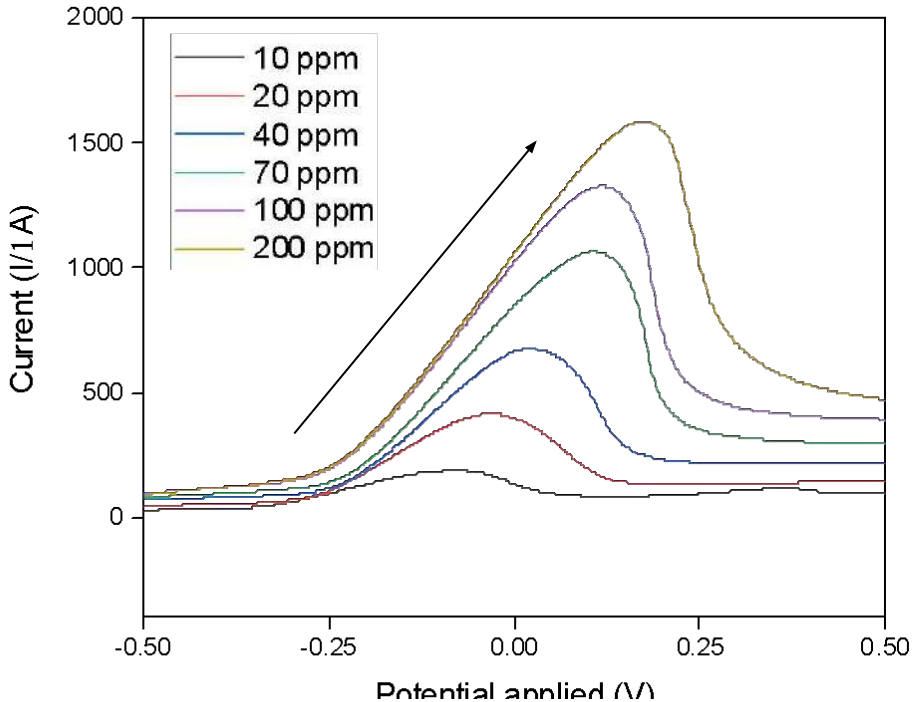
- electrochemical adsorption processes
- Higher peak current means a higher