Paper as substrate

Paper can…

Store

Filter

React

Drawbacks…

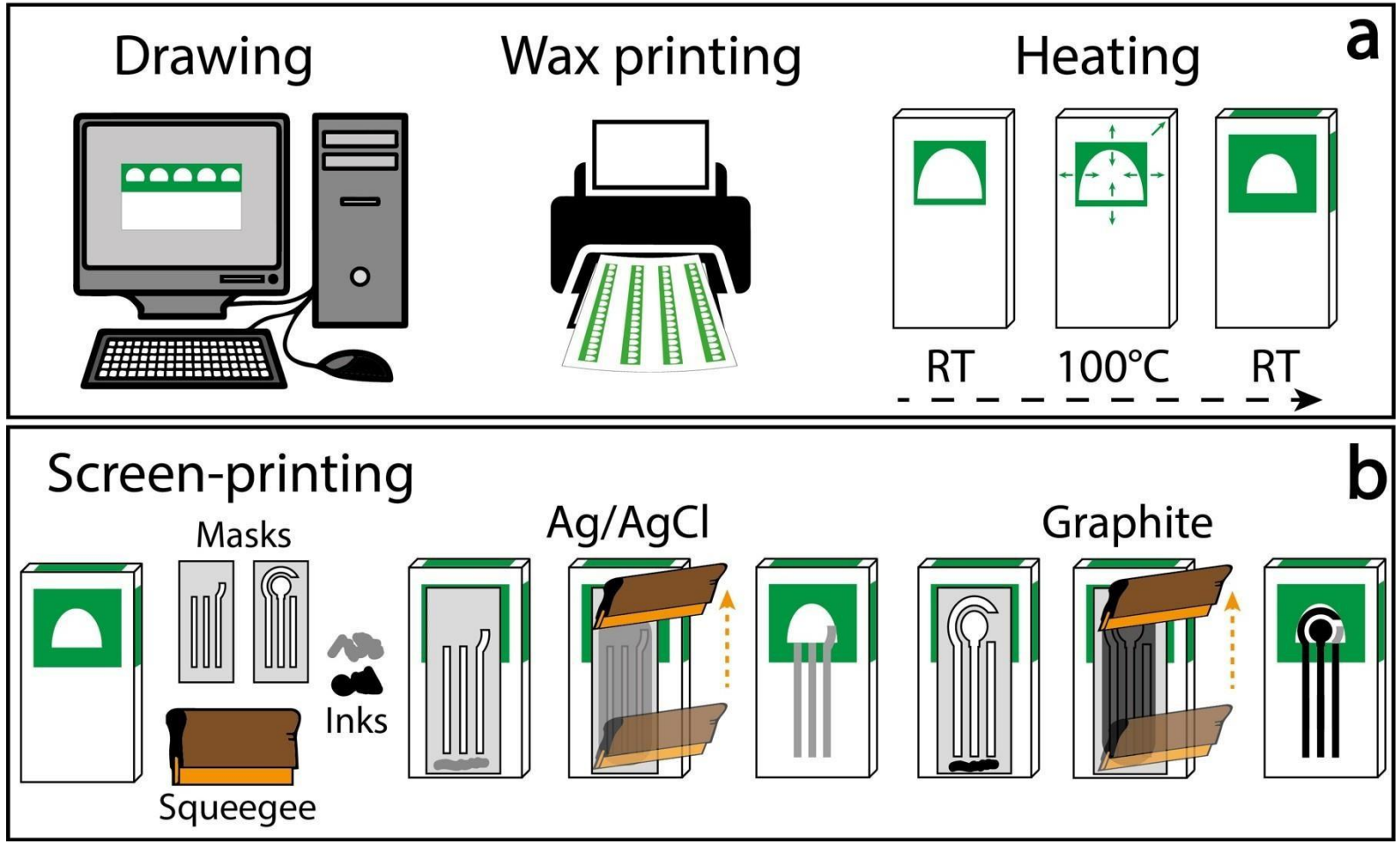
Reagents diffusion…

Electrical noise! ☹

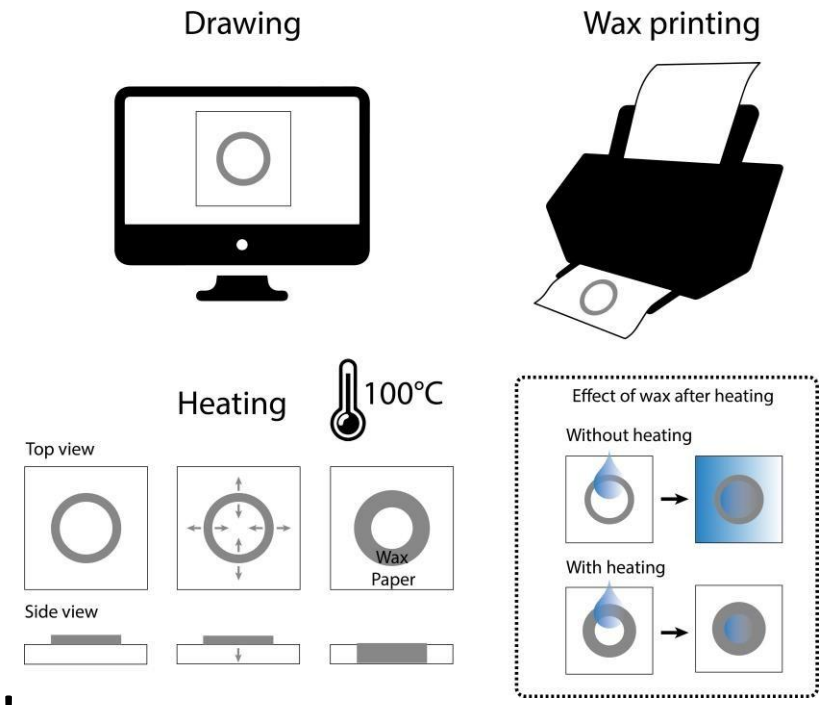
An hydrophobic barrier is needed…

From Paper to E-Paper

Few and easy steps



Hydrophilicity matters



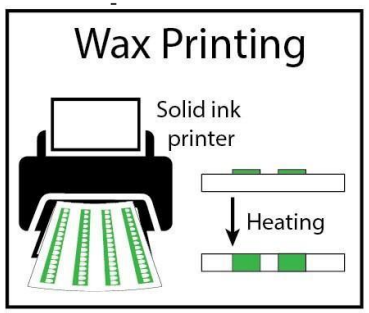
… also the cost!

Costs of the components for producing one device (all the costs have to be intended in Euro).

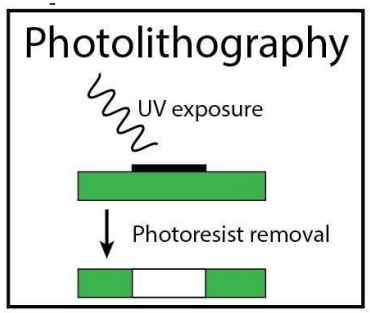
Substrate	Ag/AgCl ink	Carbon ink	Insulator	Substrate	Total cost	Saving[c]
Polyester	0.010	0.007	0.003[a]	0.013	0.033	45%
Whatman #1			0.001[b]	0.007	0.025	30%
Office paper			0.001[b]	0.0001	0.018	/

[a] Insulator ink.
[b] Wax.
[c] Calculated as 1 – [Office paper/Other] × 100.

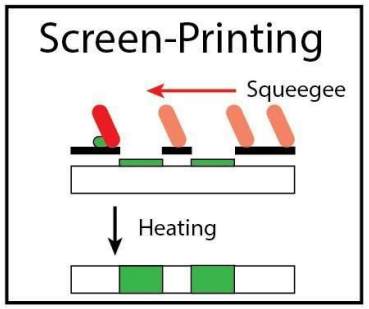
It depends on what you need and you have!



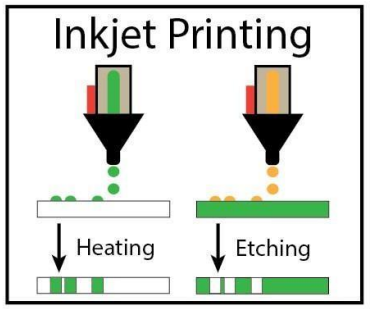
Sustainable
Low resolution



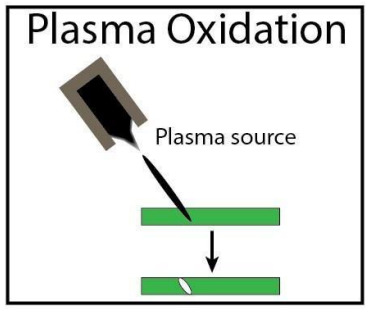
High resolution
Expensive



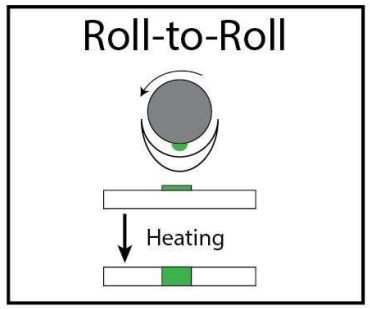
Easiness
Ad hoc masks



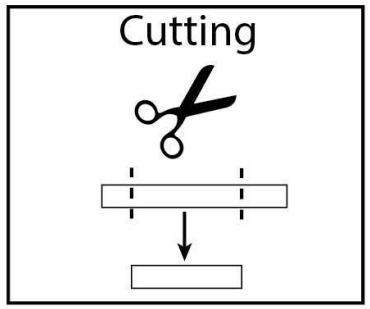
Reduced waste
Expensive printer



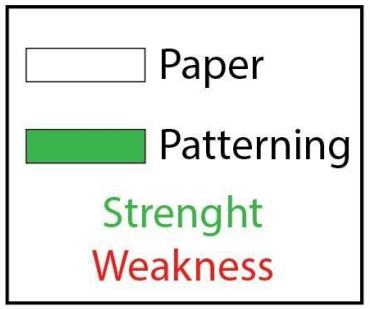
Cheap patterning
Hydrophobized paper



Mass scalable
Too many steps

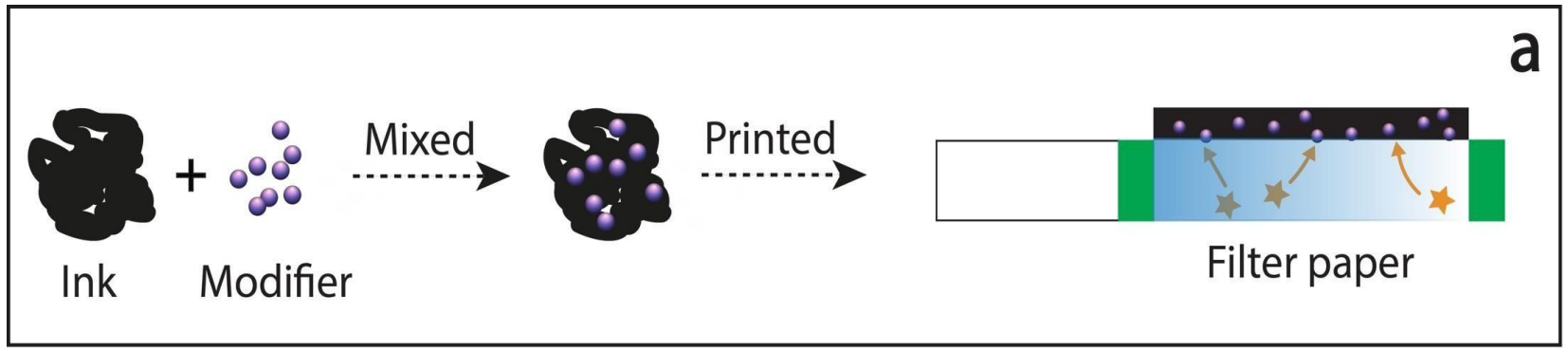


Low-cost
No channels

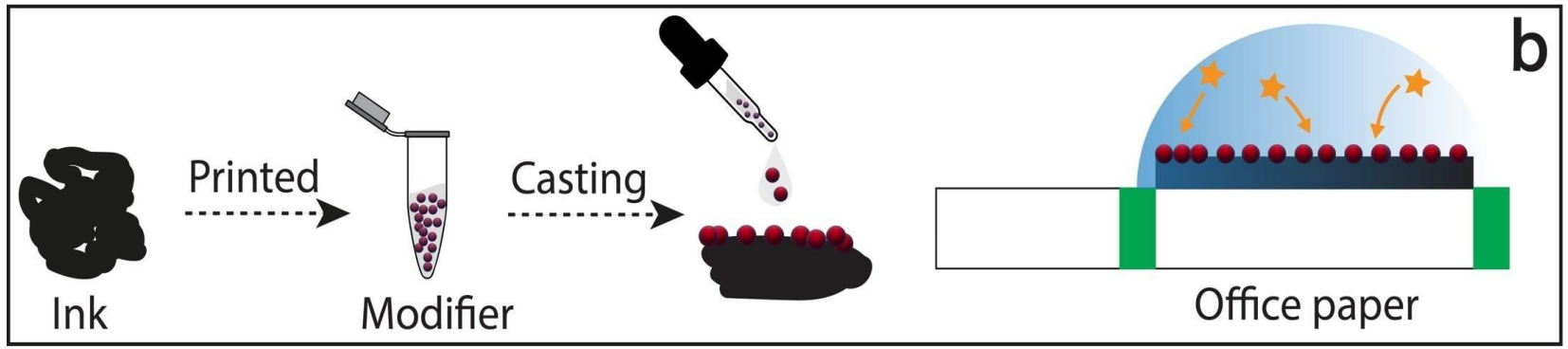


Which E-Paper?

Porous

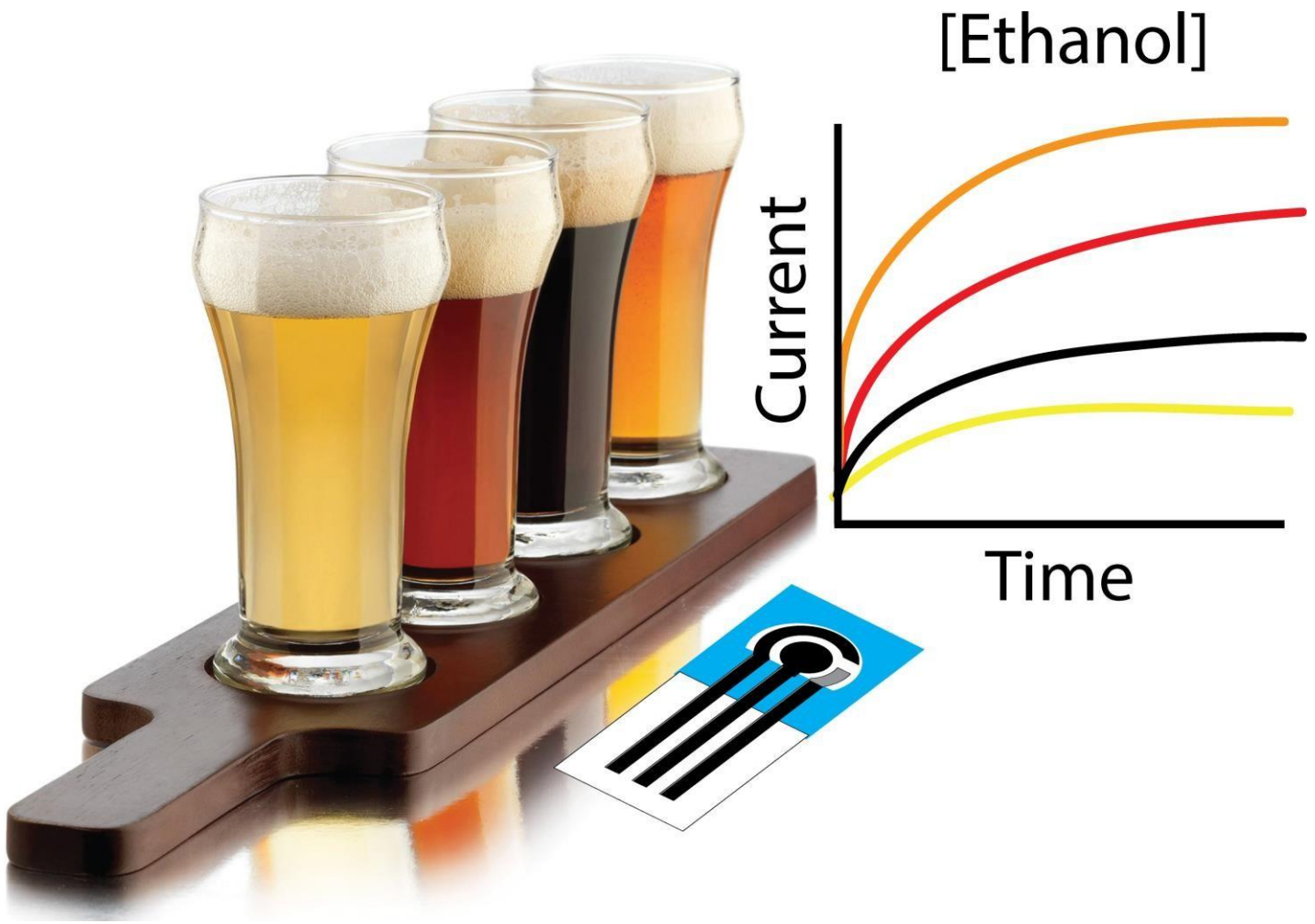


Non porous



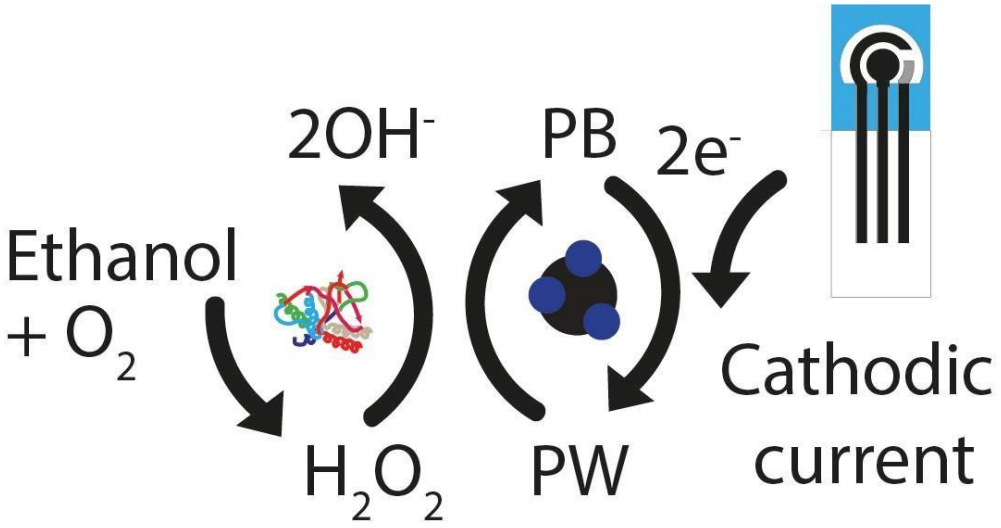
Anyway, paper is the substrate… we need to make these strips ad-hoc

Office paper for ethanol

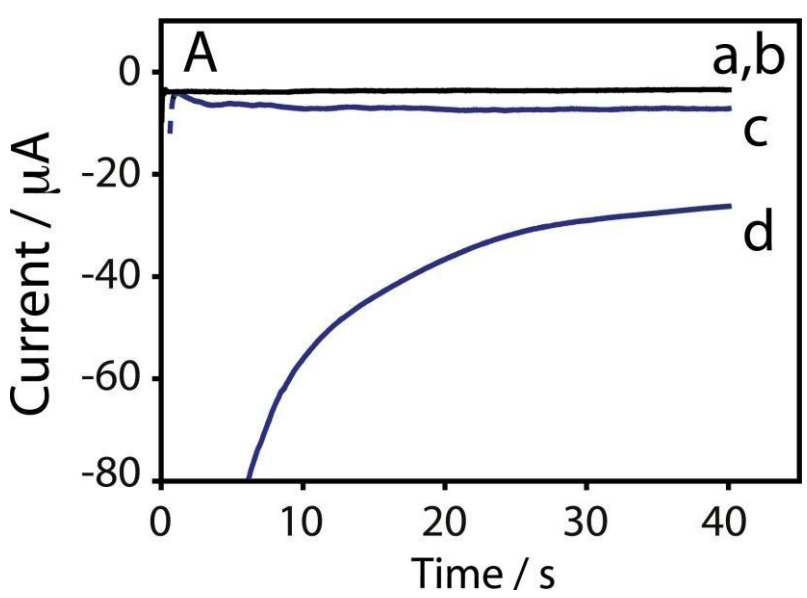


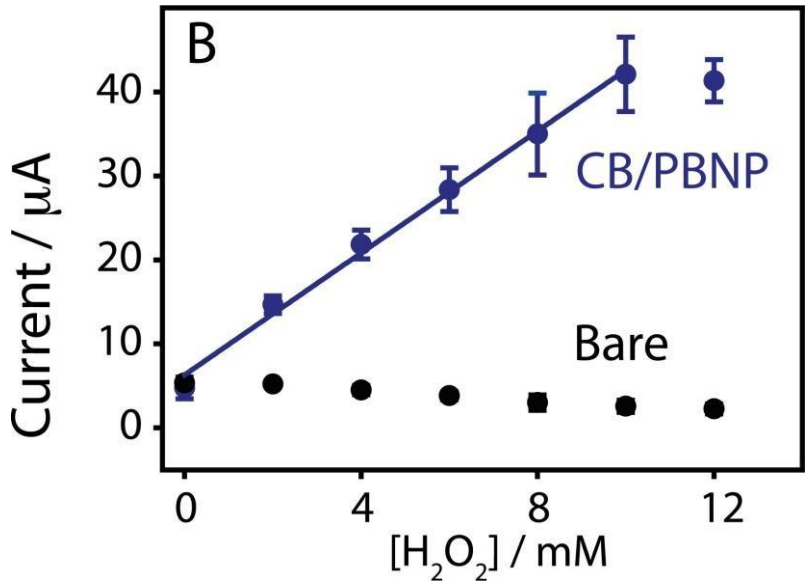
S. Cinti et al. Anal. Chim. Acta 960 (2017) 123-130

