

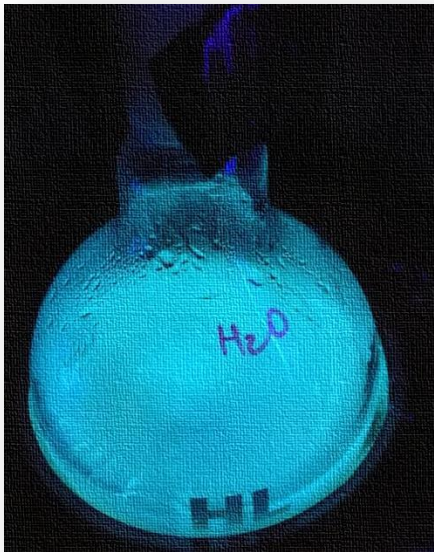


Departamento de Química

LAQAPAB laboratório

Adsorventes para análise química,
proteção de ambiente e biomedicina

Carbon nanoparticles: Synthesis, Characterization and Application Prospects

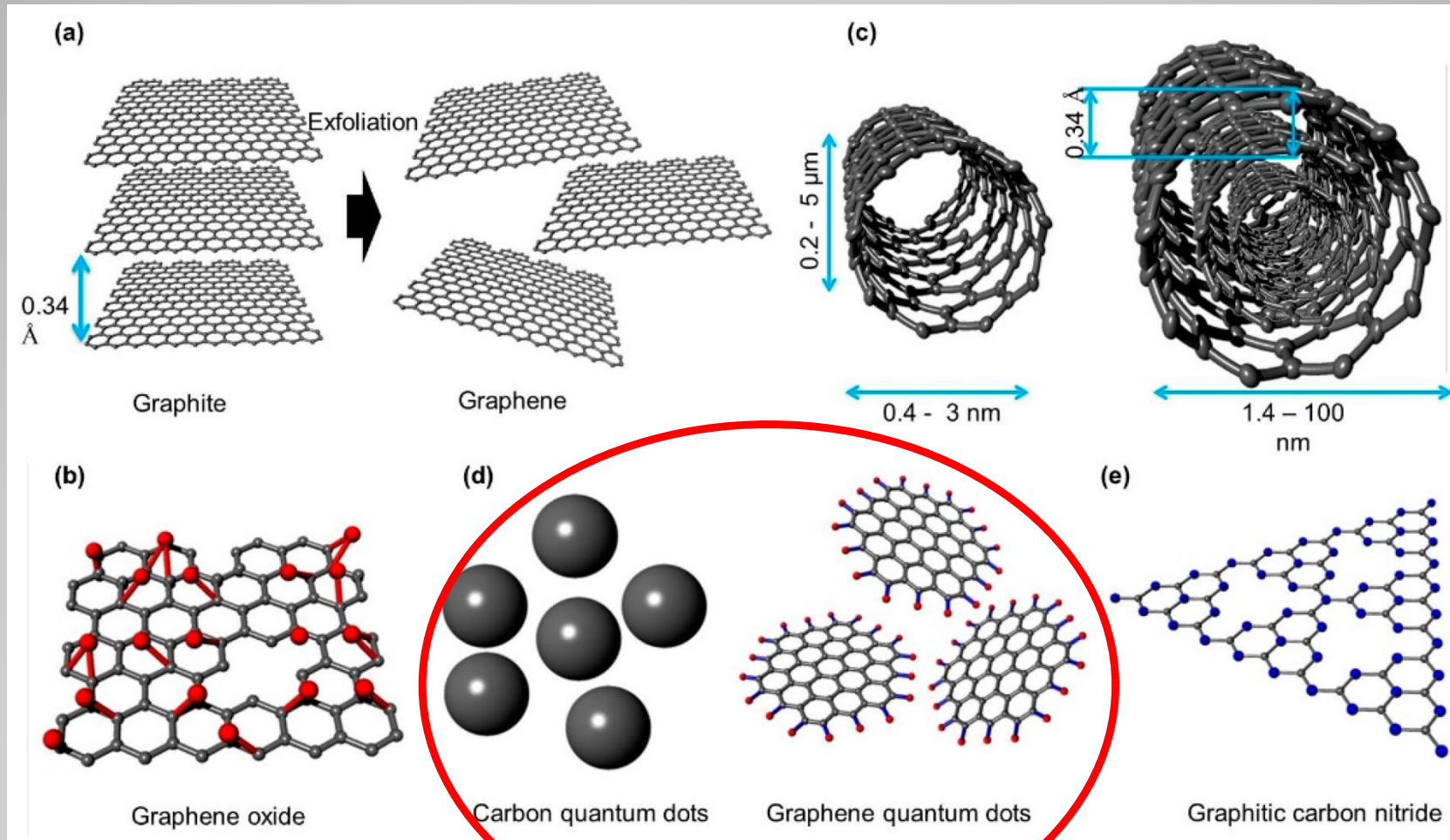


Post-Doc: Albina Mikhraliieva (DSc, Brazil)
Supervisor: prof. Volodymyr Zaitsev

Ukraine, 2021



What are Carbon Dots?

