

CUSTOMIZED SCREEN-PRINTED ELECTRODES BASED ON Ag-NANOSEEDS FOR ENHANCED ELECTROANALYTICAL RESPONSE TOWARDS Cd(II), Pb(II) AND As(V) IN AQUEOUS SAMPLES

KARINA TORRES-RIVERO*, CLARA PÉREZ-RÀFOLS, JULIO BASTOS-ARRIETA, NÚRIA SERRANO, VICENÇ MARTÍ AND ANTONIO FLORIDO



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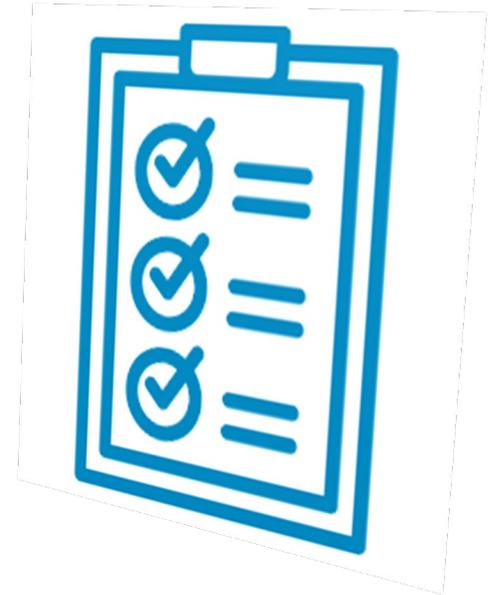


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2021

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AGENDA

- Heavy Metals contamination problematic
- Objectives
- Methodology
- Results
- Conclusions



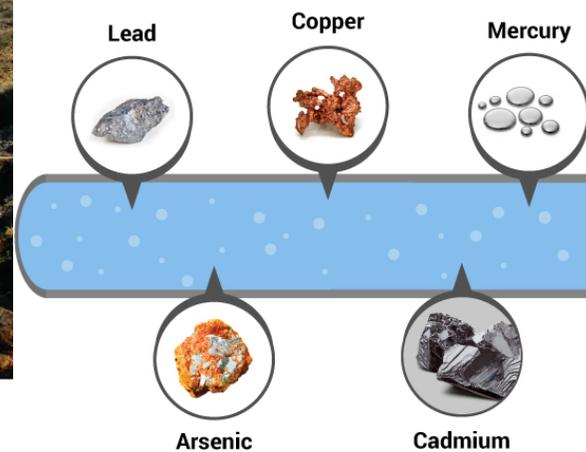
HEAVY METAL WATER CONTAMINATION

Heavy metal ions

- ✓ High toxicity
- ✓ Non-biodegradability
- ✓ Bioaccumulation
- ✓ Adverse health effects in humans



Waste water from a gold mine flows into the forest. Guyana, South America. kakteen / shutterstock



	WHO	EU Directive 2020/2184
Pb(II)	10 µg/L	10 µg/L
Cd(II)	3 µg/L	5 µg/L
As(V)	10 µg/L	10 µg/L

Source: <https://theconversation.com/acid-drainage-the-global-environmental-crisis-youve-never-heard-of-83515>

Source: <http://water-purifiers.com/heavy-metal-contamination-in-drinking-water/>

HEAVY METAL WATER CONTAMINATION

Heavy Metal Detection Methods

- ✓ Flameless atomic adsorption spectrometry (FAAS)
- ✓ Inductively coupled plasma mass spectrometry (ICP-MS)
- ✓ Hydride generation atomic fluorescence spectrometry (HG-AFS)
- ✓ **ELECTROCHEMICAL TECHNIQUES: ANODIC STRIPPING VOLTAMMETRY (ASV)**