Detection mechanism

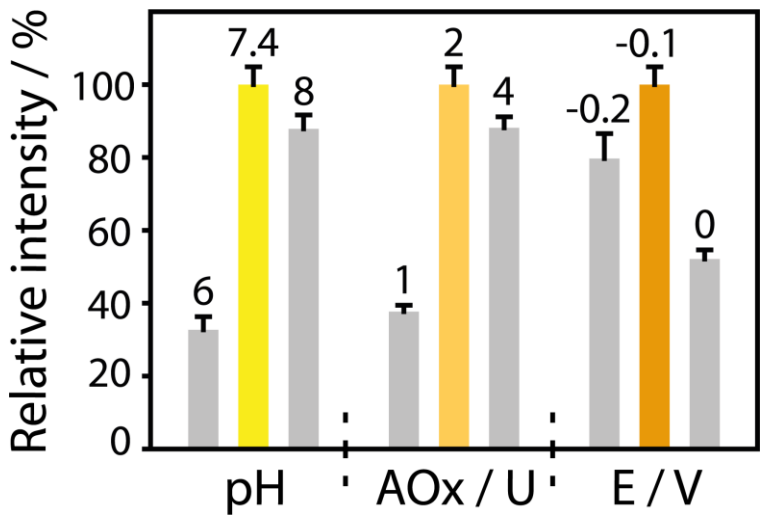


- Office paper
- Carbon Black
- Prussian Blue
- Alcohol oxidase

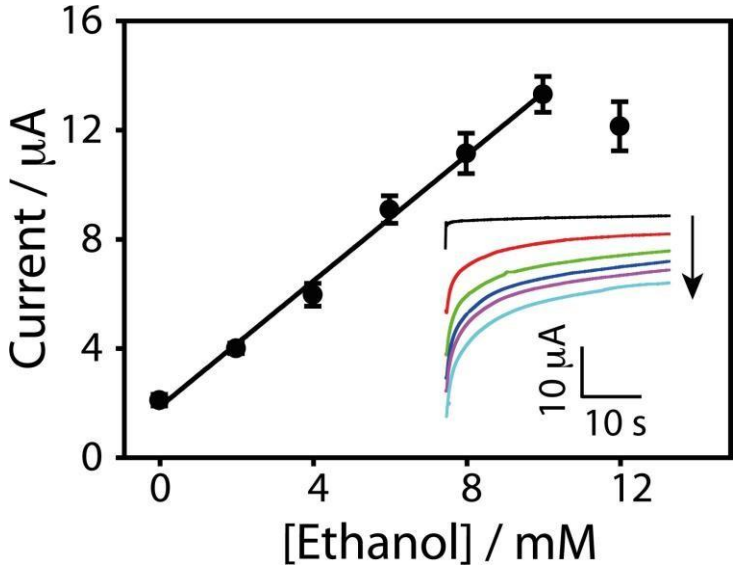




Optimization



Calibration curve



LOD = 0.5 mM

Linear range up to 10 mM

RSD = 8 %

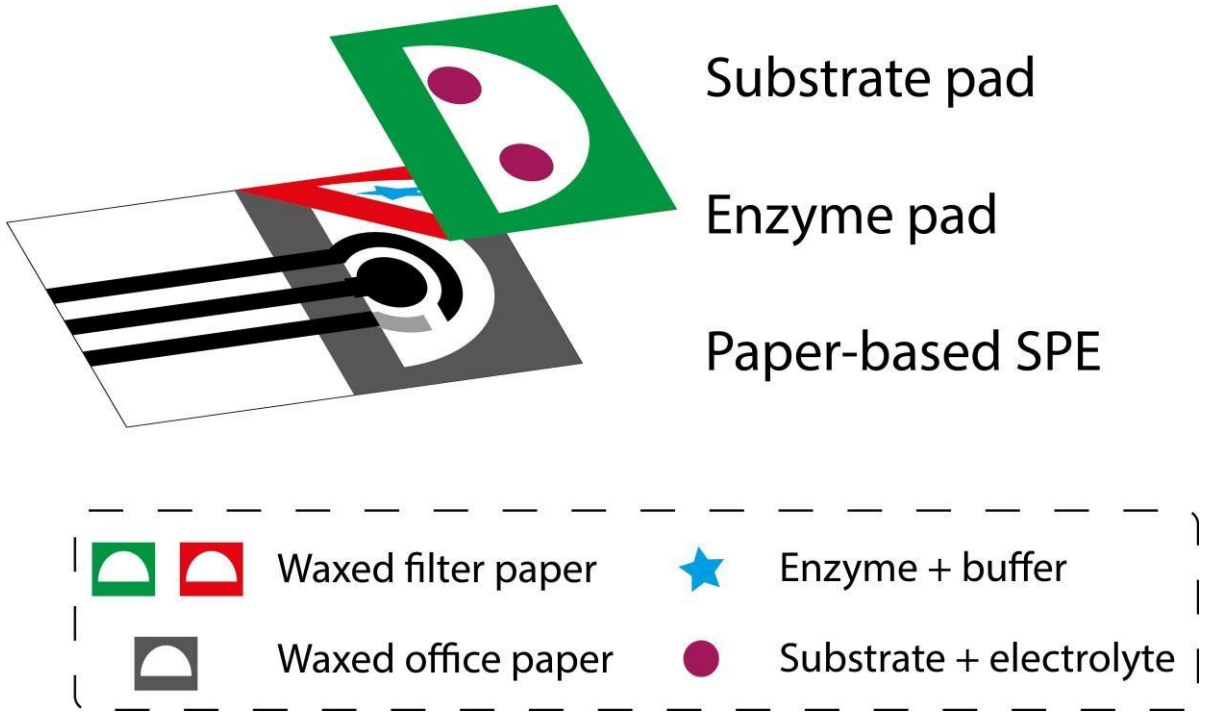
Accordance with label

Detection of ethanol in commercial beers.

Beer	Lager Best Bräu, Poland	Weiss Franziskaner, Germany	Pilsner Ceres, Denmark	Alcohol free Tourtel, Italy
				
Label [ethanol]/%$_{vol}$ (M)	4.7% (0.805 M)	5% (0.856 M)	4.6% (0.787 M)	<0.5% (0.086 M)
Found [ethanol]/%$_{vol}$ (M)	4.7 ± 0.4 (0.805 ± 0.075)	5.0 ± 0.4 (0.86 ± 0.07)	4.4 ± 0.2 (0.75 ± 0.04)	0.34 ± 0.03 (0.059 ± 0.004)
RSD/%	9.3	8.1	5.3	6.8

3-D paper origami for pesticides

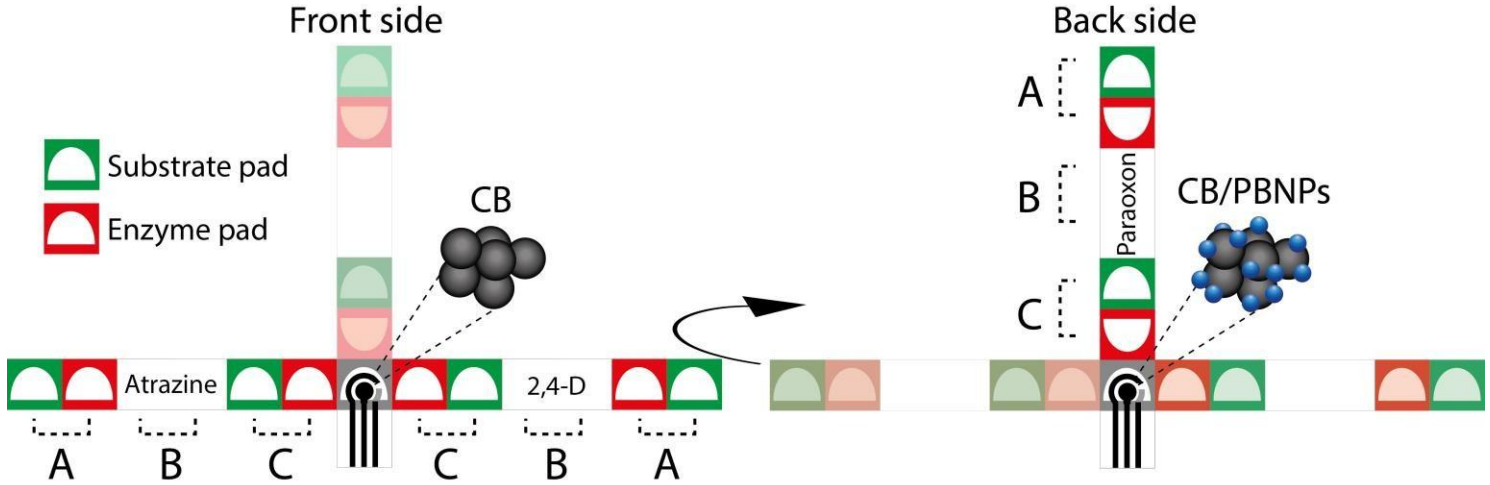
Filter paper + office paper



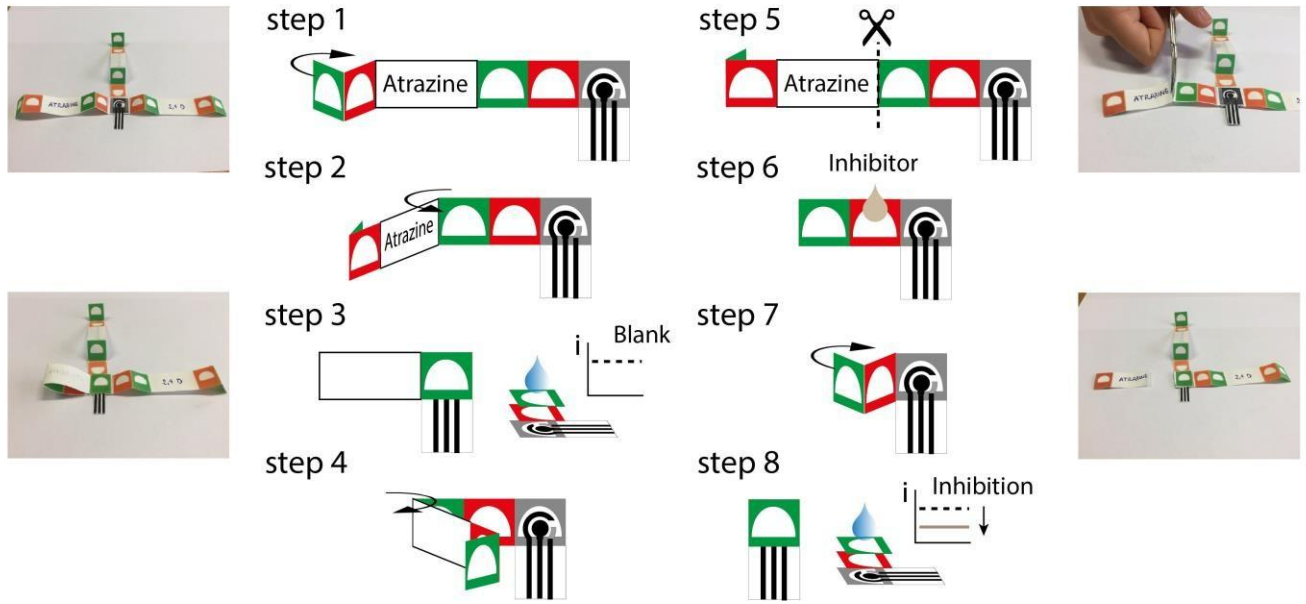
Paraoxon, **2,4-dichlorophenoxyacetic acid**, and **atrazine** by inhibition of **butyrylcholinesterase**, **alkaline phosphatase,** and **tyrosinase**

F. Arduini et al. Biosens. Bioelectron. (2018) DOI:10.1016/j.bios.2018.10.014

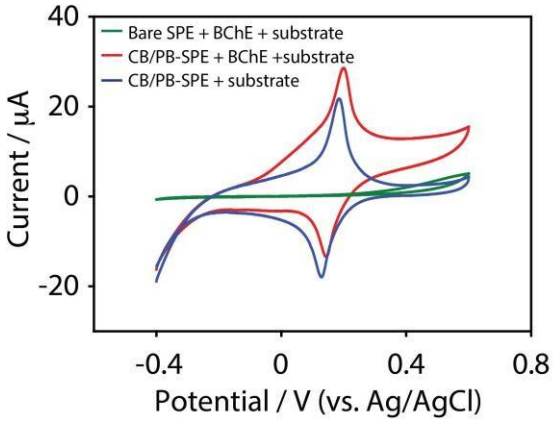
Configuration

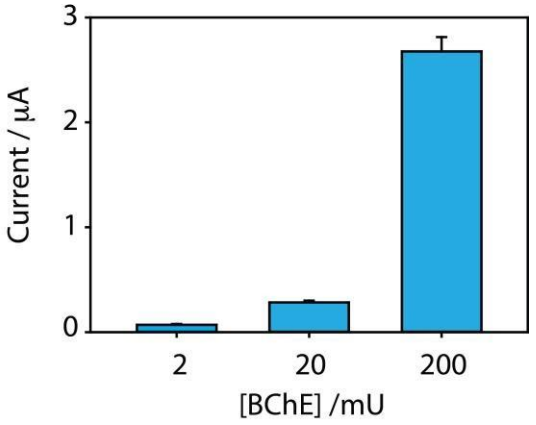


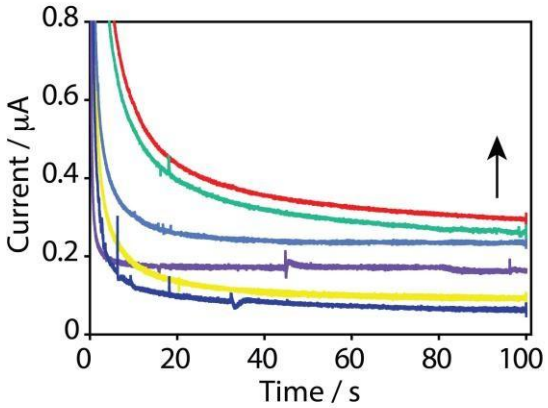
Measurements, e.g. Atrazine

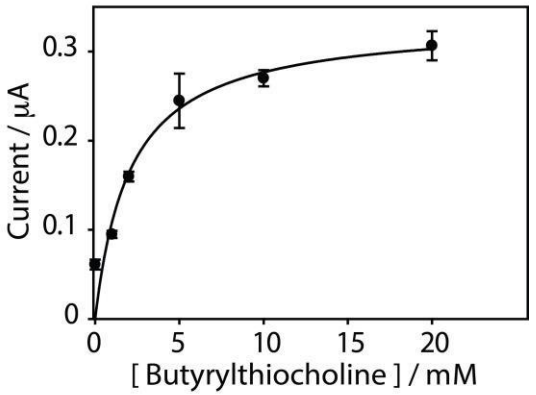


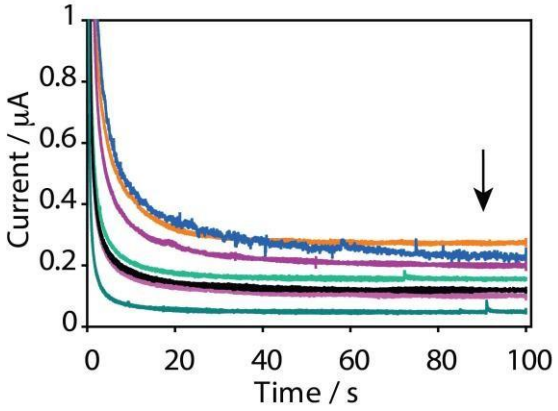
E.g. paraoxon detection

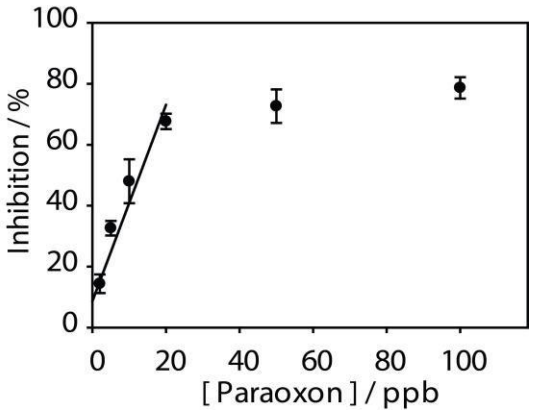












LOD = 2 ppb

Linear range up to 30 ppb

RSD = 11%

<u>Real sample: River water</u>

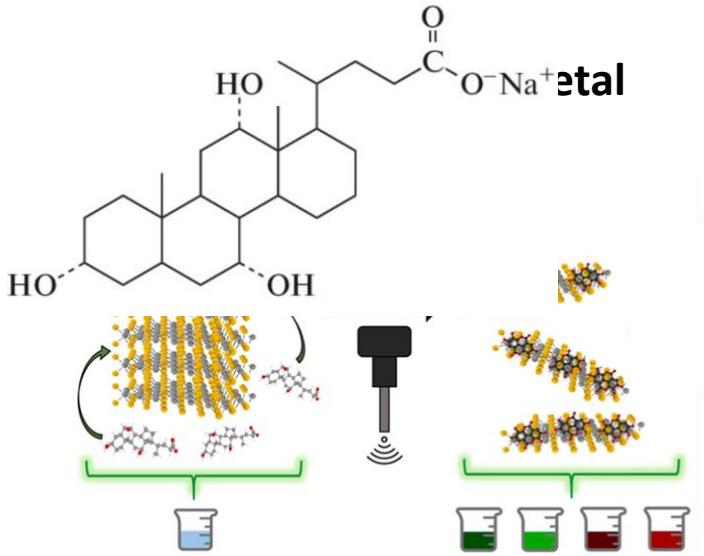
Linear up to 30 ppb

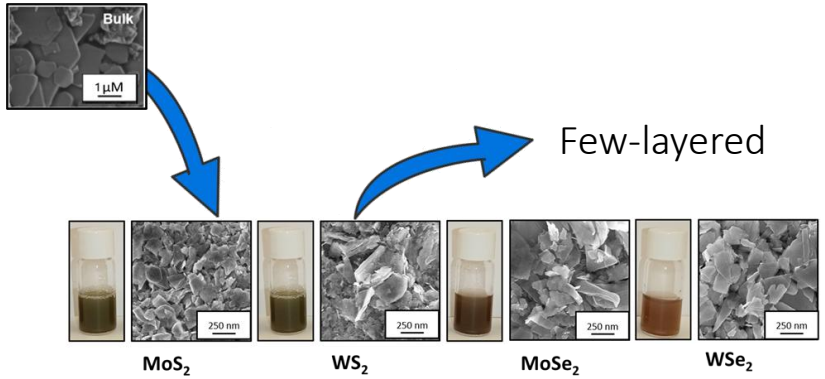
Recoveries: 90 and 88% (10 and 20 ppb)

Green and sustainable production of nanomaterials

Sodium cholate as redox-inert exfoliating agent for nanomaterials synthesis/preparation in water

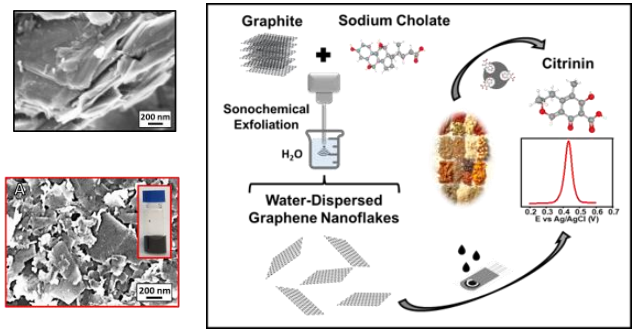
UNITE UNIVERSITÀ DEGLI STUDI DI TERAMO





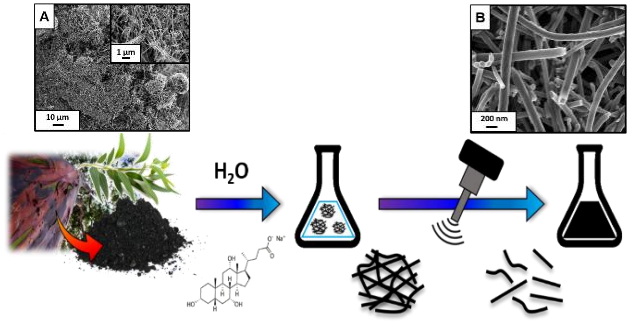
Rojas, D., Della Pelle, F., Del Carlo, M., Compagnone, D., & Escarpa, A. (2020). Electrochemistry Communications, 115, 106718.

2D Graphene nanoflakes



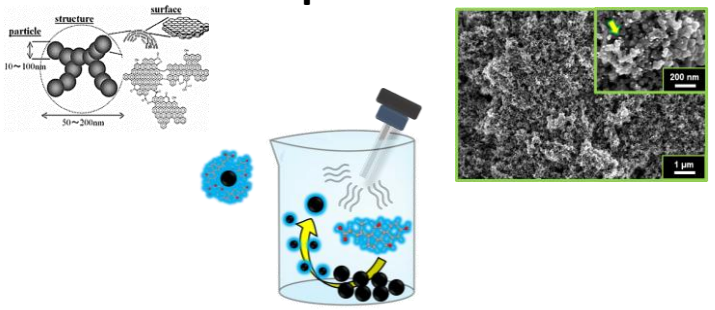
Elfadil, D., Silveri, F., Palmieri, S., Della Pelle, F.*, Sergi, M, Del Carlo, M., Amine, A.**, Compagnone, D. (2022). Talanta. 124010

1D Biochar nanofibers



Bukhari, Q.U.A; Silveri, F.; Della Pelle, F.*; Scroccarello, A.; Zappi, D.; Cozzoni, E.; Compagnone, D.*. (2021) ACS Sustainable Chem. & Eng, 9, 41

OD Carbon Black and mesoporous carbon

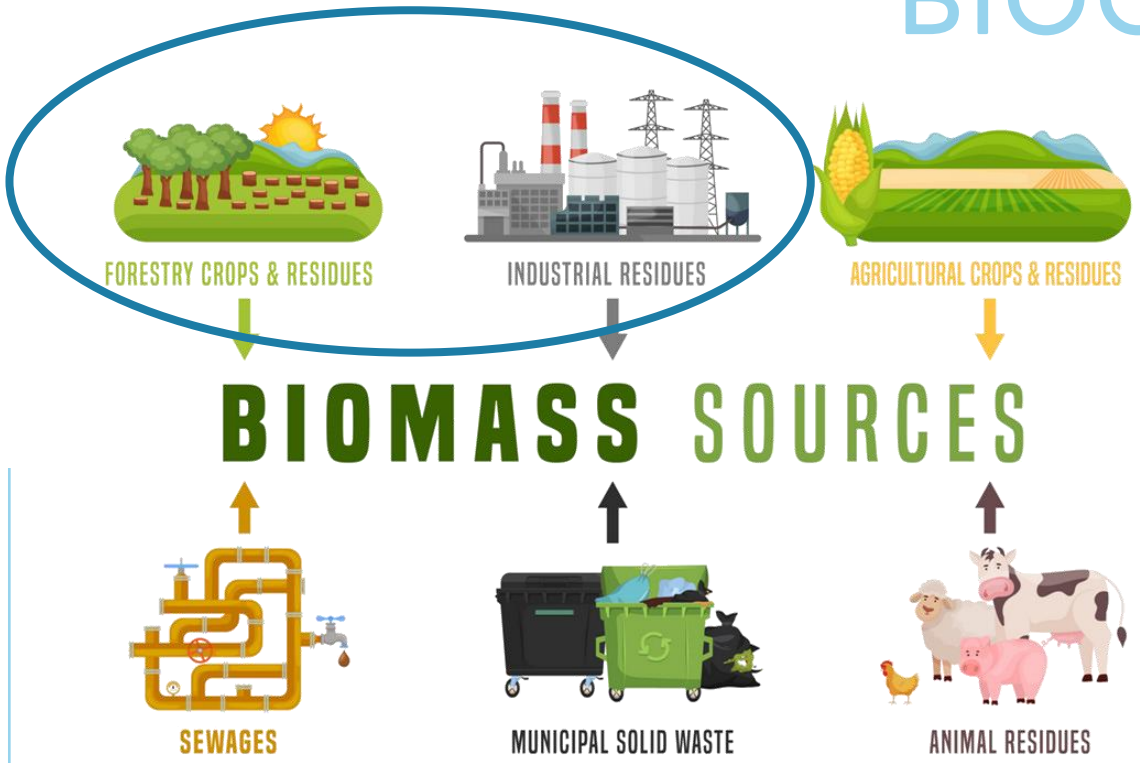


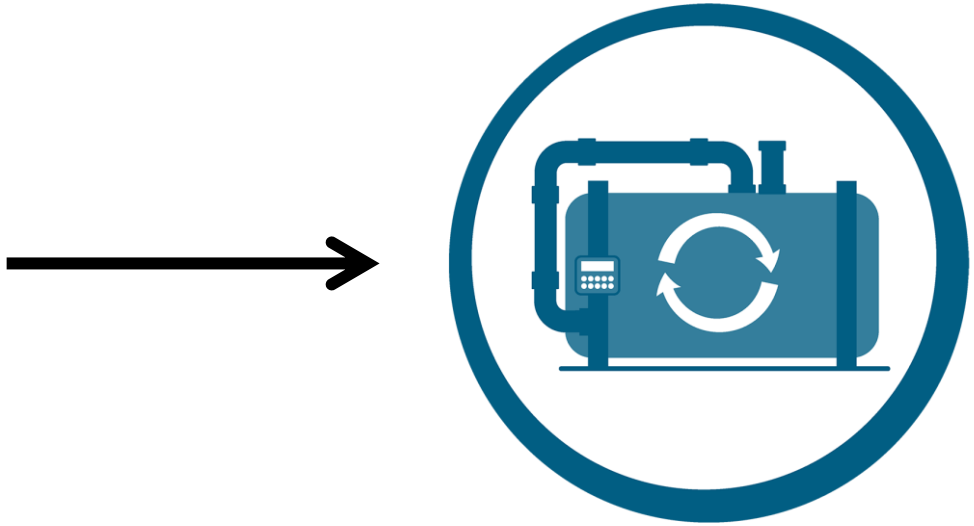
Silveri, F., Della Pelle, F. *, Scroccarello, A., Mazzotta, E., Di Giulio, T., Malitesta, C., Compagnone, D. * (2022). Antioxdants,11, 2008

Sodium cholate as E.A. and S.A.