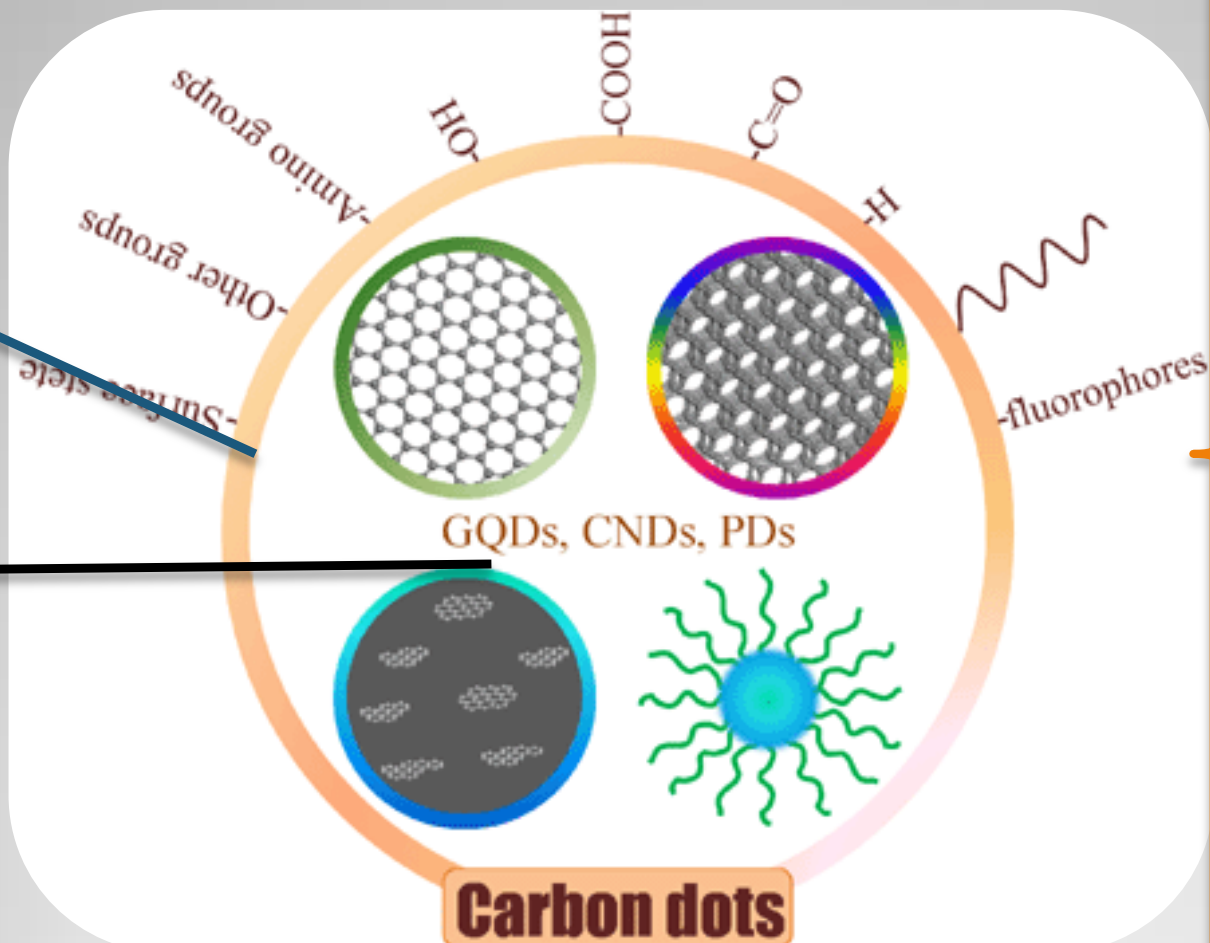


What are Carbon Dots?



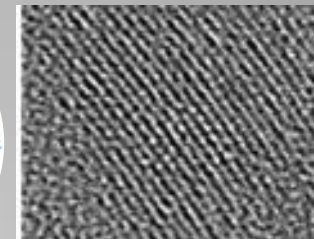
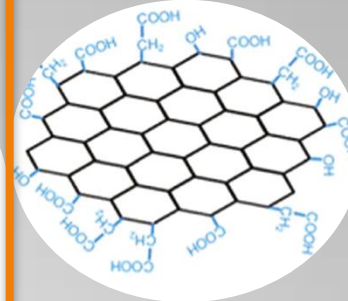
Surface state

Carbon core

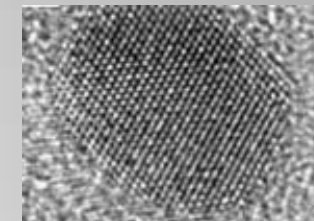
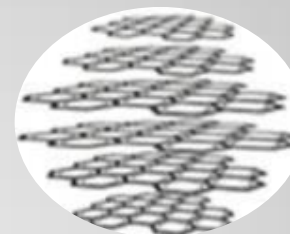


Average size <10 nm

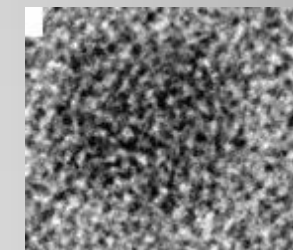
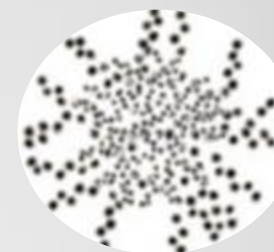
Graphene Quantum Dots



