

# Plasmonic nanostructures employed for optical and colorimetric (bio)sensing strategies

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#### Is this Nano?



Nanotechnology is a "system of innovative methods to control and manipulate matter at near-atomic scale to produce new materials, structures, and devices".

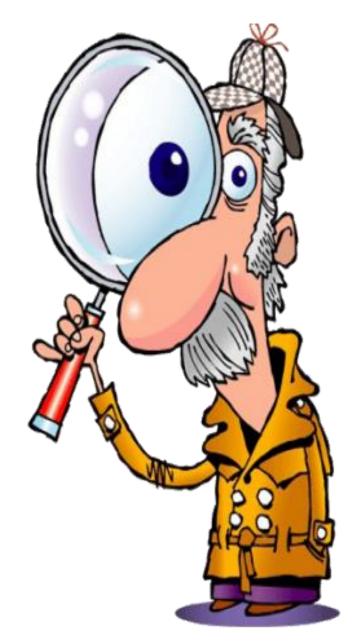
Nanomaterials (NMs)

Materials in the range of 100 nm are considered to be nanoparticles. They exhibit a wide range of properties, including optical, electrical, catalytic, magnetic, and biological activity.



Considerations on the EU definition of a nanomaterial: Science to support policy making

Eric A.J. Bleeker<sup>\*</sup>, Wim H. de Jong, Robert E. Geertsma, Monique Groenewold, Evelyn H.W. Heugens, Marjorie Koers-Jacquemijns, Dik van de Meent, Jan R. Popma, Anton G. Rietveld, Susan W.P. Wijnhoven, Flemming R. Cassee, Agnes G. Oomen

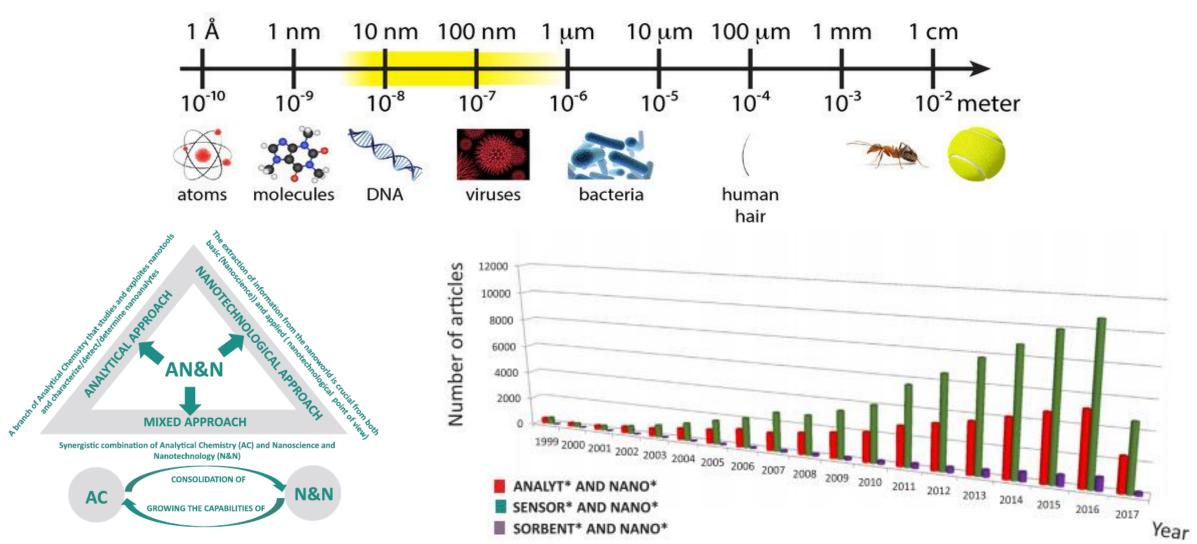


## Is this Nano?

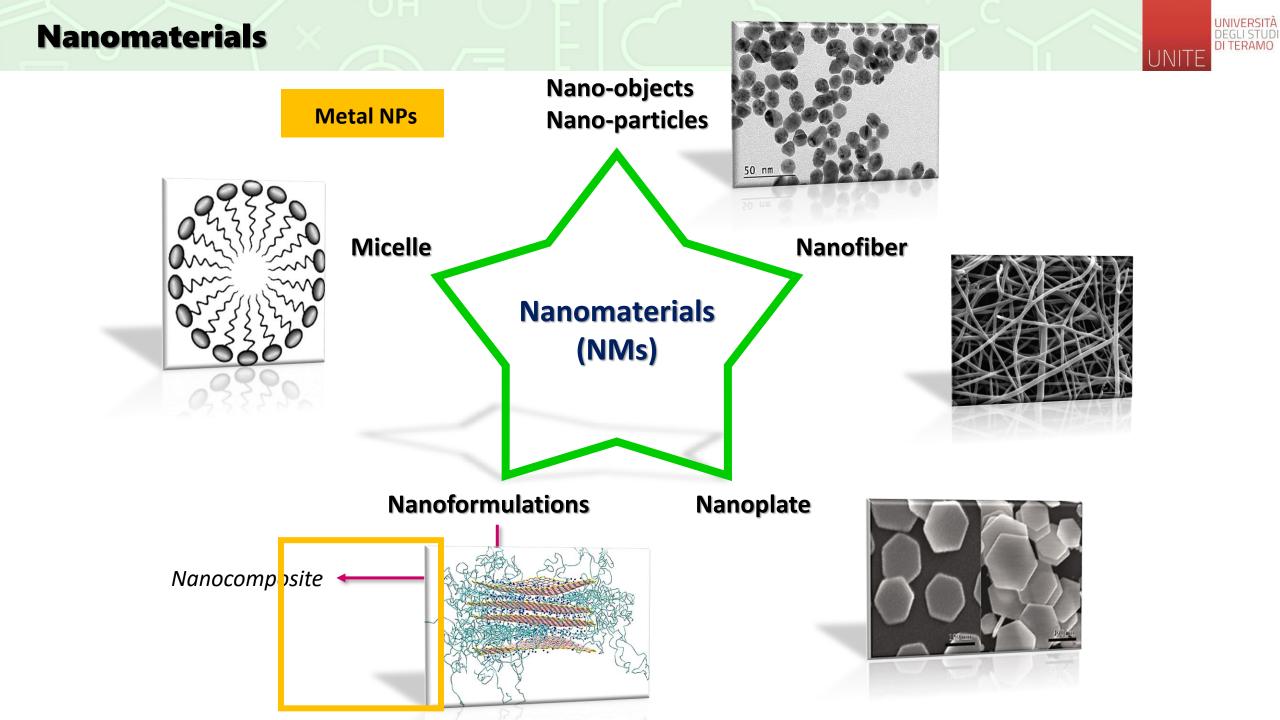




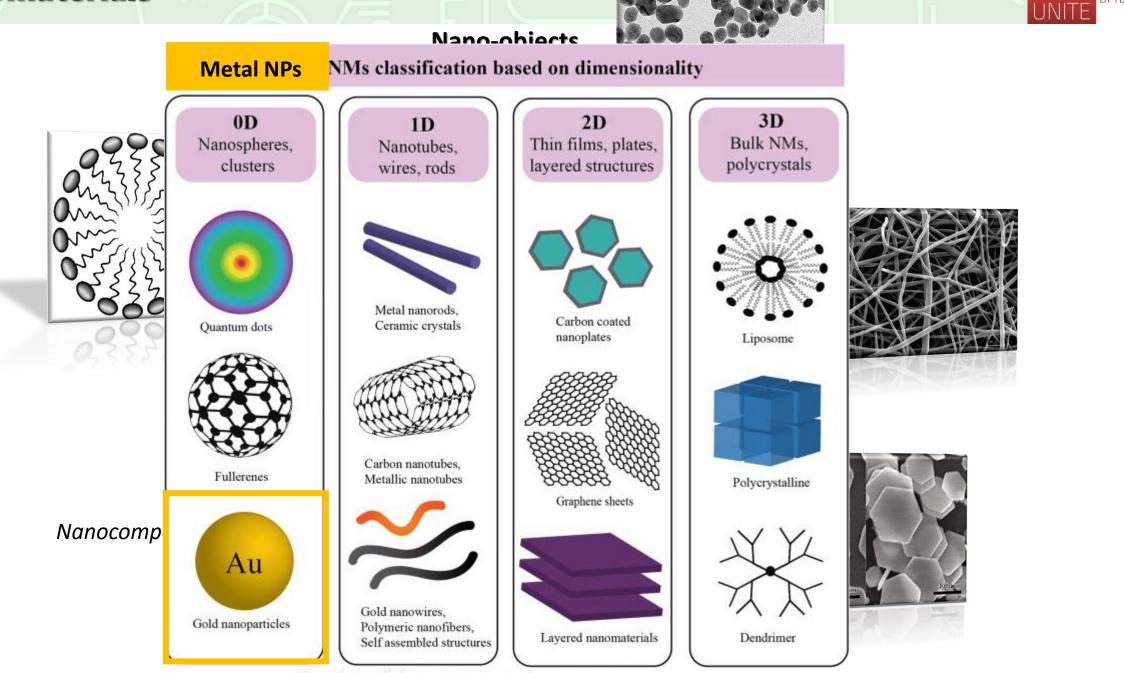




European Commission, Commission Recommendation of 18 October 2011 on the definition of nanomaterial, Official Journal of the European Union. 2011/696/EU: 38-40, 2011



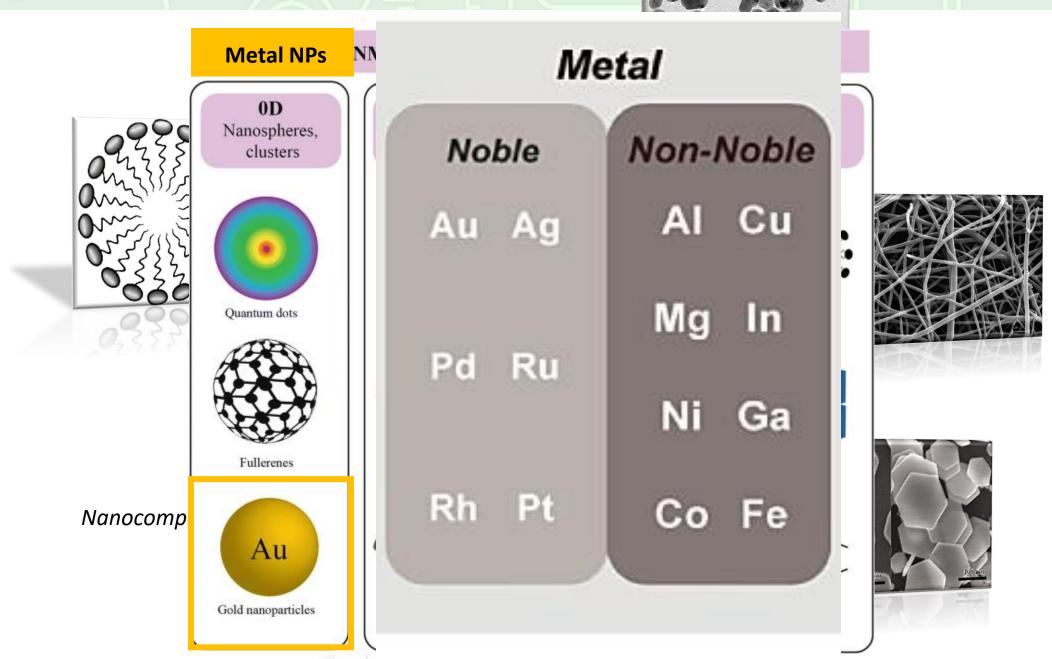
### Nanomaterials



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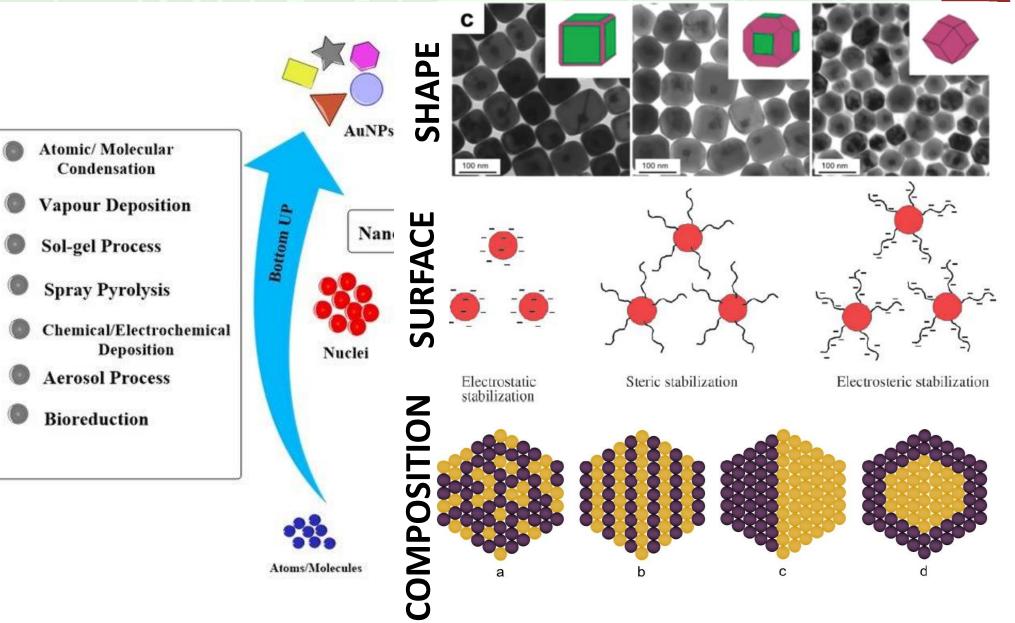
#### Nanomaterials





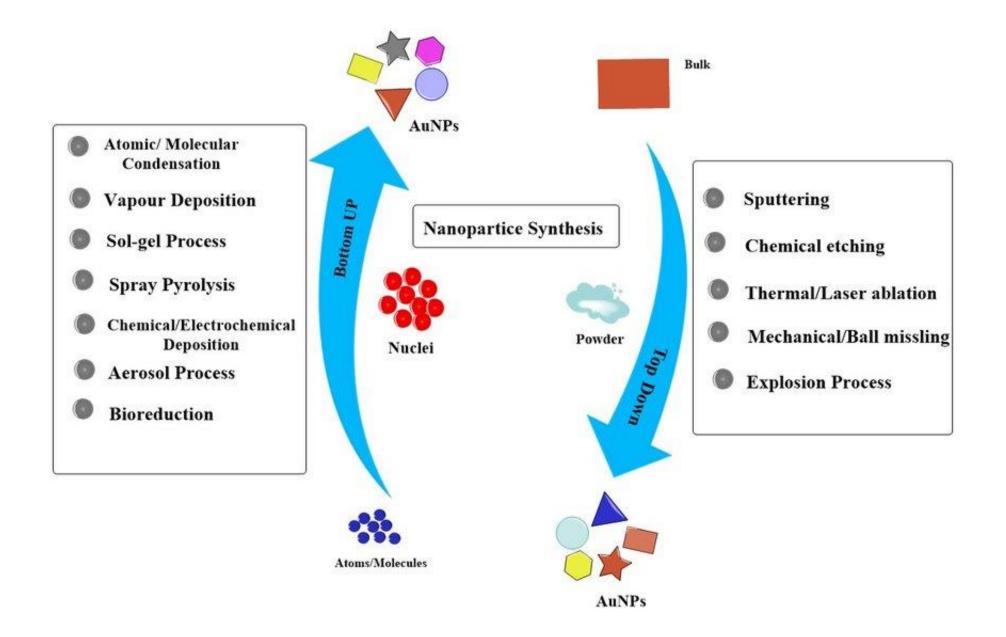
#### **Metal based Nanoparticles**





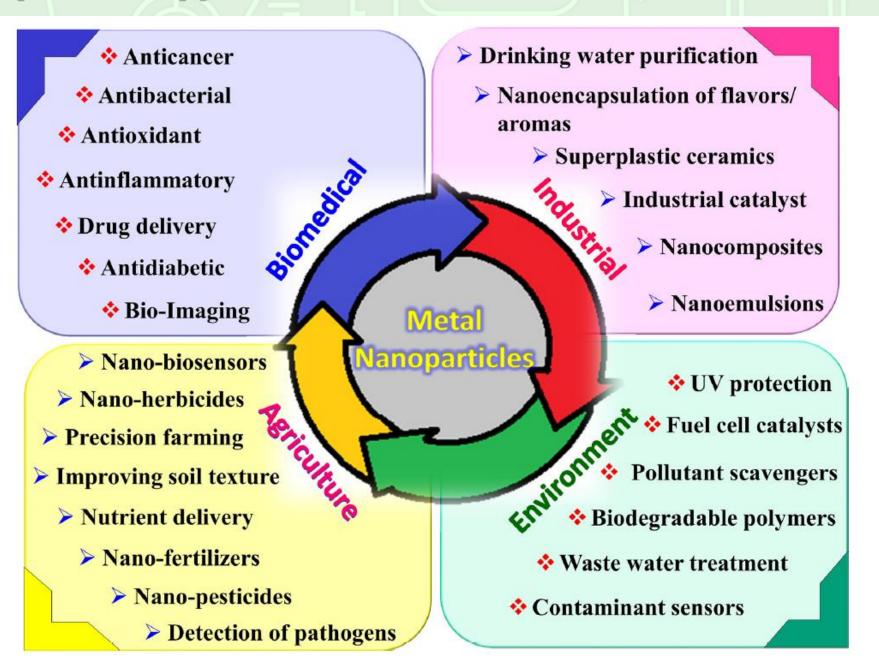
#### **Metal based Nanoparticles**





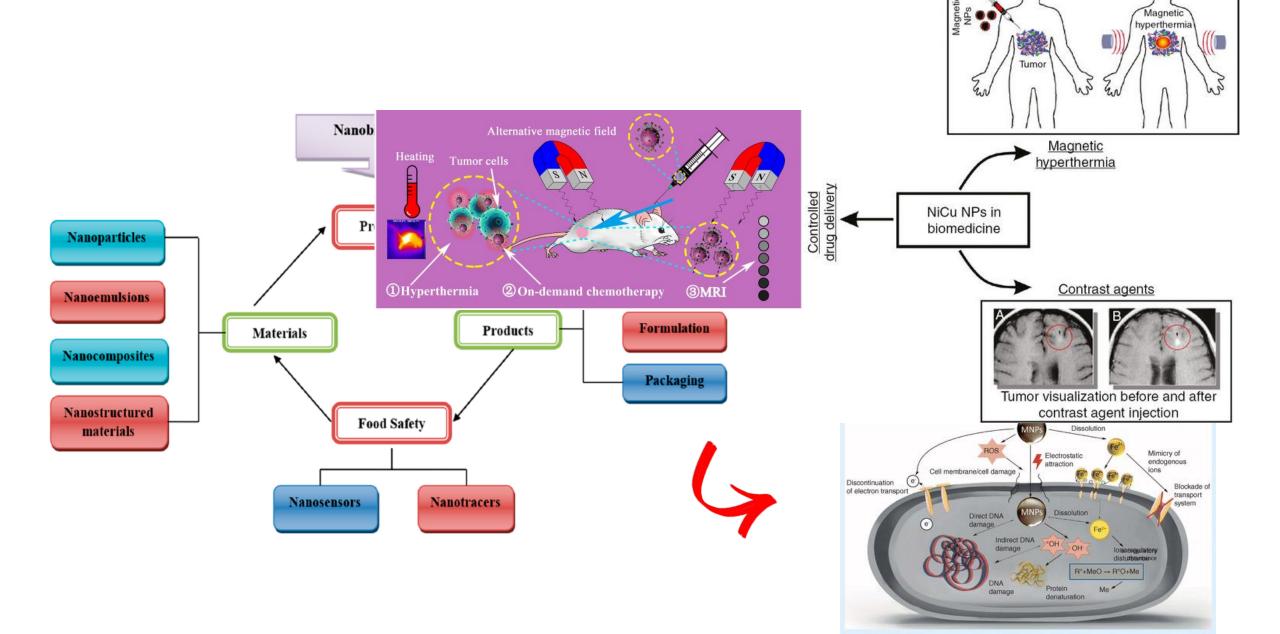
#### **Metal Nanoparticles application fields**