Effective exfoliation/nanodispersion
NMs easily purifiable
NMs Stable
Low cost
Redox-inert

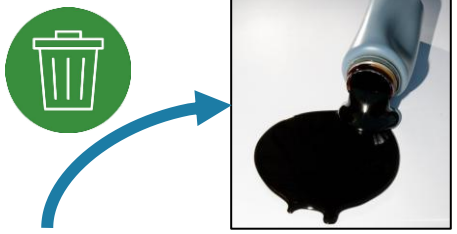
BIOCHAR





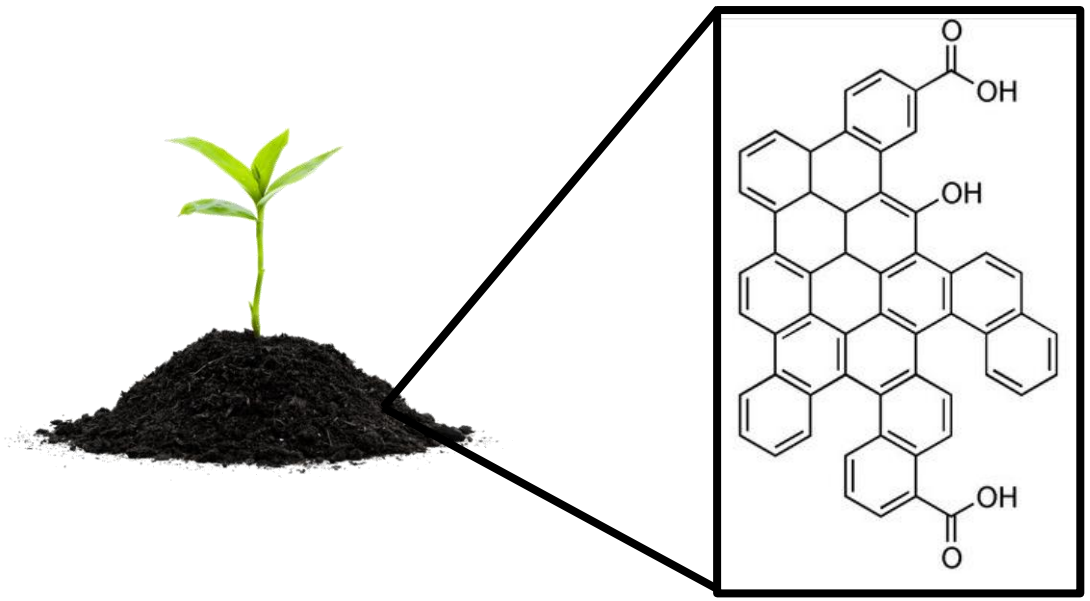
Pyrolysis
controlled temperature and atmosphere

PAPER INDUSTRY

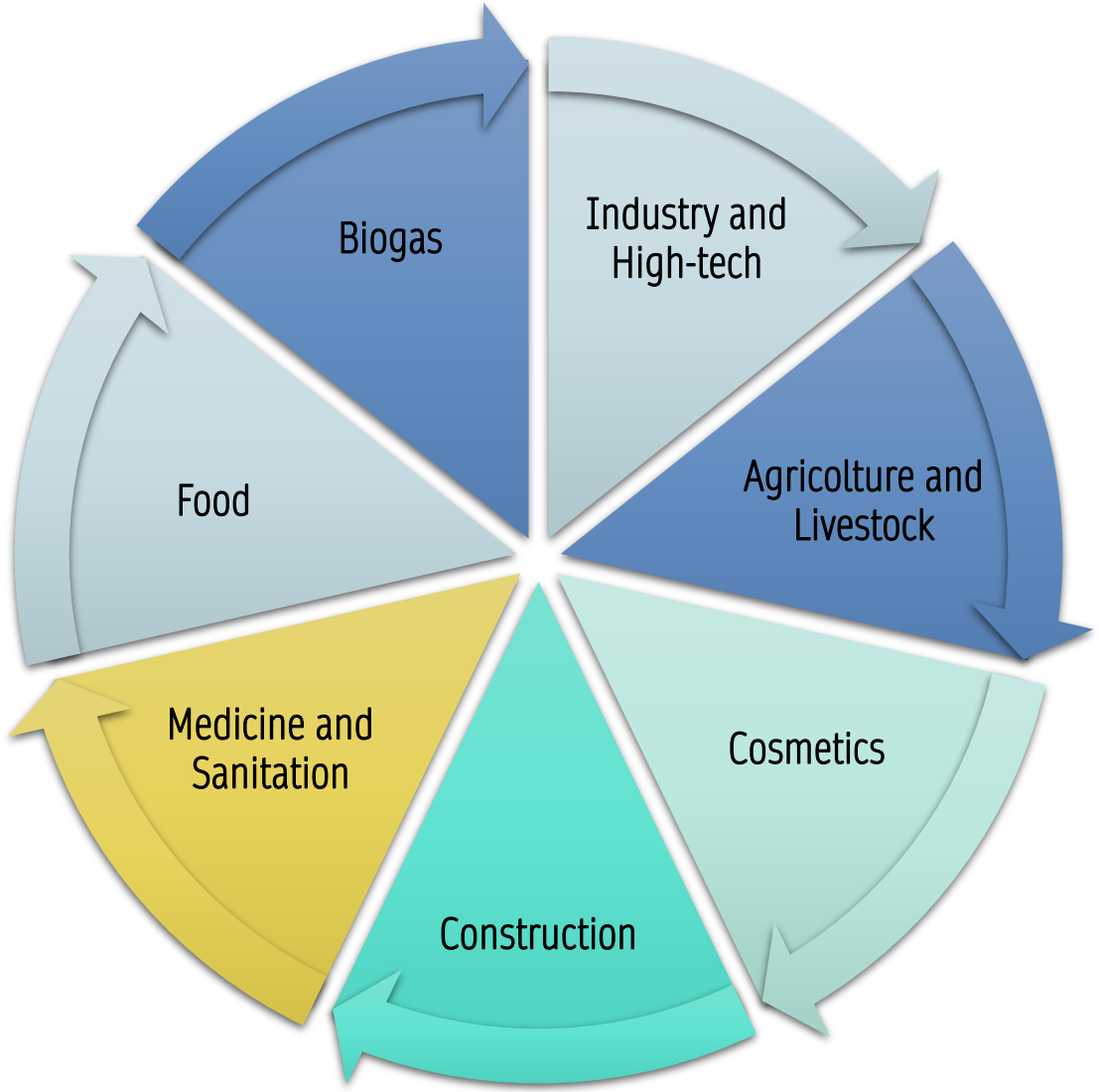


Black liquor

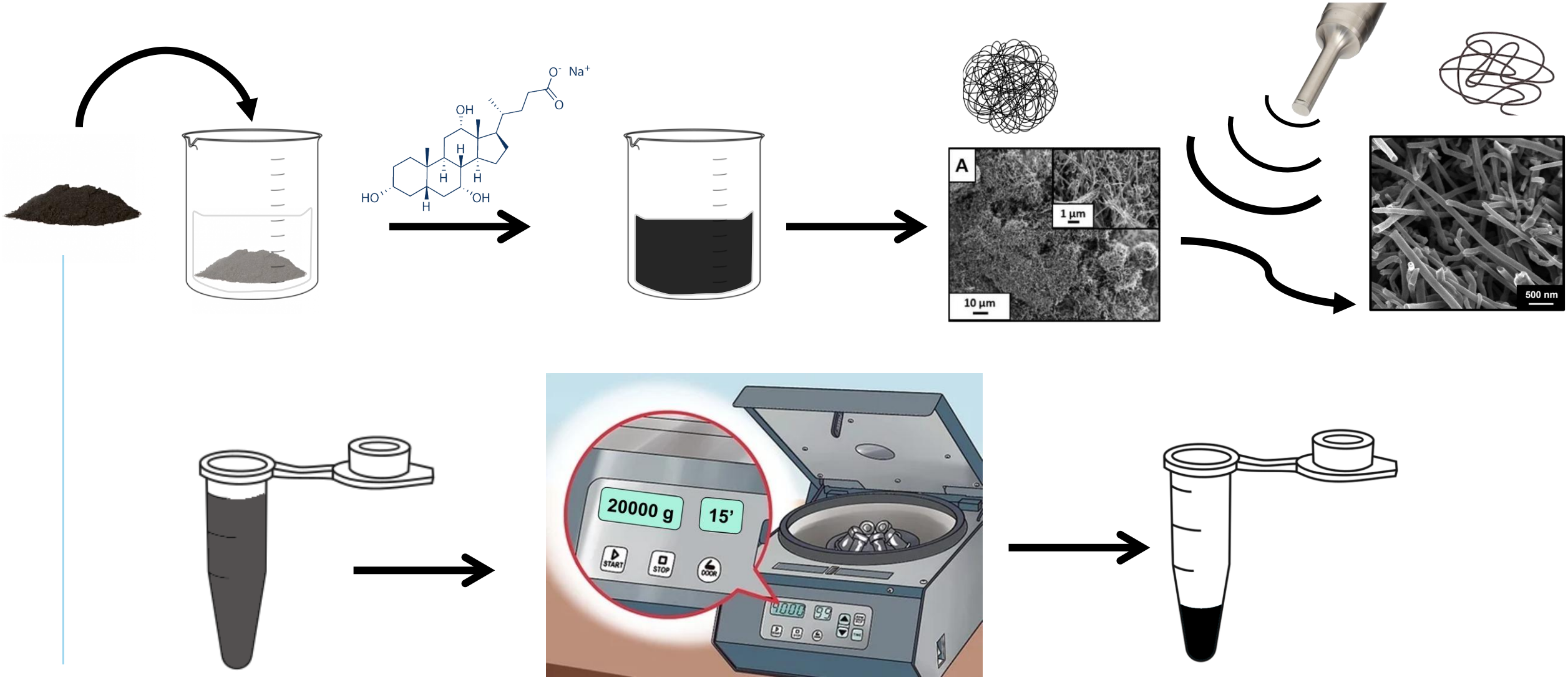
BIOCHAR



- Oxygen-rich functionalization, amorphous carbons, mostly hybridized sp^3, and crystalline areas with sp^2 graphene-like conjugated carbons
- Large surface area, good stability, and properties similar to those of other carbon-based materials

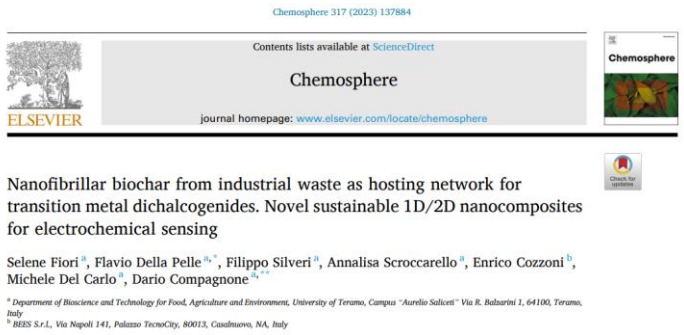


BIOCHAR: WATER-PHASE 'EXFOLIATION'



Bukhari, Q. U. A., Silveri, F., Della Pelle, F., Scroccarello, A., Zappi, D., Cozzoni, E., & Compagnone, D.. *ACS Sustainable Chemistry & Engineering*, 2021, 9(41), 13988-13998.

Nanofibrillar biochar from industrial waste as hosting network for transition metal dichalcogenides. Novel sustainable 1D/2D nanocomposites for electrochemical sensing


Chemosphere 317 (2023) 137884

Contents lists available at ScienceDirect

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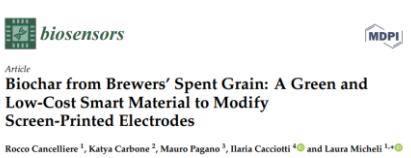
journal homepage: www.elsevier.com/locate/chemosphere

Nanofibrillar biochar from industrial waste as hosting network for transition metal dichalcogenides. Novel sustainable 1D/2D nanocomposites for electrochemical sensing

Selene Fiori [a], Flavio Della Pelle [a,*], Filippo Silveri [a], Annalisa Scroccarello [a], Enrico Cozzoni [b], Michele Del Carlo [a], Dario Compagnone [a,**]

[a] Department of Bioscience and Technology for Food, Agriculture and Environment, University of Teramo, Campus "Aurelio Saliceti" Via R. Balzarini 1, 64100, Teramo, Italy

[b] BEES S.r.l., Via Napoli 141, Palazzo TecnoCity, 80013, Casalnuovo, NA, Italy


biosensors

MDPI

Article

Biochar from Brewers' Spent Grain: A Green and Low-Cost Smart Material to Modify Screen-Printed Electrodes

Rocco Cancelliere [1], Katya Carbone [2], Mauro Pagano [3], Ilaria Cacciotti [4] and Laura Micheli [1,*]


ACS Sustainable Chemistry & Engineering

Water-Phase Exfoliated Biochar Nanofibers from Eucalyptus Scraps for Electrode Modification and Conductive Film Fabrication

Qurat Ul Ain Bukhari, Filippo Silveri, Flavio Della Pelle, Annalisa Scroccarello, Daniele Zappi, Enrico Cozzoni, and Dario Compagnone


Green Chemistry

ROYAL SOCIETY OF CHEMISTRY

CRITICAL REVIEW

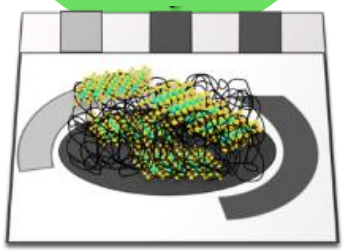
State-of-the-art and perspectives in the use of biochar for electrochemical and electroanalytical applications

grado zero innovation

Nanofibrillar BH from eucalyptus scraps

Exfoliated Transition Metal Dicalchogenides (TMD)

Water-dispersed nanocomposites BH-TMD


Screen-printed electrode modification

Dopamine

Serotonin

Quercetin

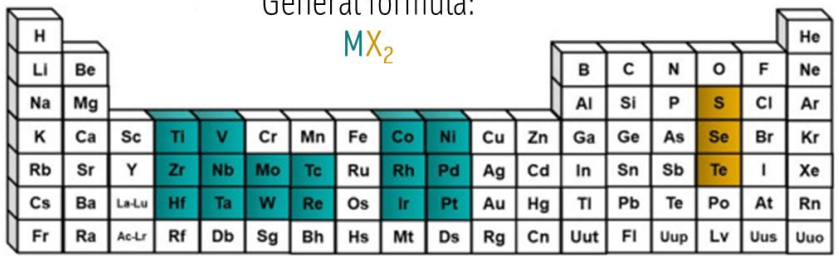
Rutin

Food supplements, biological fluids and drugs

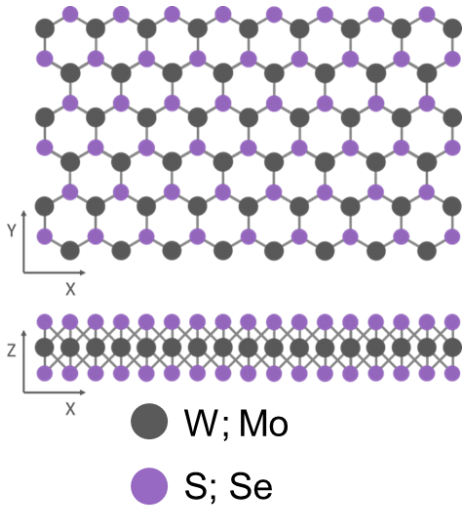


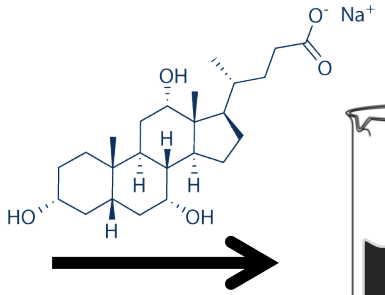

General formula:

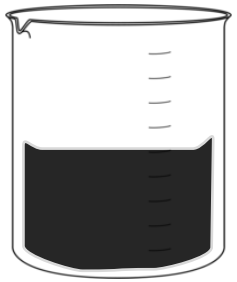
MX_2

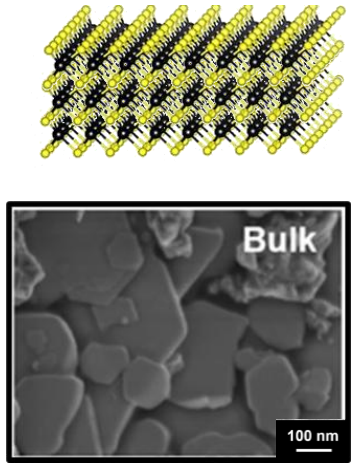


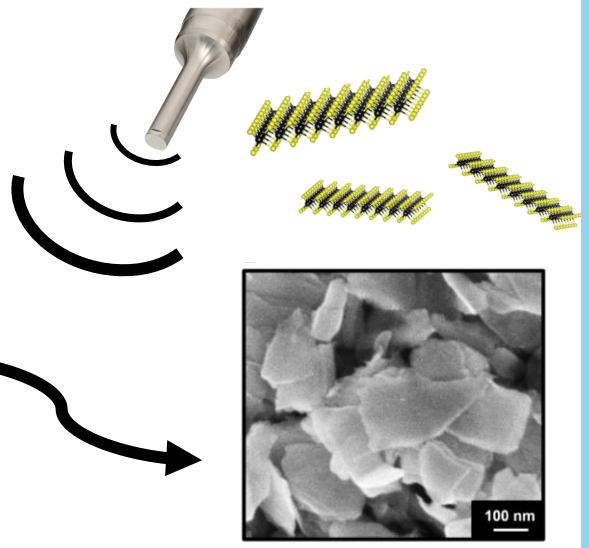
TMD: LIQUID-PHASE EXFOLIATION

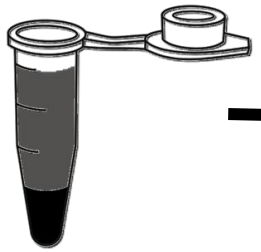


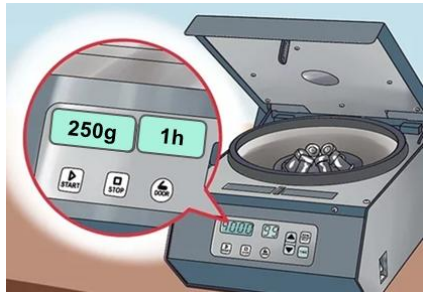


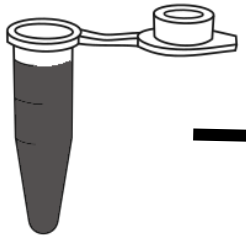


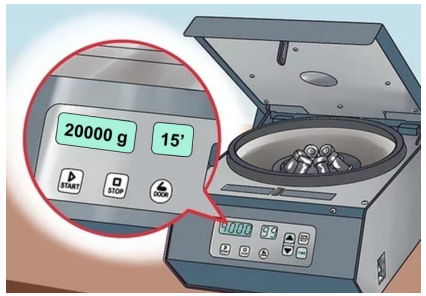


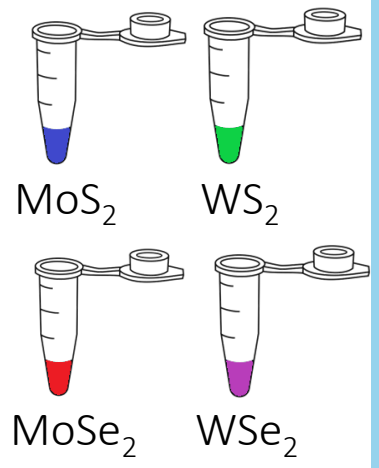




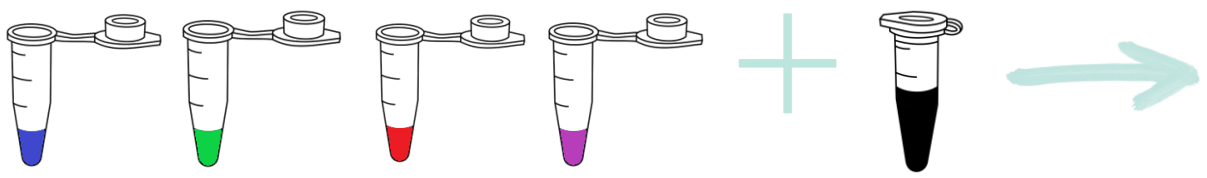


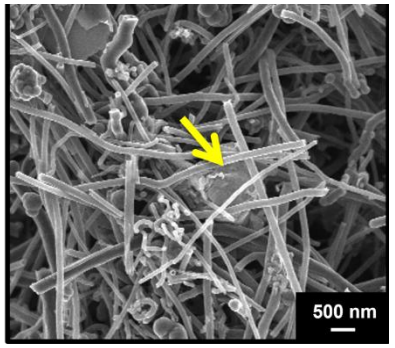




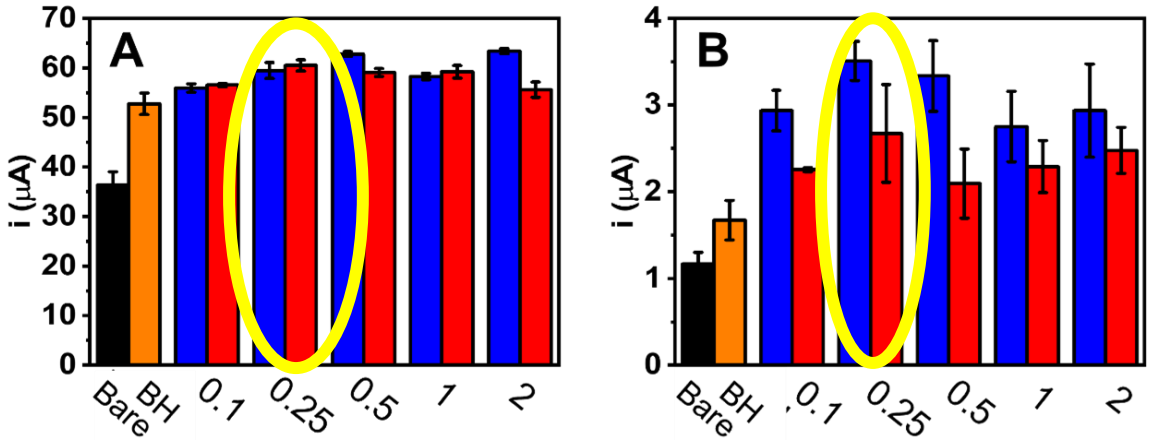


Nanocomposites formation





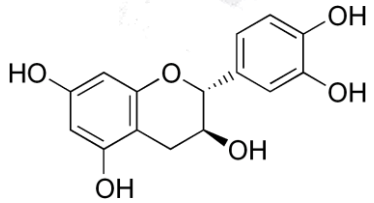
Four BH-TMDs NCs in water used to modify commercial screen-printed electrodes via drop-casting