Carbon Quantum Dots



Carbon Nanodots

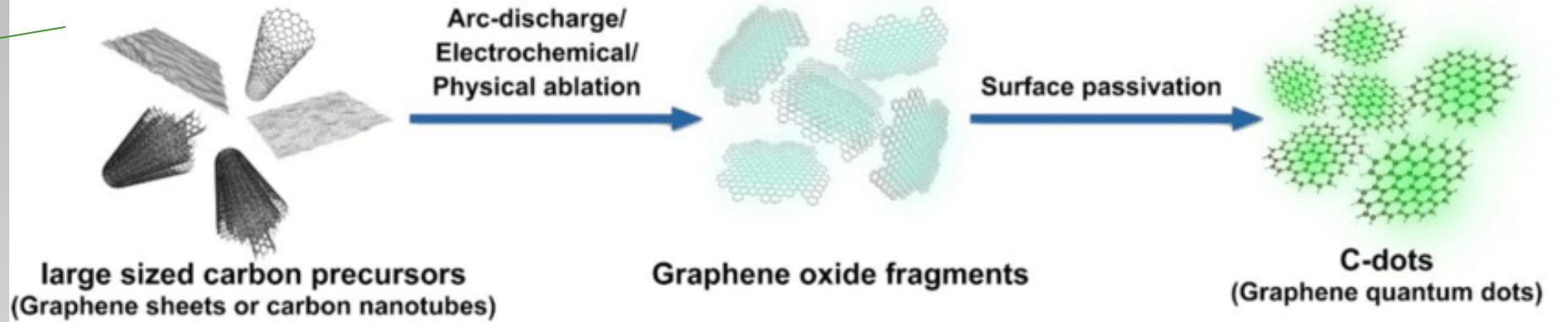




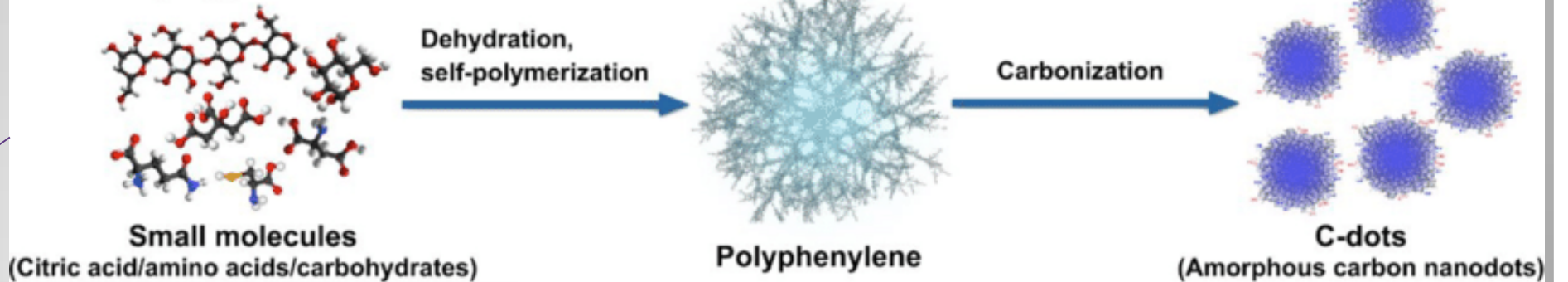
Synthesis

Breaking down larger carbon structures

Top-down approaches



Bottom-up approaches

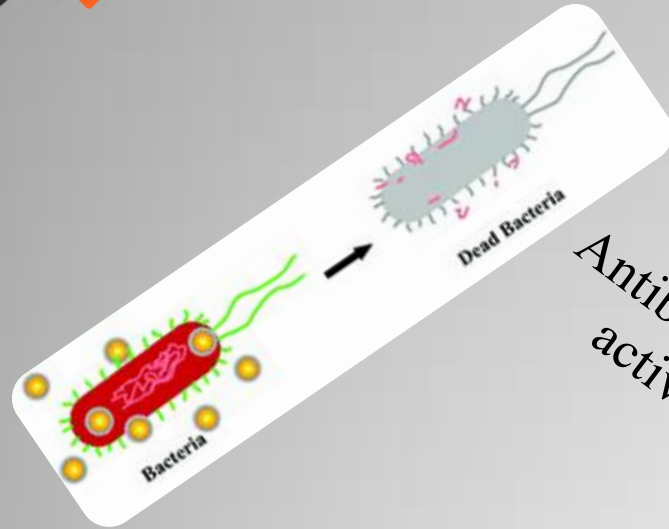


- Pyrolysis
- Ultrasonic and microwave synthesis
- Support approach
- Hydrothermal/solvothermal
- Passivation agents
- Biomass



Application

modification
photoluminescence
stability
biocompatibility

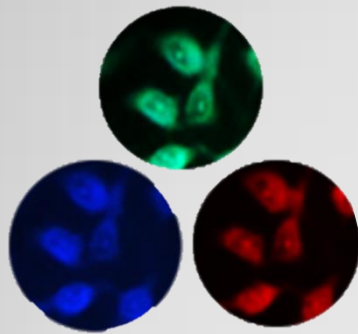


Antibacterial activity



CDs

Catalysis



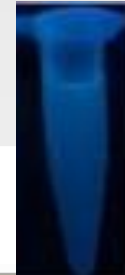
Bioimaging

Optoelectronic

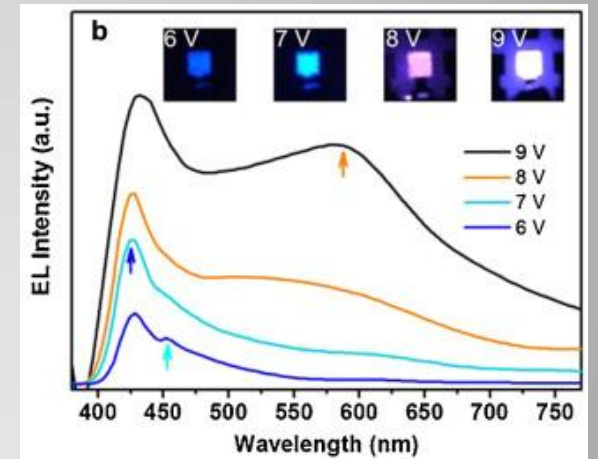
Sensor



Cu^{2+}



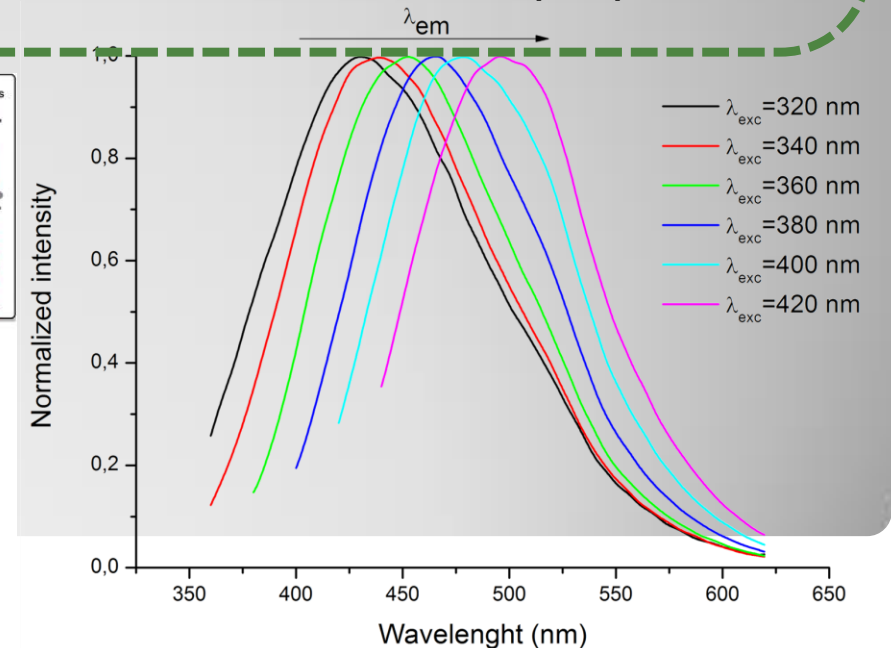
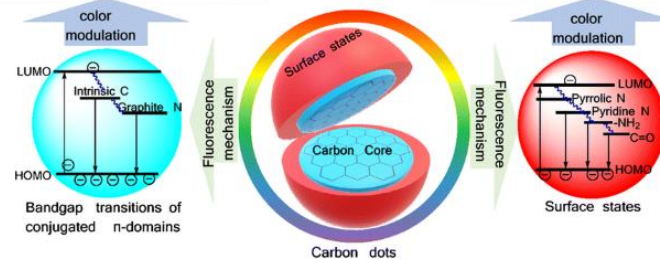
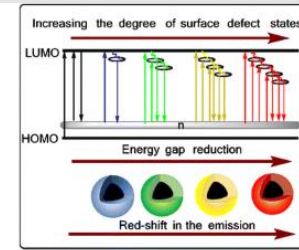
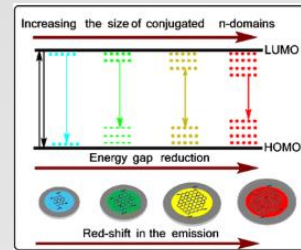
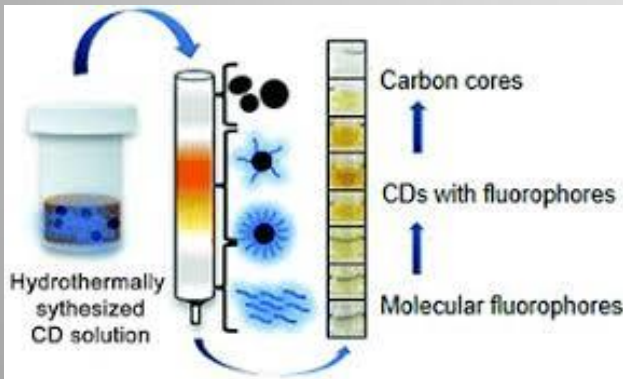
quenching



? Problems

Control of the morphology and size distribution of CDs are the principal challenges.