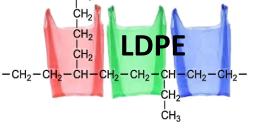
### Nanotechnology



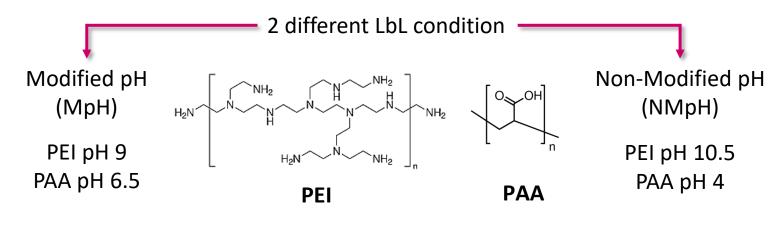


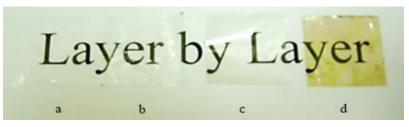
### Nanoparticles application in food technology



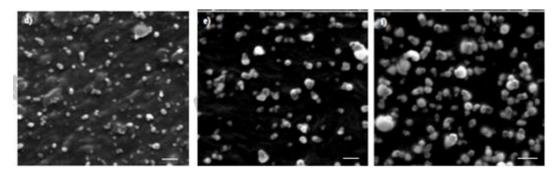


Commercial low-density polyethylene (LDPE) films coated using a layer-by-layer (LbL) technique by alternating the deposition of polyethyleneimine (PEI), poly(acrylic acid) polymer (PAA) solutions and antimicrobial silver nanoparticles (Ag).





The colour changes of a) LDPE films and b) LDPE LbL coated (3 coatings) films without Ag and c) AgNPs presence on LDPE LbL coated (3 coatings) films immersed in 0.5% AgNO<sub>3</sub>, or d) AgNPs presence on LDPE LbL coated with (3 coatings) film immersed in 5% AgNO<sub>3</sub> and UV/ozone treated for 20 min.

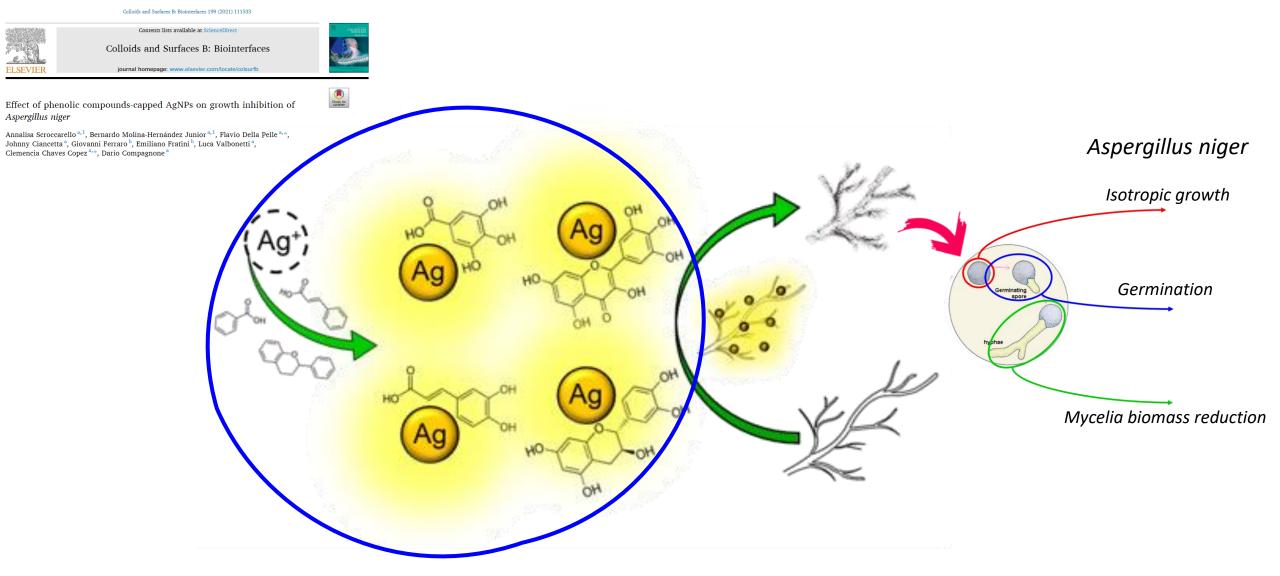


(d) LbL coated (3 coating) immersed in 0,5% AgNO<sub>3</sub>, (e) LbL coated (3 coatings) immersed in 2% AgNO<sub>3</sub>; and (f) LbL coated (3 coatings) immersed in 5% AgNO<sub>3</sub>. Scale bar = 500nm

d		Inibition zone (mm²)	
	Film	S. aureus	P. fluorescens
	LPDE films	0.00	0.00
	LPDE + PEI/PAA (MpH)	350.4 ± 13.30	694.8 ± 19.15
	LPDE + PEI/PAA (NMpH)	460.0 ± 25.41	737.0 ± 15.08

## MNPs as antimicrobial agent





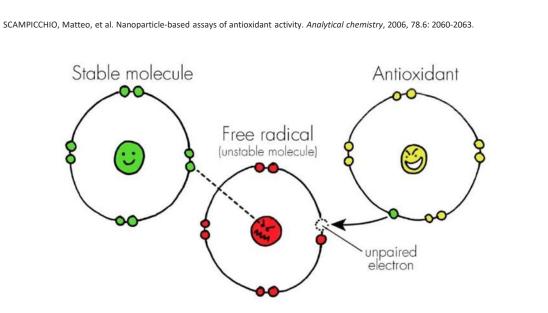
#### Phenolic content and antioxidant capacity evaluation trough Au and AgNPs formation

Nanomaterial-Based Sensing and Biosensing of Phenolic Compounds and Related Antioxidant Capacity in Food

sensors

... Nanomaterial-based method for estimating the antioxidant activity relies on the polyphenol-mediated growth of MNPs (AuNPs and AgNPs), and optical monitoring of the corresponding plasmon absorption bands...

MDPI



Α OH = H Ag⁺ Aq<sup>0</sup> В CH 0.5 AU iii Abs / AU 700 500 600 300 400 800  $\lambda / nm$ -----ÓН Secondaricizetino

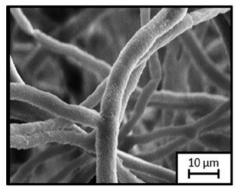
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### MNPs as antimicrobial agent: AgNPs' antifungal performances

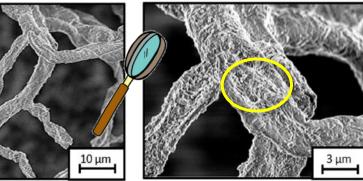
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#### Hyphae interaction

#### Control

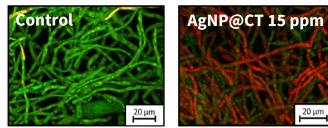


AgNP@CT 15 ppm

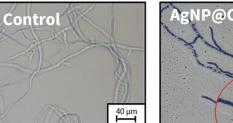


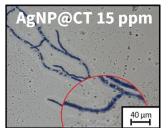
## HYPOTHESIS: - AgNPs adhesion - Membrane damages - Hyphae necrosis

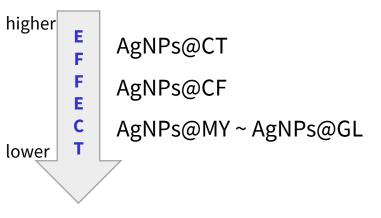
#### Live and death kit assay



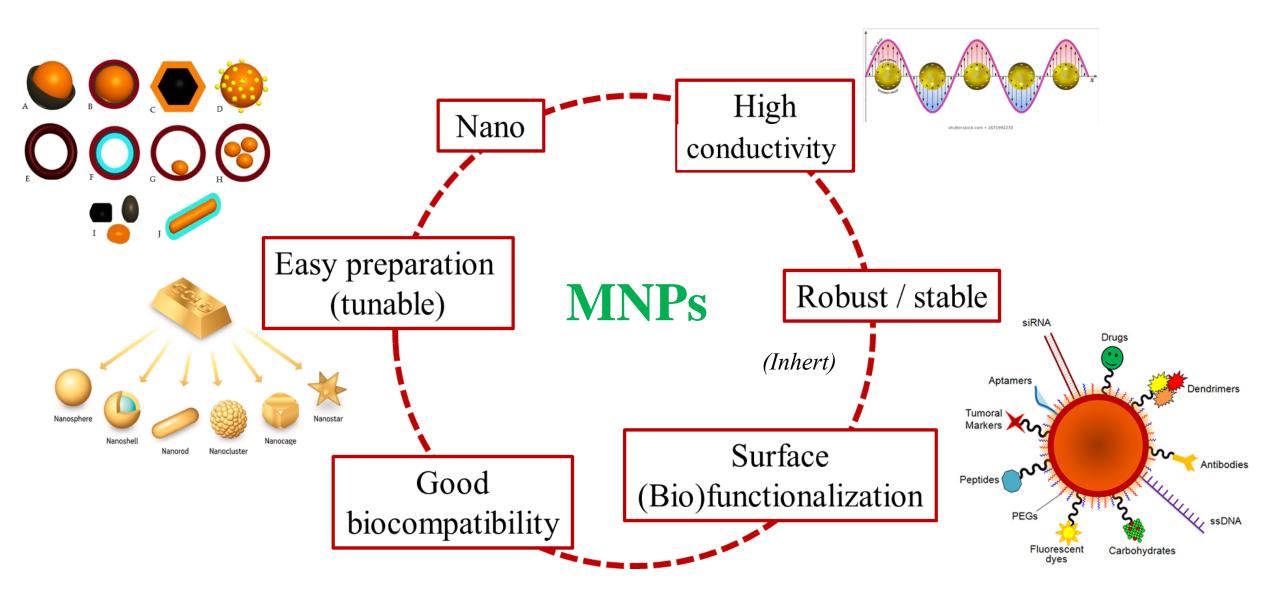
#### Evans blue assay







#### Advantages of metal nanoparticles for analyitical purposes

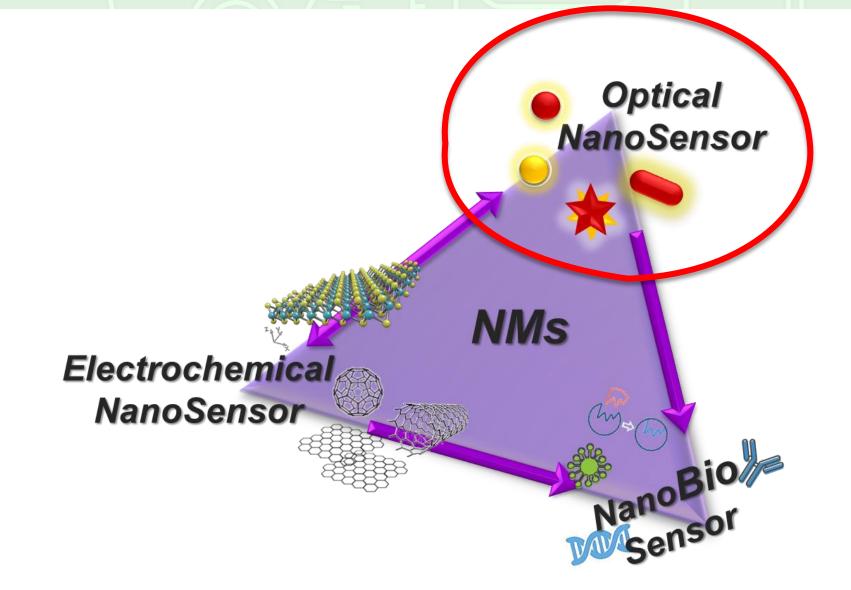


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#### Nanoparticles in analytical chemistry and food analysis





## **MNPs and (L)SPR**



#### Metal nanoparticles

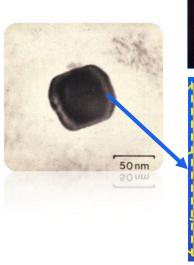
#### From Romans...

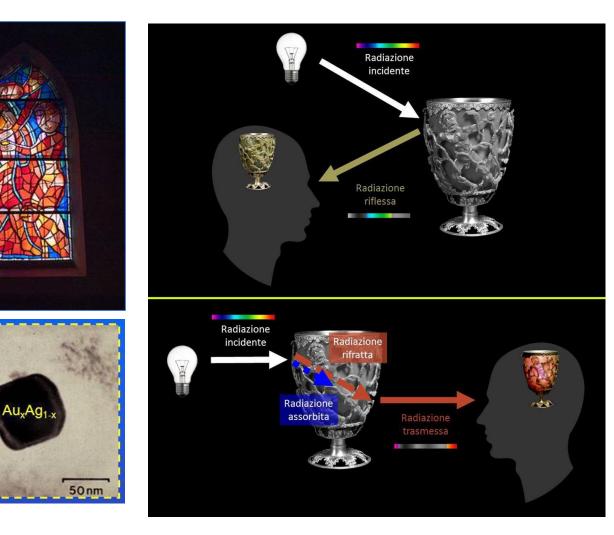


#### Nanotecnologia romana

I colori cangianti della Coppa di Licurgo, datata IV secolo a.C., sono dovuti a nanoparticelle di oro e argento disperse nella matrice vetrosa.

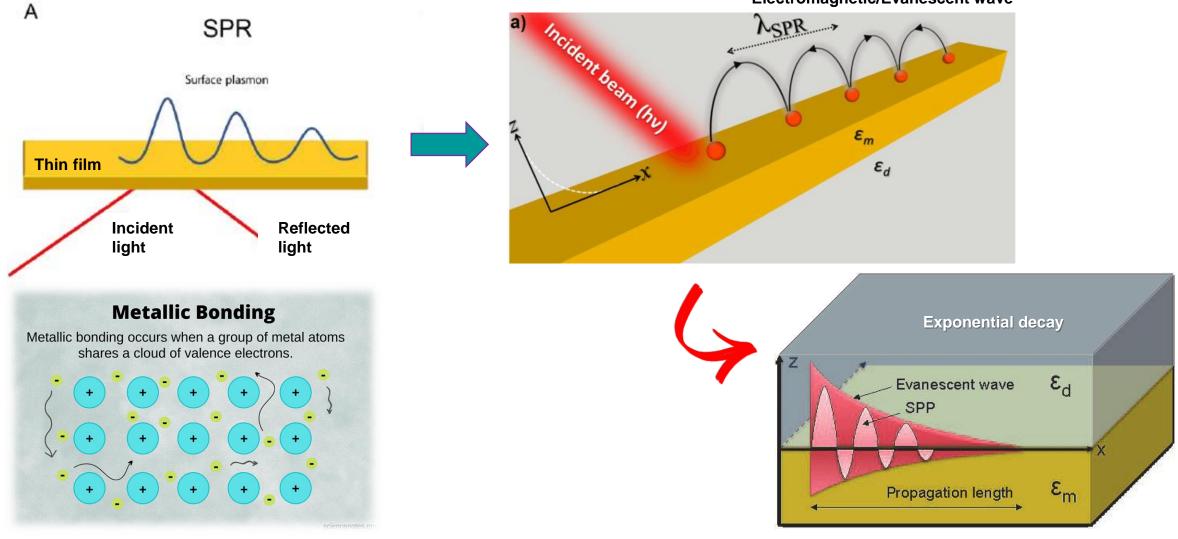






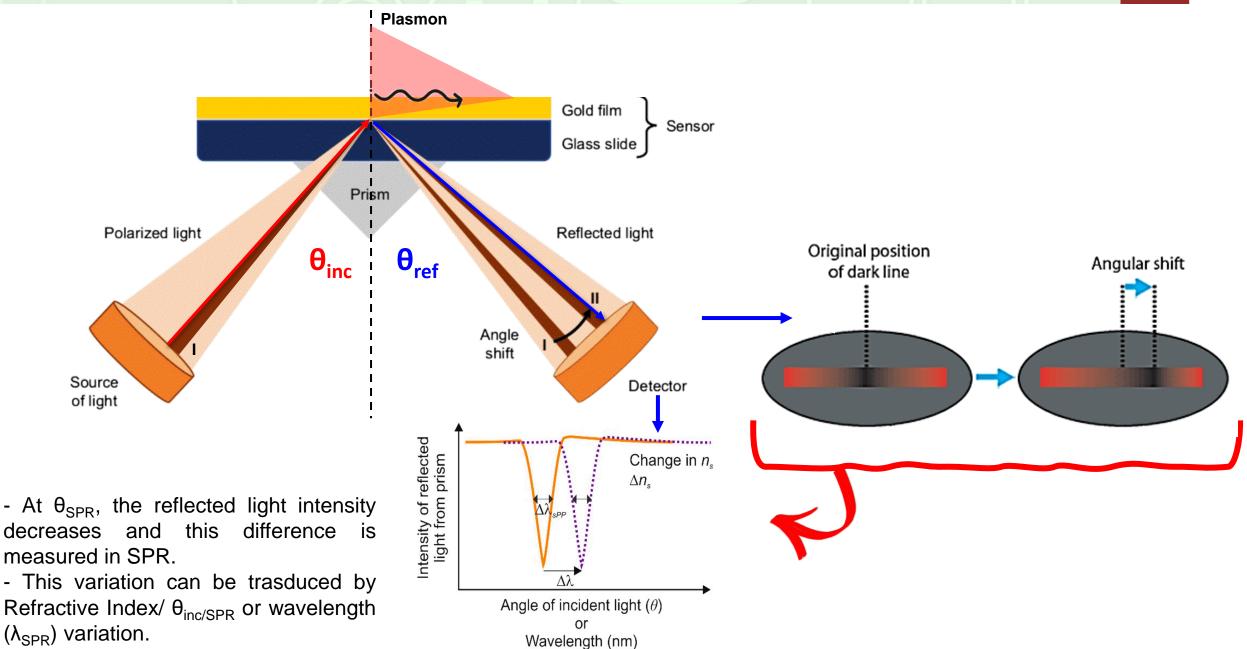
Novoselov, K. S., Geim, A. K., Morozov, S. V., Jiang, D. A., Zhang, Y., Dubonos, S. V., ... & Firsov, A. A. (2004). science, 306(5696), 666-669.