BH → 1 mg/mL
TMD → 0.25 mg/mL

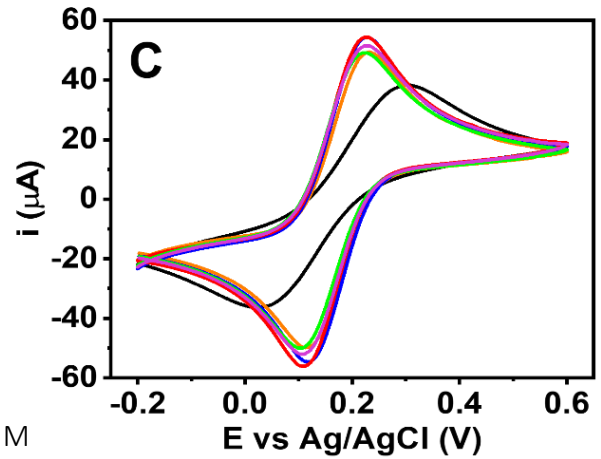
A, C, D- $[Fe(CN)_6]^{3-/4-}$

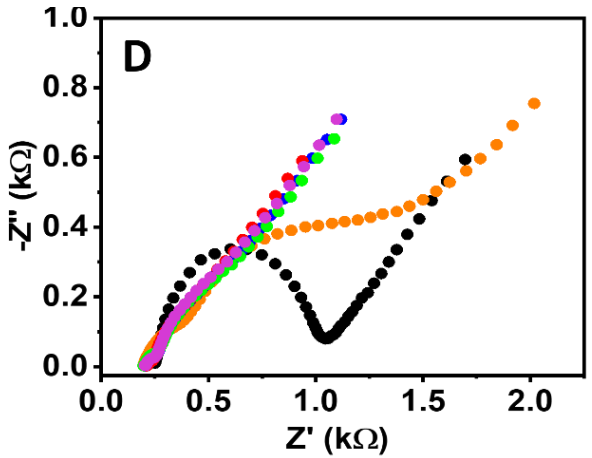
B-



BH-MoS_2

BH-$MoSe_2$

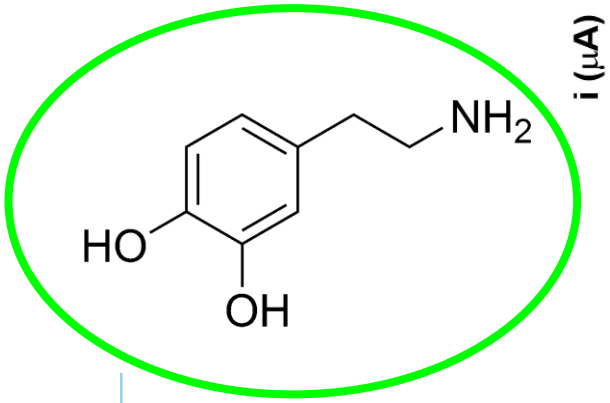


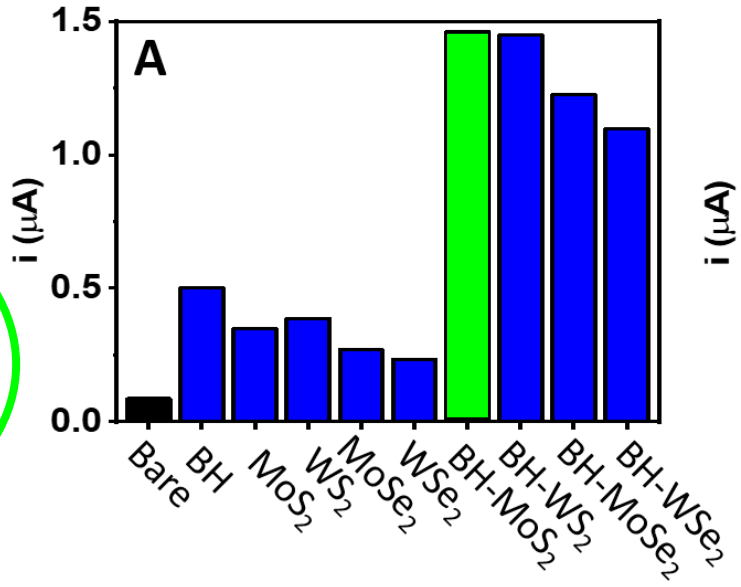


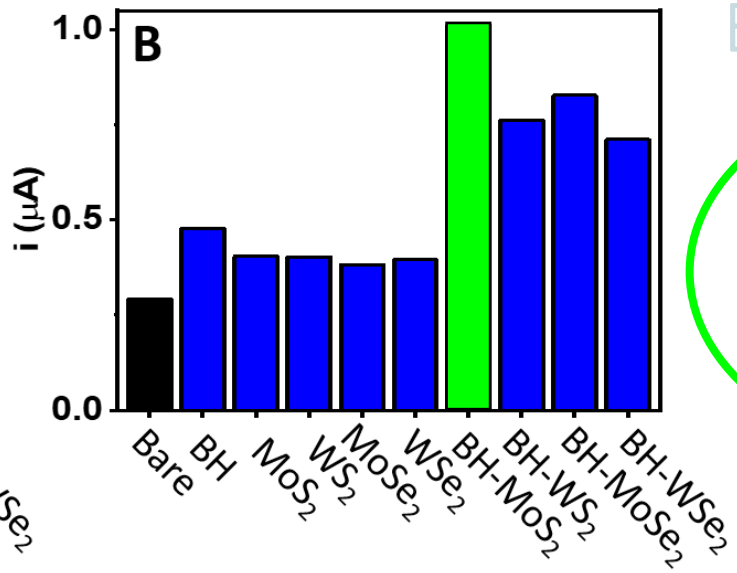
Anodic peak intensity extrapolated from CVs carried out at 25 mV s^{-1} with 5 mM $[Fe(CN)_6]^{3-/4-}$ in 0.1 M KCl (A, C) and 100 μM catechin (B). EIS performed with 5 mM $[Fe(CN)_6]^{3-/4-}$ in 0.1 M KCl (D)

Electrosensing ability

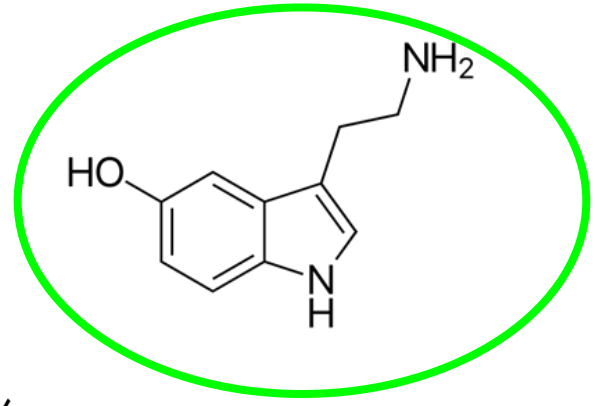
A DOPAMINE 5 µM



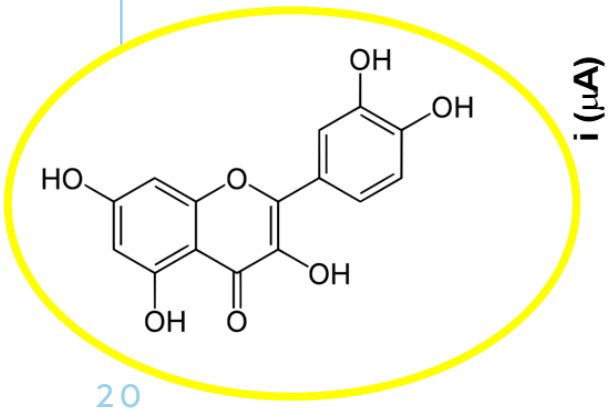


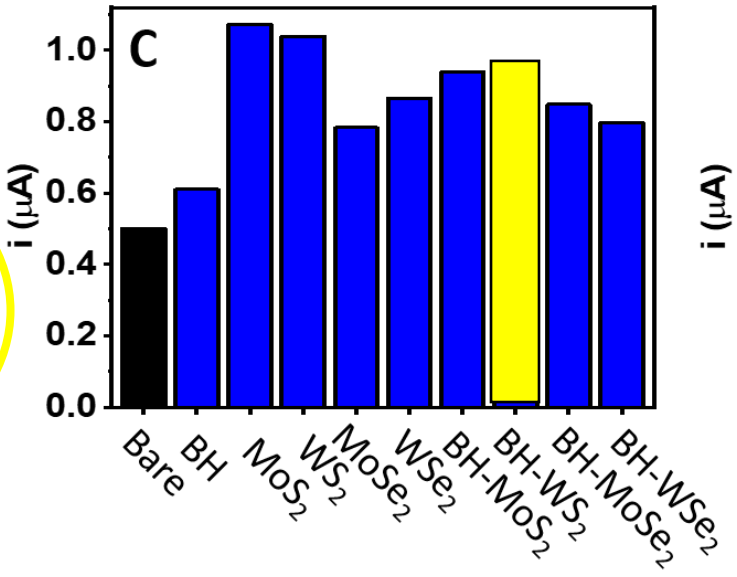


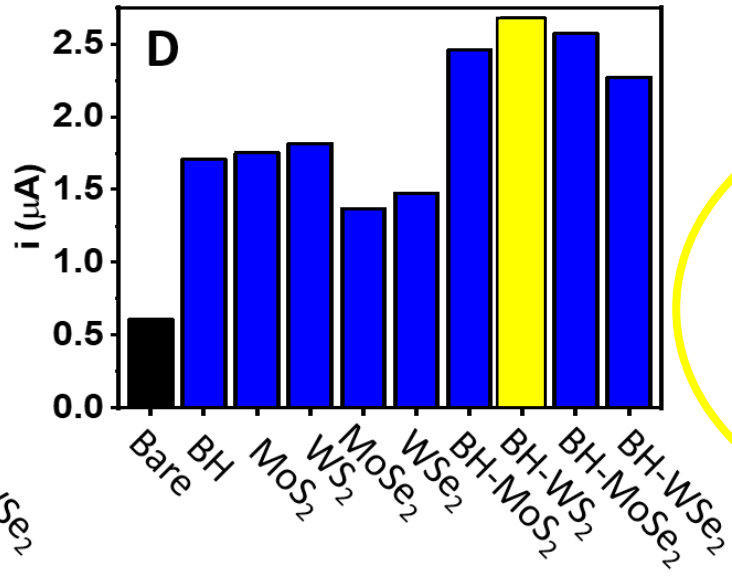
B SEROTONIN 10 µM



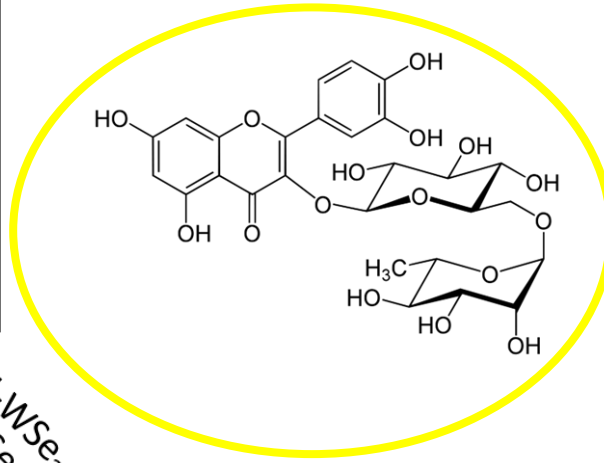
C QUERCETIN 5 µM





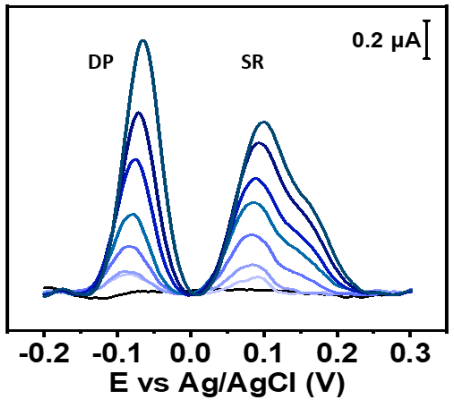


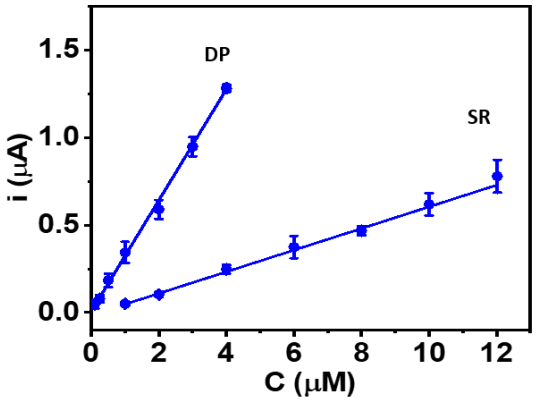
D RUTIN 5 µM



Current intensity extrapolated from DPV performed in PB pH 7

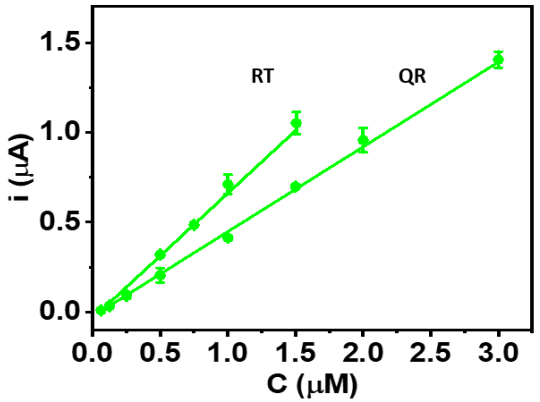
Calibration and sample analysis

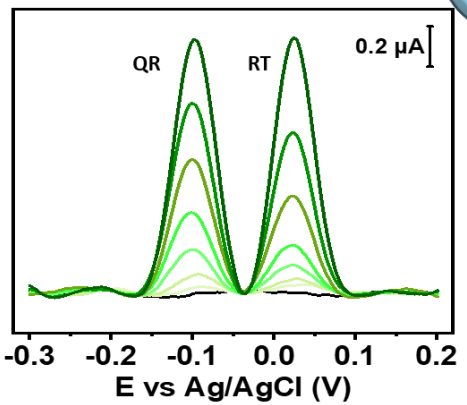




Dopamine
LOD = 0.02 µM
L.R. 0.1-4 µM
RSD≤ 5% (n=3)

Serotonin
LOD=0.21 µM
L.R. 1-12 µM
RSD≤ 5% (n=3)

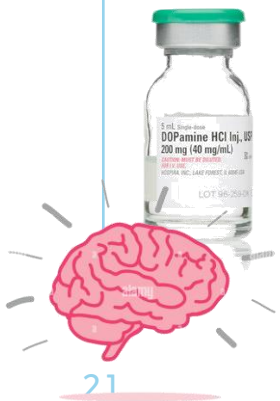


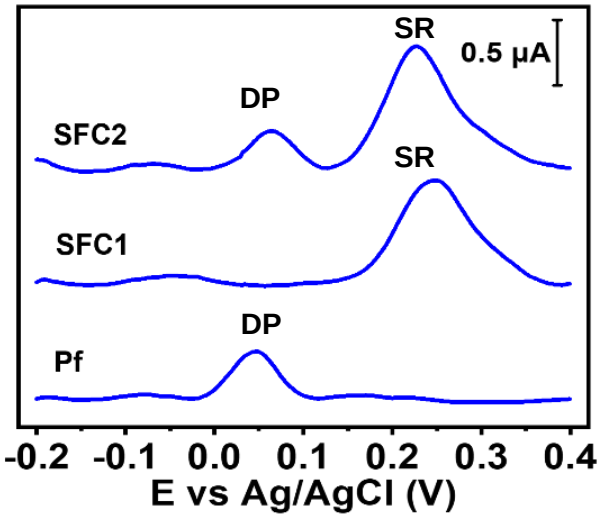


Quercetin
LOD=0.03 µM
L.R. 0.12-3 µM
RSD≤ 4% (n=3)

Rutin
LOD=0.01 µM
L.R. 0.06-1.5 µM
RSD≤ 5% (n=3)

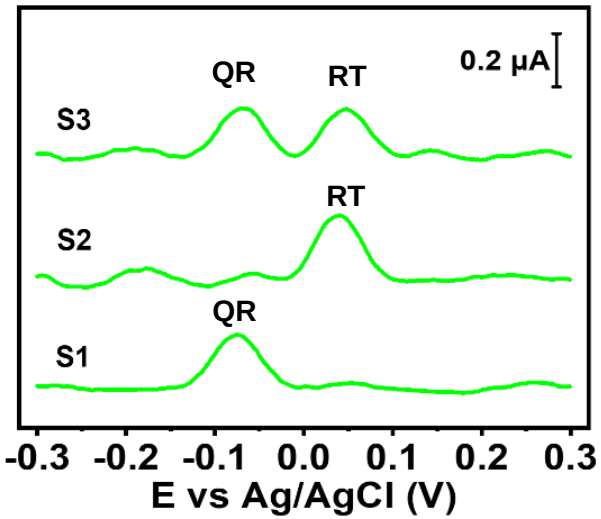
BH-MoS_2





DOPAMINE
Rec = 90-100%
SEROTONIN
Rec = 96-102%
QUERCETIN
Rec = 93-112%
RUTIN
Rec = 90-102%
RSD≤ 6% (n=3)

BH-WS_2

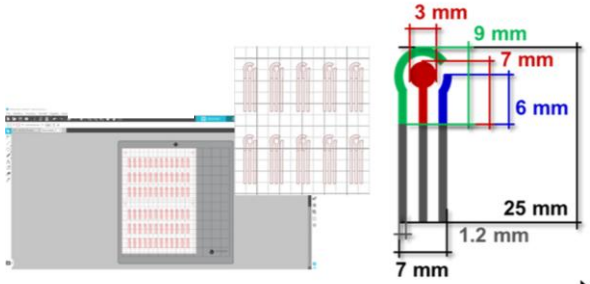


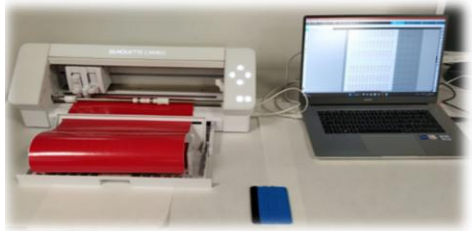
Current intensity extrapolated from DPV performed in PB pH 7

In House Sensors production

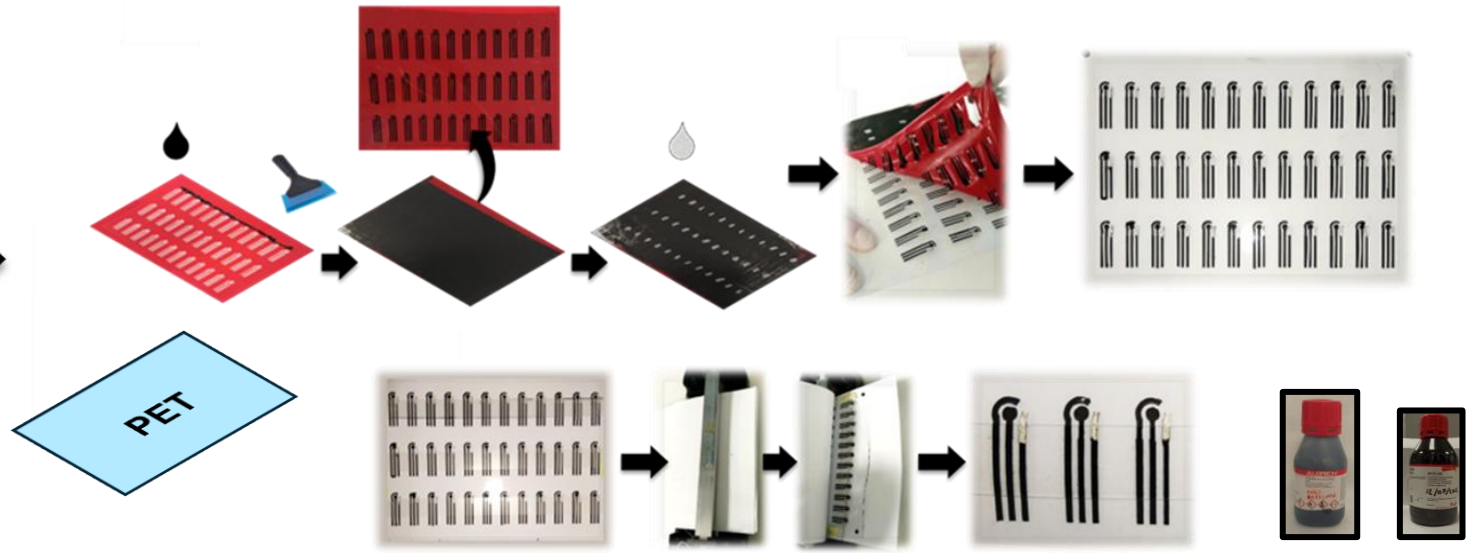
Stencil-printing manufacturing

Flexible polymers

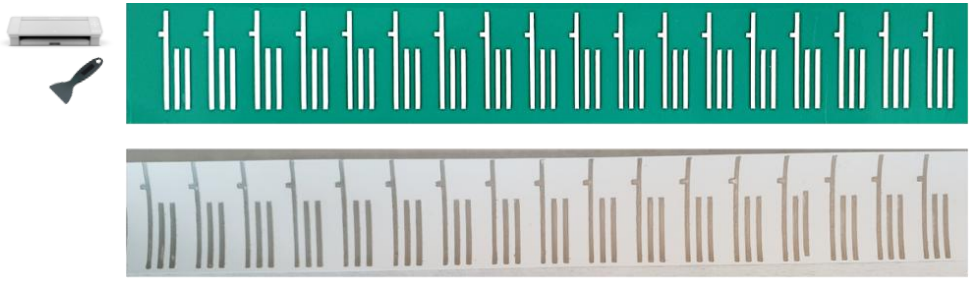




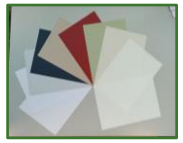
Cutting plotter

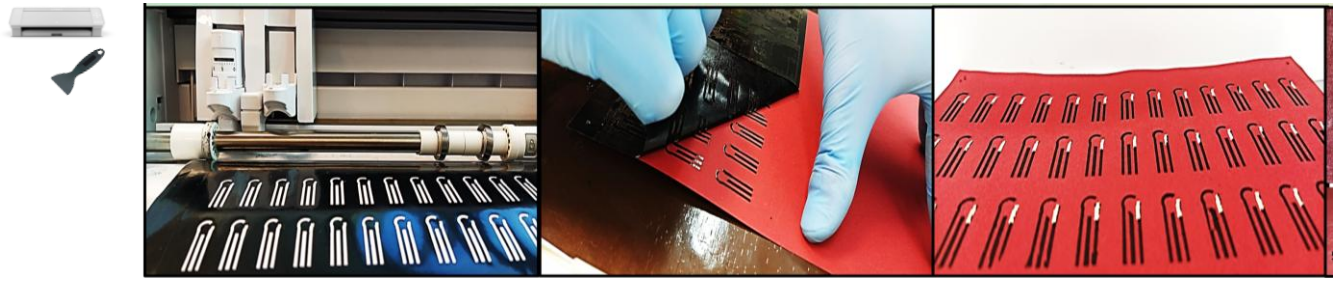


Nitrocellulose



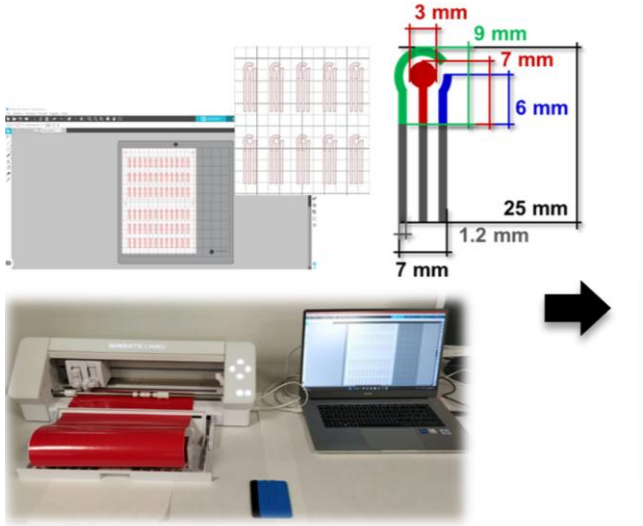
Paper





Silveri et al. (2023). **Biosensors and Bioelectronics**, 237, 115450.
Della Pelle et al. (2023). **Nanoscale**, 15(15), 7164-7175.