amount of species adsorbed

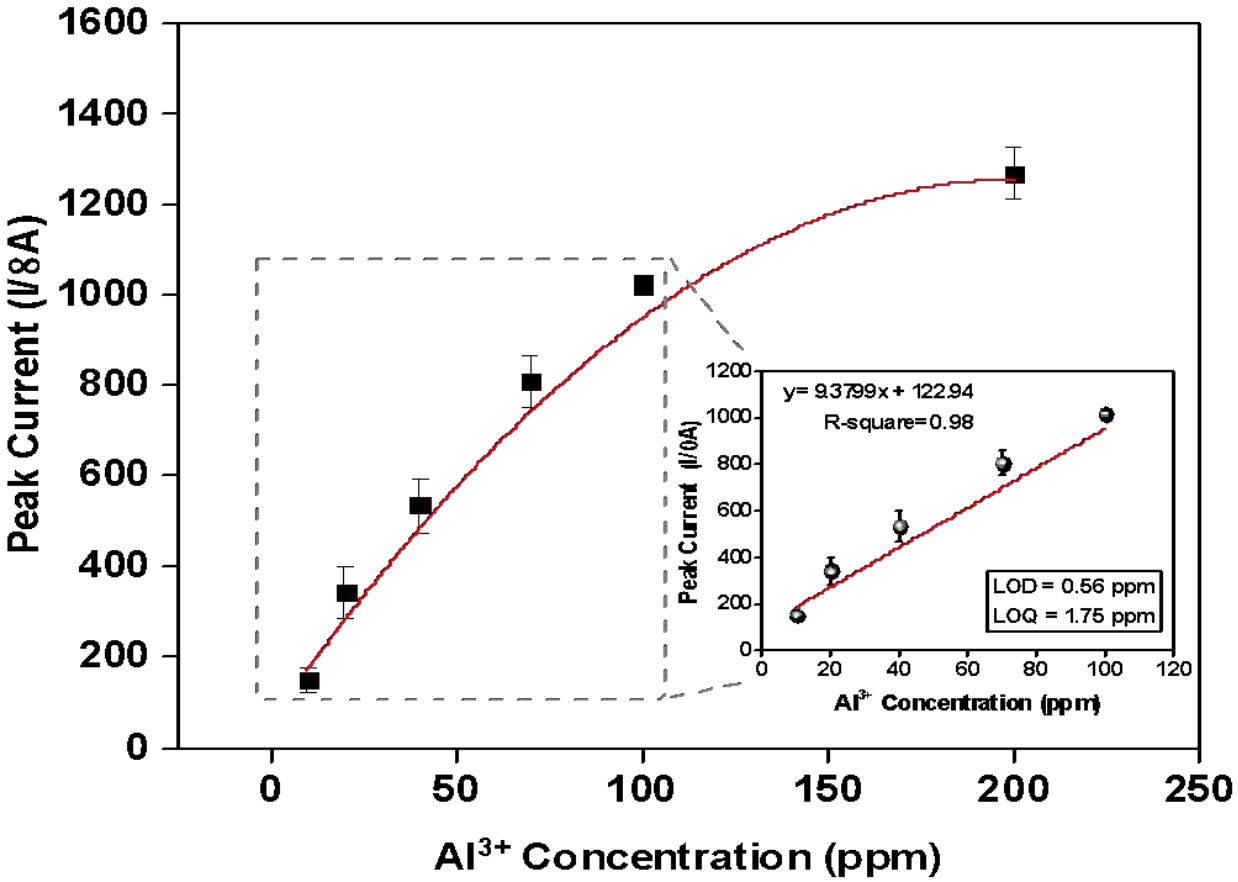
$$I_p = \frac{n^2F^2}{4RT}\, vA\Gamma^*$$