- Structure, composition and photo-physics of CDs are not fully understood.
- Carbon source and synthesis conditions are crucial criteria of fabrication
- Unclear mechanism of CDs synthesis
- Aggregation of the pyrolytic products, most of the bottom-up synthesis results in the mixture of CDs with different particle sizes and surface properties and thus require chromatographic separation
- Tedious purification of C-dots
- **Excitation-dependent** behavior → the broad size distribution of the prepared particles.

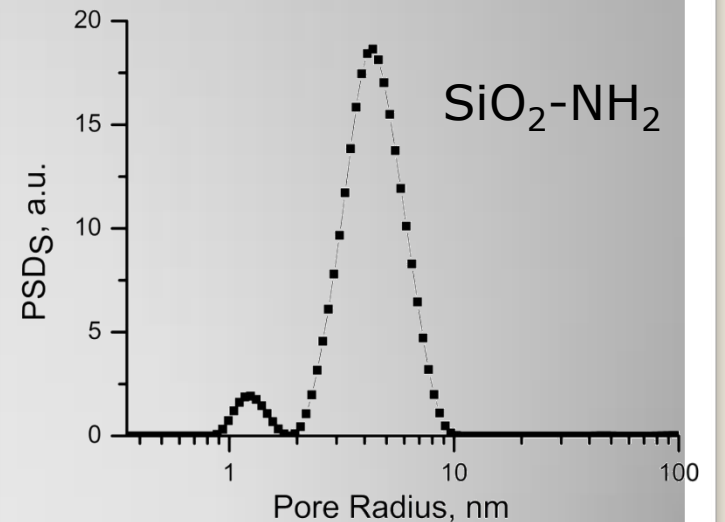
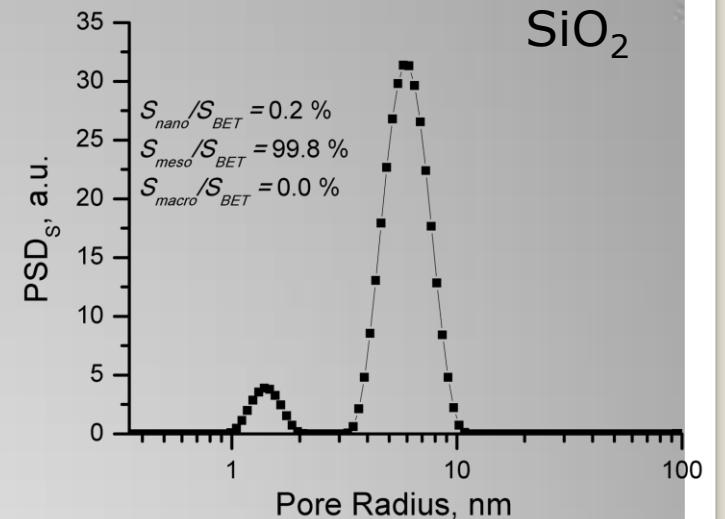


Solution



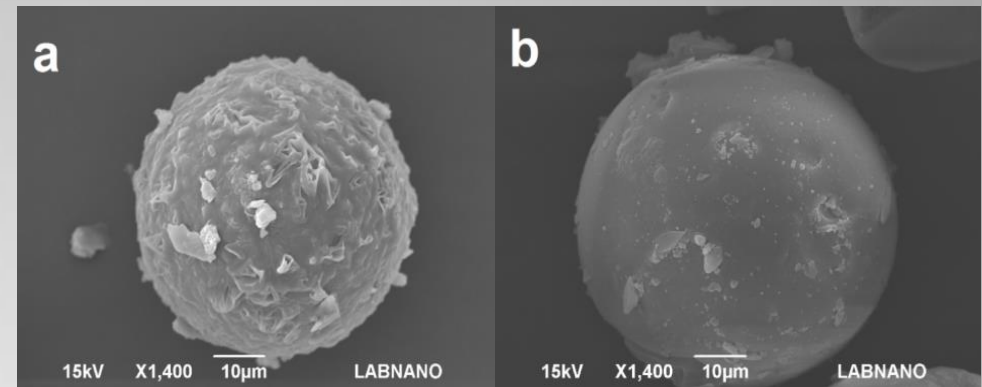
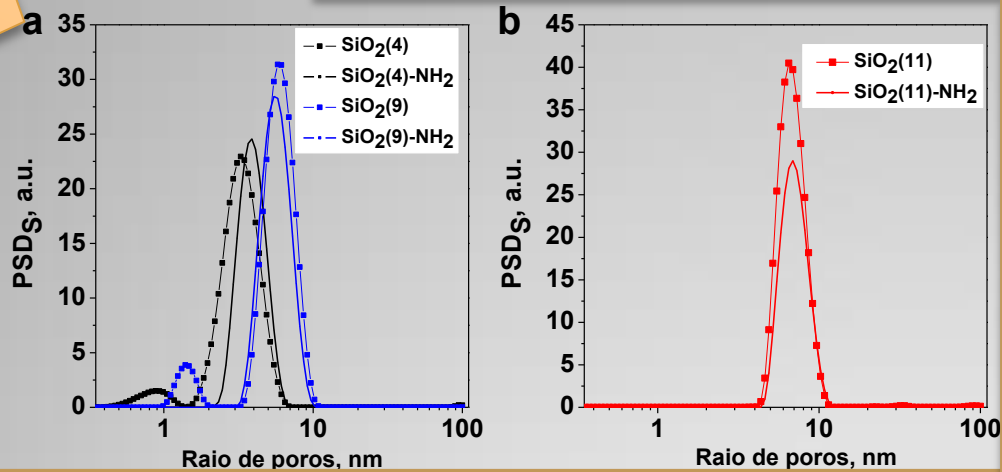
Combination of a traditional preparation method of CDs with pyrolysis of citric acid in a size-limiting reactor was used to overcome these difficulties and obtain a homogeneous

- Nanoreactor is the porous material with high thermal resistance
- Nanoporous silica gel is a suitable material due to monomodal pore size distribution varying in the range from 2 to 15 nm and has high thermal and chemical stability