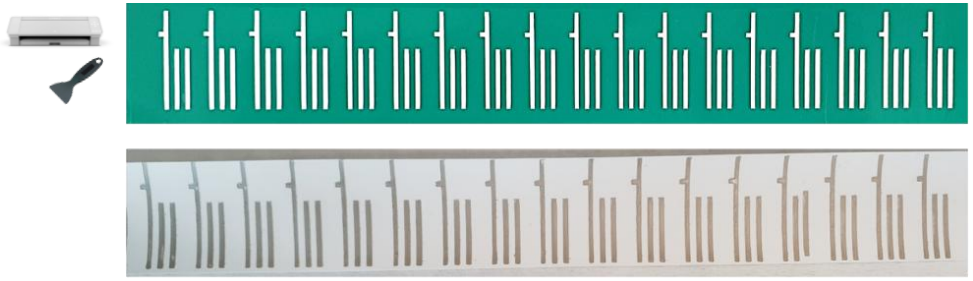
Stencil-printing manufacturing

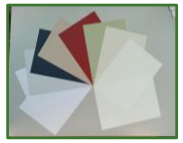


Cutting plotter

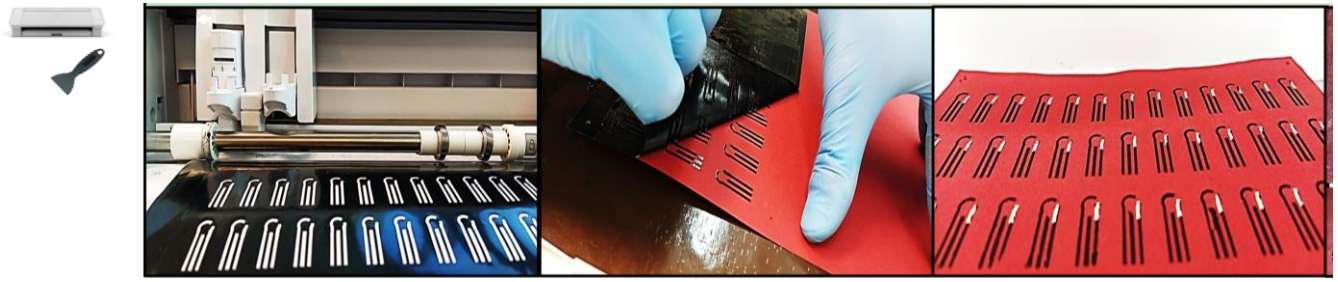
Flexible polymers

Nitrocellulose





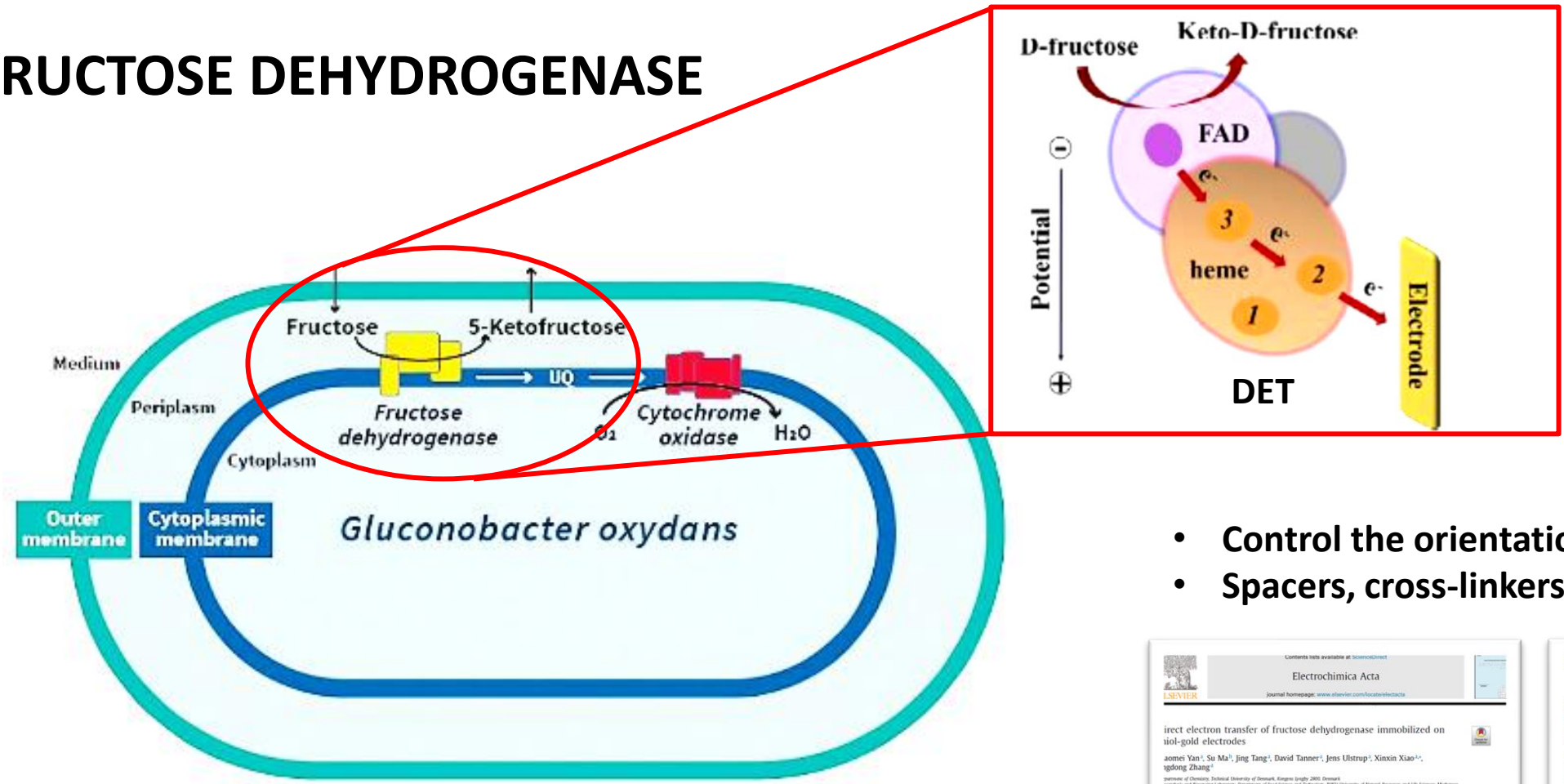
Paper



Silveri et al. (2023). **Biosensors and Bioelectronics**, 237, 115450.
Della Pelle et al. (2023). **Nanoscale**, 15(15), 7164-7175.

INTRODUCTION

FRUCTOSE DEHYDROGENASE



Gluconobacter japonicus (native type EC 1.1.99.11 from NCBR 3260)

- **Control the orientation of the enzyme.**
- **Spacers, cross-linkers, nanomaterials**

Electrochimica Acta

irect electron transfer of fructose dehydrogenase immobilized on iiol-gold electrodes


analytical chemistry

Highly Sensitive Membraneless Fructose Biosensor Based on Fructose Dehydrogenase Immobilized onto Aryl Thiol Modified Highly Porous Gold Electrode: Characterization and Application in Food Samples


ChemPubSoc Europe

DOI: 10.1002/celc.201700861

CHEMELECTROCHEM Articles

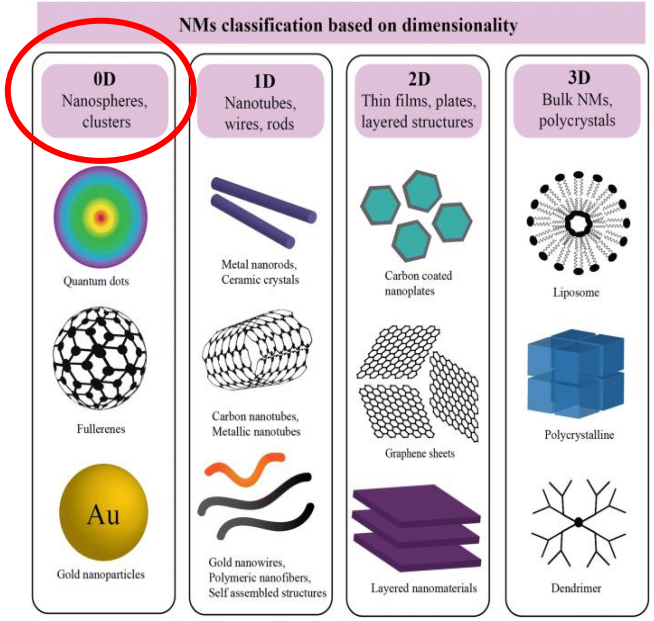
Fructose Dehydrogenase Electron Transfer Pathway in Bioelectrocatalytic Reactions

Michal Kizling and Renata Bilewicz

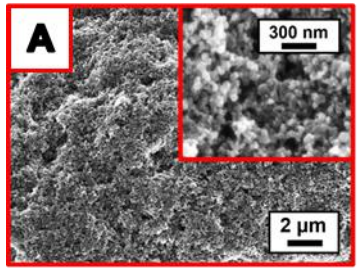
Prof. Keisei Sowa

NANOMATERIALS (NMs)

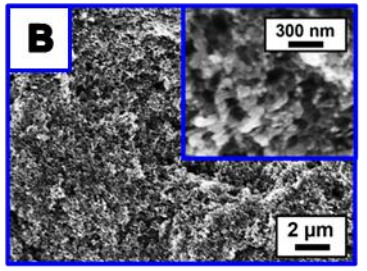
1 nm ≤ NMs ≤ 100 nm



Carbon Black



Mesopourus Carbon

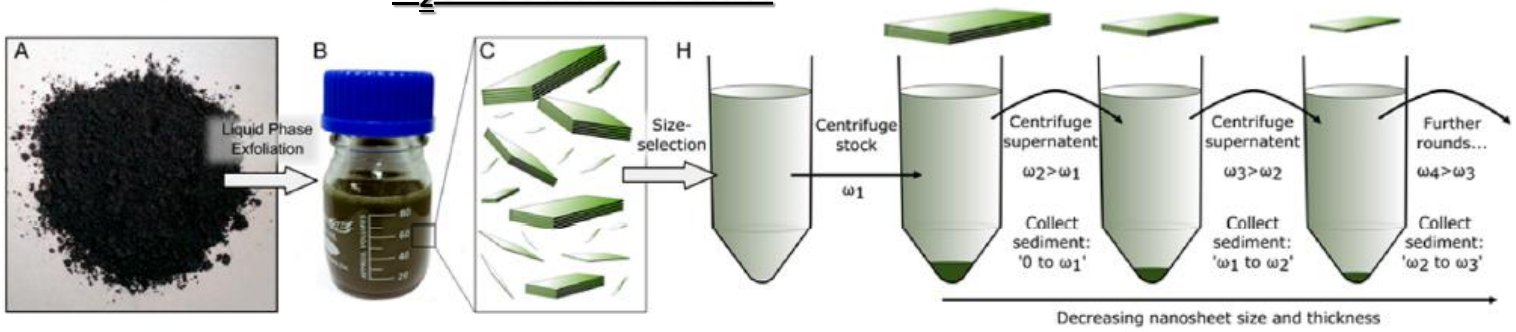


LIQUID PHASE EXFOLIATION/DISPERSION (LPE/D)

Breaking bulk material interaction

Stabilization: H_2O +Sodium Cholate

Purification



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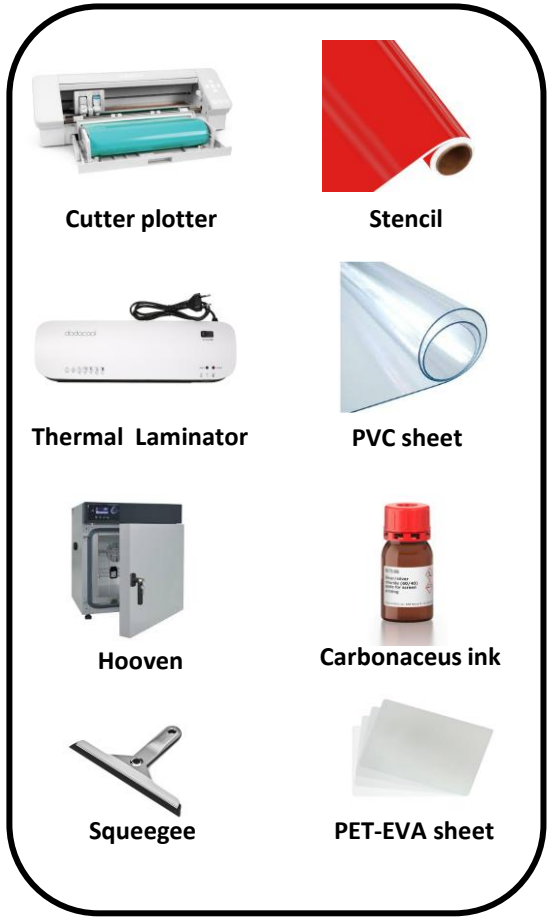
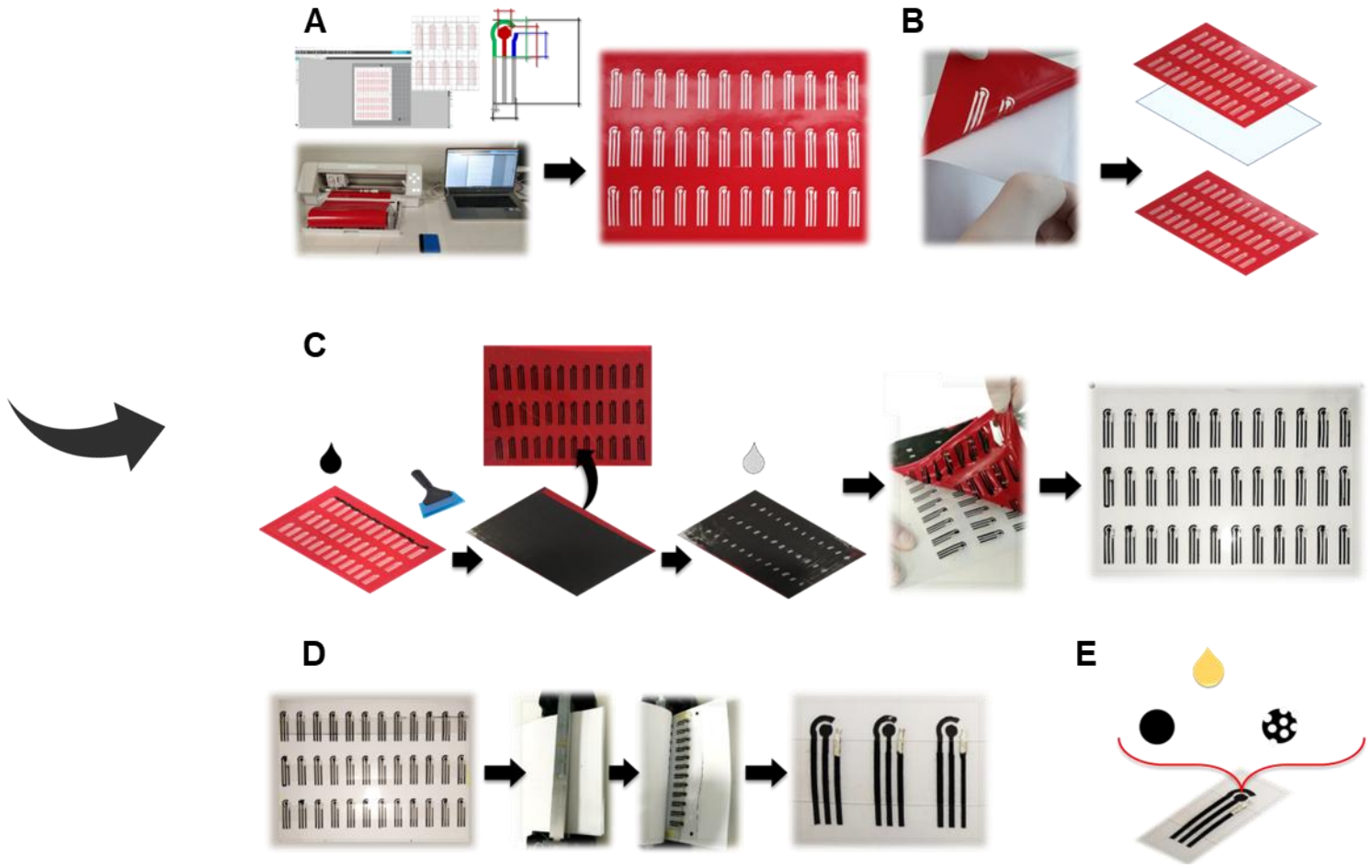
SENSORS MANUFACTURING





Sketch of the nano-StEPs biosensors manufacturing. (A) Stencil-printing mask design and production via cutting plotter engraving. (B) Stencil-mask peeling-off and alignment onto PVC substrate. (C) Stencil printing of electrode contacts with carbon ink and reference electrodes finalization with silver ink. (D) Electrodes contact insulation by thermal lamination. (E) Biosensor assembling via 0D-NMs and FDH modification.

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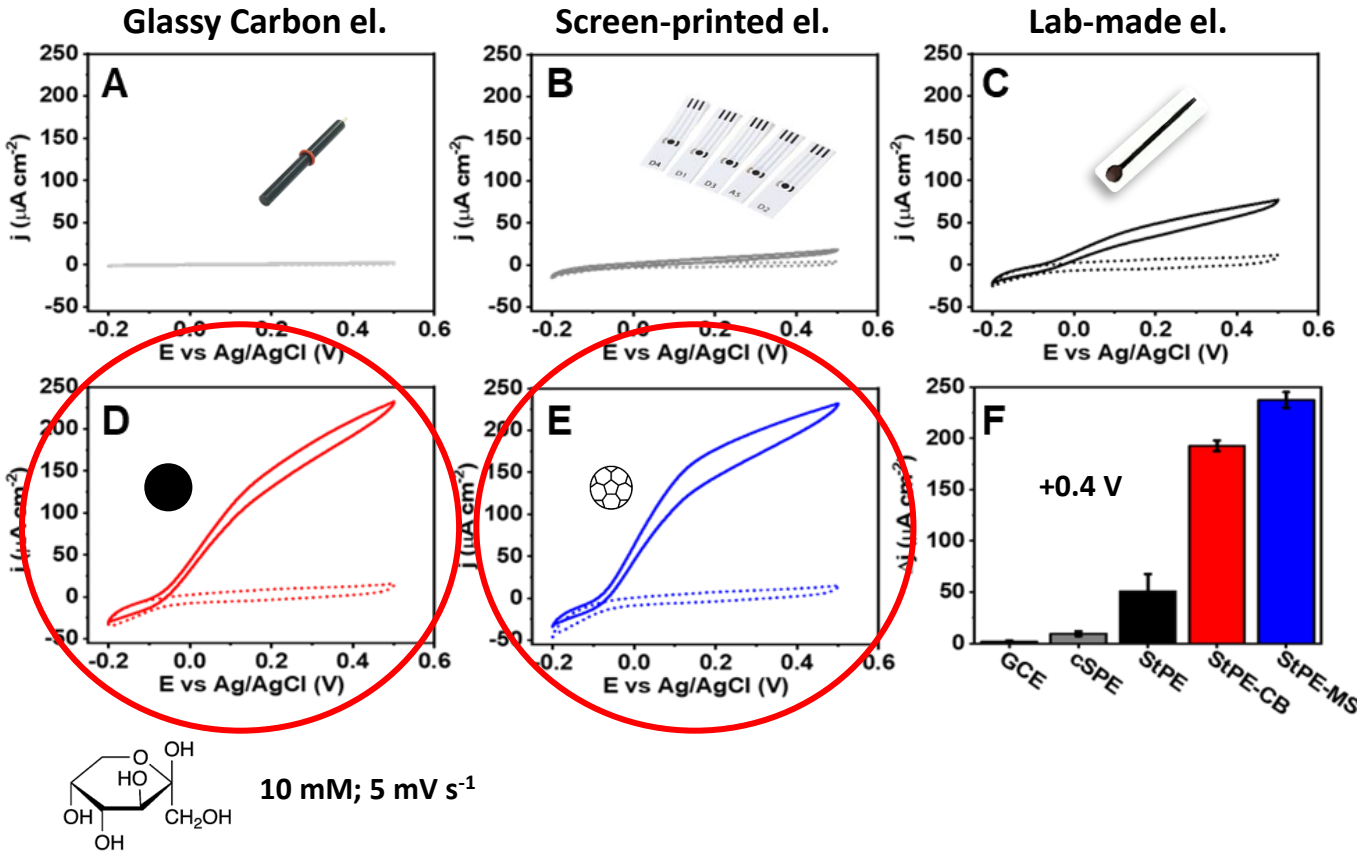
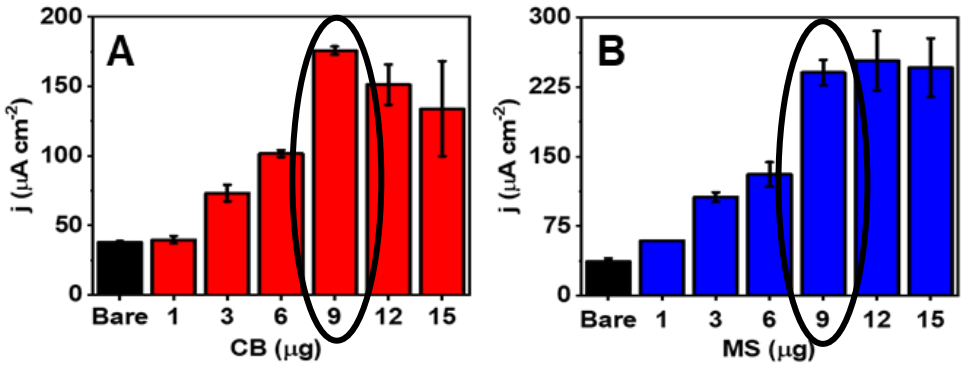
BIOCATALITYC BEHAVIOR