- 3 electrodes per concentration (single measurement)

Ayala et al. J. Electroanal. Chem., vol. 919, no. April, p. 116552, 2022.

Calibration curve: Incremental measurements





Sensors and Actuators B 133 (2008) 462–466

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Cyclic voltammetry on electrode surfaces covered with porous layers: An analysis of electron transfer kinetics at single-walled carbon nanotube modified electrodes

Ian Streeter, Gregory G. Wildgoose, Lidong Shao, Richard G. Compton*

Physical and Theoretical Chemistry Laboratory, Oxford University, South Parks Road, Oxford OX1 3QZ, United Kingdom

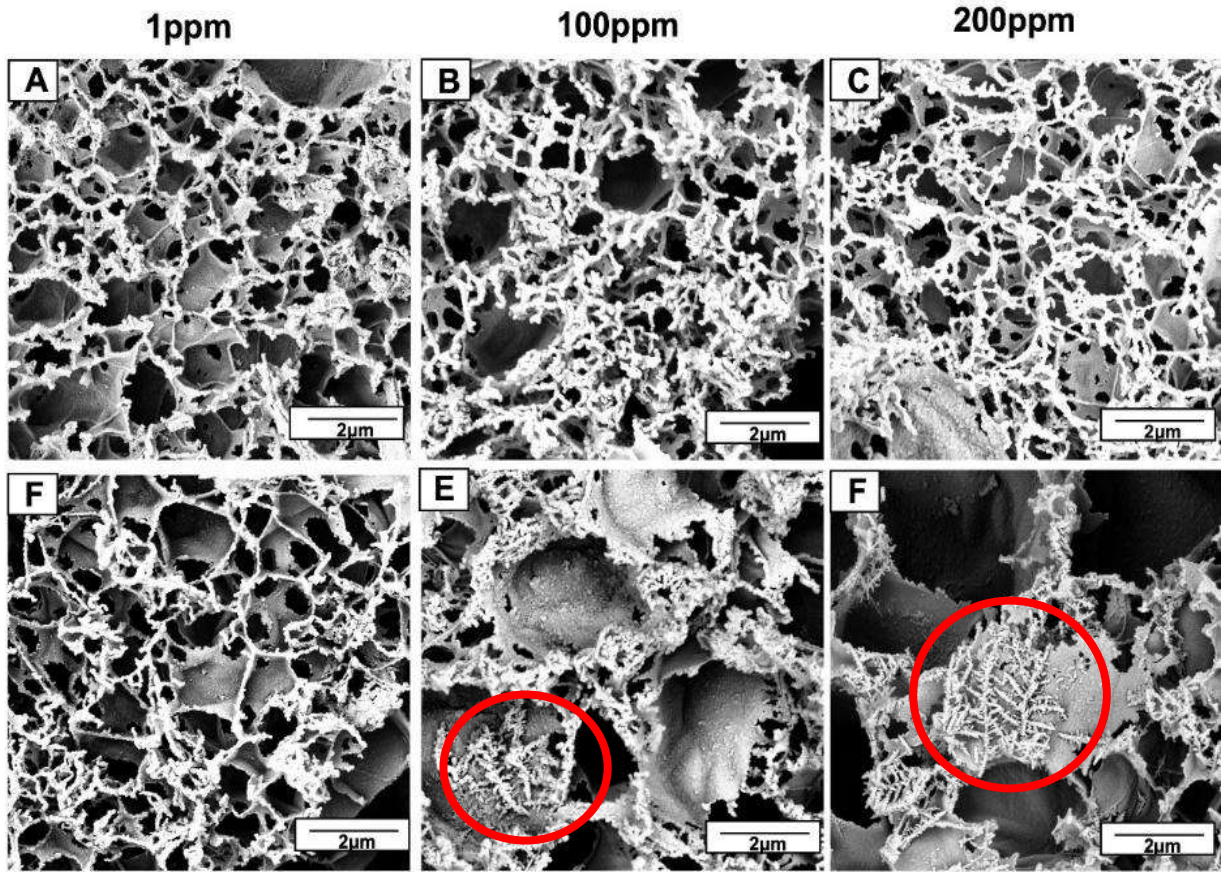
$$E_{p=E_f^\theta} + \frac{RT}{\alpha F} \ln\left(\frac{\alpha F v}{RTlK_0}\right)$$