Surface plasmon resonance (SPR) is a phenomenon where the electrons in the metal surface layer are excited by photons of incident light with a certain angle of incidence, and then propagate parallel to the metal surface (Zeng et al., 2017)



#### Electromagnetic/Evanescent wave

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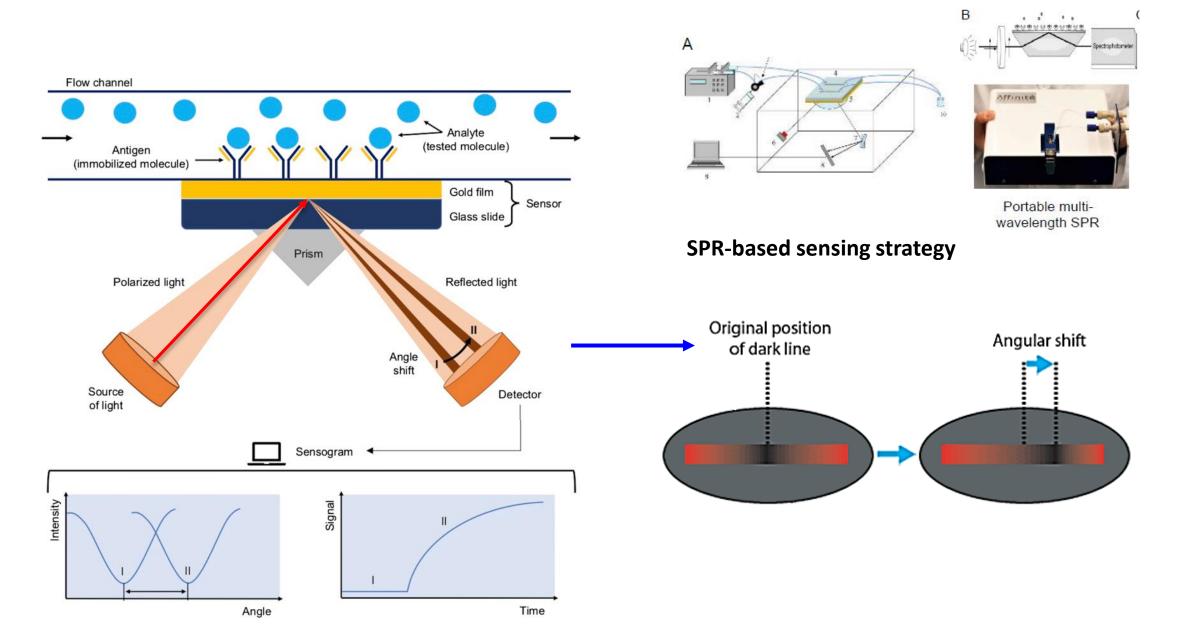


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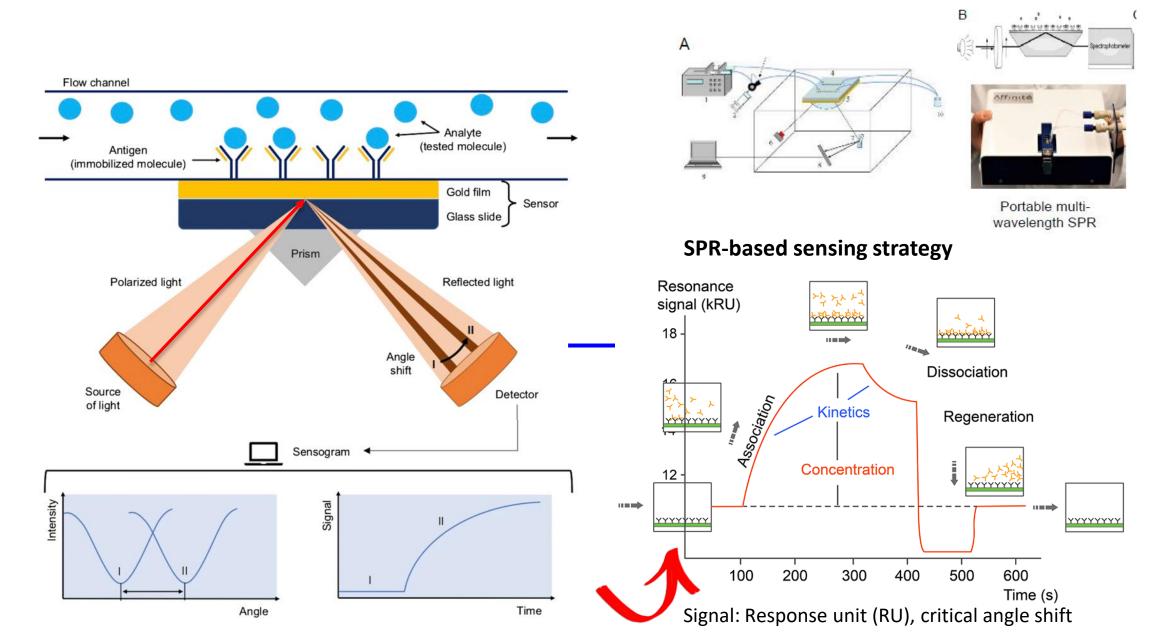
UNIT

 $(\lambda_{SPR})$  variation.









#### **MNPs and (L)SPR**



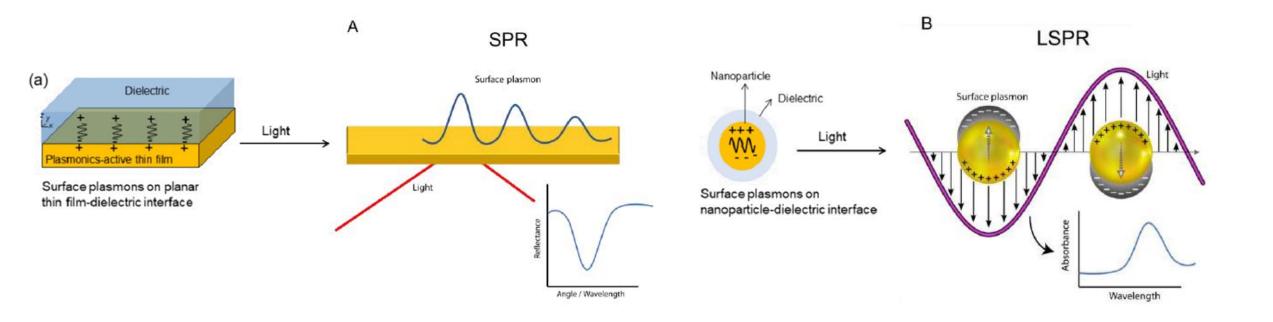
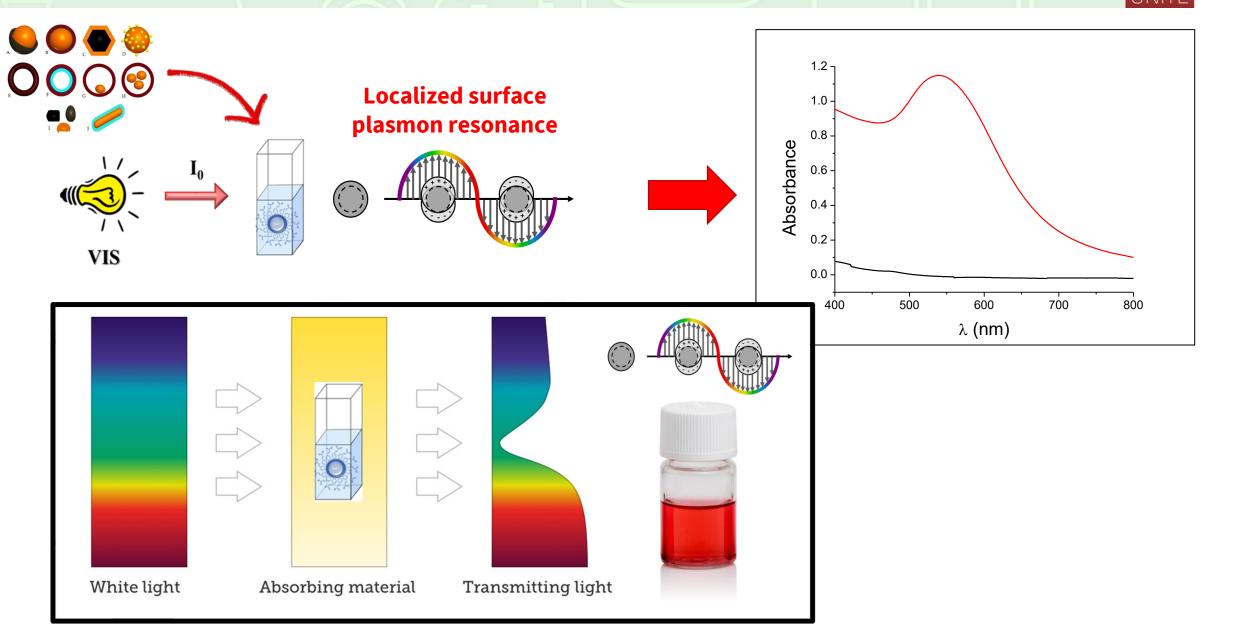


Figure 1. A) Prism coupling configuration of SPR, where a light beam impinges on a thin metallic film deposited on a prism. P-polarized light absorbed by the surface plasmon is seen from a minimum in the reflection spectra. B) Representation of the localized surface plasmon on nanoparticles and absorbance spectra obtained for binding events on nanoparticles.

#### Localized Surface Plasmon Resonance (LSPR)



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### **MNPs and LSPR**



#### Metal nanoparticles: their camaleontic features

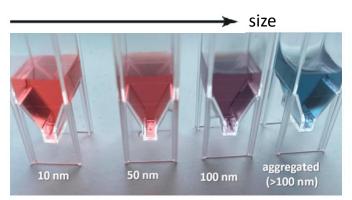


MNPs can interact in different ways VIS-electromagnetic radiation depen on their shapes, sizes, and composition

#### Gold nanoparticles

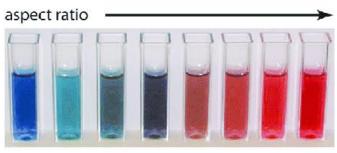
Diameter

10 nm

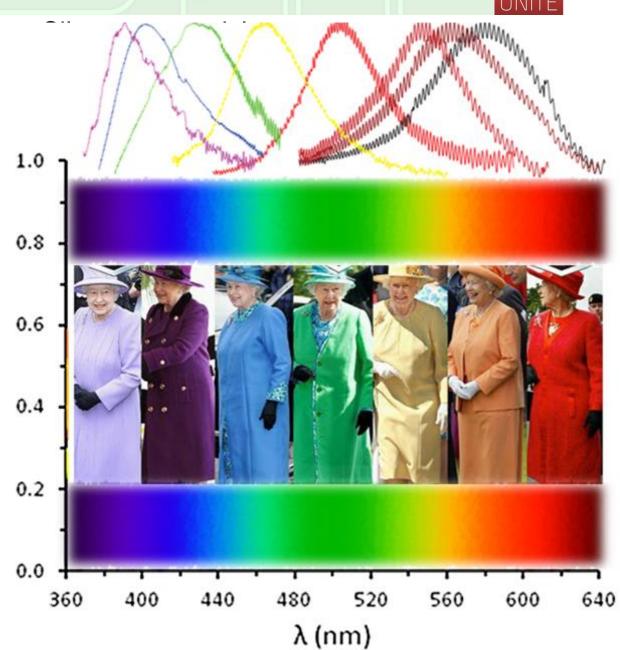


#### Gold nanorods



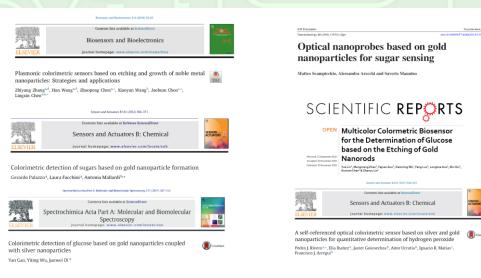


Absorbance





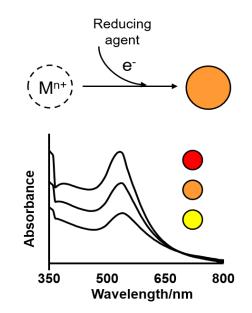
Colloidal metal nanoparticles based assays

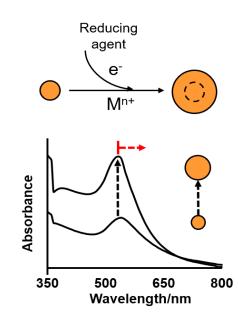


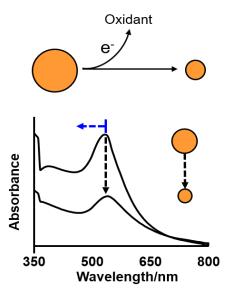


Benediktas Brasiunas 🛱, Anton Popov 🛱, Arunas Ramanavicius 🛱, Almira Ramanaviciene 🔍 🛱

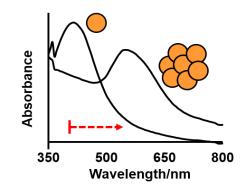
#### **Localized Surface Plasmon Resonance**