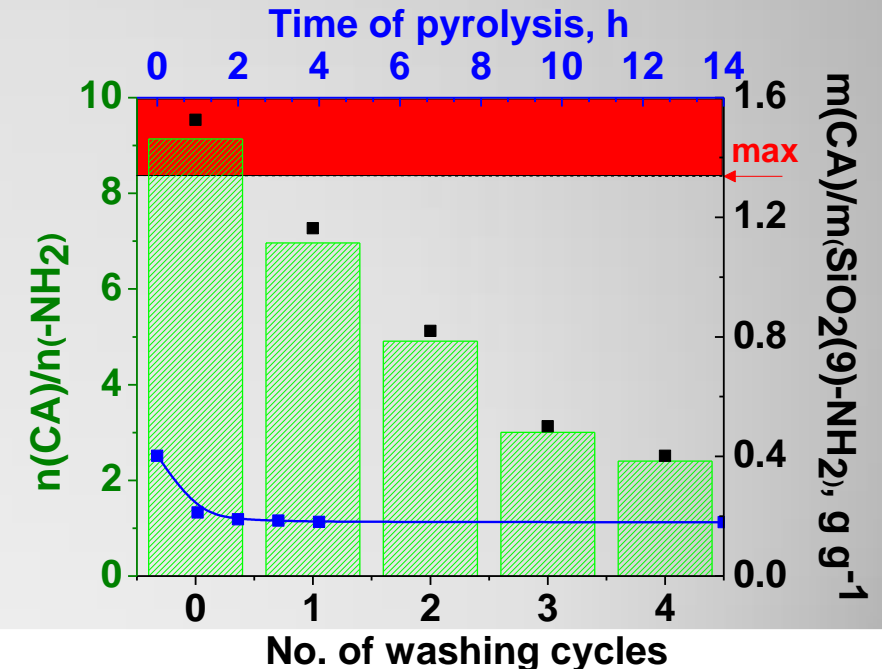


SiO₂
SiO₂-NH₂

Solid support



Silicas	S _{BET} (m ² g ⁻¹)	S _{meso} /S _{BET} (%)	Volume pore (cm ³ g ⁻¹)	Concentration of immobilized groups* (mmol g ⁻¹)	Maximum loading of CA on SiO ₂ -NH ₂ , (g g ⁻¹)
SiO ₂ (4)	360.3	94.9	0.72	0	1.12
SiO ₂ (4)-NH ₂	278.0	100	0.47	0.8±0.2	0.73
SiO ₂ (6)	—[b]	—	0.80	0	1.24
SiO ₂ (6)-NH ₂	278.6	100	0.59	0.8±0.1	0.91
SiO ₂ (9)	387.4	99.8	1.01	0	1.57
SiO ₂ (9)-NH ₂	325.8	99.9	0.86	0.9±0.1	1.35
SiO ₂ (11)	400.2	99.6	1.25	0	1.94
SiO ₂ (11)-NH ₂	290.0	99.4	0.95	0.7±0.2	1.47

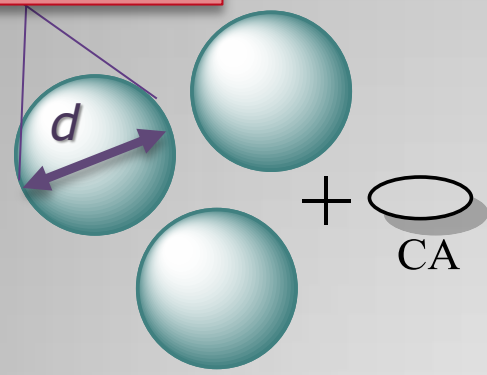




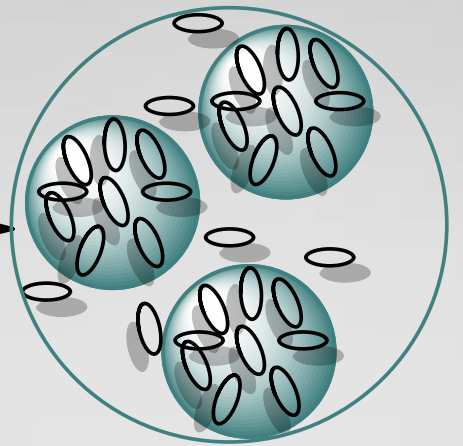
Realization

SiO₂
SiO₂-NH₂

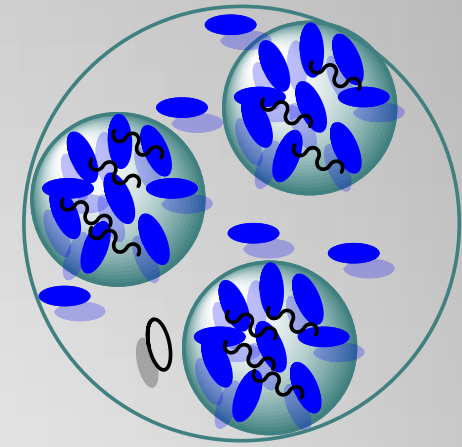
4, 6, 9, 11 nm



Impregnation

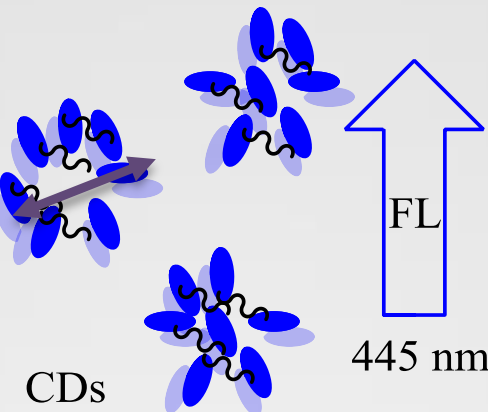
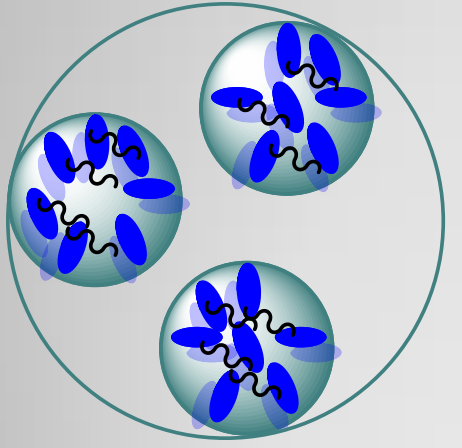


170 °C
N₂

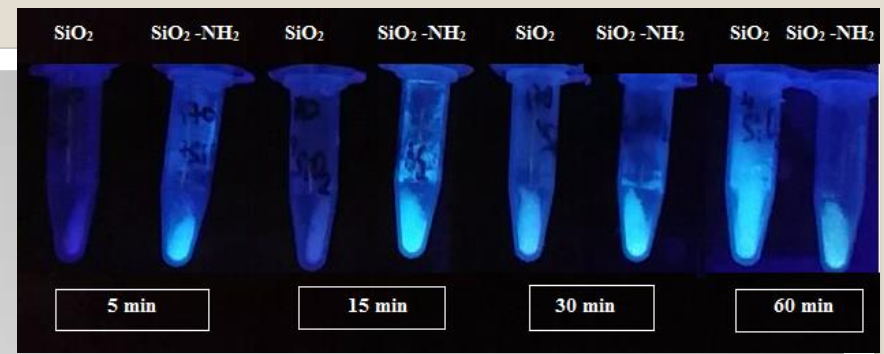


Lavagem

Elimination of silica support

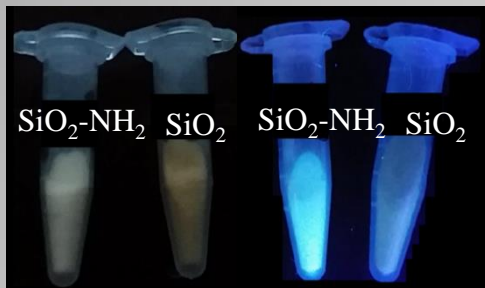


- = silica pore
- = CA, citric acid
- = Silica@CDs (composite)