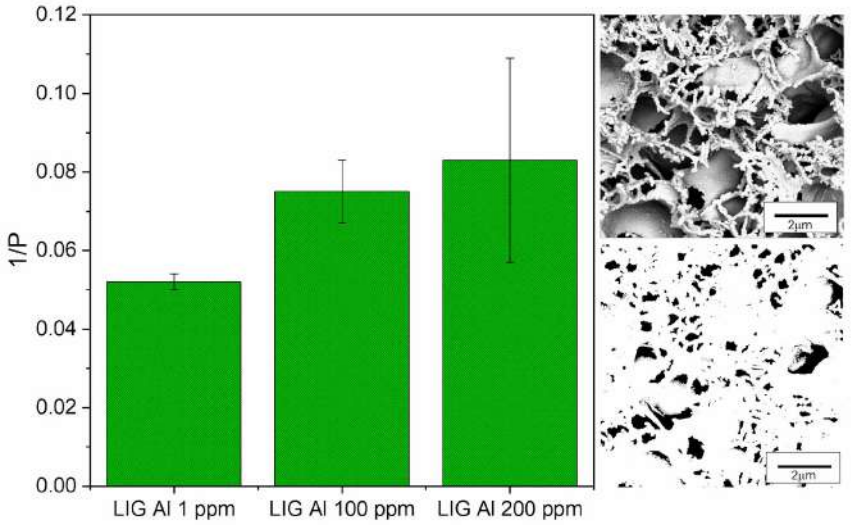
Hubbard equation

- more time is required to reduce the layer of electroactive species

l Layer thickness

Morphological characterization





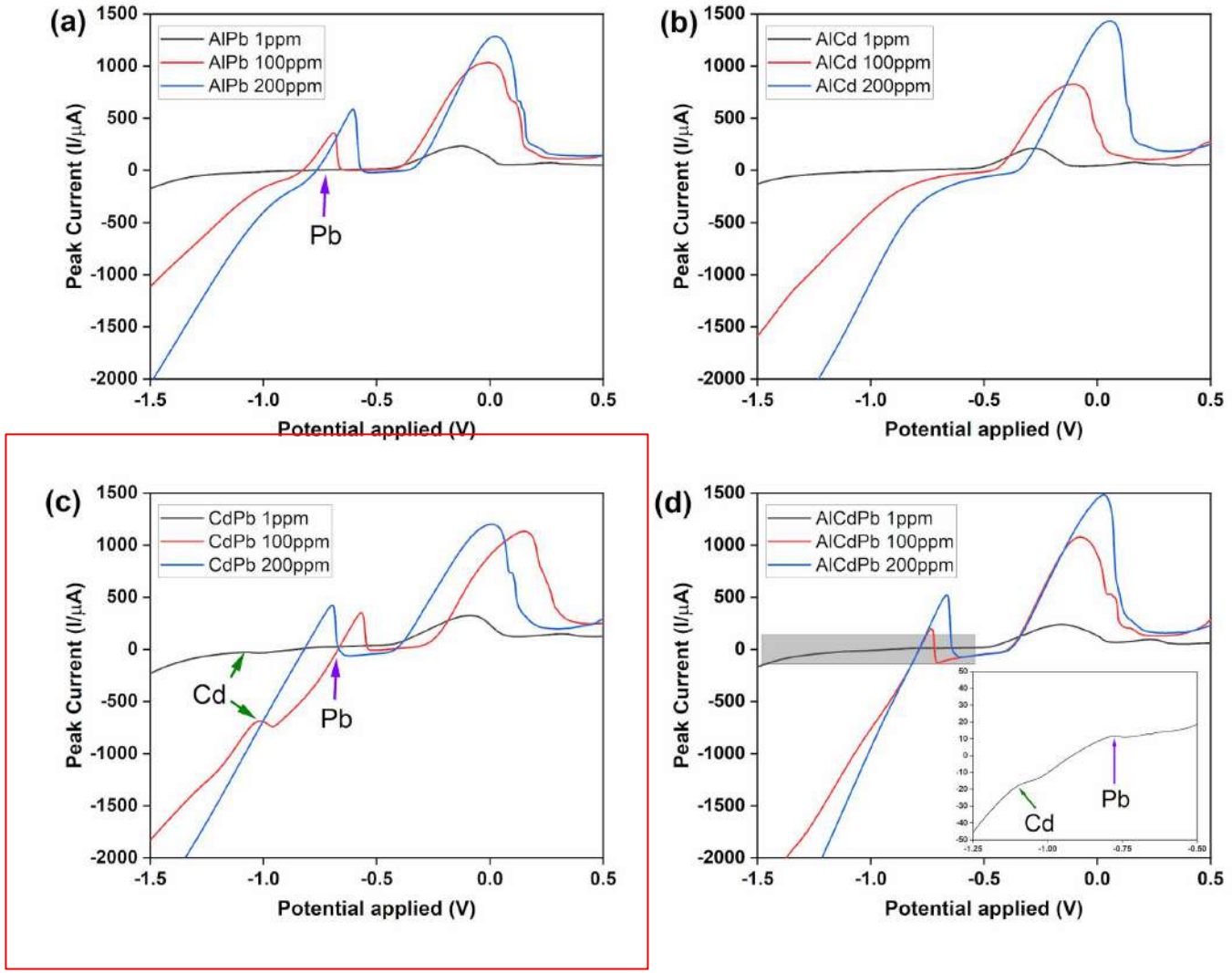
- Non-uniform thickness, grows in the already deposited patches high local current densities. Related to the electrochemical response