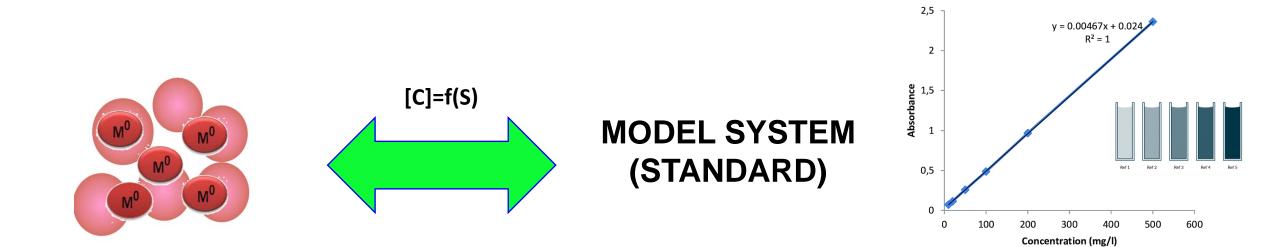


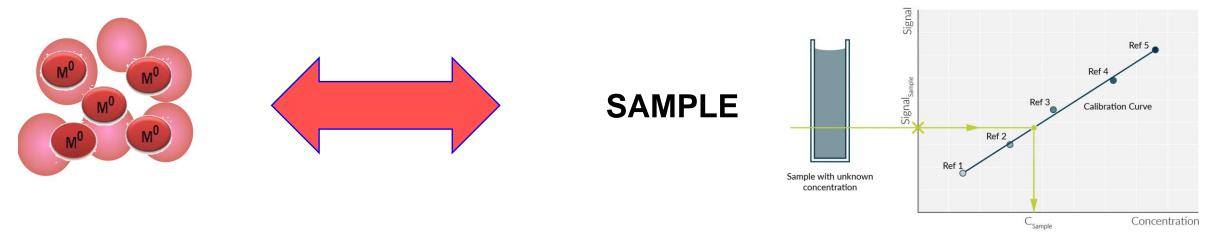




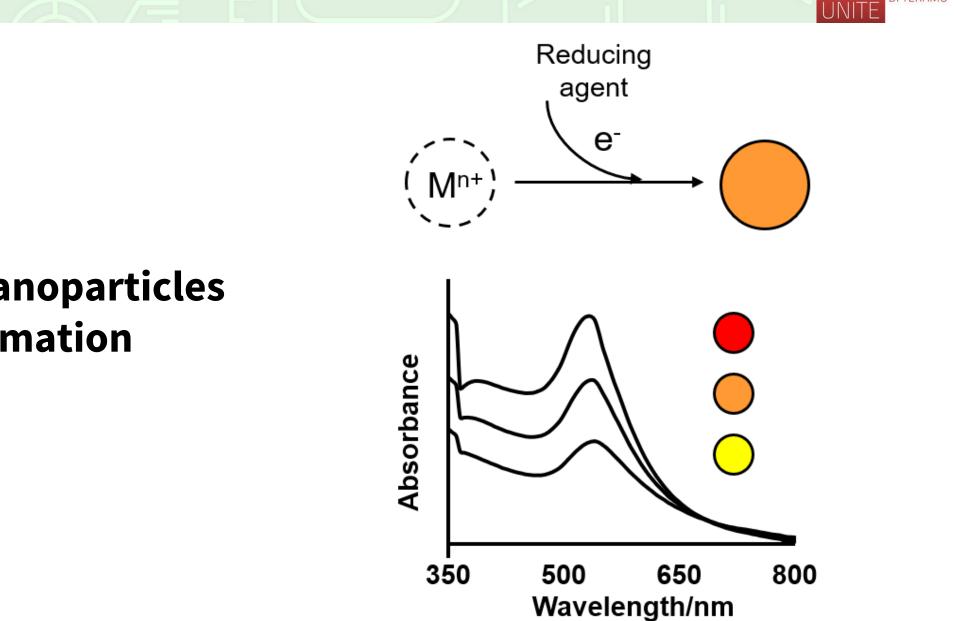








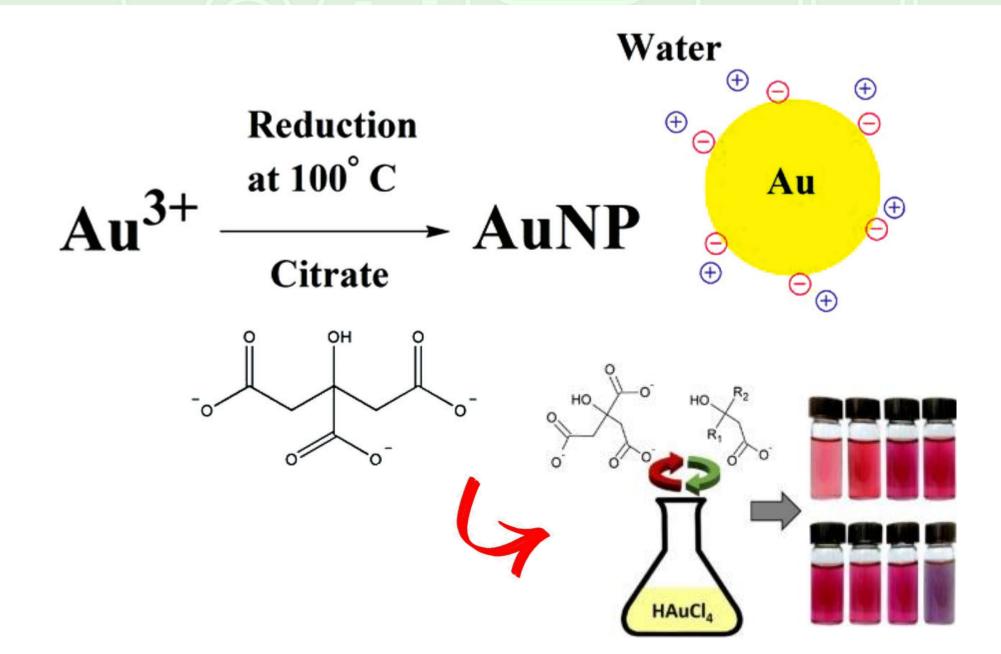
Concentration



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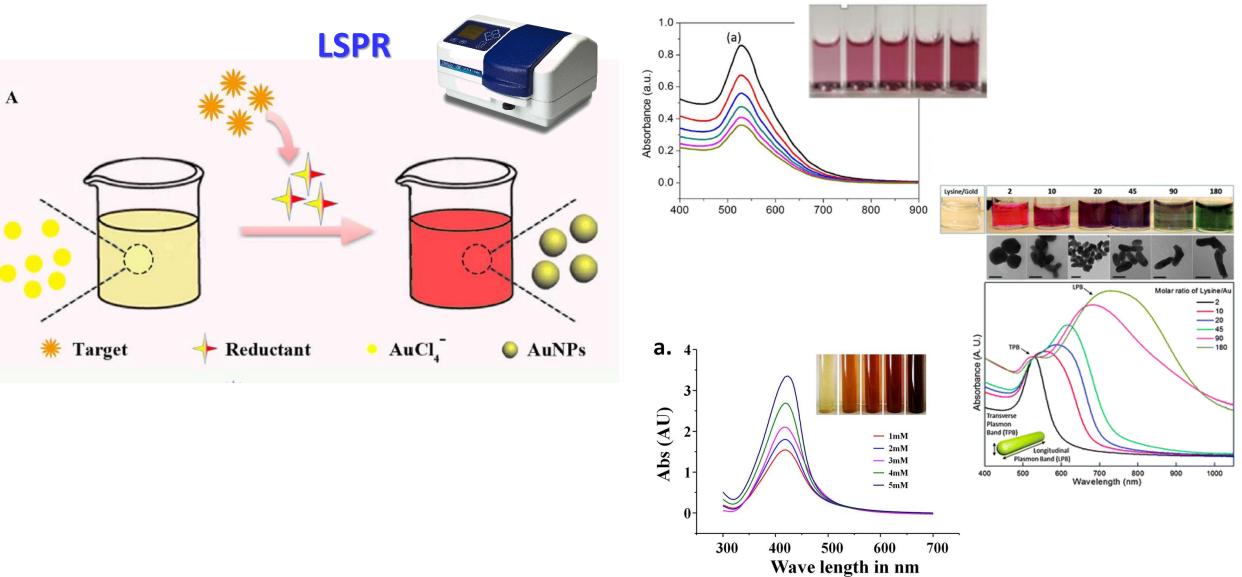
## Metal nanoparticles formation





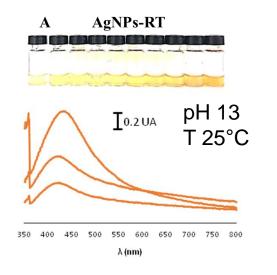
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#### Main strategy



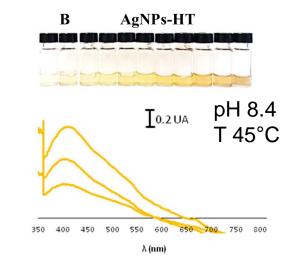
**Analytical signal** 

Phenolic content and antioxidant capacity evaluation trough Au and AgNPs formation

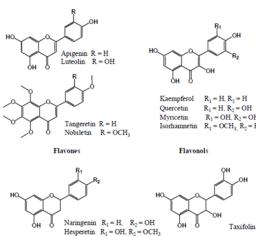


**Antioxidant Capacity** 





**Class Selective** 

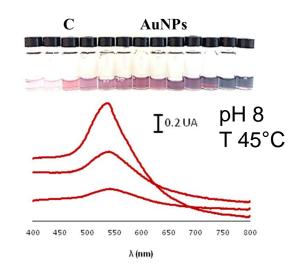




Flavanones

Taxifolin

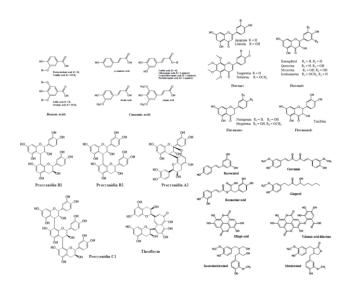
Flavanonols



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#### Total phenolic content





#### Phenolic content and antioxidant capacity evaluation trough Au and AgNPs formation

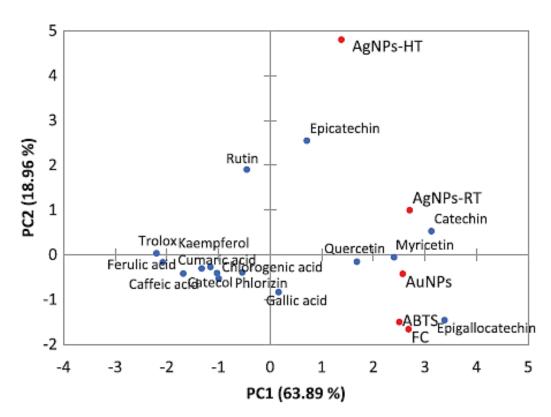
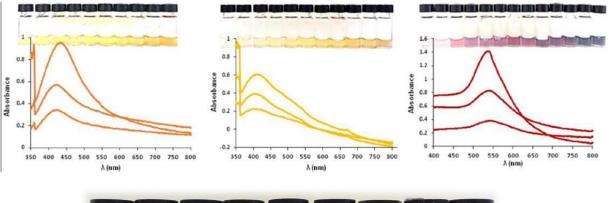


Fig. 2. PCA of the ABTS, FC, AuNPs and the proposed AgNPs-based methods reactivity vs. polyphenolic compounds. The biplot (Score and loading) of the first two principal components showed 82.85% of the cumulative variance. Rows normalization were applied to the dataset. Data were autoscaled before PCA.

R	ABTS	FC	AgNPs-HT	AgNPs-RT	AuNPs
ABTS	1	0.876	0.891	0.956	0.977
FC	0.876	1	0.733	0.913	0.801
AgNPs-HT	0.891	0.733	1	0.770	0.826
AgNPs-RT	0.956	0.913	0.770	1	0.950
AuNPs	0.977	0.801	0.826	0.950	1

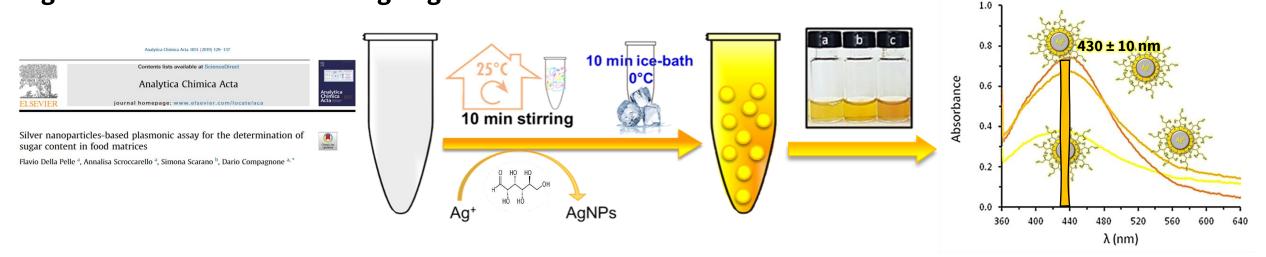


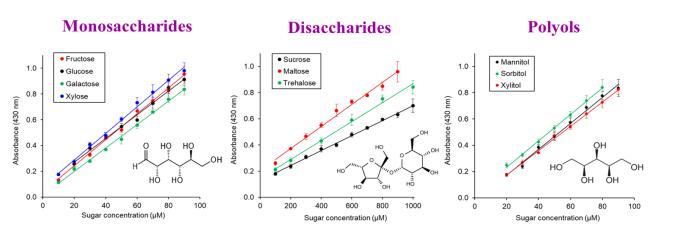


VT: Vanilla Tea
TG: Green Tea
TC: Classic Tea
SD: sogni d'oro infused
RE: Relax infused
RB: Rosa di bosco Infused
LT: Lemon Tea
IN: Finocchio infused
DIG: Digestiva infused



#### Sugars content evaluation trough AgNPs formation





AgNPs assay Sample RSD Ion chromatography RSD **Rel. error**  $(g 100 \text{ mL}^{-1}, \text{Glu}, \text{Eq.})$  (%, n = 5)  $(g 100 \text{ mL}^{-1}, \text{Glu} + \text{Fru})$  (%, n = 3) (%) Peach tea  $2.98 \pm 0.14$ 4.74 2.71 +5.0 $3.13 \pm 0.08$ Black tea  $3.56 \pm 0.23$ 6.51  $3.05 \pm 0.11$ 3.56 - 14.3 Coconut water  $4.72 \pm 0.13$ 2.84  $4.93 \pm 0.05$ 0.98 +4.4 $3.50 \pm 0.25$ 7.23  $3.53 \pm 0.04$ 1.21 Gaseous +0.9Cedrata  $9.36 \pm 0.27$ 8.9 2.85  $8.74 \pm 0.78$ - 6.6  $6.11 \pm 0.07$ Tonic water 1.15  $5.62 \pm 0.12$ 2.11- 8.0 Apple 1  $1.67 \pm 0.02$ 1.12  $1.50 \pm 0.02$ 1.11 - 10.2 Apple 2 1.78 0.0  $1.14 \pm 0.04$ 3.41  $1.14 \pm 0.02$ Apple 3  $3.27 \pm 0.04$ 1.30  $3.12 \pm 0.10$ 3.10 - 4.6 Apple 4  $2.43 \pm 0.23$ 9.42  $2.67 \pm 0.11$ 4.10 +9.9Apple 5  $2.43 \pm 0.02$ 0.74  $2.31 \pm 0.9$ 3.80 - 5.0

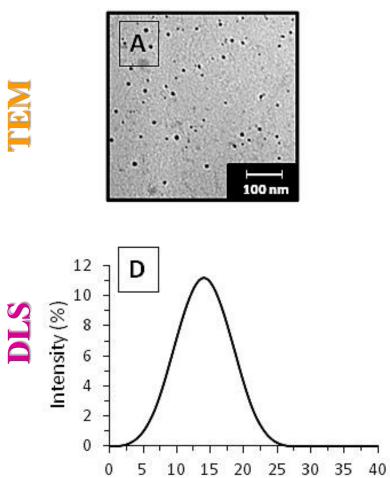
Recovery: 86-118%

 $R^2 \ge 0.991$ Monosaccharides and polyols LOD=  $8.7 \pm 0.4 \mu M$ Disaccharides average LOD=  $120 \pm 0.1 \mu M$ RSD  $\le 8\%$ 

#### AgNPs Morphological study

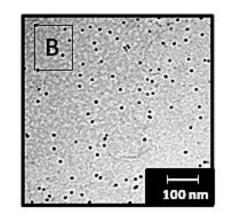
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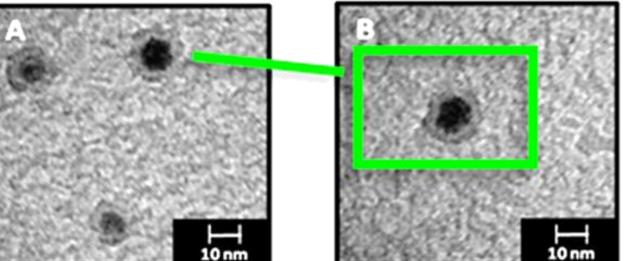


AgNPs size (d, nm)

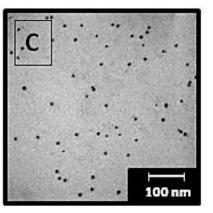
Sucrose



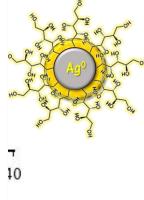
Intensity (%)



Xylitol

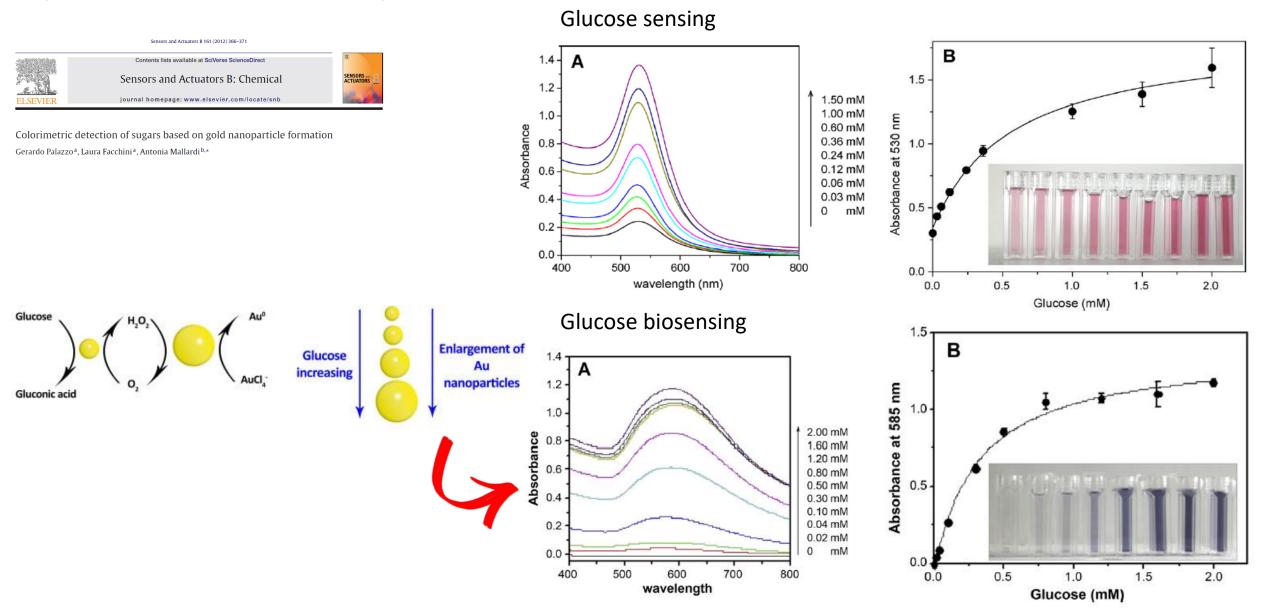


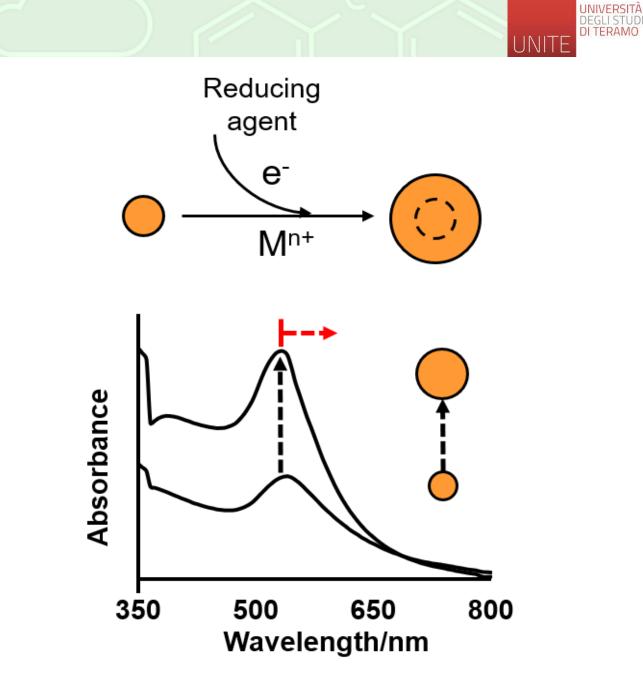




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#### Sugars content evaluation trough AuNPs formation