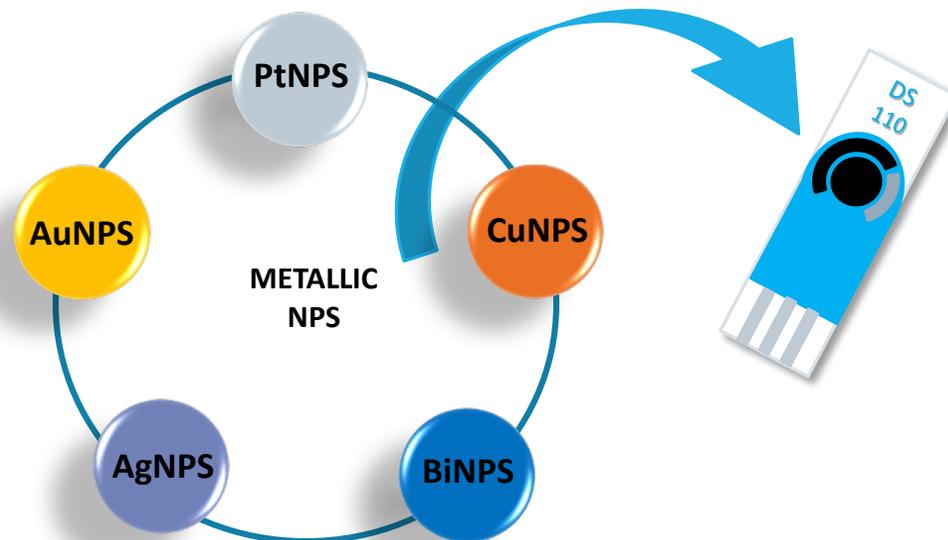
LOWER
DETECTIO
LIMITS

LOW COST



FAST
ANALYSIS

Nanoparticles/Screen-printed electrodes¹

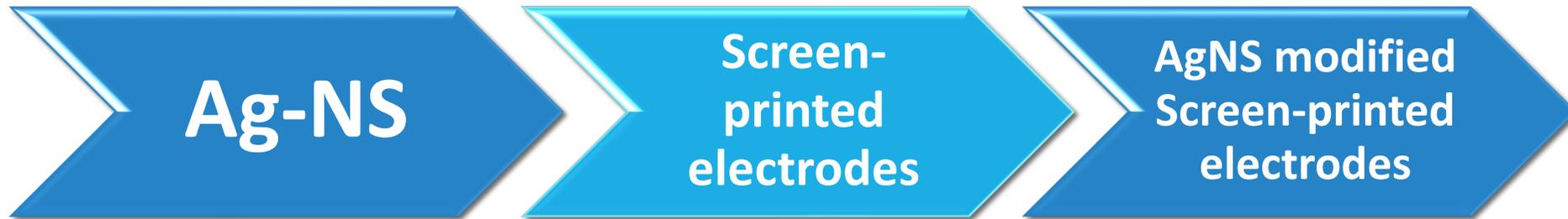


- ✓ Electrochemical response improvement
- ✓ ↓ Electron Transfer Resistance
- ✓ Catalyzing the electrode's response at low analyte concentrations

¹Torres-Rivero, K.; Florido, A.; Bastos-Arrieta, J. Recent Trends in the Improvement of the Electrochemical Response of Screen-Printed Electrodes by Their Modification with Shaped Metal Nanoparticles. *Sensors* **2021**, *21*, 2596.

OBJECTIVES

Voltammetric determination of HMIs based on the use of carbon-nanofiber-based screen-printed electrodes (SPCNFEs) modified with silver nanoparticles (Ag-NPs)



Synthesize Ag-NS

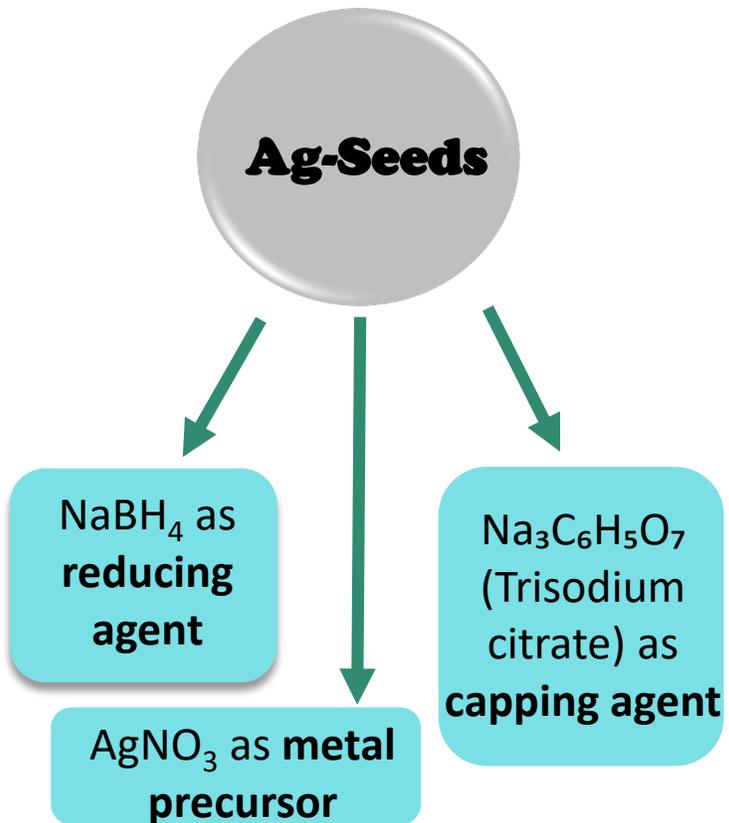
Modify SPCNFE with the dropcasting technique