- Unpaired electrodes
- Highly active catalytic sites

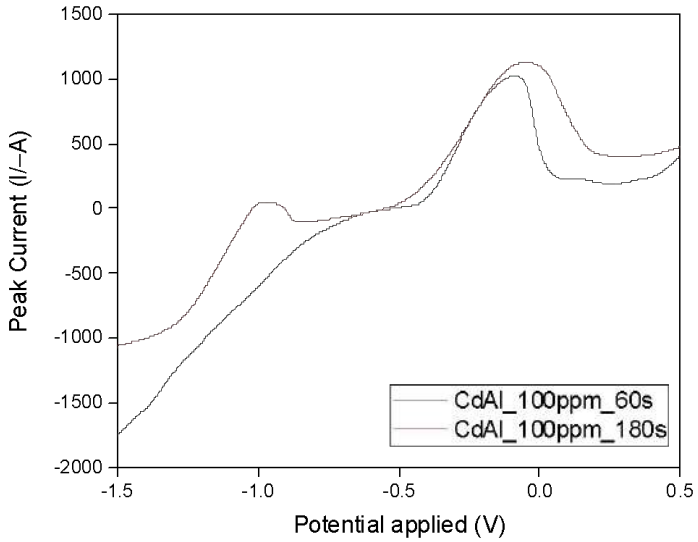
Dendrites

- Non-uniform metal deposition on the electrode Surface
  - Anisotropy in the system, Crystalline structure

Interference analysis



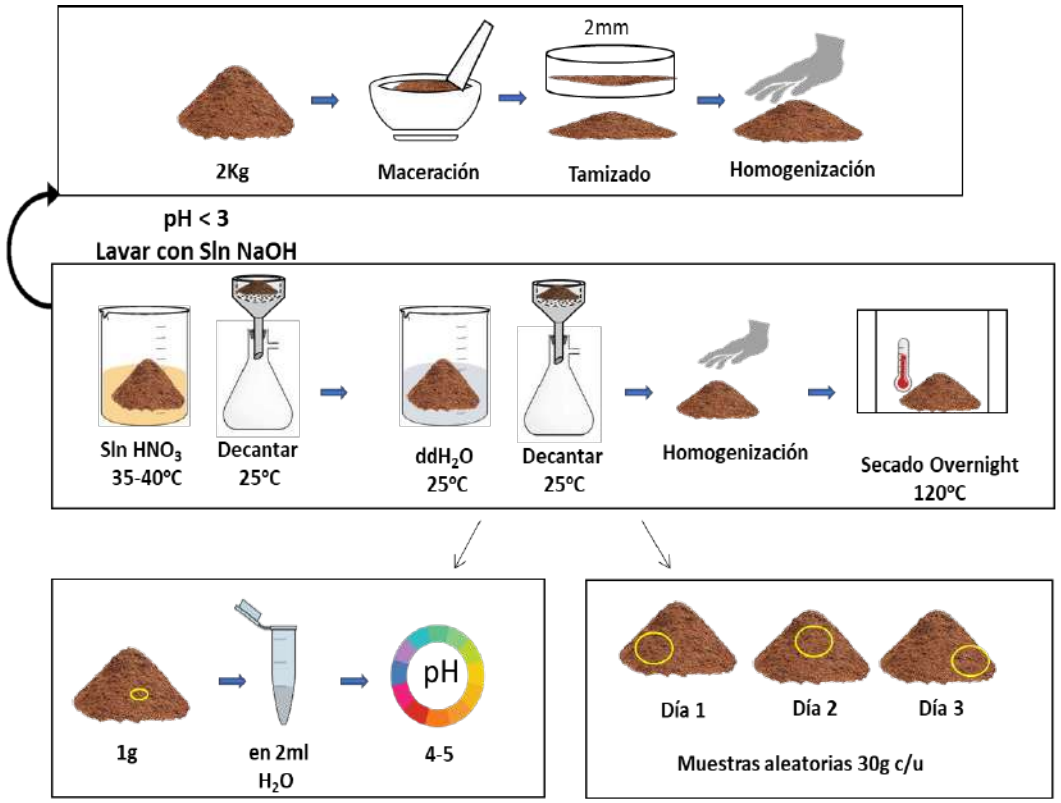
- Differential stripping response in relation to the Potential