Sensor comparison



10 mM; 5 mV s^{-1}

0D-NMs amount optimization



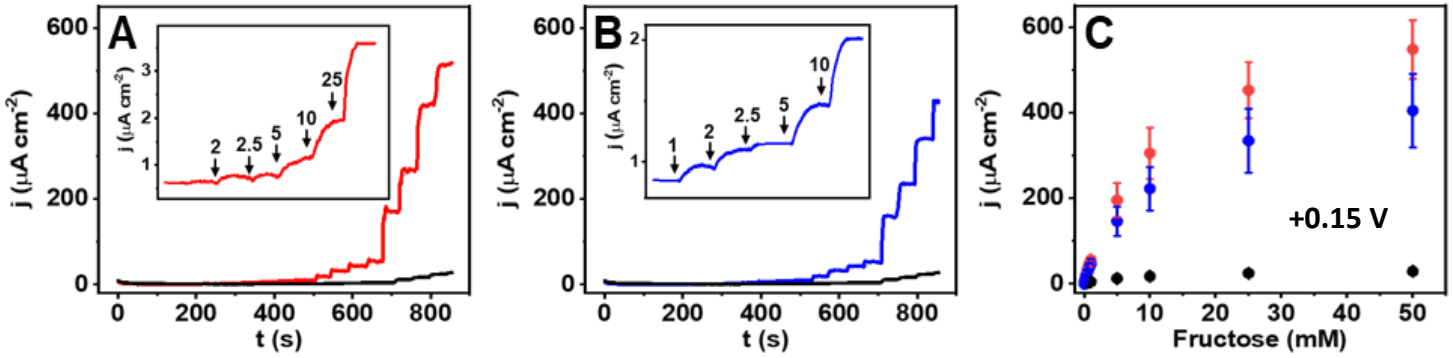
Catalytic current density at +0.4 V for increasing amounts of 0D-nanomaterials

RESULTS

KINETIC AND ANALYTICAL PERFORMANCE

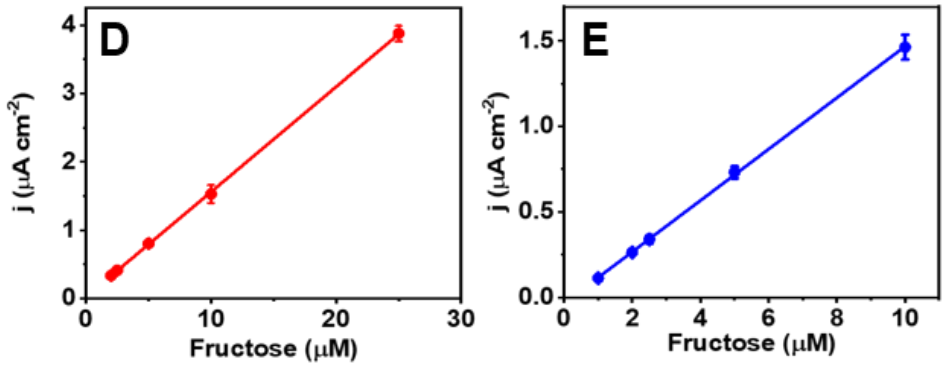
- Km_{app} → **CB-SC**: 12.14±0.32 mM
  MS-SC: 11.87±0.54 mM

- j_{max} → **CB-SC**: 677.1±6.5 µA cm^{-2}
  MS-SC: 496.5±8.2 µA cm^{-2}



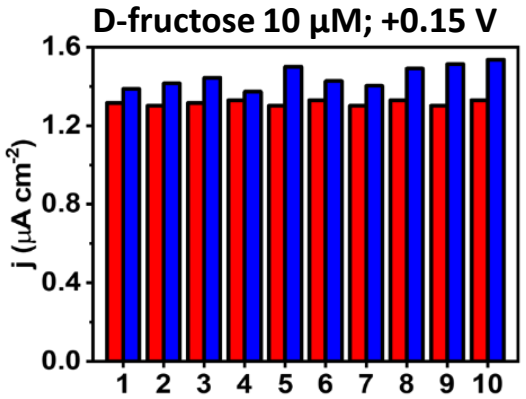
- **Linear range** → **CB-SC**: 1-10 µM (R^2=0.999)
  MS-SC: 2-25 µM (R^2=0.999)

- **LOD** → **CB-SC**: 0.35 µM
  MS-SC: 0.16 µM



RESULTS

REPEATABILITY AND REPRODUCIBILITY

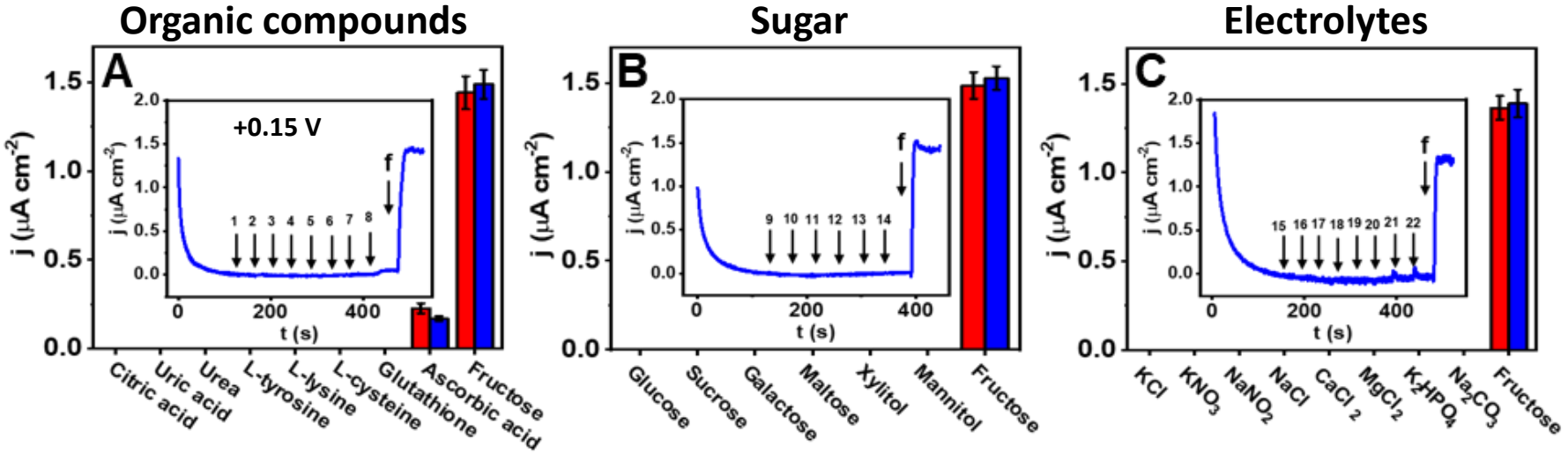


- **CB-SC**: RSD = 1.0 %
- **MS-SC**: RSD = 3.9 %

CB-SC slope: 0.153 ± 0.003 µA cm^{-2} μM^{-1}, RSD = 1.9 %

MS-SC slope: 0.150 ± 0.006 µA cm^{-2} μM^{-1}, RSD = 4.3 %

SELECTIVITY STUDY



(A) Organic compounds legend: 1 (100 µM citric acid), 2 (100 µM uric acid), 3 (1 mM urea), 4 (100 µM L-tyrosine), 5 (100 µM L-lysine), 6 (100 µM L-cysteine), 7 (100 µM glutathione), 8 (10 µM ascorbic acid). (B) Monosaccharides and disaccharides legend: 9 (1 mM D-glucose), 10 (1 mM sucrose), 11 (1 mM D-galactose), 12 (1 mM maltose), 13 (1 mM xylitol), 14 (1 mM Mannitol). (C) Electrolytes legend: 15 (1 mM KCl), 16 (1 mM KNO_3), 17 (1 mM $NaNO_2$), 18 (1 mM NaCl), 19 (1 mM $CaCl_2$), 20 (1 mM $MgCl_2$), 21 (1 mM K_2HPO_4), 22 (1 mM Na_2CO_3).

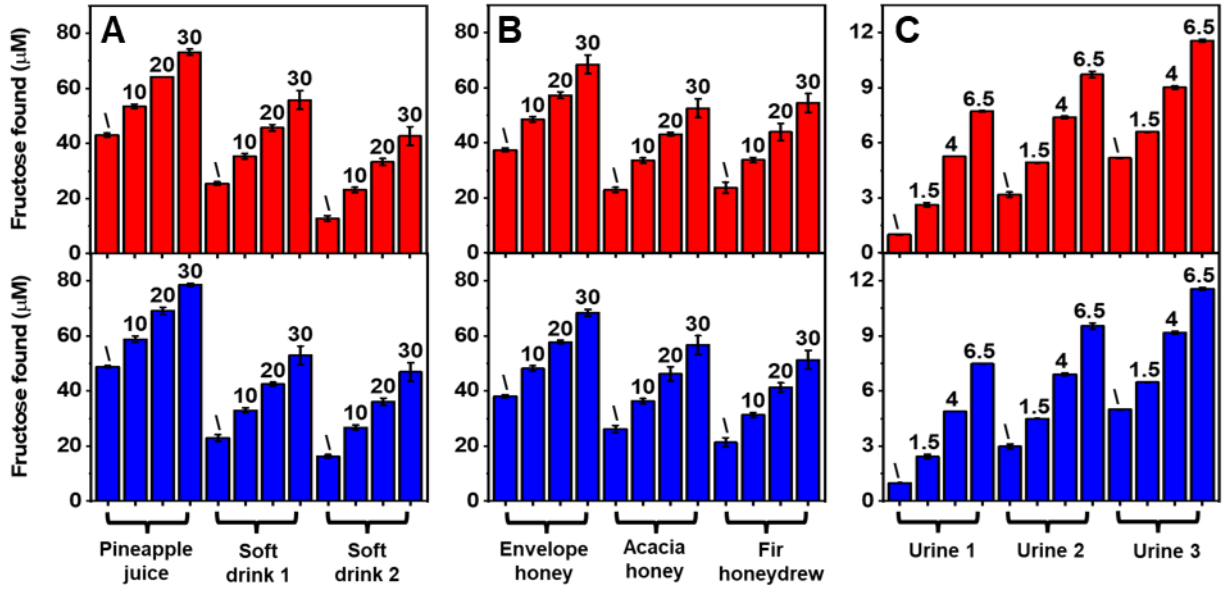
RESULTS

APPLICATION

Honey

Beverage

Urine



CB-SC → **Rec**: 116.1% - 94.9%, **RSD** ≤ 9%

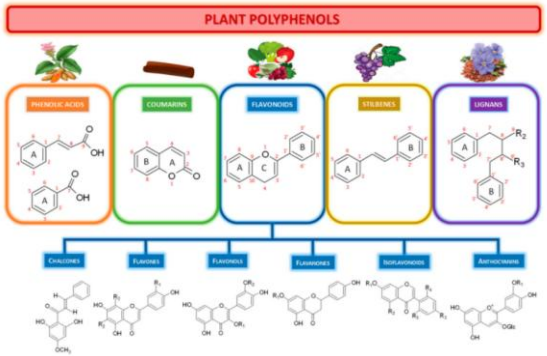
MS-SC → **Rec**: 105.0% - 95.9%, **RSD** ≤ 8%

FUNCTIONALISED NANOMATERIALS

Phenolic compounds as redox-active exfoliating agent for nanomaterials synthesis/preparation in water

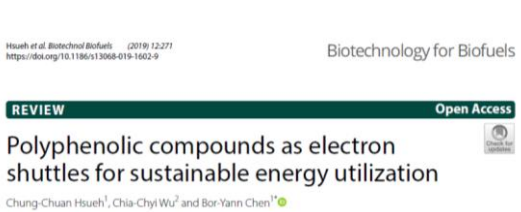
UNITE UNIVERSITÀ DEGLI STUDI DI TERAMO

What about phenols/polyphenols as stabilizing agents?

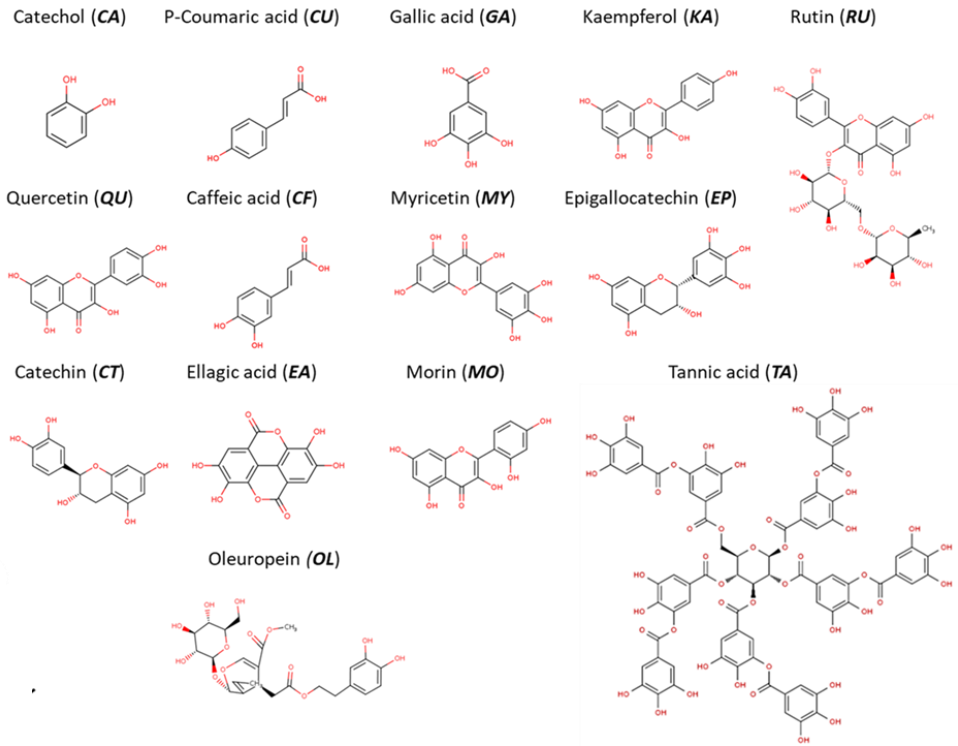


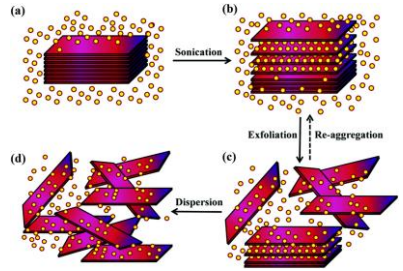
Do they work as stabilizing agents?

Can they confer particular properties to NMSs?

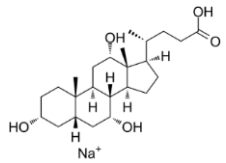


(Poly)phenols investigated



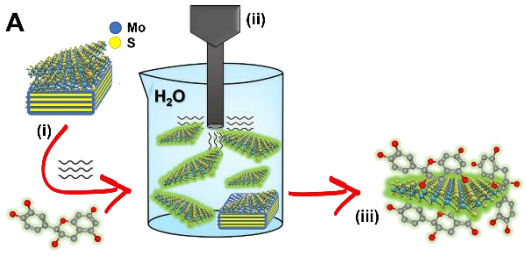


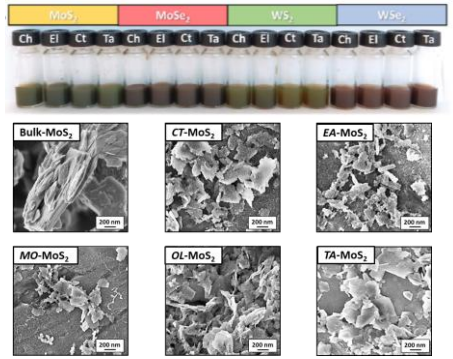
Sodium Cholate: control

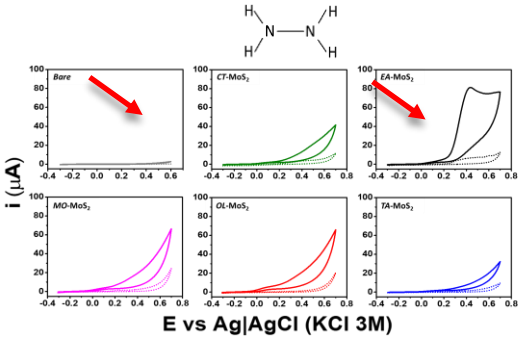


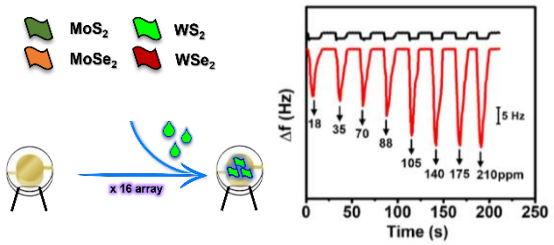
Phenolic compounds as redox-active exfoliation agents

2D Redox-active Group VI TMDs









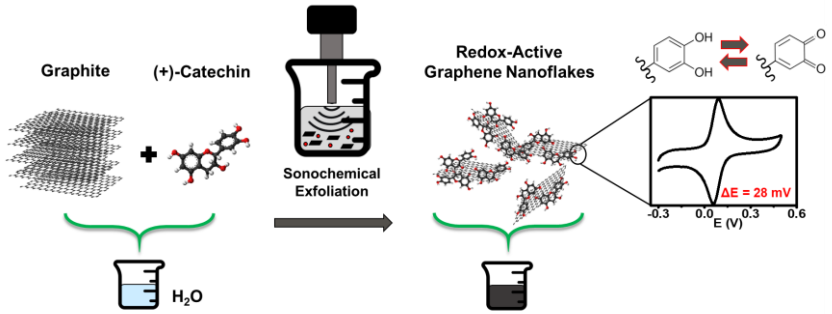
Hydrazine
Dashed line: *PPs*-MoS_2 0.1 M PB (pH 7);
Solid line: *PPs*-MoS_2 in presence of 5 mM of N_2H_4 ;
Scan rate: 25mVs^{-1};

Isopentyl acetate.
Black-line: bare-QCM;
red-line: Tn-WS_2 modified QCM.


Materials Today Chemistry 26 (2022) 101122
Contents lists available at ScienceDirect
Materials Today Chemistry
journal homepage: www.journals.elsevier.com/materials-today-chemistry/
Phenolic compounds as redox-active exfoliation agents for group VI transition metal dichalcogenides
D. Rojas [a], F. Della Pelle [a,*], F. Silveri [a], G. Ferraro [b], E. Fratini [b], D. Compagnone [a,**]

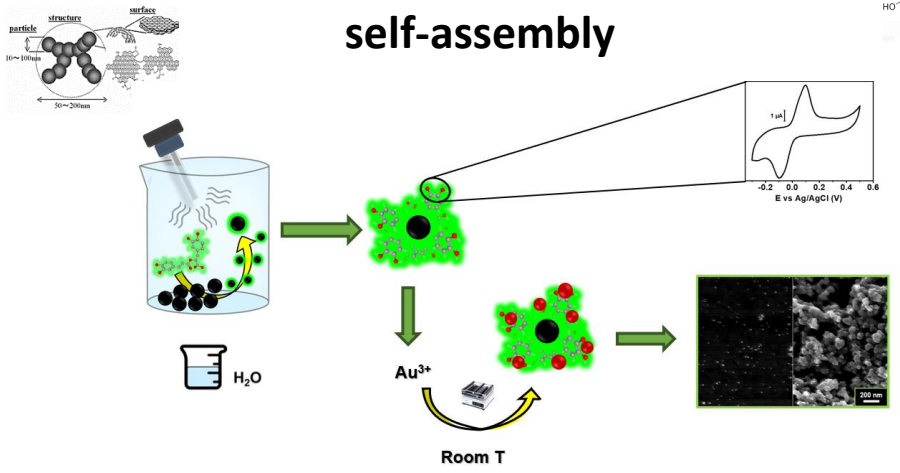

Biosensors and Bioelectronics 210 (2022) 114749
Contents lists available at ScienceDirect
Biosensors and Bioelectronics
journal homepage: www.elsevier.com/locate/bios
An electronic nose based on 2D group VI transition metal dichalcogenides/ organic compounds sensor array
Sara Gaggiotti [a,b,1], Annalisa Scroccarello [b,1], Flavio Della Pelle [b,*], Giovanni Ferraro [c], Michele Del Carlo [d], Marcello Mascini [a], Angelo Cichelli [b], Dario Compagnone [b,**]

2D redox-active Graphene nanoflakes



Silveri, F., Della Pelle, F.*, Rojas, D., Bukhari, Q. U. A., Ferraro, G., Fratini, E., & Compagnone, D. (2021). Microchimica Acta, 188(11), 1-13.