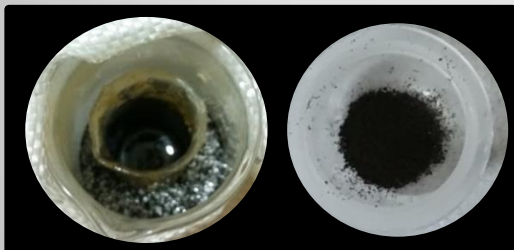
Composite



With silica gel

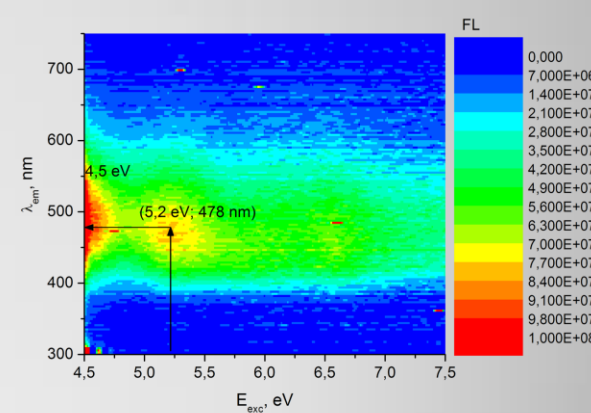
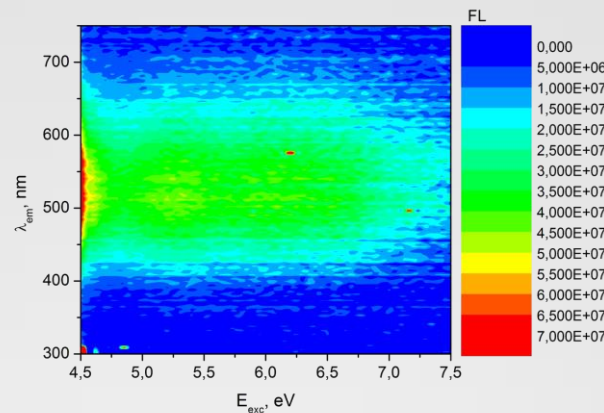
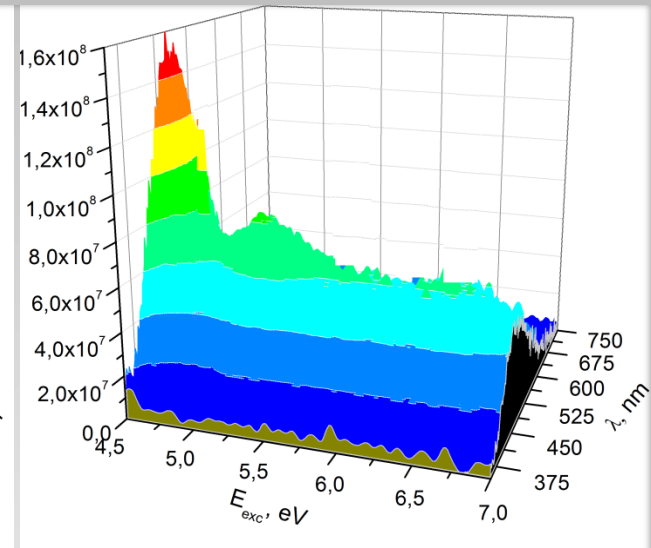
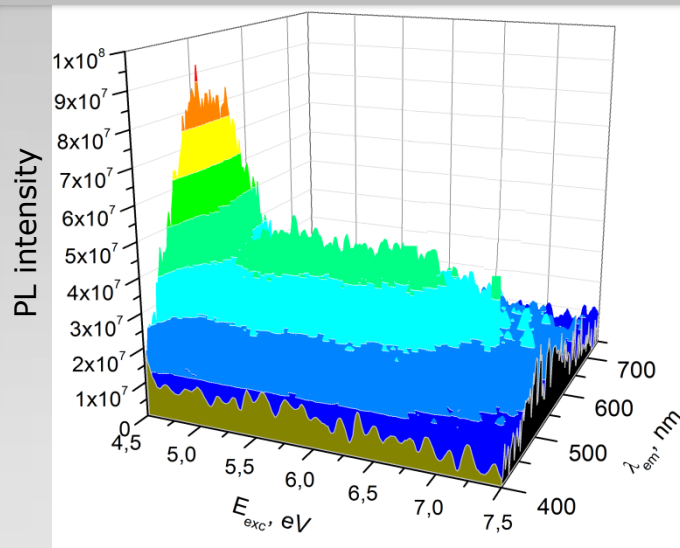
170°C, 180 min