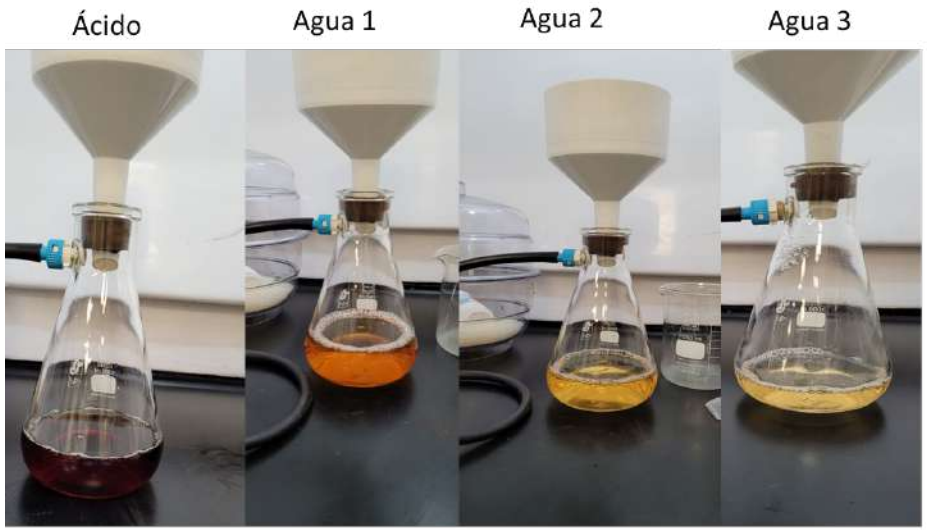


- Increase in deposition time is needed to detect Cd in presence of Al at high concentrations

Application in soils

Spiked Soil Sample preparation





2 Suelo enriq. Aluminio

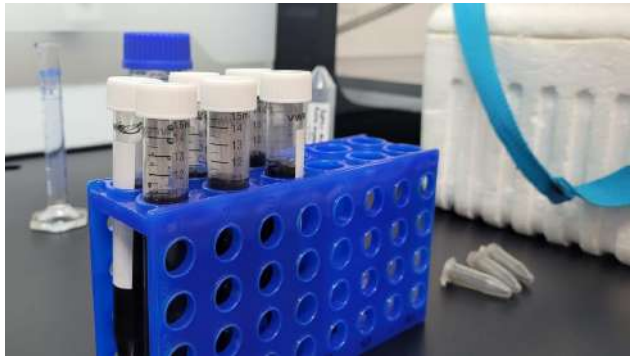
	Conc.	Abs.	BG	ActualConc.	Actual Conc. Unit	%RSD
	17.0069	0.1327	0.0020	17.0069	ppm	2.44

SD	C#	SG#
0.0032	01	1

Atomic Absorption

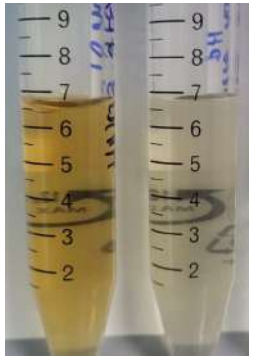
PRELIMINARY

Extraction from Soil



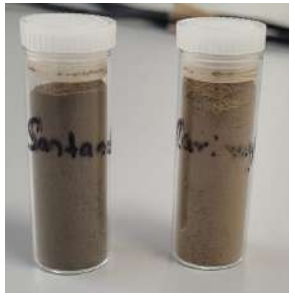
2 min asentar

2 min: 7ml



HNO3 B.acetato

- Ácido acético 0.11 M pH 2.85
- Agitación 1h
- Centrifugación
- Filtración
- Ajuste pH NaOH



Recuperación del 64.7%

Int. J. Electrochem. Sci., 11 (2016) 1840 - 1851

International Journal of ELECTROCHEMICAL SCIENCE
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Electrochemical Determination of Trace Cadmium in Soil by a Bismuth Film/Graphene-β-cyclodextrin-Nafion Composite Modified Electrode