- Characterize the modified electrode surface.
- Characterize the modified electrode electrochemically using DPASV
- Study the applicability of the modified electrode in As(V) spiked tap water samples



METHODOLOGY

1. Ag-Seeds Synthesis²



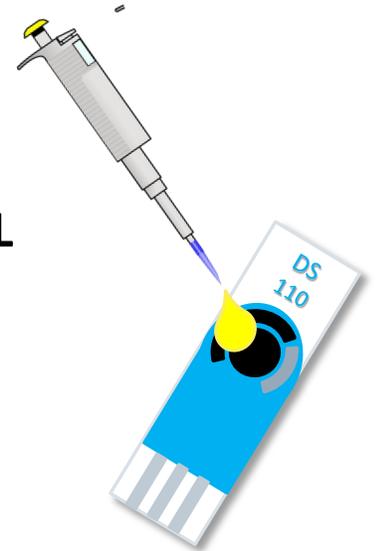
2. Ag-Nanoseeds characterization

- ✓ Scanning electron microscopy (SEM)
- ✓ Transmission electron microscopy (TEM)

3. SPCNFE Modification

Dropcasting

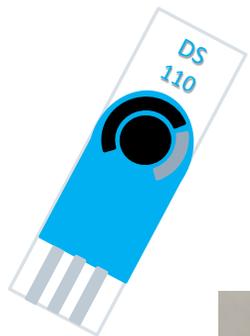
1 drop of 40 μL



- ✓ Heat at 50 °C for 30 minutes

METHODOLOGY

4. Modified SPCNFE Surface characterization



Scanning Electron Microscopy (SEM)

5. SPEs Electrode



Characterization



SPCNFE electrode
Potentiostat

	Pb(II)	Cd(II)	As(V)
Buffer	0.1 mol/L Acetate buffer pH 4.5	0.1 mol/L Acetate buffer pH 4.5	0.01 mol/L HCl pH 2.0
Standard	1 mg/L	1 mg/L	1 mg/L

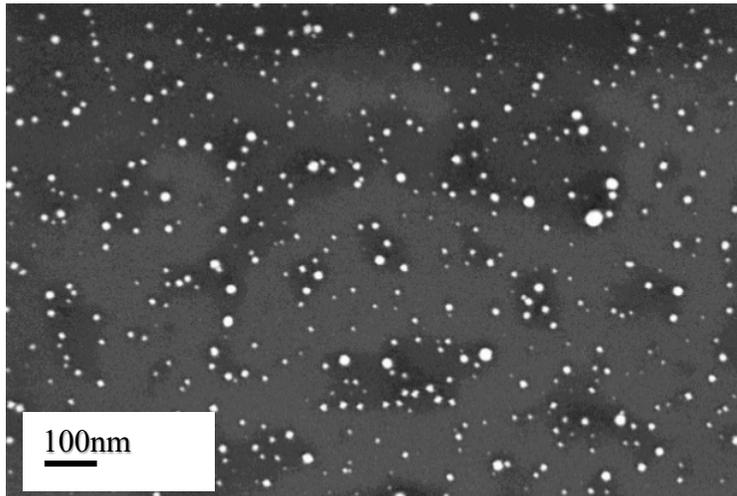
DPASV Parameters

	Pb(II)	Cd(II)	As(V)
E_d	-1.4 V	-1.4 V	-1.3 V
T_d	180 s	180 s	120 s
Scanning Potential	-1.4 V - 0.0 V	-1.4 V - 0.0 V	-1.3 V - 0.65 V