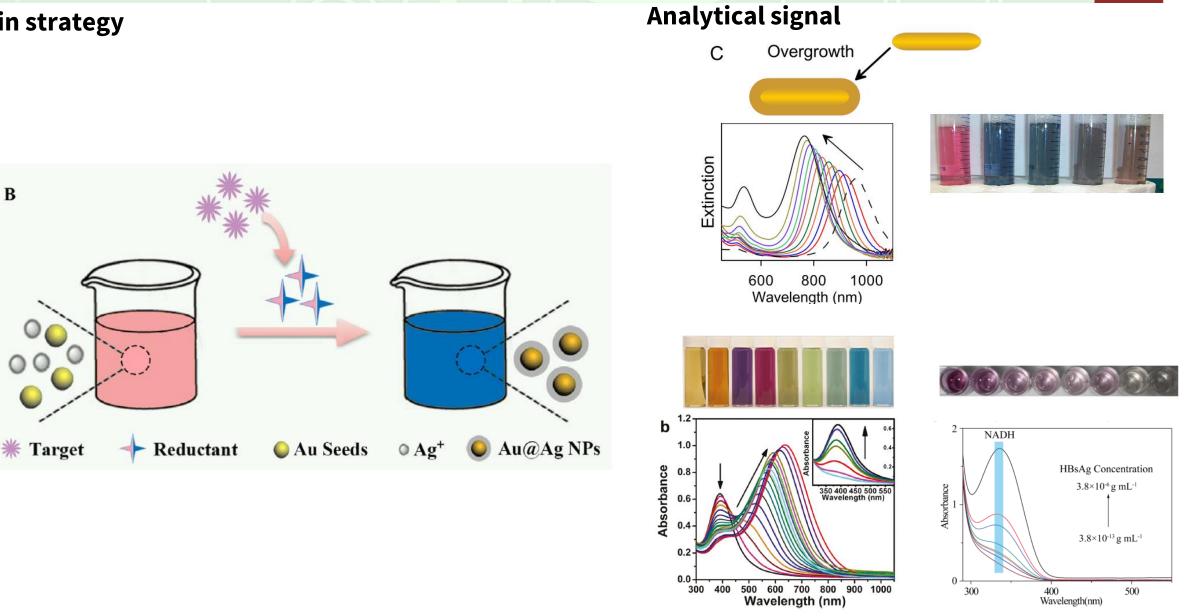




## Metal nanoparticle-based seed-growth strategies

### Metal nanoparticles growth

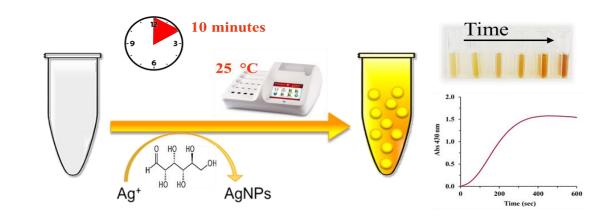
#### Main strategy

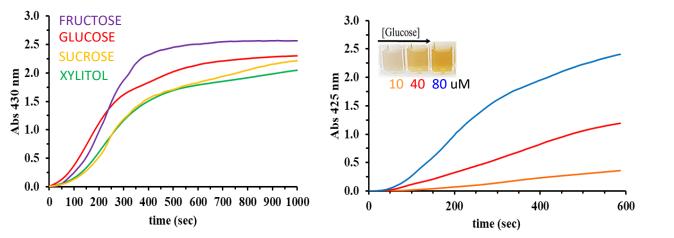


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# Metal nanoparticles growth

Reducing sugars monitoring trough AgNPs growth







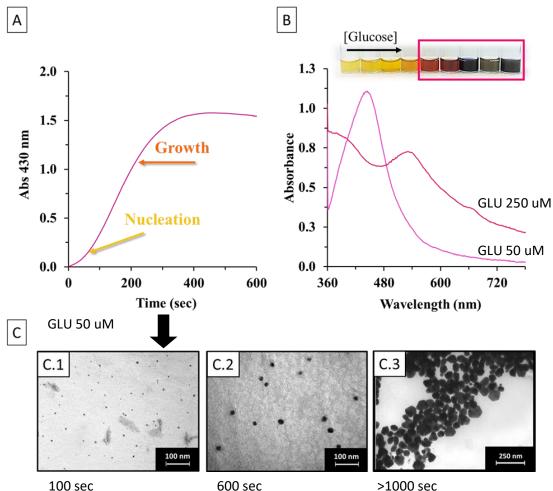
#### Studies on Silver Nanoparticles Production Mediated by Sugars

Annalisa Scroccarello, Flavio Della Pelle, Simona Scarano and Dario Compagnone

© Springer Nature Switzerland AG 2020

G. Di Francia et al. (eds.), Sensors and Microsystems, Lecture Notes

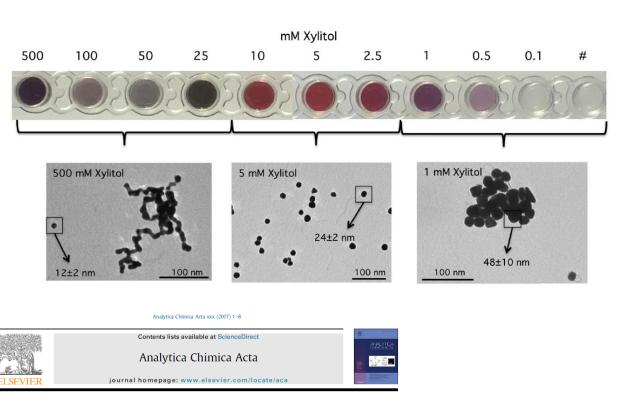
in Electrical Engineering 629, https://doi.org/10.1007/978-3-030-37558-4\_5





#### Metal nanoparticles growth

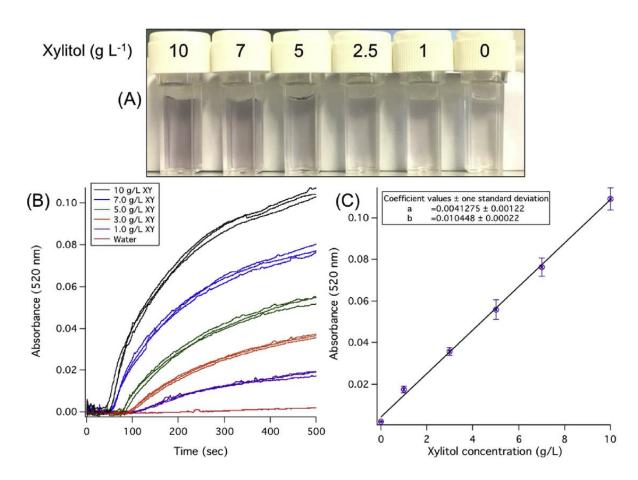
#### Xylitol monitoring trough AuNPs growth



Seed formation and growth phenomena study

The early nucleation stage of gold nanoparticles formation in solution as powerful tool for the colorimetric determination of reducing agents: The case of xylitol and total polyols in oral fluid

#### **Dose-response kinetic and curve**





#### Metal nanoparticles growth

Absorbance (520 nm)

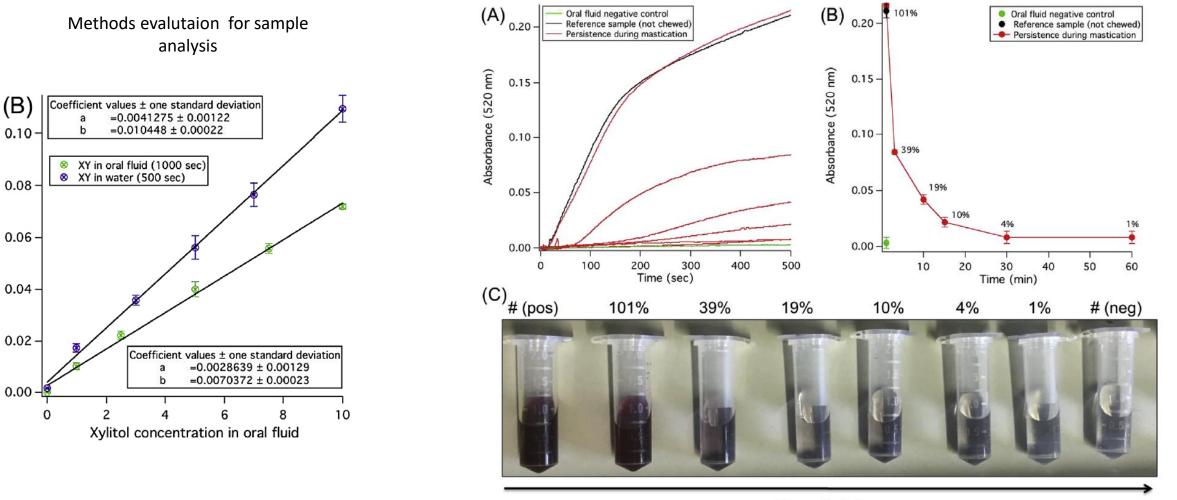


**Recovery study** 

#### Xylitol montoring along 1 hours of chewin-gum mastication

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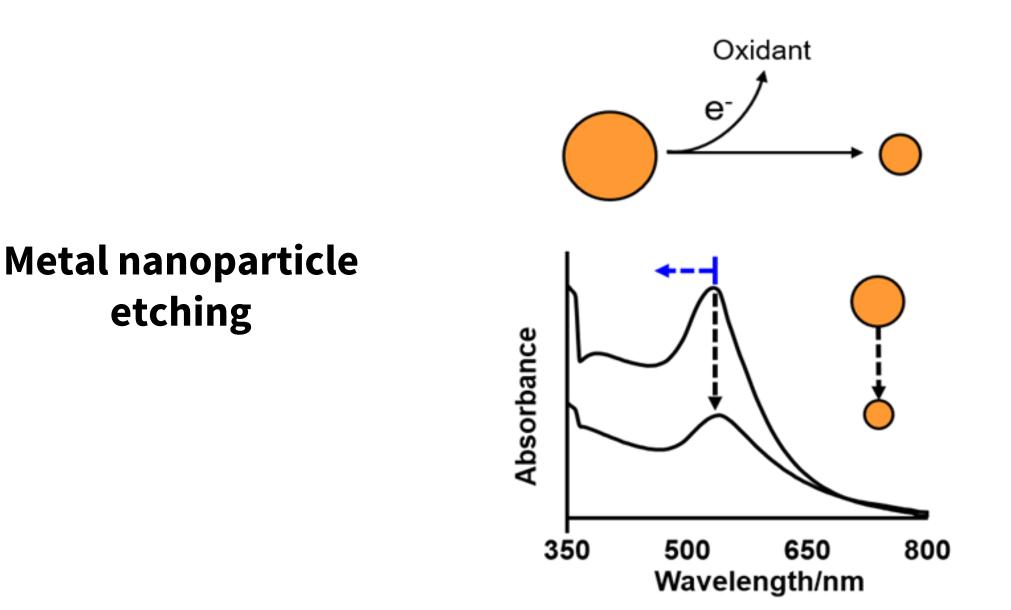
UNIT



**Xylitol decay** 

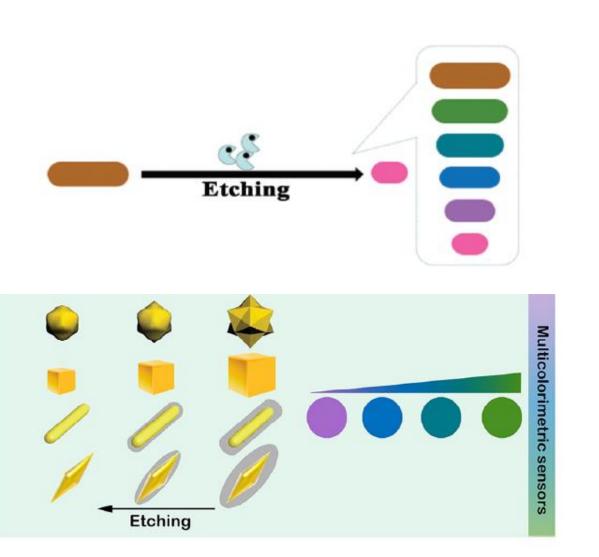
Time (min)



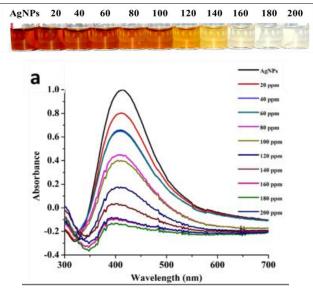


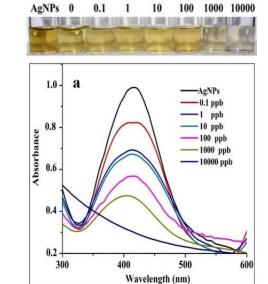


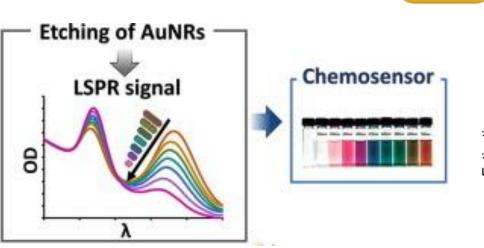
#### Main strategy

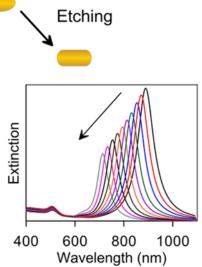


#### **Analytical signal**



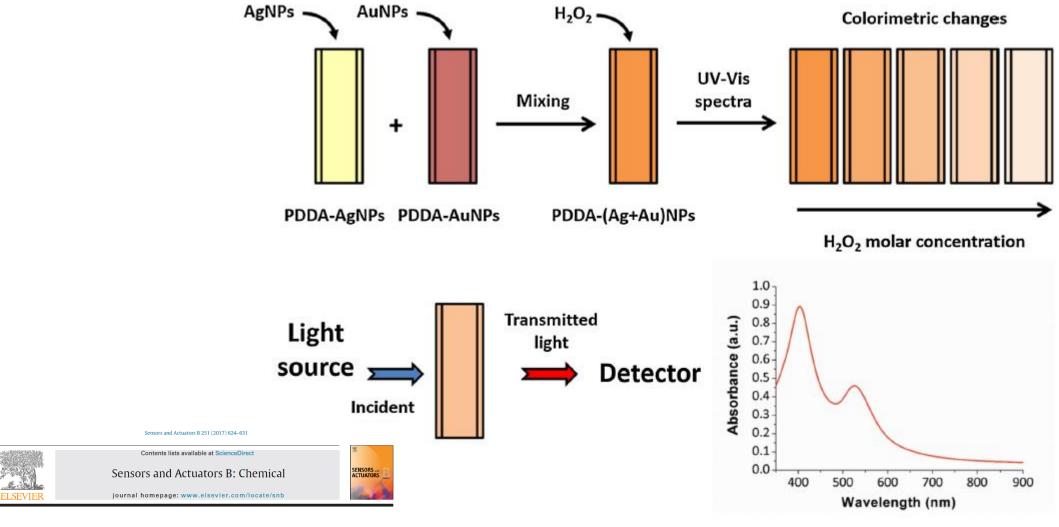








H<sub>2</sub>O<sub>2</sub> determination trough MNPs etching

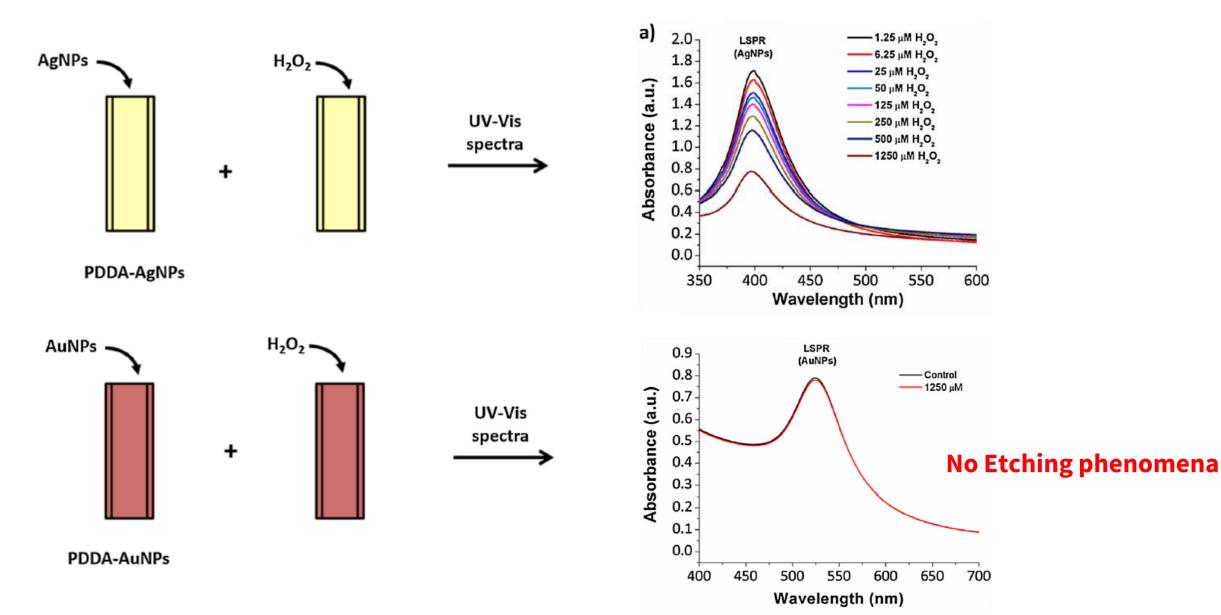


A self-referenced optical colorimetric sensor based on silver and gold nanoparticles for quantitative determination of hydrogen peroxide