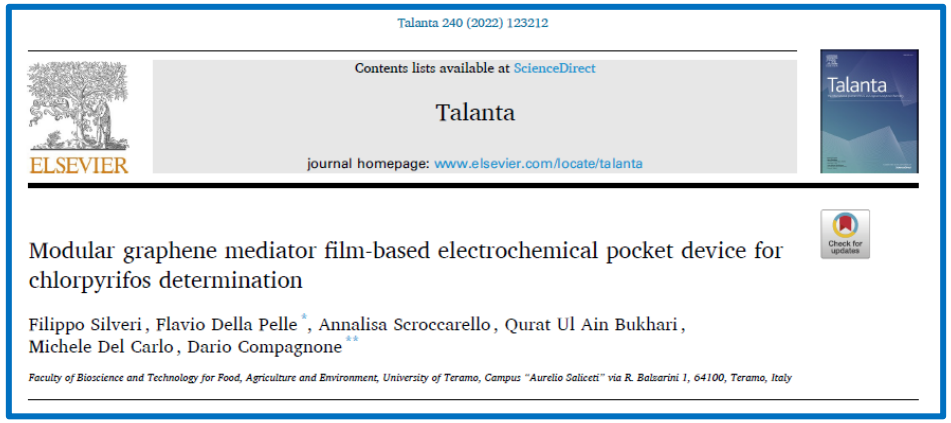
OD Redox-active CB for AuNPs self-assembly



Silveri, F., Della Pelle, F. *, Scroccarello, A., Mazzotta, E., Di Giulio, T., Malitesta, C., Compagnone, D. * (2022). Antioxdants, 11, 2008

Redox-graphene based device

Device assembling



Talanta 240 (2022) 123212

Contents lists available at ScienceDirect

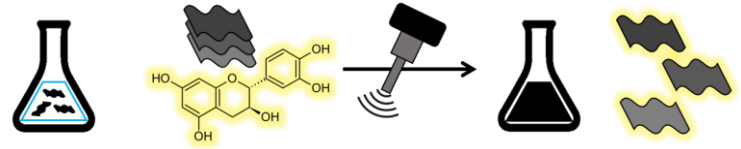
Talanta

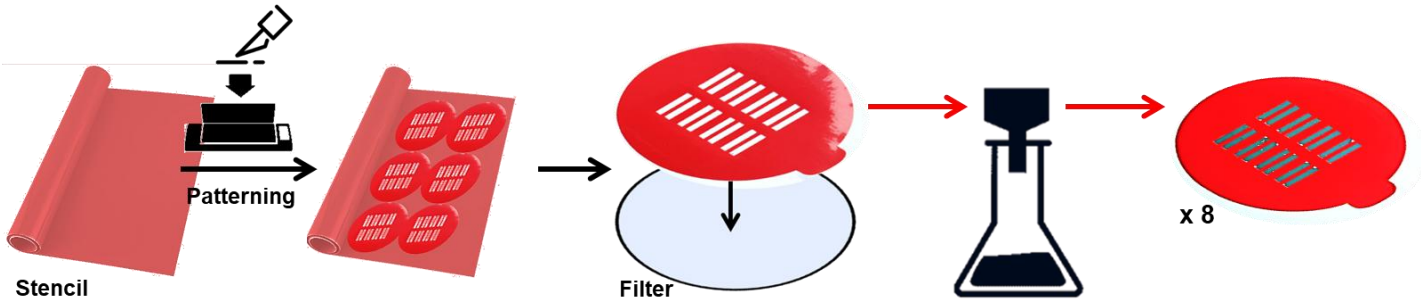
journal homepage: www.elsevier.com/locate/talanta

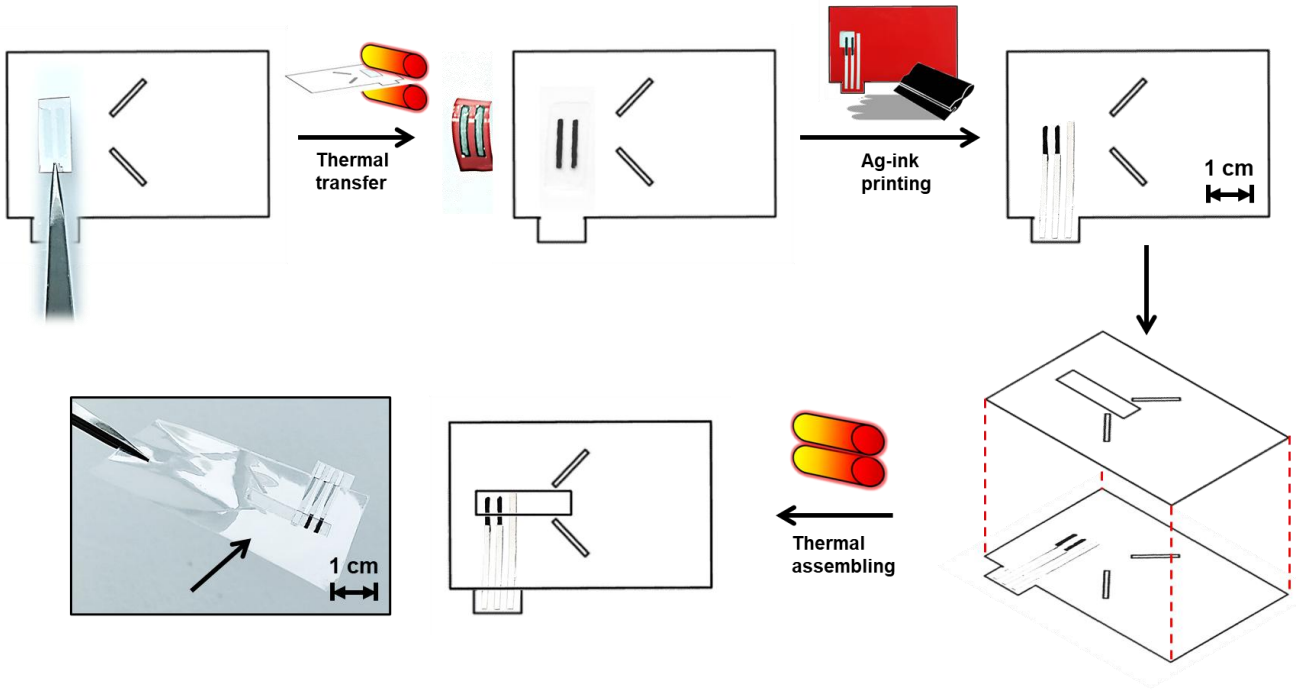
Modular graphene mediator film-based electrochemical pocket device for chlorpyrifos determination

Filippo Silveri, Flavio Della Pelle *, Annalisa Scroccarello, Qurat Ul Ain Bukhari, Michele Del Carlo, Dario Compagnone **

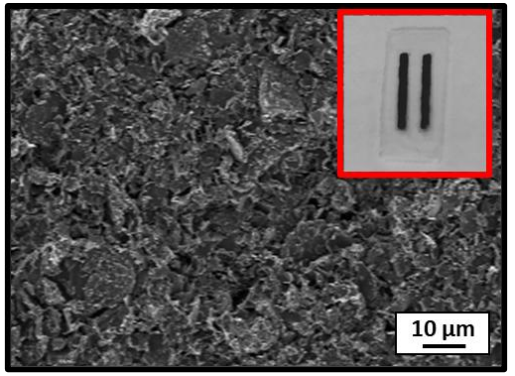
Faculty of Bioscience and Technology for Food, Agriculture and Environment, University of Teramo, Campus "Aurelio Saliceti" via R. Balsarini 1, 64100, Teramo, Italy

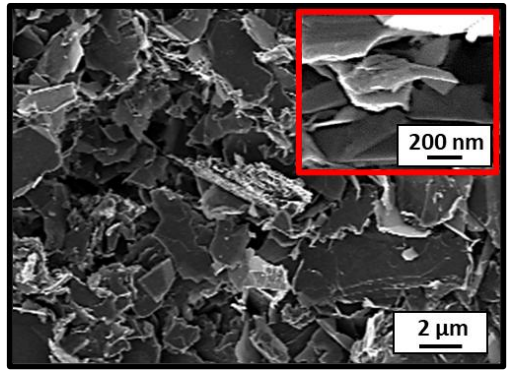






SEM

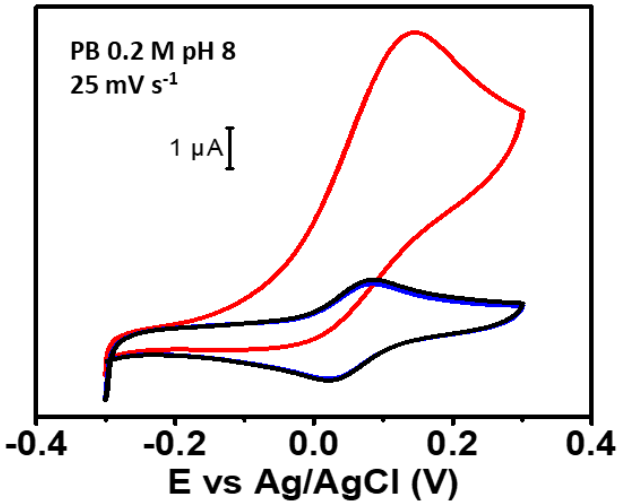




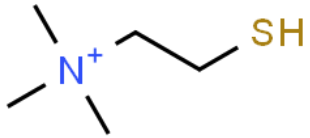
Redox-graphene based device

Sensor development

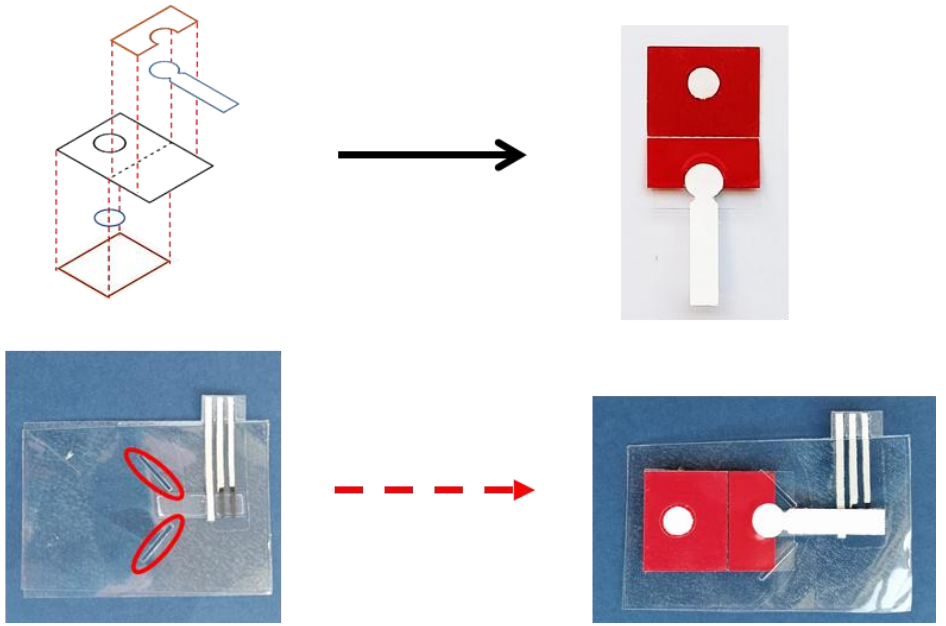
1



Thiocholine
2.5 mM



2



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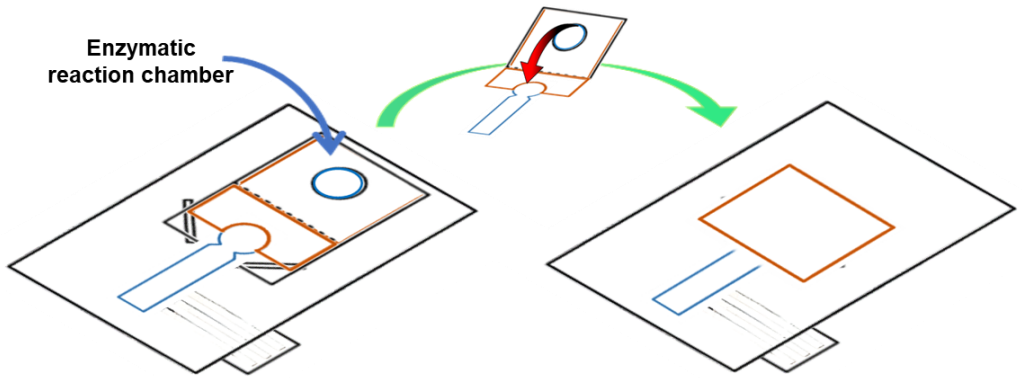

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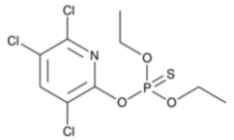
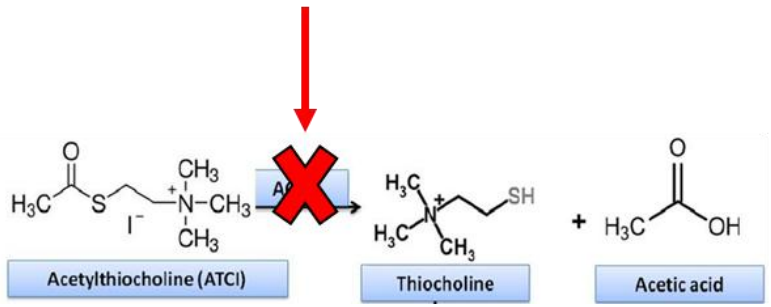
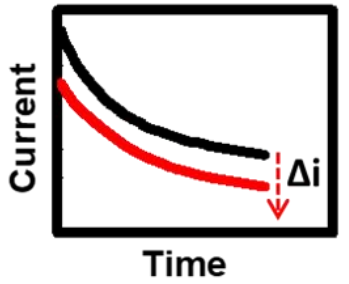


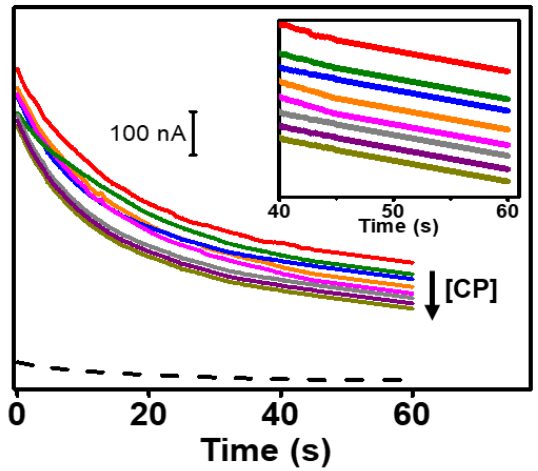
Chlorpyrifos (**inhibitor**)

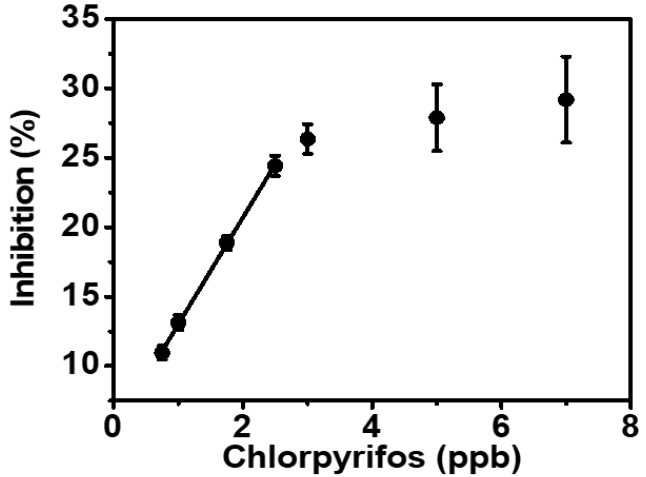




Redox-graphene based device

Analytical performance

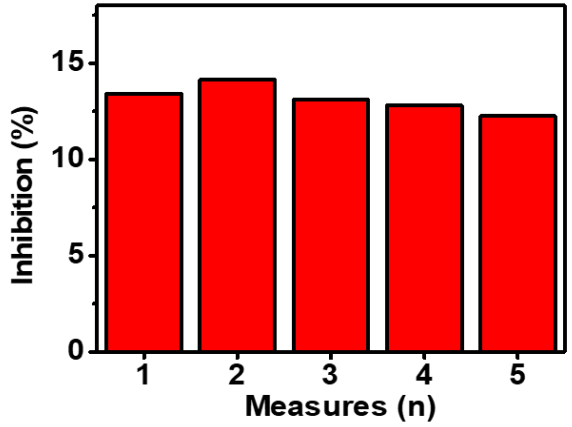




- **LOD** = 0.2 ppb (6.9 % of inhibition)
- **L.R**. = 0.7 – 2 ppb
- I%= 7.65[CP]+5.37 (**R^2** = 0.9995)

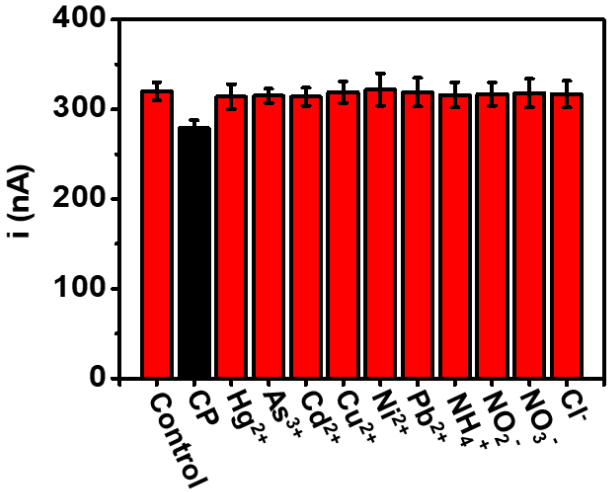
Slope = 7.65 ± 0.19
RSD = 2.5 % (n = 3)

Consecutive measures

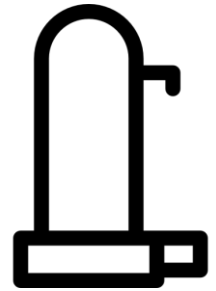


RSD = 5.4 % (n = 5)

Interference study



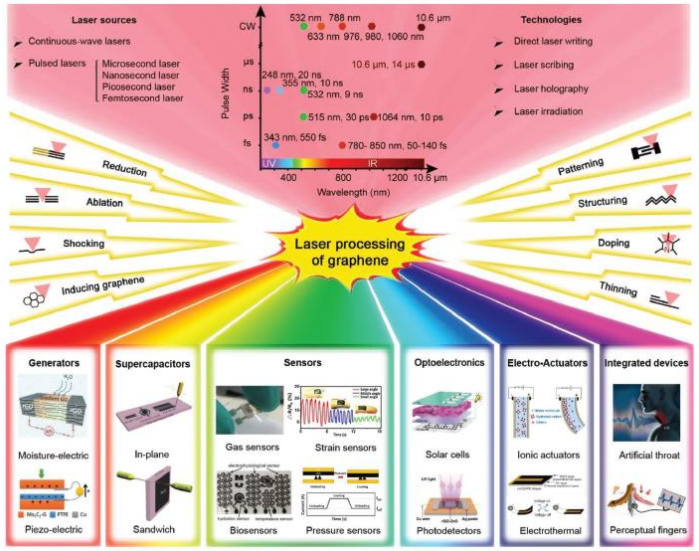
Samples analysis

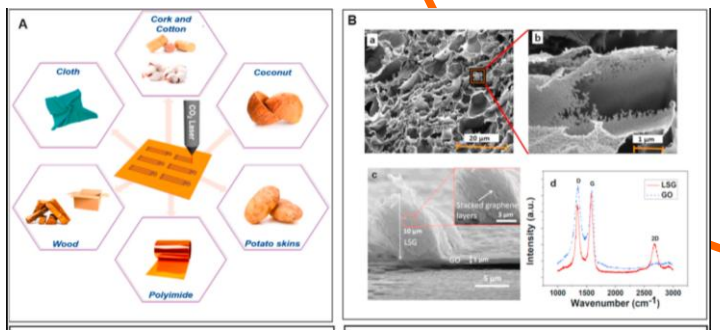


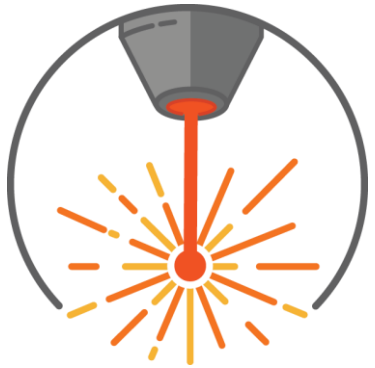
River and well water

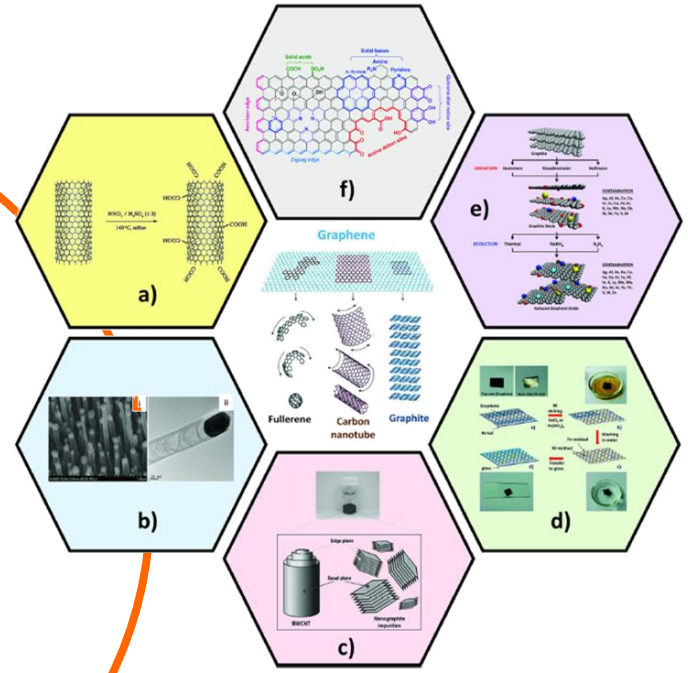
Recoveries: 94.0 – 113.0 %
RSD < 4.0 % (n = 3)

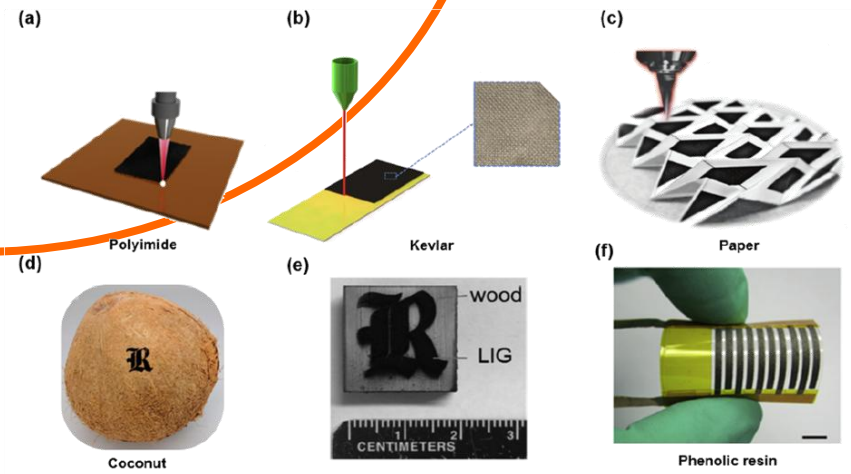
CO_2 laser-plotter for conductive nanostructured films formation





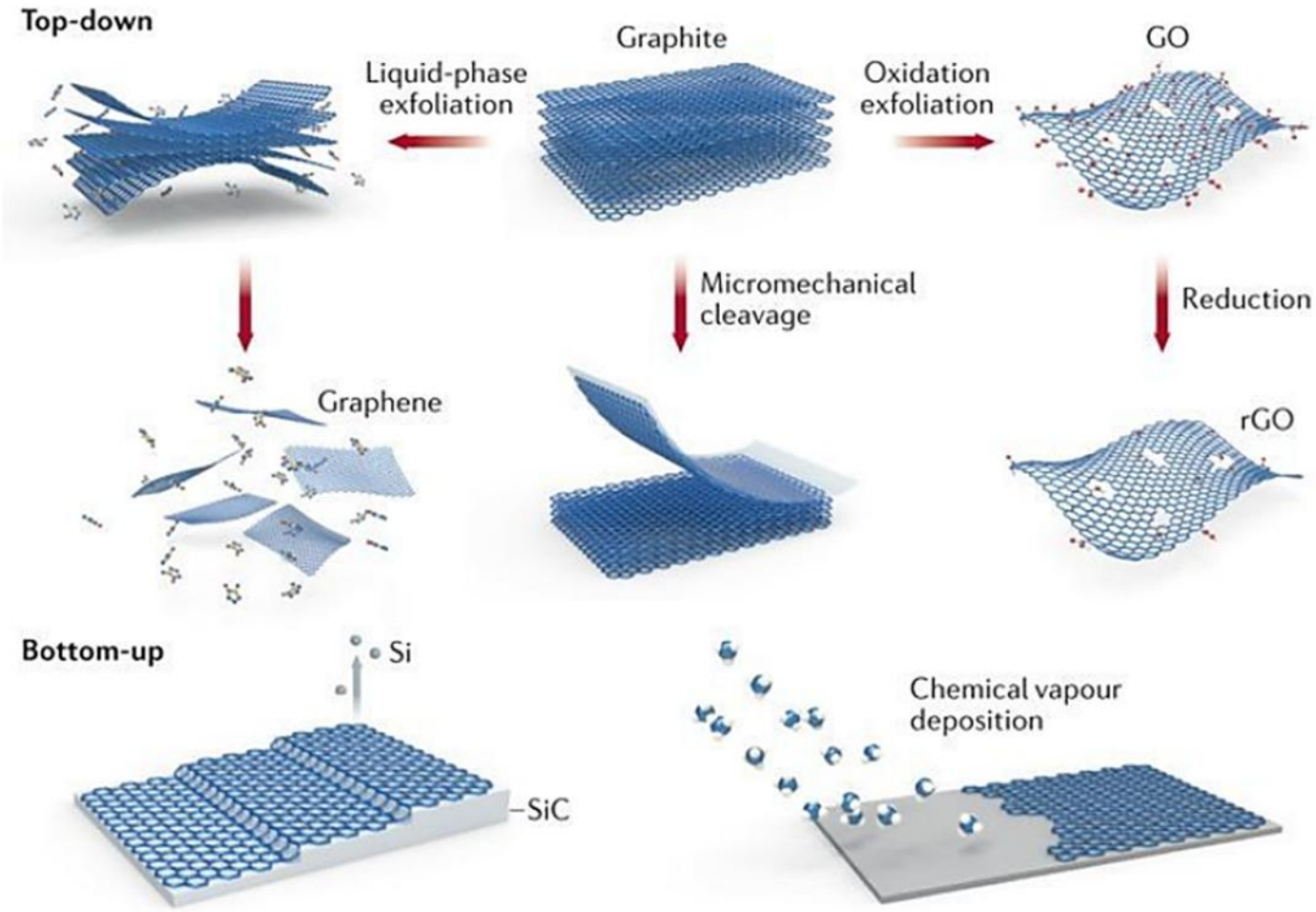






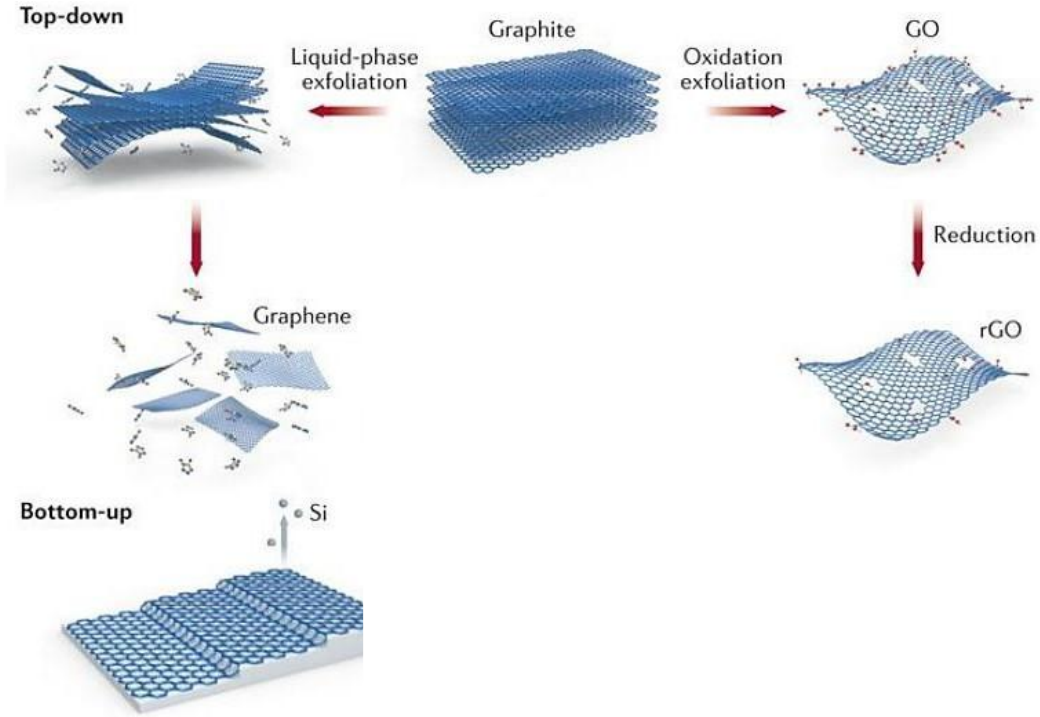
You et al. (2020). Advanced Materials, 32(15), 1901981.
Lahcen et al. (2020). Biosensors and Bioelectronics, 168, 112565
Wang et al. (2022). Biosensors, 12(2), 55.
Simsek, M., & Wongkaew, N. (2021). Analytical and Bioanalytical Chemistry, 413(24), 6079-6099.