Without silica gel



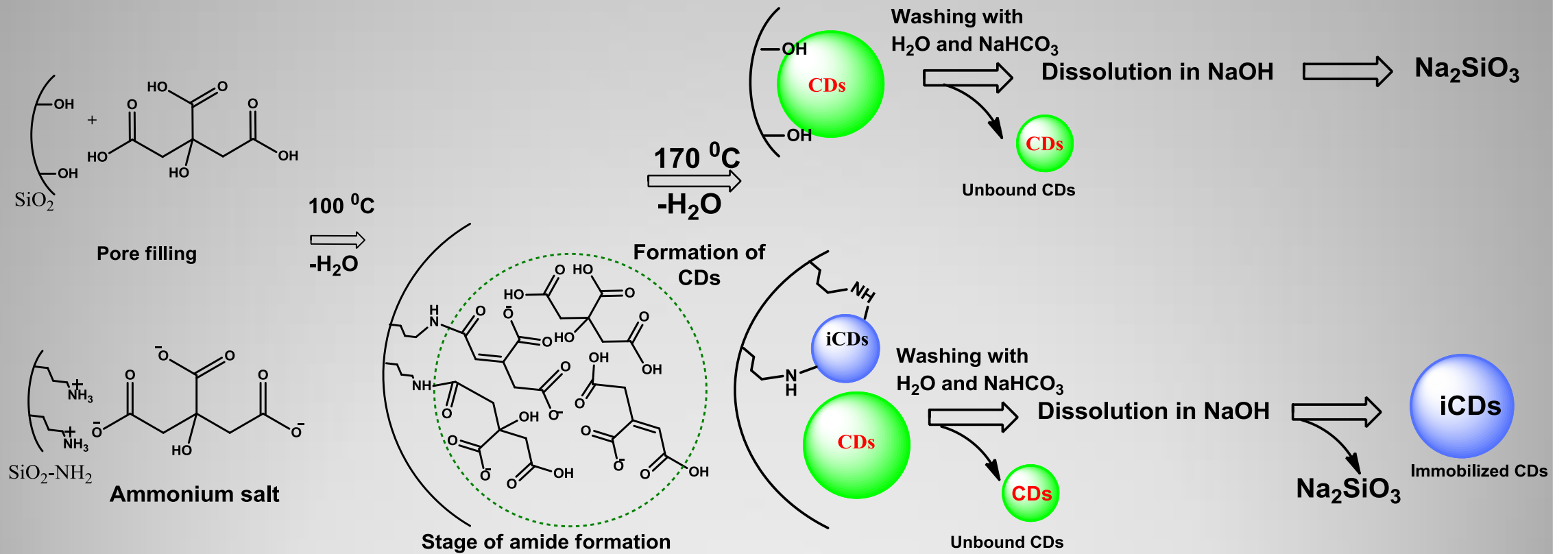
SiO₂@CDs – 180 min

SiO₂-NH₂@CDs – 180 min





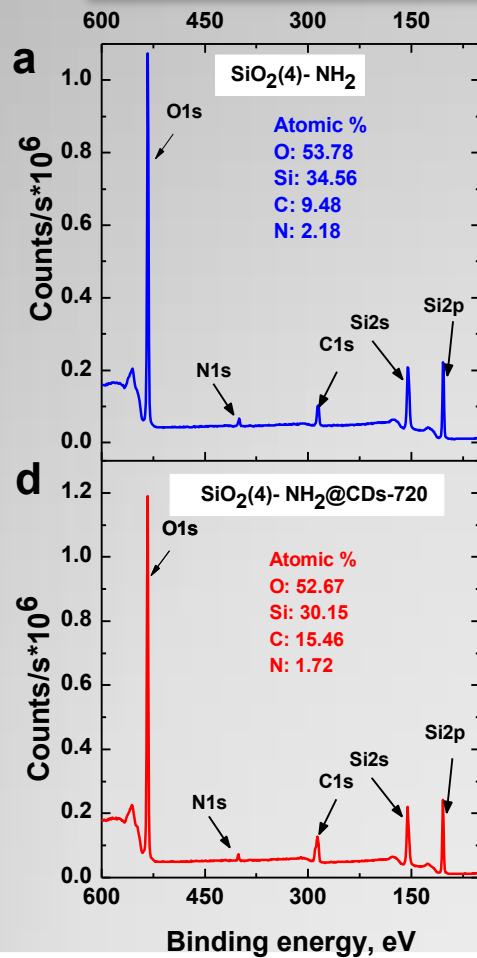
Bottom-up approach



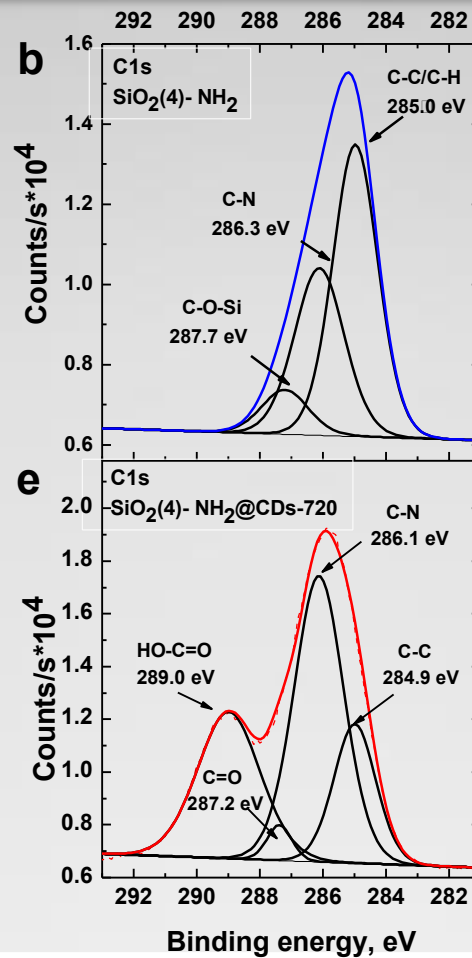
Composite characterization. XPS



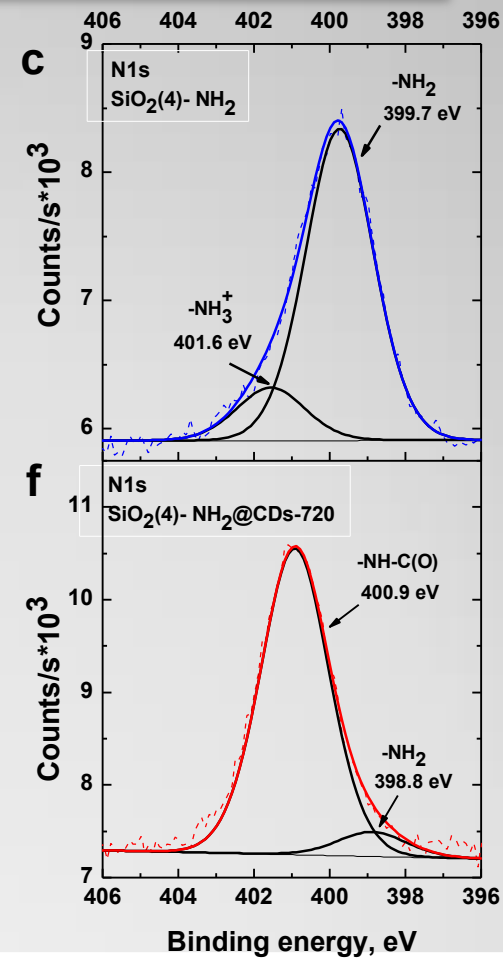