RESULTS

1. Ag-Nanoseeds characterization³

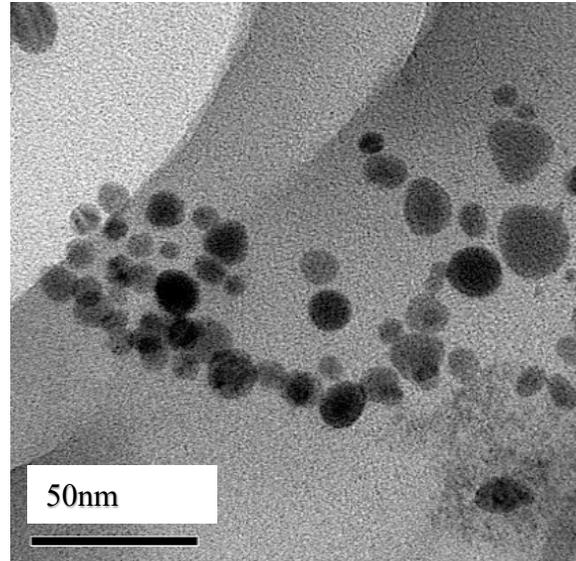
SEM micrograph



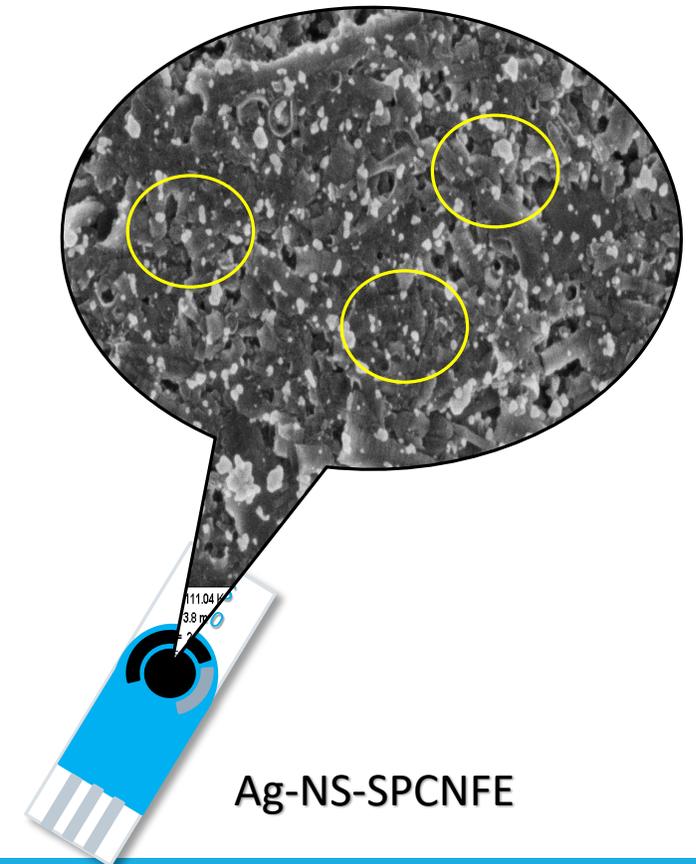
Ag-Nanoseeds

- Spherical shape
- 12.2 ± 0.4 nm

TEM micrograph



2. Ag-NS-SPCNFE Surface characterization⁴

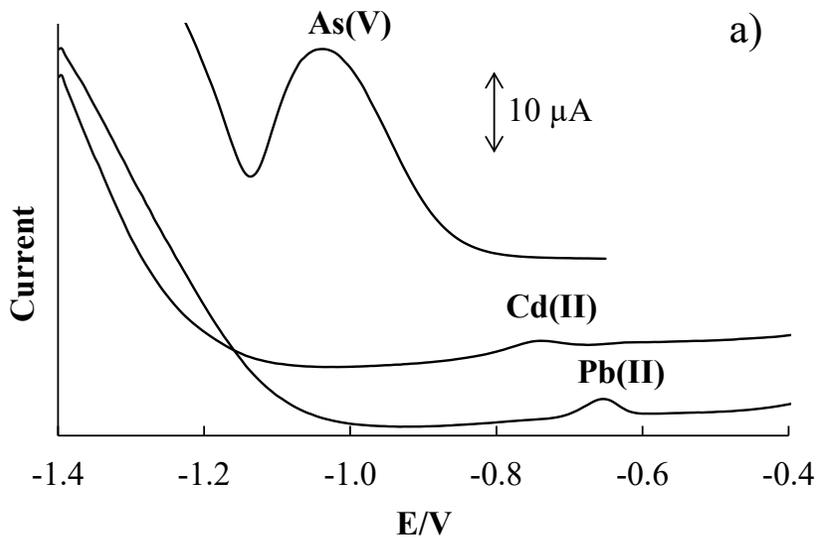


³Torres-Rivero, K.; Torralba-Cadena, L.; Espriu-Gascon, A.; Casas, I.; Bastos-Arrieta, J.; Florido, A. Strategies for Surface Modification with Ag-Shaped Nanoparticles: Electrocatalytic Enhancement of Screen-Printed Electrodes for the Detection of Heavy Metals. *Sensors* 2019, 19, 1–14