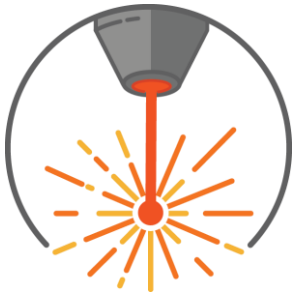
Graphene production



Laser induced graphene

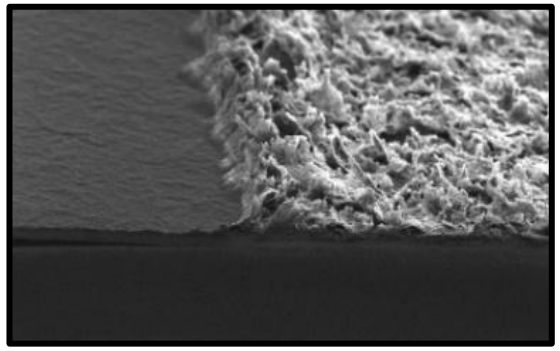
Graphene





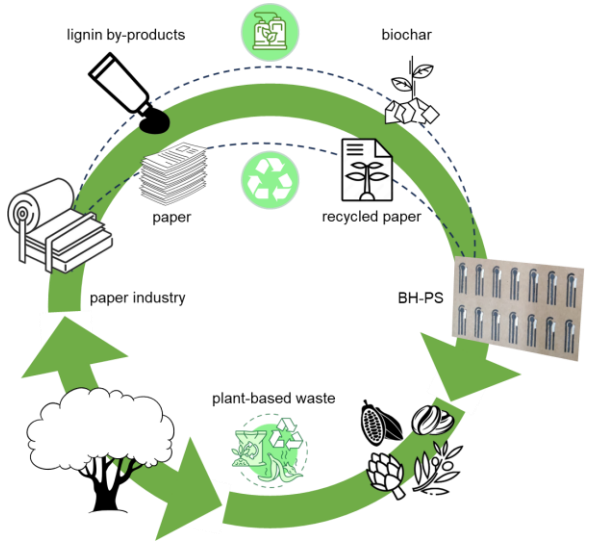
Laser induced graphene

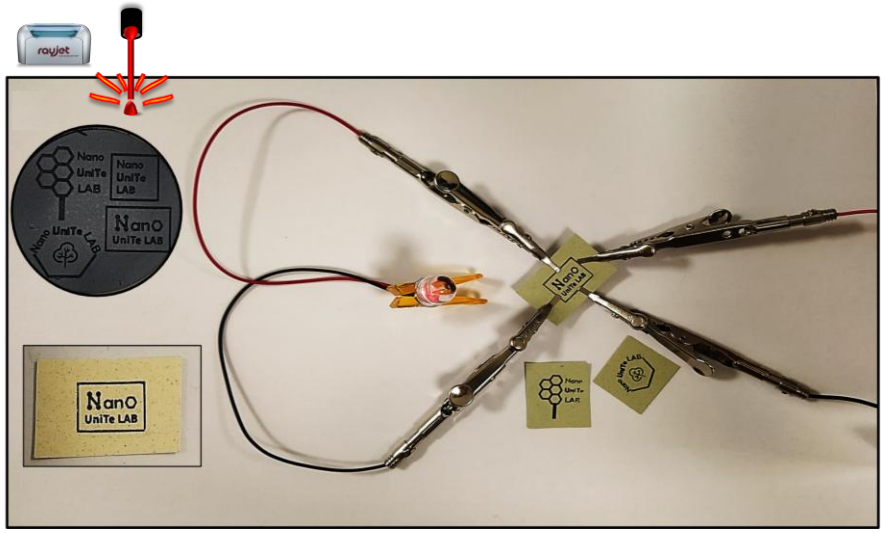
Laser-induced rGO transferable conductive films

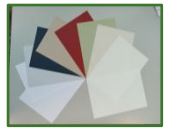


Scroccarello et al. (2023). **ACS sensors**, 2023, 8, 2, 598–609
Zhao et al. (2023). **ACS Appl. Mater. Interfaces,** 15, 7, 9024–9033

Recycled and by-products derived papers for L-rGO sensors







Cellulosic substrates

Office paper

NT — Navigator

RL — Rismaluce

15% textile industry

CW — Refit Cotton White

WB — Refit Wool Blue

RO — Remake Oyster

15% agro-industry by-product

CO — Crush Cocoa

CA — Crush Cherrys

CK — Crush Kiwi

75% bamboo 100% recycle

FB — Free Tree Bamboo Cream

TW — Tokyo White

Laser-designed Paper/Graphene 3D pop-up for carbaryl analysis

Sensors & Actuators: B. Chemical 399 (2024) 134768

Contents lists available at ScienceDirect

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journal homepage: www.elsevier.com/locate/snb

Integrated paper/graphene 3D pop-up device for the quantitative sensing of carbaryl

Selene Fiori [1], Annalisa Scroccarello [1], Flavio Della Pelle *, Michele Del Carlo, Dario Compagnone *

Department of Bioscience and Technologies for Food, Agriculture and Environment, University of Teramo, Via R. Balzarini, 1, 64100 Teramo, TE, Italy

CARBARYL (CBR)

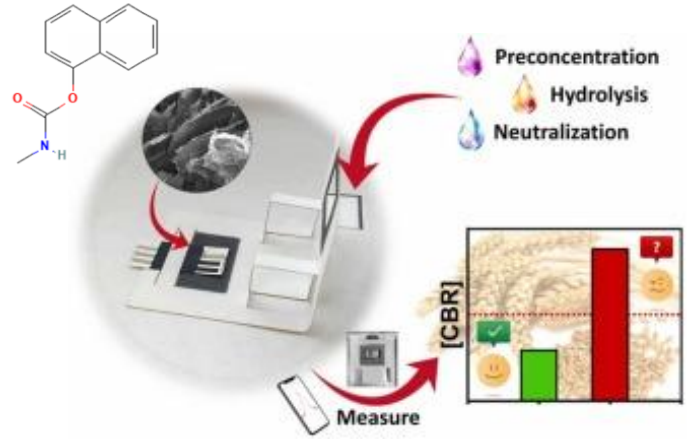
European Commission

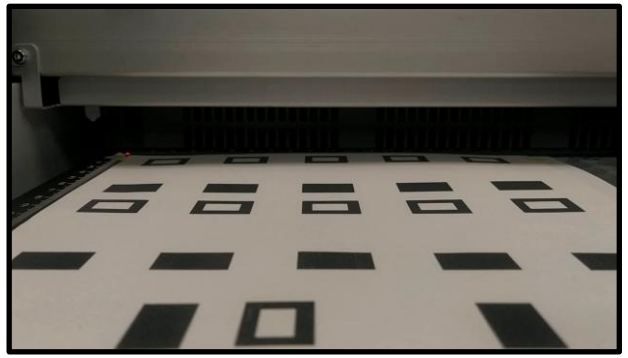
Minimum residue levels (MRL)

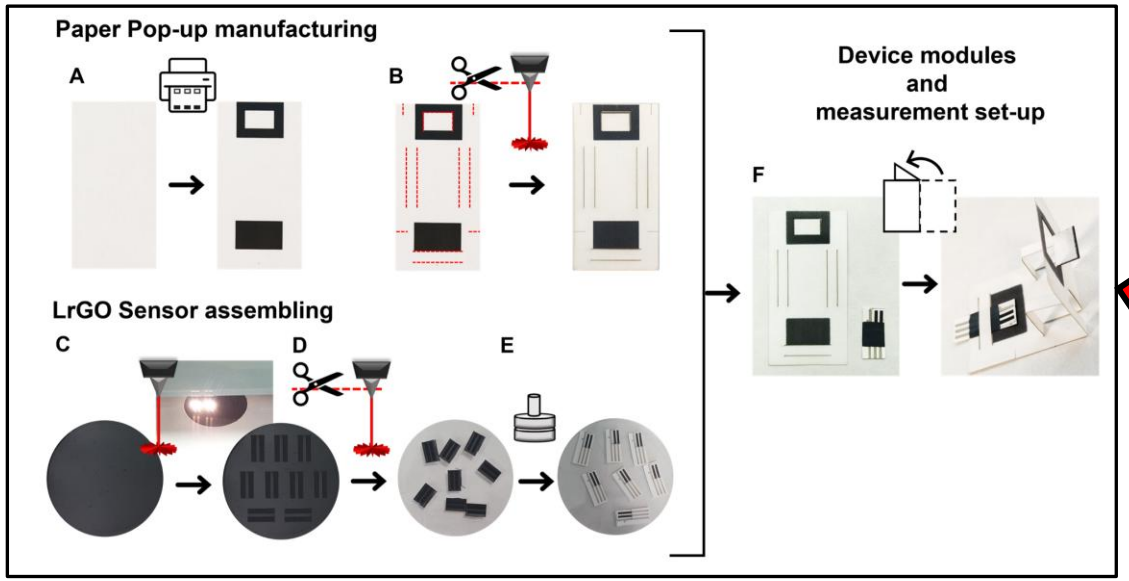
MRL 0.5 mg/Kg

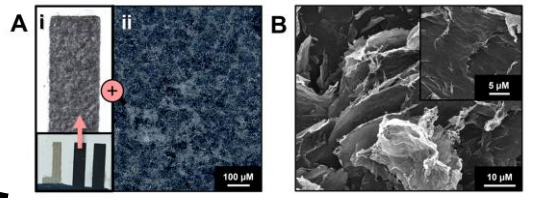
MRL 0.8 mg/Kg

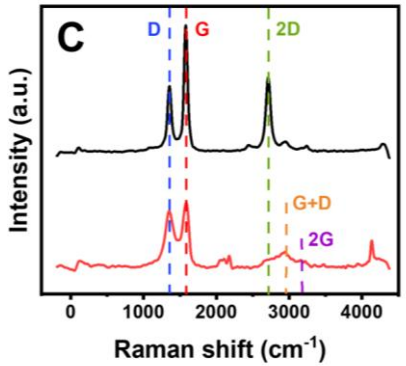
MRL 0.1 mg/Kg







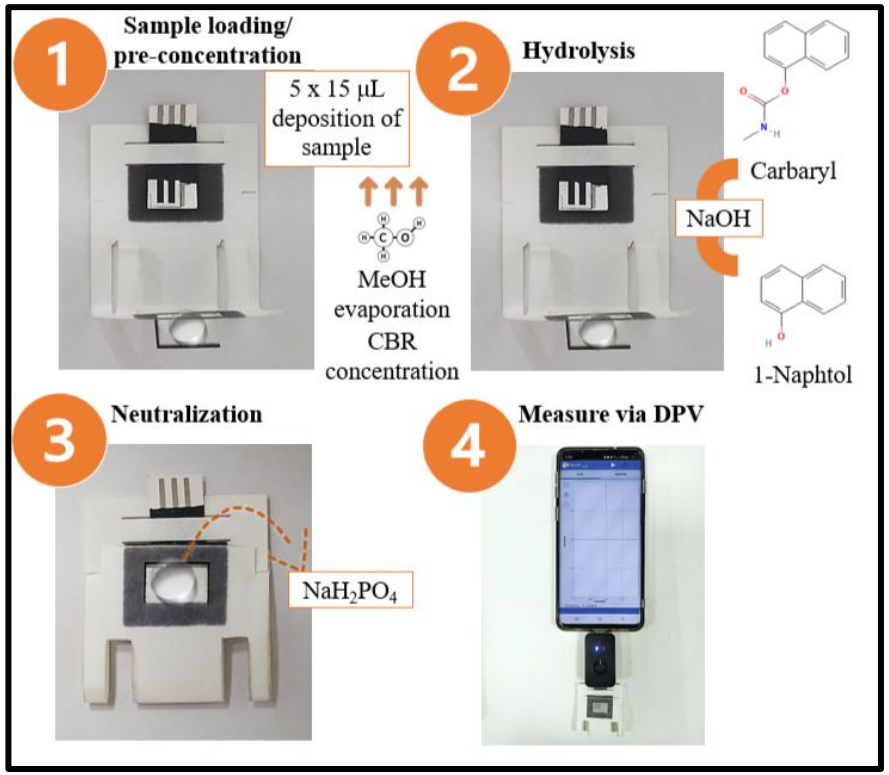


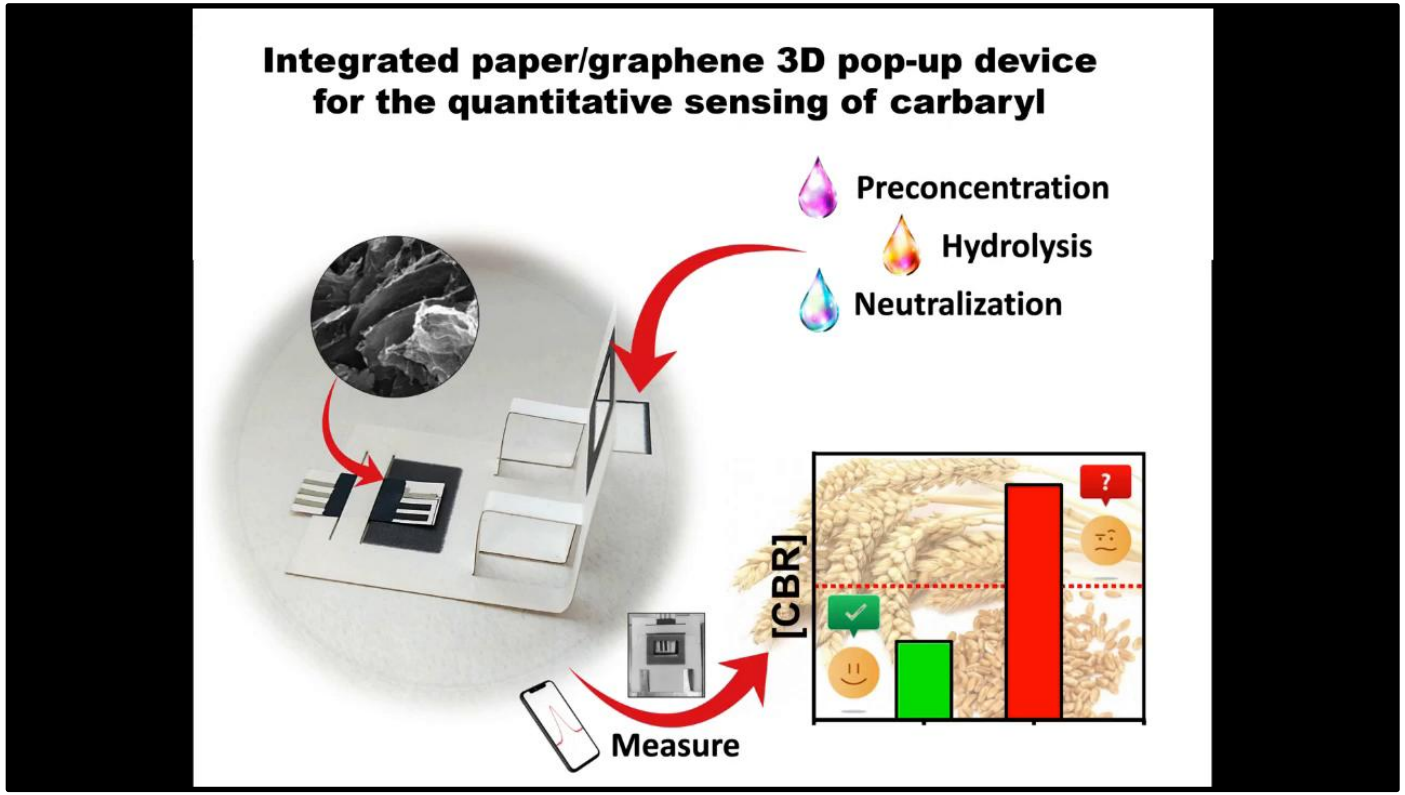


Fiori et al. (2024). **Sensors and Actuators B: Chemical**, 399, 134768.

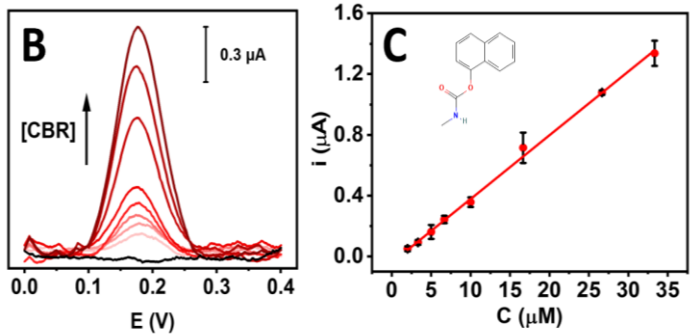
Laser-designed Paper/Graphene 3D pop-up for carbaryl analysis

Assay format





LOD = 0.4 µM
L.R.: 1.5-33 µM (R^2 =0.995)
Slope RSD = 8% (n = 3)



Fiori et al. (2024). **Sensors and Actuators B: Chemical**, 399, 134768.

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Sample and interferences analysis

GRAINS SAMPLE ANALYSIS

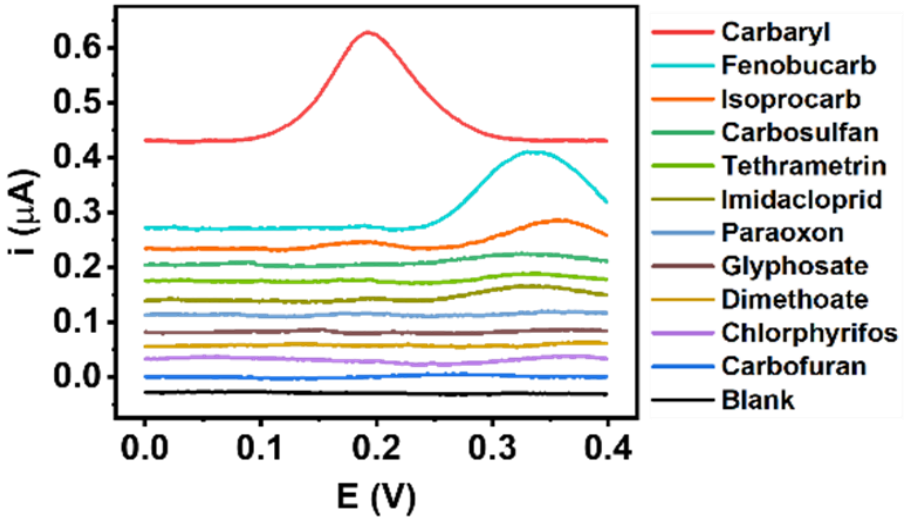
5 g of grain extracted in 10 mL of methanol analyzed directly with the pop-up device

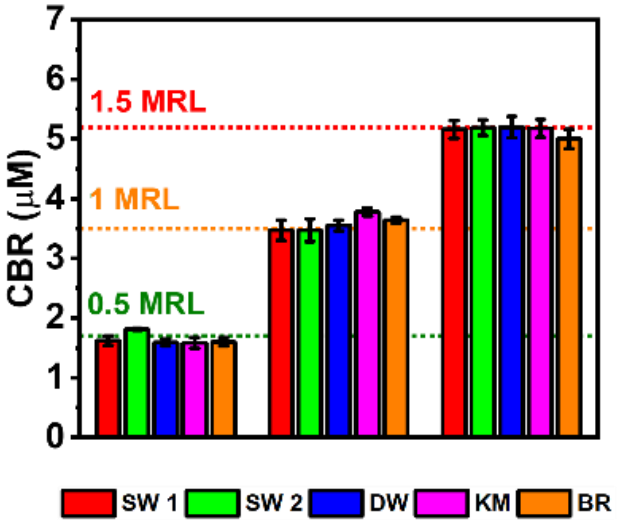
European Commission

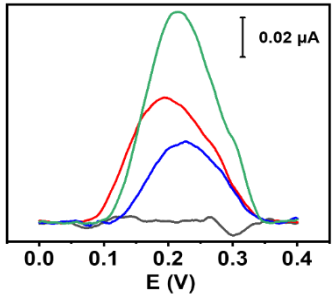
Minimum residue level (MRL) 0.5 mg Kg^{-1}

Samples fortified at **0.5**, **1** and **1.5** MRL value

Soft wheat 1 (**SW1**)
Soft wheat 2 (**SW2**)
Durum wheat (**DW**)
Kamut (**KM**)
Barley (**BR**)







Rec: 93 – 108 **%**
RSD ≤ 6 **%** (n = 3)

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