Pedro J. Rivero<sup>a,\*</sup>, Elia Ibañez<sup>b</sup>, Javier Goicoechea<sup>b</sup>, Aitor Urrutia<sup>b</sup>, Ignacio R. Matias<sup>c</sup>, Francisco J. Arregui<sup>b</sup>

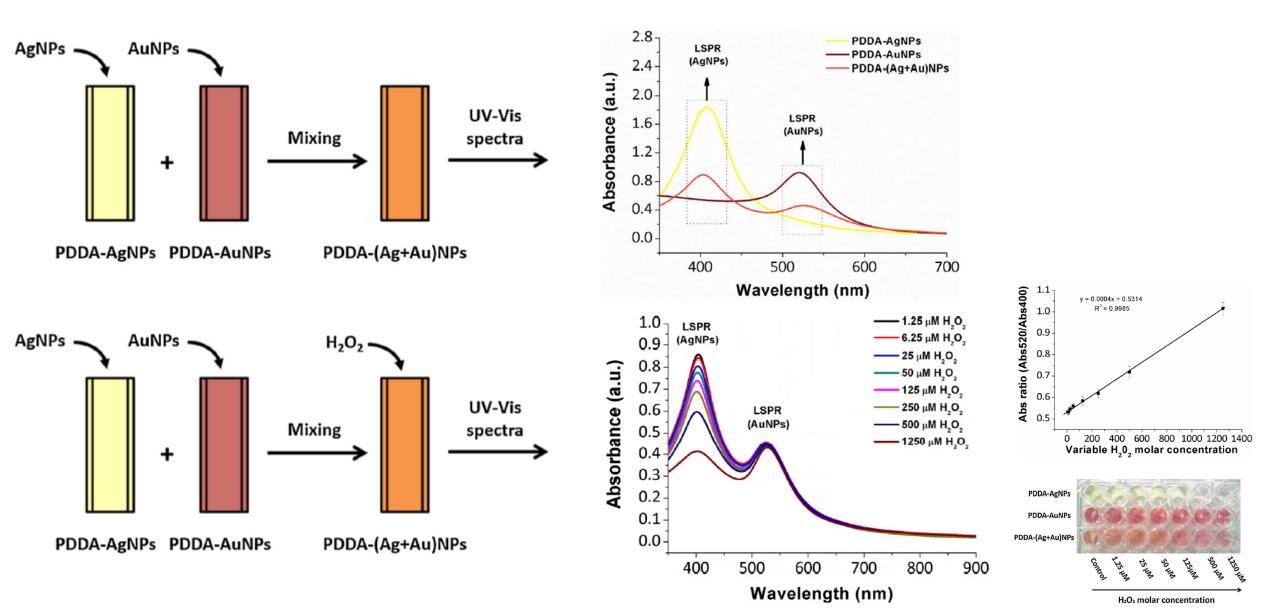


#### **Etching phenomena study**



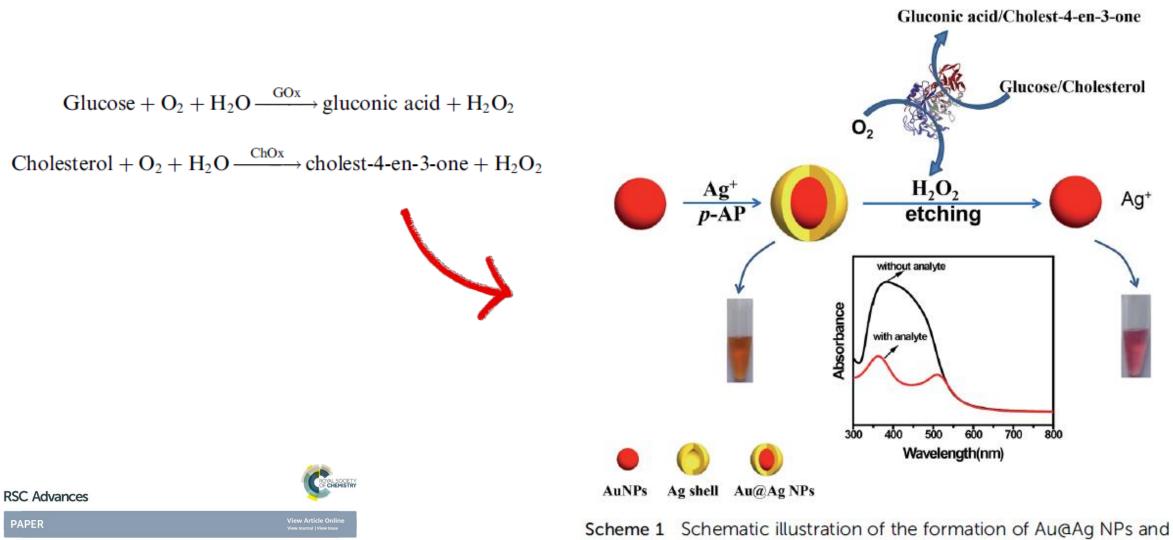


#### **Etching phenomena study.** H<sub>2</sub>O<sub>2</sub> determination





#### Glucose and cholesterol evaluation trough MNPs etching

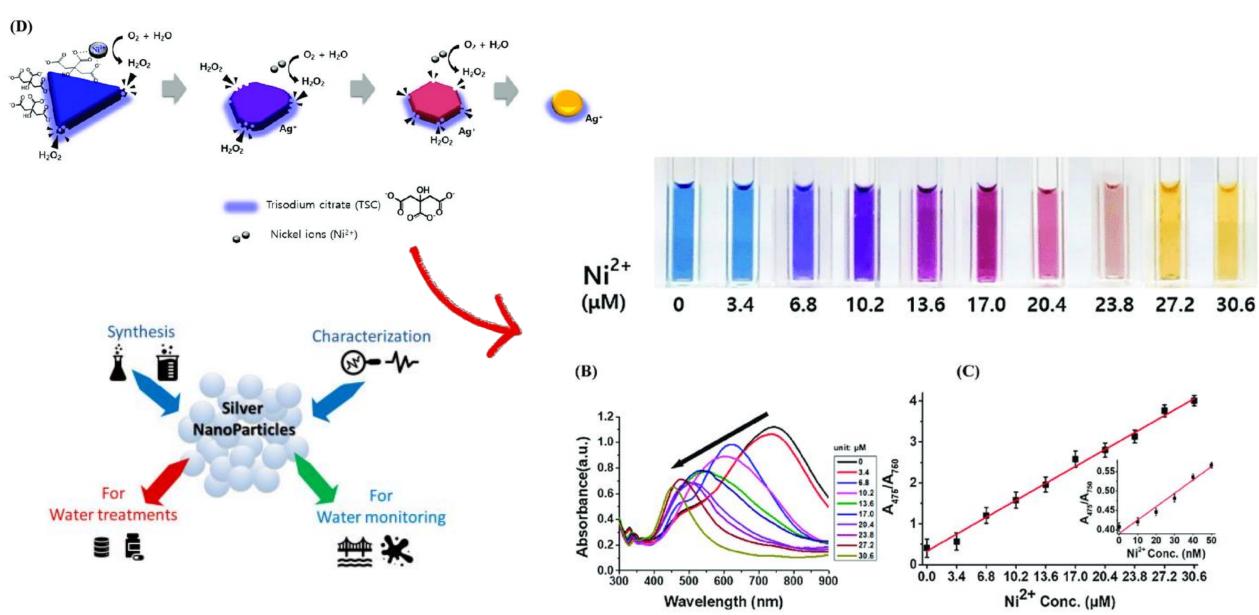


CrossMark S Cite this: RSC Adv., 2016, 6, 35001

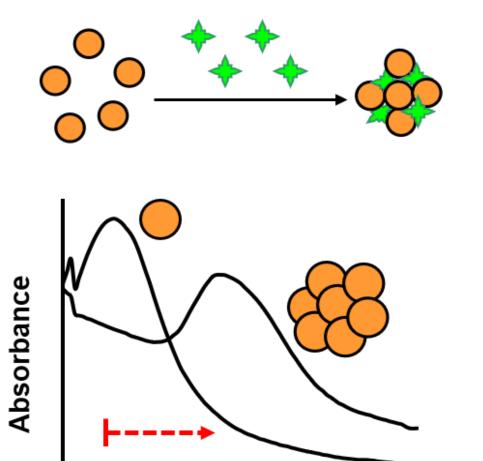
Sensitive colorimetric detection of glucose and cholesterol by using Au@Ag core-shell nanoparticles† Scheme 1 Schematic illustration of the formation of Au@Ag NPs and its application for the colorimetric detection of  $H_2O_2$  and glucose/ cholesterol.



#### **Nickel determination**





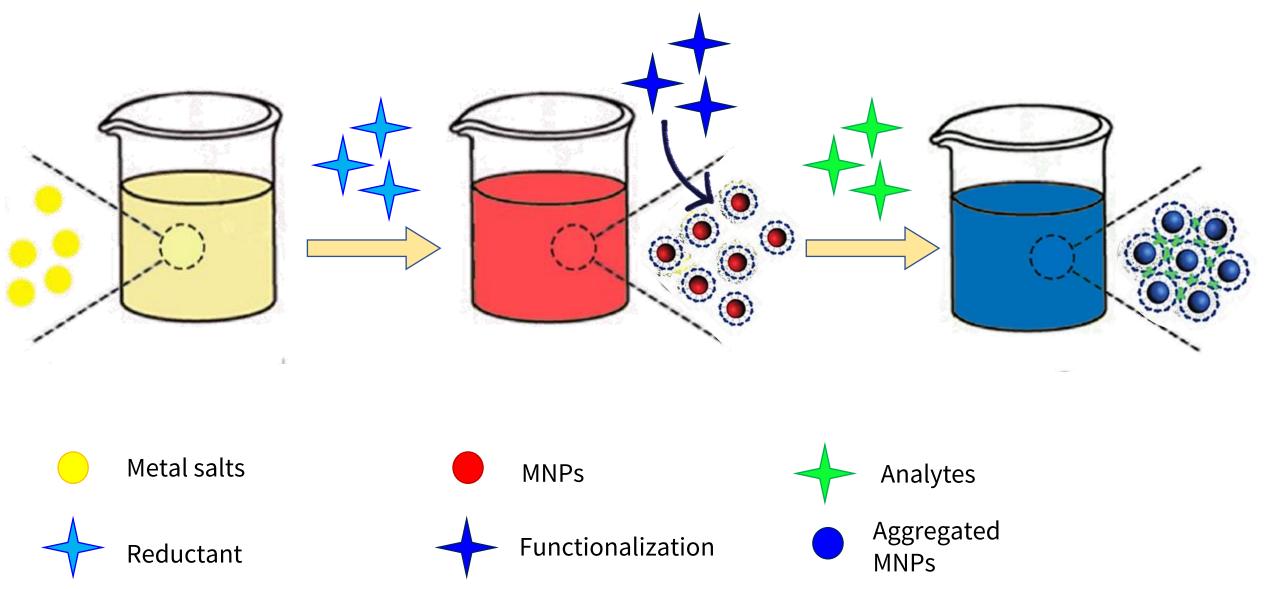


Wavelength/nm

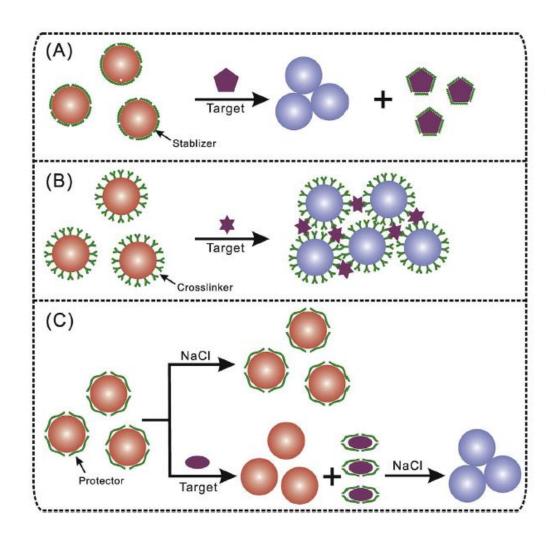
# Metal nanoparticle aggregation

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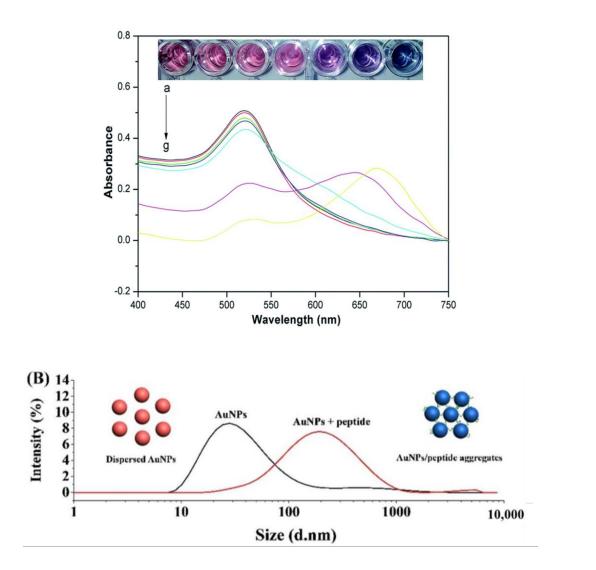
#### Main strategy



#### Main strategy



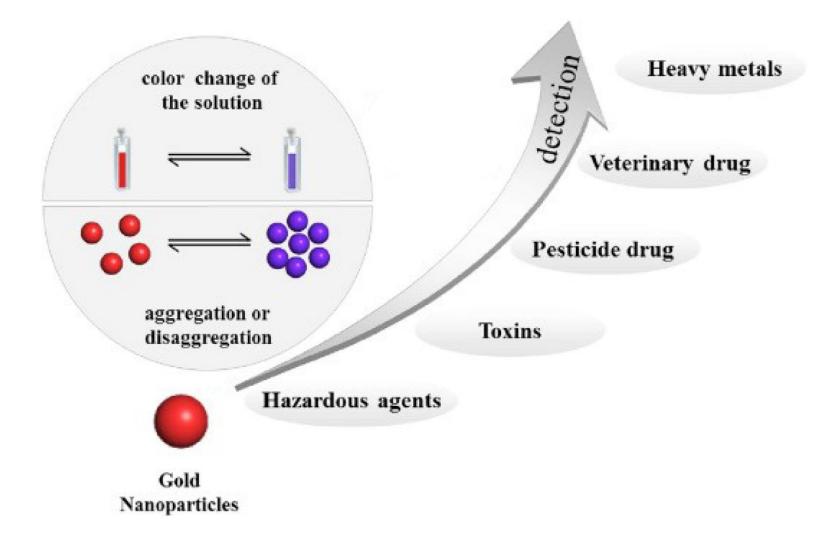
#### **Analytical signal**



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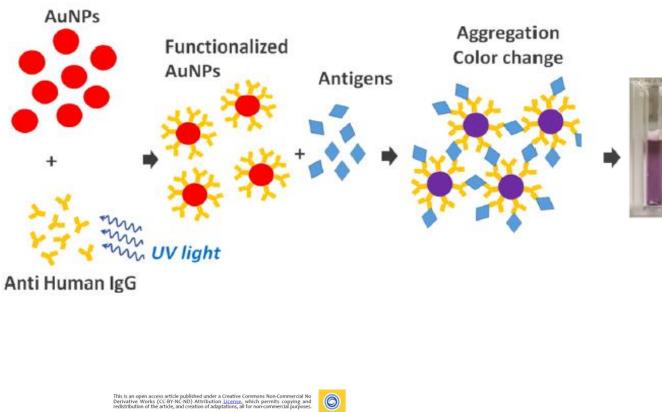
#### Main strategy







#### Immuno-based determination of HIgG

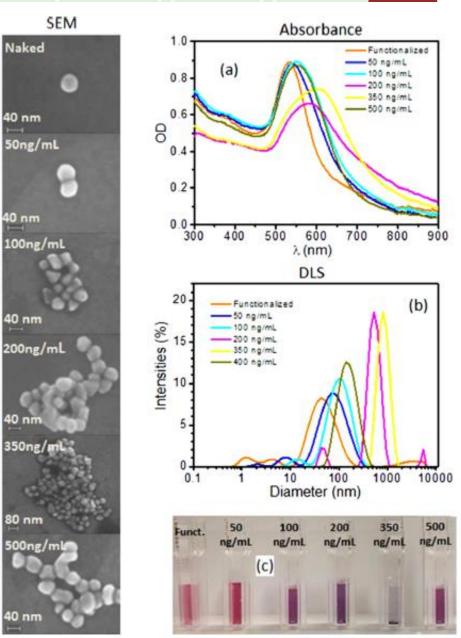


Article



#### Colorimetric Immunosensor by Aggregation of Photochemically **Functionalized Gold Nanoparticles**

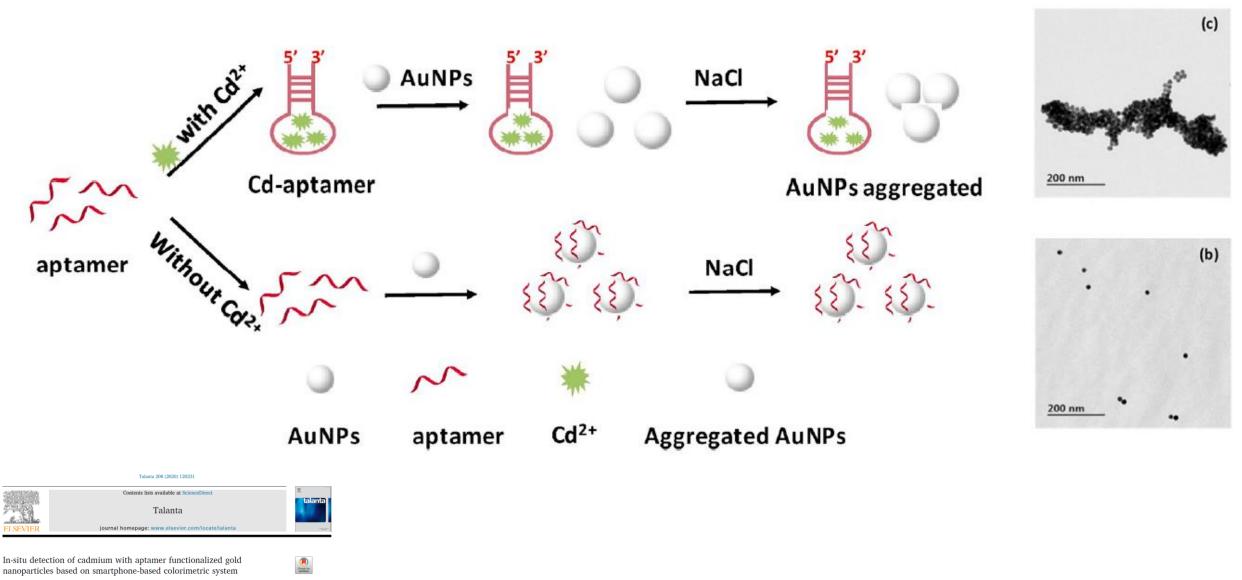
Marzia Iarossi,<sup>†,||</sup> Chiara Schiattarella,<sup>†,‡</sup> Ilaria Rea,<sup>‡</sup> Luca De Stefano,<sup>‡</sup> Rosalba Fittipaldi,<sup>§</sup> Antonio Vecchione,<sup>§</sup> Raffaele Velotta,<sup>\*,†,©</sup> and Bartolomeo Della Ventura<sup>†</sup>