⁴Torres-Rivero, K.; Pérez-Ràfols, C.; Bastos-Arrieta, J.; Florido, A.; Martí, V.; Serrano, N. Direct As(V) determination using screen-printed electrodes modified with silver nanoparticles. *Nanomaterials* 2020, 10, 1–10

RESULTS

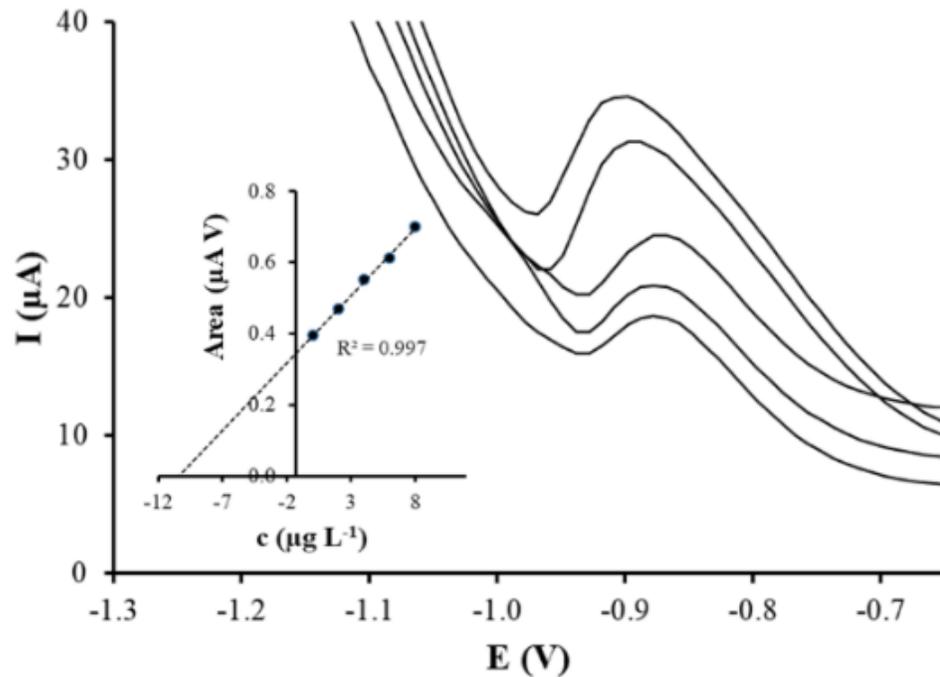
Ag-NS-SPCNFE Electrochemical Characterization



Analyte	LOD ($\mu\text{g}\cdot\text{L}^{-1}$)	Linear Range ($\mu\text{g}\cdot\text{L}^{-1}$)	R^2	Sensitivity ($\text{nA}\ \mu\text{g}^{-1}\ \text{L}$) (SD)
Pb(II)	3.3	10.9-99.6	0.999	103 (3)
Cd(II)	3.7	12.2-73.4	0.992	22 (1)
As(V)	2.6	8.9 - 40.0	0.991	260 (10)

RESULTS

Ag-NS-SPCNFE-Application in real sample



	As(V)	
	CAs(V) ($\mu\text{g.L}^{-1}$)	SD ($\mu\text{g.L}^{-1}$)
DPASV	10.04	0.37
ICP-MS	10.70	0.20

Tap water samples were collected from the local water distribution network managed by Aigües de Barcelona Company
<https://www.aiguesdebarcelona.cat/>

CONCLUSIONS

- ✓ Ag-Nanoseeds were **synthesized**, microscopically **characterized**, and **used for the modification of SPCNFE**.
- ✓ Ag-NS-SPCNFE was **suitable for determining Pb(II), Cd(II), and As(V)** at low $\mu\text{g}\cdot\text{L}^{-1}$ levels.
- ✓ Electrochemical characterization showed **wider linear ranges for Pb(II) and Cd(II)** determination but **lower sensitivities as compared to As(V)**.
- ✓ **LODs** achieved in this investigation **are equal or lower** than other **LODs previously reported**.
- ✓ The suitability of Ag-NS-SPCNFE for the **determination of As(V) in spiked water samples**, achieving **comparable results with ICP-MS** with **good reproducibility**.

Acknowledgments

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Thank you for your attention!

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