Guo Zhao[1,2], Hui Wang[1,2], Gang Liu[1,2,]*

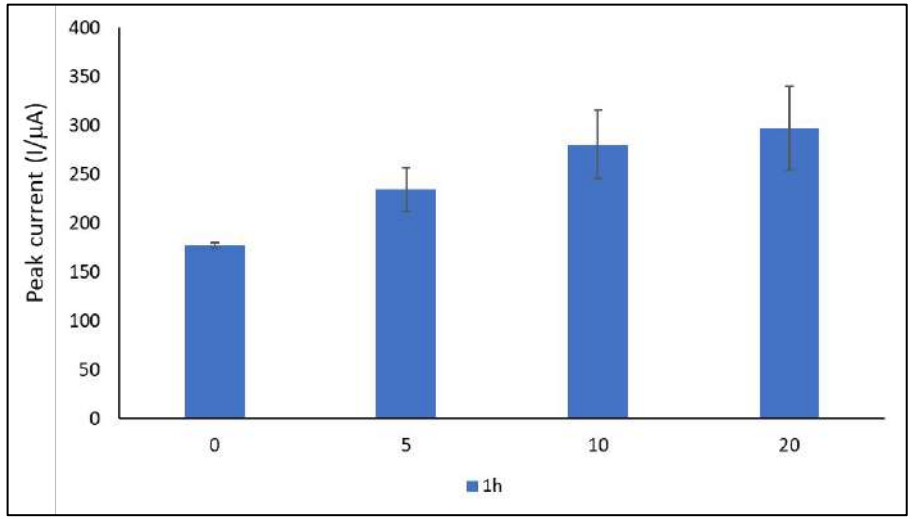
ASABE

An ASABE Meeting Presentation

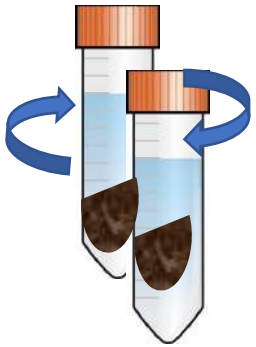
Paper Number: 12-1341188

On-Site Determination of Heavy Metal in Soil Using Electrochemical Stripping Analysis

Wang Zhiqiang, Lecturer, Ph.D.Student
Key Lab of Modern Precision Agriculture System Integration Research, China Agricultural University, 17 Qinghua Donglu, Beijing China, zbwzq77@sina.com
Key Lab of Agricultural Information Acquisition Technology (Beijing), China Agricultural University, 17 Qinghua Donglu, Beijing China
College of Computer Science and Technology, Shandong University of Technology, 12 Zhangzhou Road, Zibo China
Liu Gang, Professor, Ph.D.Supervisor

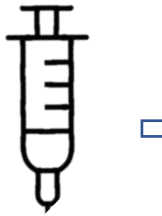


Goal



1g in 40ml CH_3COOH 0.11M / stir manually x t

Filtration



Neutralized Aliquot + Standard solution ([Al] + Bi+ Acetate Buffer)

Aliquot 40ml over the LIG

Conclusions

Here, we demonstrate an electrochemical-based sensor using bismuth-modified laser-induced graphene (LIGs) electrodes for Al^{3+} detection and quantification, in a range relevant to agricultural practice.

Our system can be extended to other applications in agriculture. i.e., can be optimized and used for the detection of different metallic ions such as Cd^{2+} and Pb^{2+}.

Once the protocol for extraction protocol is optimized. The portability, ease of use, and cost-effectiveness of this technology offer a means for *in situ* detection of Al3+in agricultural soils and other complex matrices

References

M. Camila Ayala, L. Lorena López, A. Jaramillo-Botero, and D. Valencia, “Electrochemical modified electrode with bismuth film for ultrasensitive determination of aluminum (iii),” *J. Electroanal. Chem.*, vol. 919, no. April, p. 116552, 2022.

S. E. Jeong, S. Kim, J. H. Han, and J. J. Pak, “Simple laser-induced graphene fiber electrode fabrication for high-performance heavy-metal sensing,” *Microchem. J.*, vol. 172, no. PA, p. 106950, 2022

G. Zhao, H. Wang, and G. Liu, “Electrochemical determination of trace cadmium in soil by a bismuth film/graphene-β-cyclodextrin-nafion composite modified electrode,” *Int. J. Electrochem. Sci.*, vol. 11, no. 3, pp. 1840–1851, 2016.

L. Baldrianova, I. Svancara, M. Vlcek, A. Economou, and S. Sotiropoulos, “Effect of Bi(III) concentration on the stripping voltammetric response of in situ bismuth-coated carbon paste and gold electrodes,” Electrochim. Acta, vol. 52, no. 2, pp. 481–490, 2006.

¡Muchas Gracias!

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