40 nr

40 nn

Cd<sup>2+</sup> indirect determination trough AuNPs aggregation



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Ying Gan<sup>a,b</sup>, Tao Liang<sup>a</sup>, Qiongwen Hu<sup>a</sup>, Longjie Zhong<sup>a</sup>, Xinyi Wang<sup>a</sup>, Hao Wan<sup>a,b,\*\*</sup>, Ping Wang<sup>a,b,\*</sup>



Cd<sup>2+</sup> indirect determination assay format

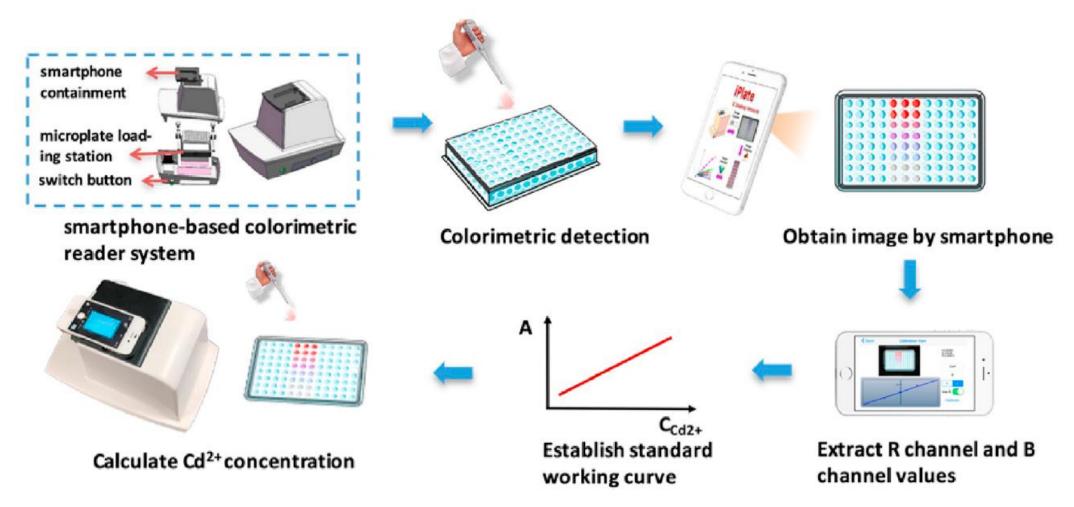
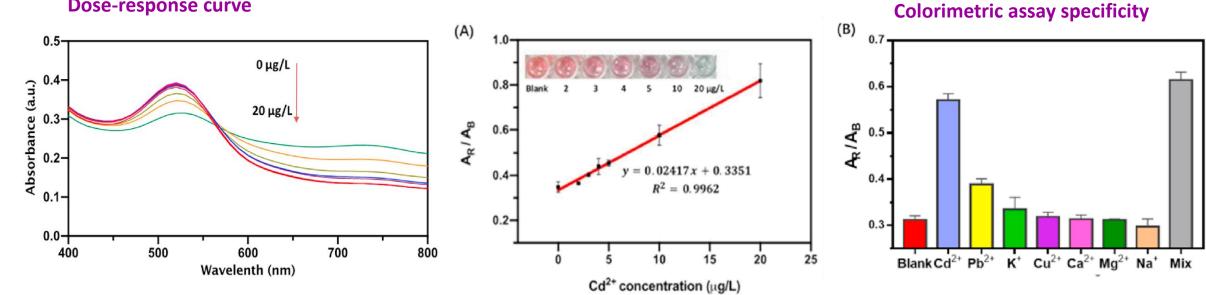


Fig. 2. Detection process of smartphone based colorimetric reader system.

#### Cd<sup>2+</sup> indirect determination

**Dose-response curve** 



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#### **Recovery study**

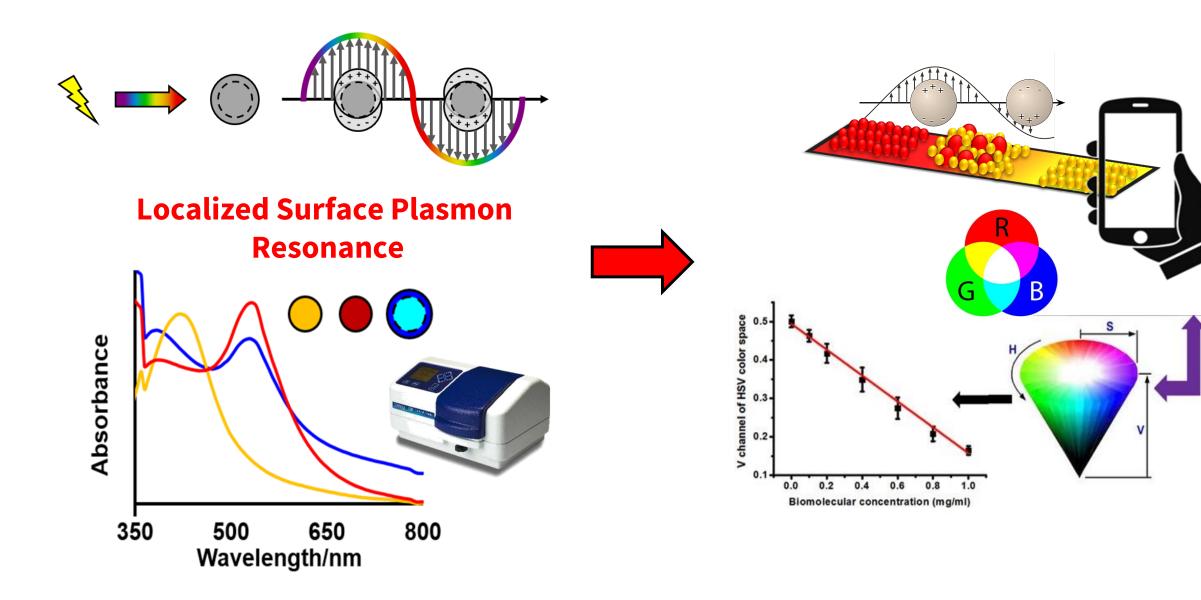
Determination of Cd <sup>2+</sup>	in tap water	samples using t	he proposed method	1.

Samples	Spiked concentration (µg/L)	This colorimetric system		Microplate reader	
		Determined conc. (µg/L)	Recovery%	Determined conc. (µg/L)	Recovery%
1	5	5.18	116.4	5.21	104.3
2	10	11.32	113.2	10.96	109.6
3	10	11.61	116.1	10.83	108.3
4	20	21.63	108.15	20.12	100.6
5	20	23.56	117.8	22.15	110.8

# **Colorimetric approach**



From plasmonic... Towards colorimetric strategies



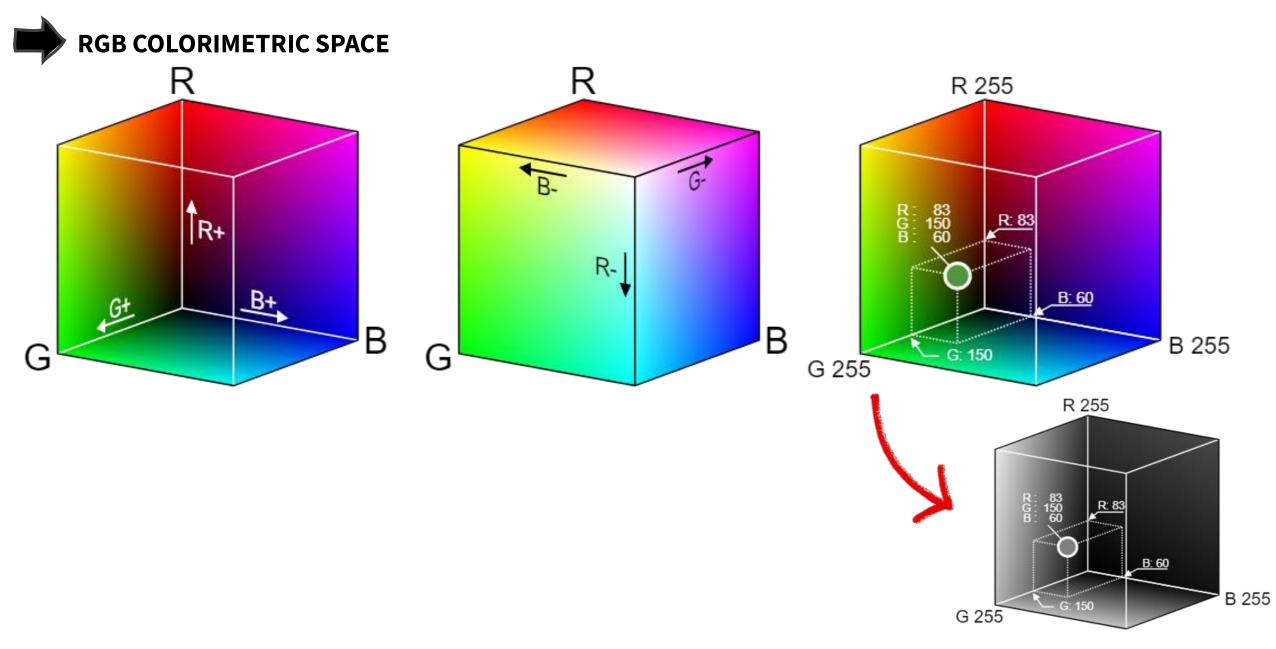


# Optical spectroscopic methods/ Spectrochemical methods. WHICH COLOR I PERCEIVE?

The Visible Spectrum					
Wavelength Region Absorbed, nm	Color of Light Absorbed	Complementary Color Transmitted			
400-435	Violet	Yellow-green			
435–480	Blue	Yellow			
480-490	Blue-green	Orange			
490–500	Green-blue	Red			
500–560	Green	Purple			
560–580	Yellow-green	Violet			
580–595	Yellow	Blue			
595–650	Orange	Blue-green			
650–750	Red	Green-blue			

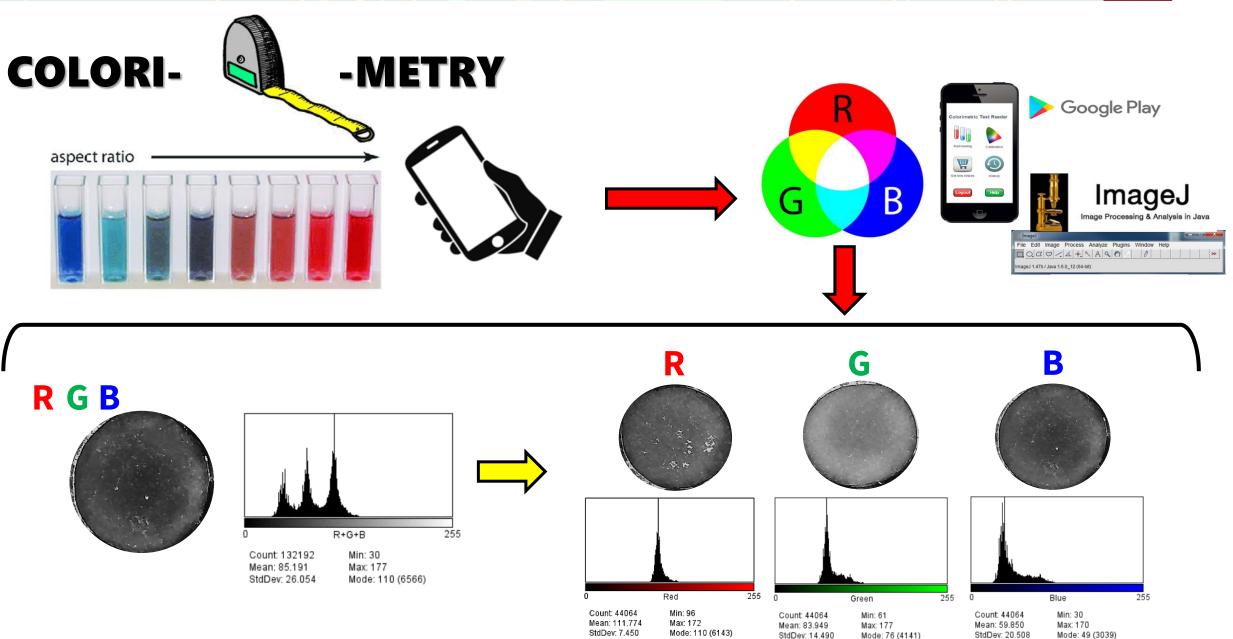
#### **COLORIMETRY PILLAR**



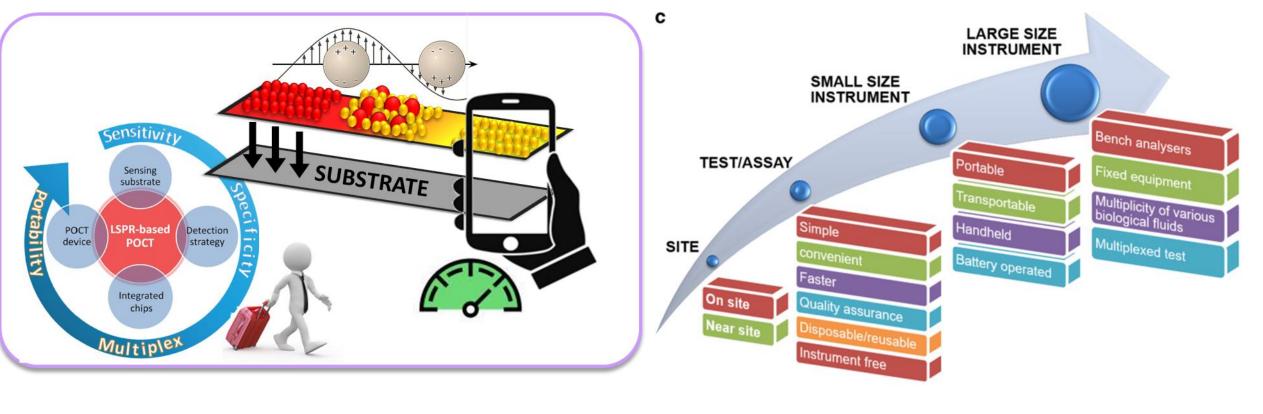


#### **COLORIMETRY PILLAR**





#### Metal nanoparticles integration onto solid substrates



# !!! Lab-on-a-strip Device !!!

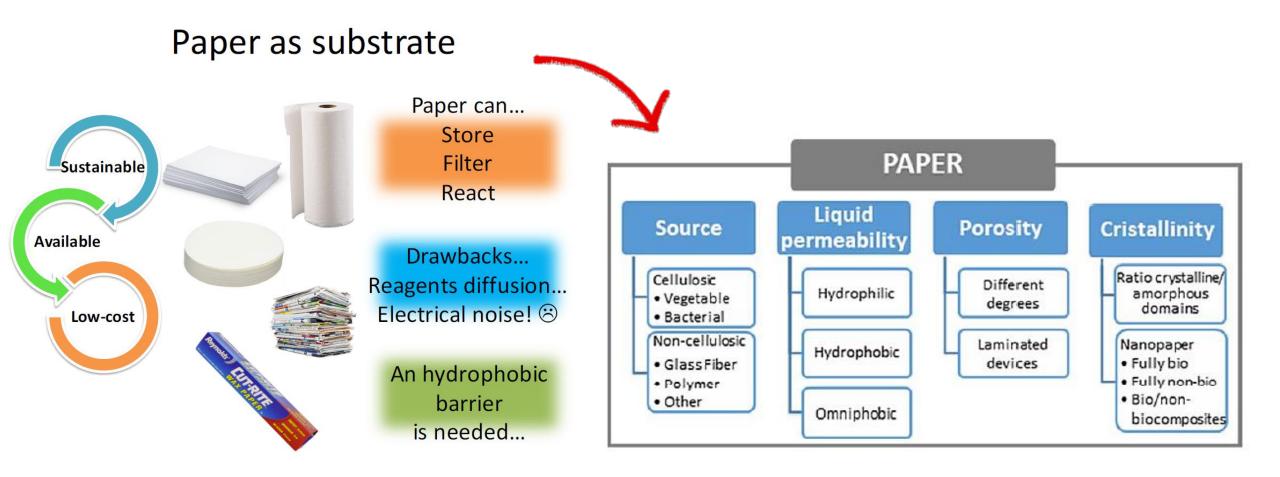


Cost performance Manufacturing Mass production UNIVERSITÀ DEGLI STUDI DI TERAMO

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#### Paper as elective substrate





#### Paper as elective substrate



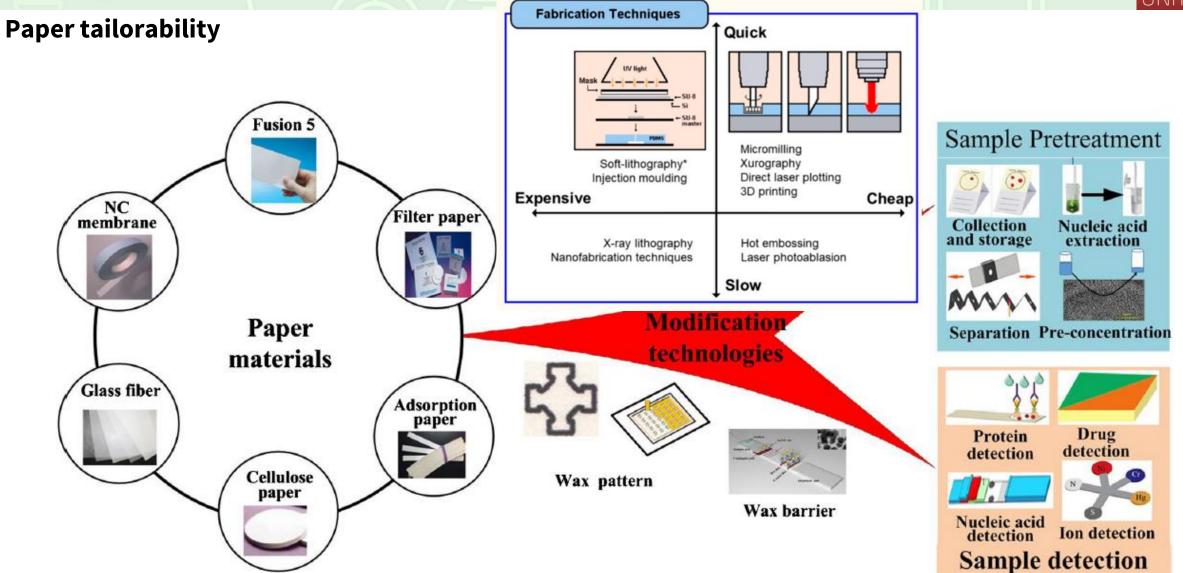


Fig. 1 Existing paper modification approaches for paper-based POCT. Different paper materials, including Fusion 5, filter paper, chromatography paper, cellulose paper, Whatman® No.1 filter paper and NC membrane, have been modified with various reagents for paper-based sample pretreatment and paper-based detection

# MNPs as colorimetric probe

#### Analytica Chimica Acta 1183 (2021) 338971



Metal nanoparticles based lab-on-paper for phenolic compounds evaluation with no sample pretreatment. Application to extra virgin olive oil samples

Check for updates

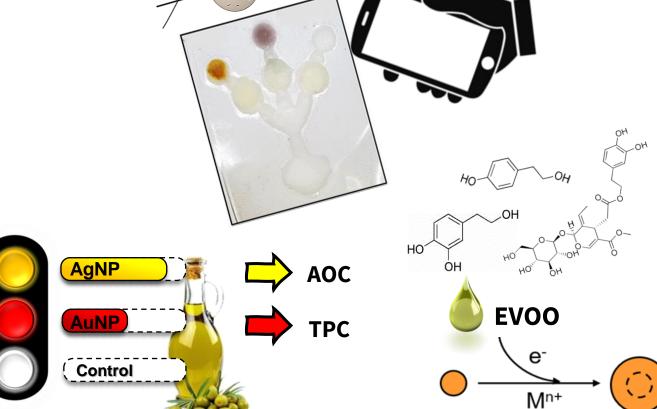
Annalisa Scroccarello <sup>a</sup>, Flavio Della Pelle <sup>a, \*</sup>, Daniel Rojas <sup>a</sup>, Giovanni Ferraro <sup>b</sup>, Emiliano Fratini <sup>b</sup>, Sara Gaggiotti <sup>c</sup>, Angelo Cichelli <sup>c</sup>, Dario Compagnone <sup>a, \*\*</sup>

#### **Office grade instruments**

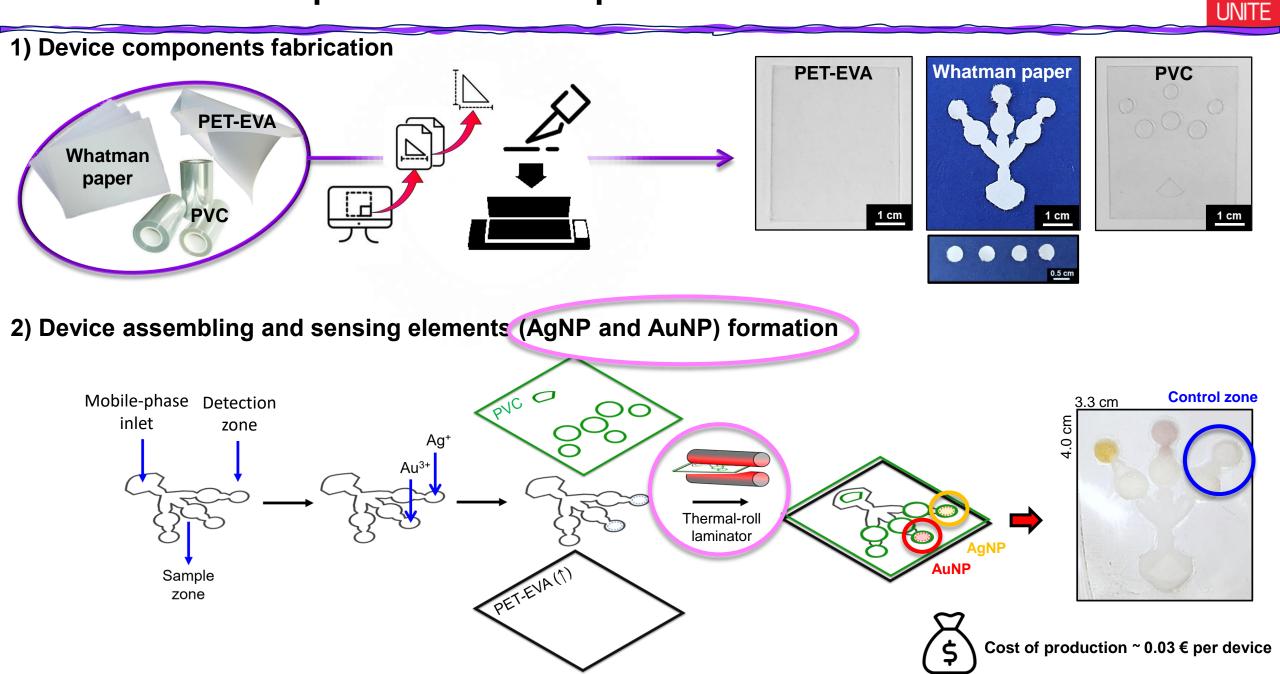


Thermal-roll laminator

# Lab-on-paper device



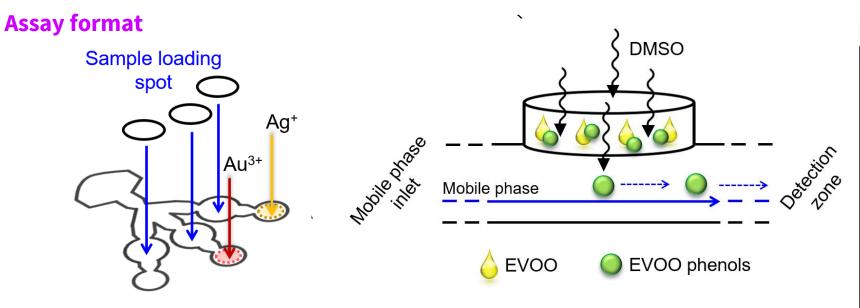
## MNPs as colorimetric probe: Device conceptualization and realization



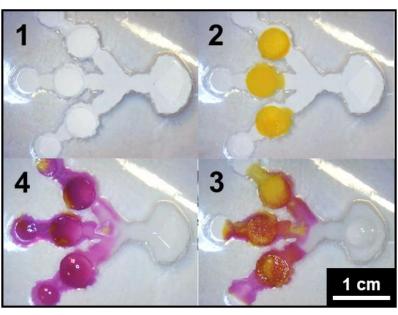
#### MNPs as colorimetric probe: Extraction-free phenolic compounds determination



Extraction-free olive oil phenolic compounds evaluation through a MNPs seed growth strategy

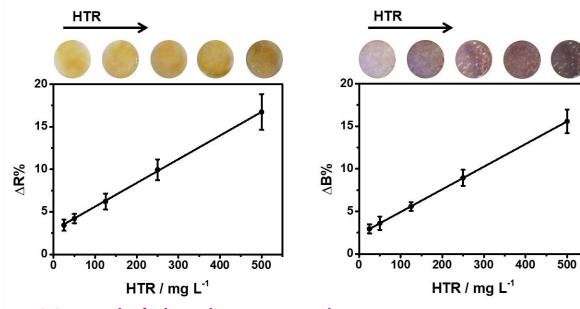


#### Assay simulation with a colorimetric dye

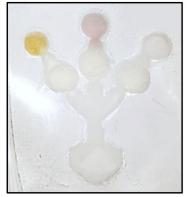


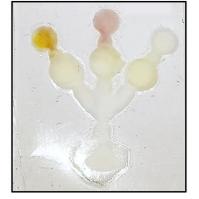
#### Extraction-free olive oil phenolic compounds evaluation trough a seed growth strategy

#### **Dose-response curve**



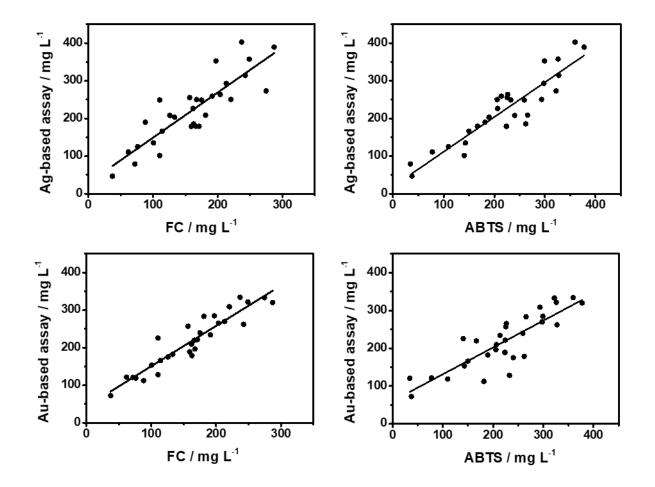
#### **EVOO samples' phenolyc compounds content**







#### Sample analysis, analytical performances



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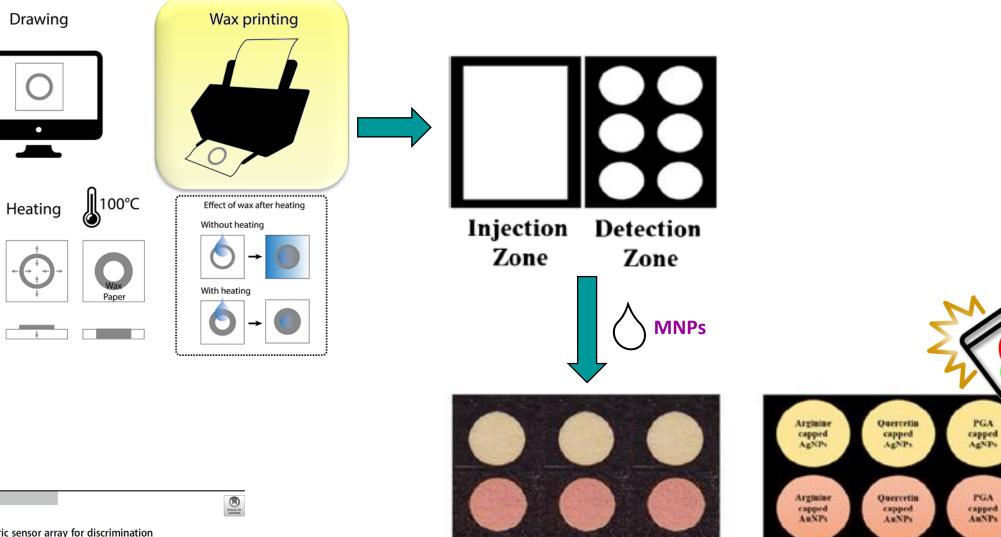
#### No interterferences by compounds commonly present in EVOO



Mean

High

Pesticides determination trough MNPs aggregation integrated in a paper-based device



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A paper-based colorimetric sensor array for discrimination and simultaneous determination of organophosphate and carbamate pesticides in tap water, apple juice, and rice

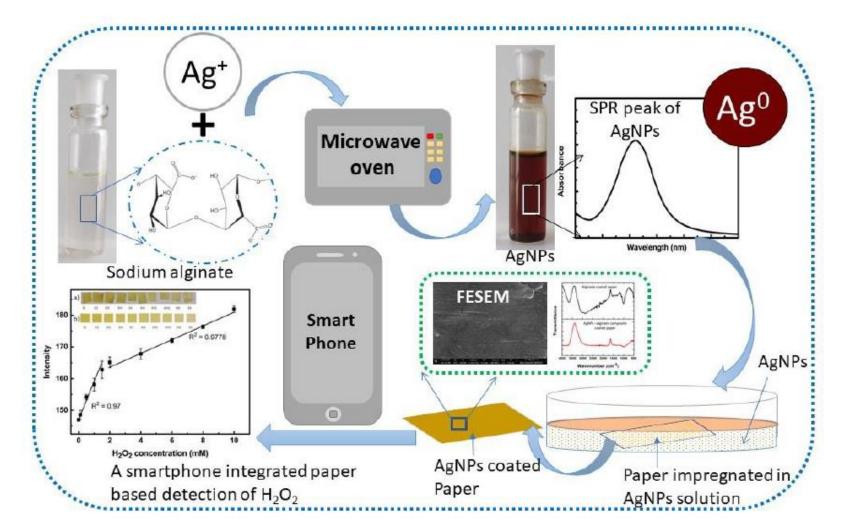
Top view

Side view

Microchimica Acta (2020) 187:621 https://doi.org/10.1007/s00604-020-04596->

**ORIGINAL PAPER** 

H<sub>2</sub>O<sub>2</sub> determination trough MNPs etching integrated in a paper-based substrate

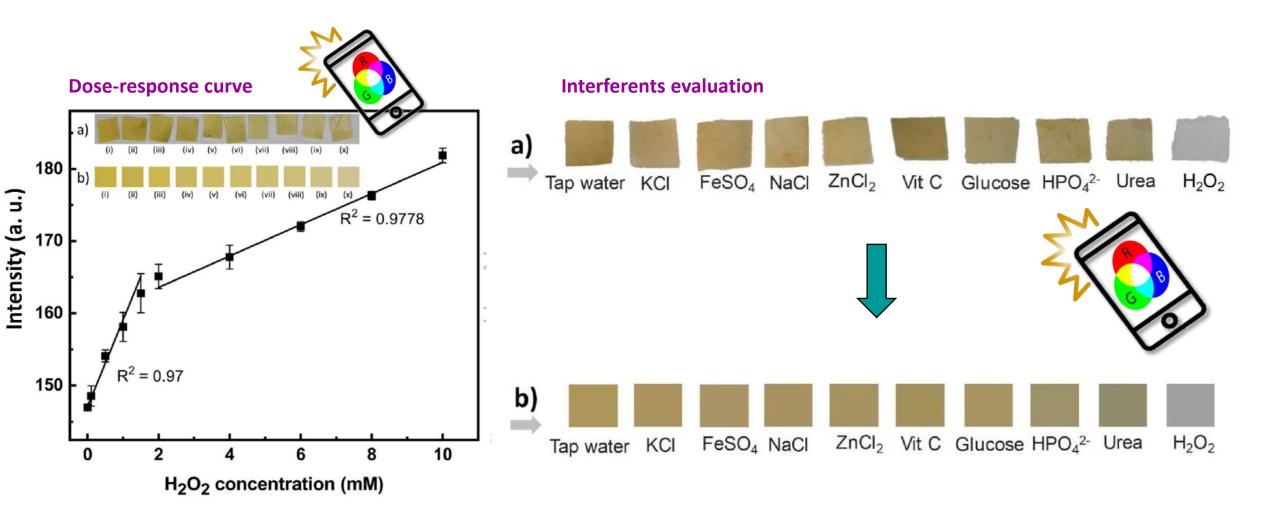


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**Development of a surface-modified paper-based** colorimetric sensor using synthesized Ag NPs-alginate composite

Lokesh Sharma, Shubhankar Gouraj, Pranit Raut & Chandrakant Tagad 🔽 🗓

H<sub>2</sub>O<sub>2</sub> determination trough MNPs etching integrated in a paper-based substrate



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#### Pesticides determination trough MNPs aggregation integrated in a paper-based device

#### **Analytes screening**







Carbaryl

Paraoxon



Parathion

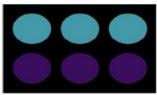
Chlorpyrifts



Malathion



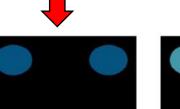
Carbaryl



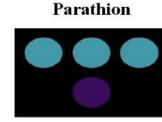
Malathion

Diazinon



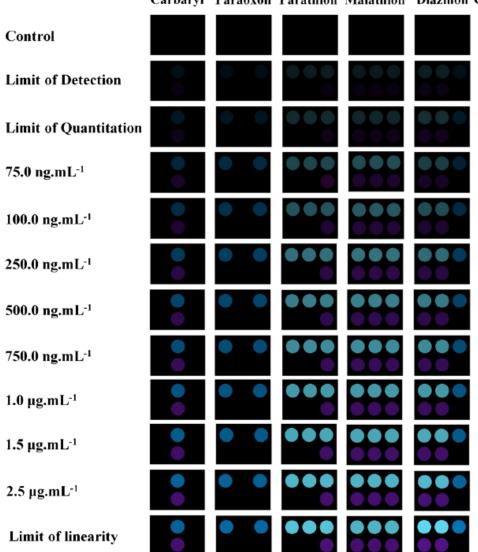


Paraoxon



Chlorpyrifos

#### **Dose-response curve and analytical parameters**



Carbaryl Paraoxon Parathion Malathion Diazinon Chlorpyrifos

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Della Pelle, F., Scroccarello, A., Sergi, M., Mascini, M., Del Carlo, M., & Compagnone, D. (2018). Simple and rapid silver nanoparticles based antioxidant capacity assays: Reactivity study for phenolic compounds. Food chemistry, 256, 342-349.

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