Survey spectra



C1s spectra



N1s spectra



**Silica-based@
CDs**

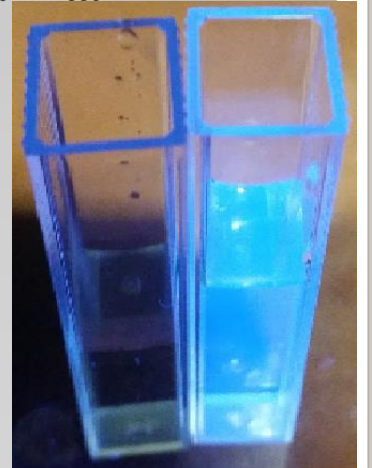
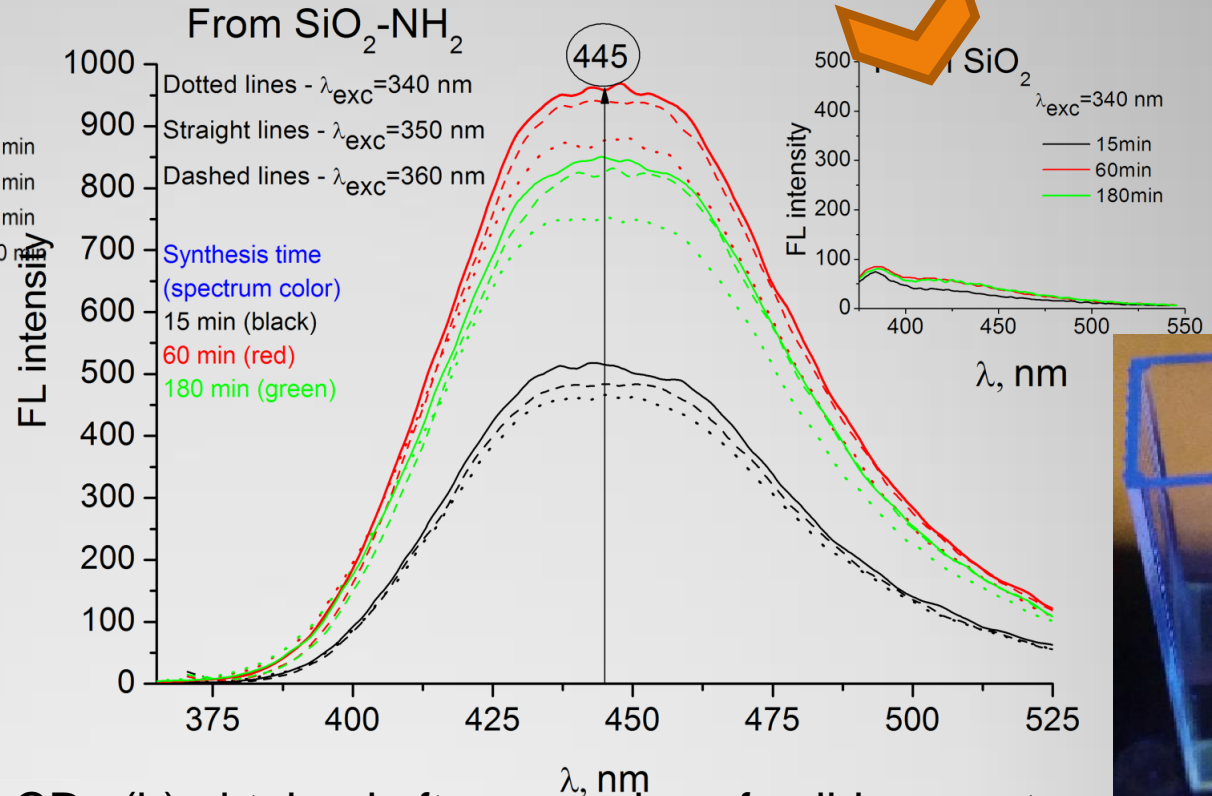
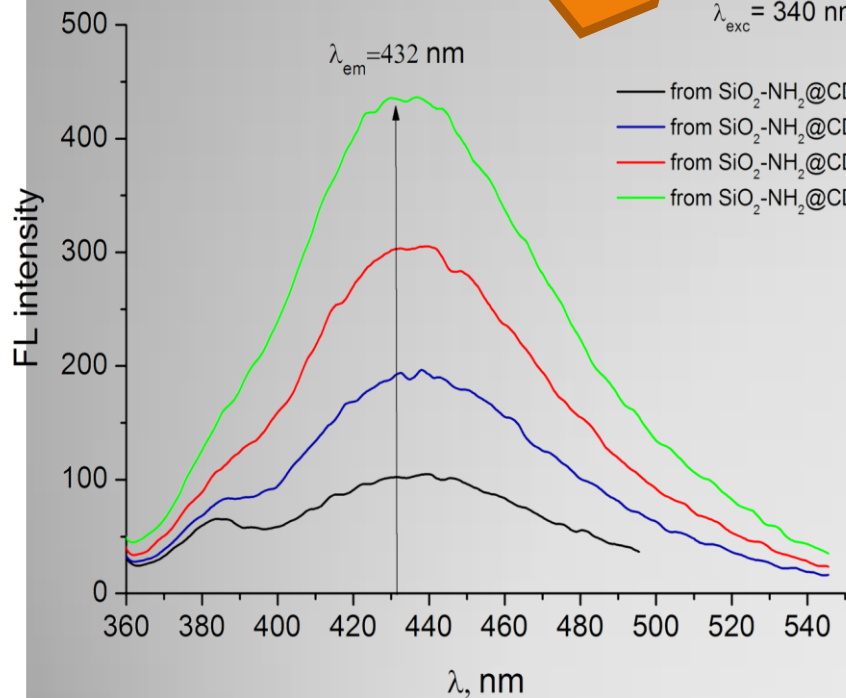


Washing – SiO₂-NH₂@CDs

Agua

NaHCO₃

NaOH

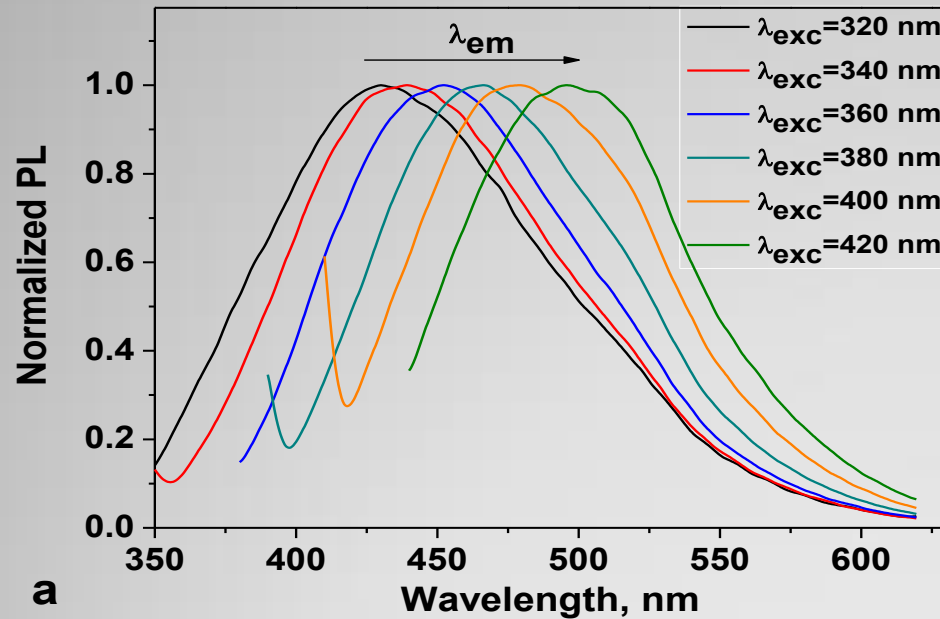


SiO₂ SiO₂-NH₂

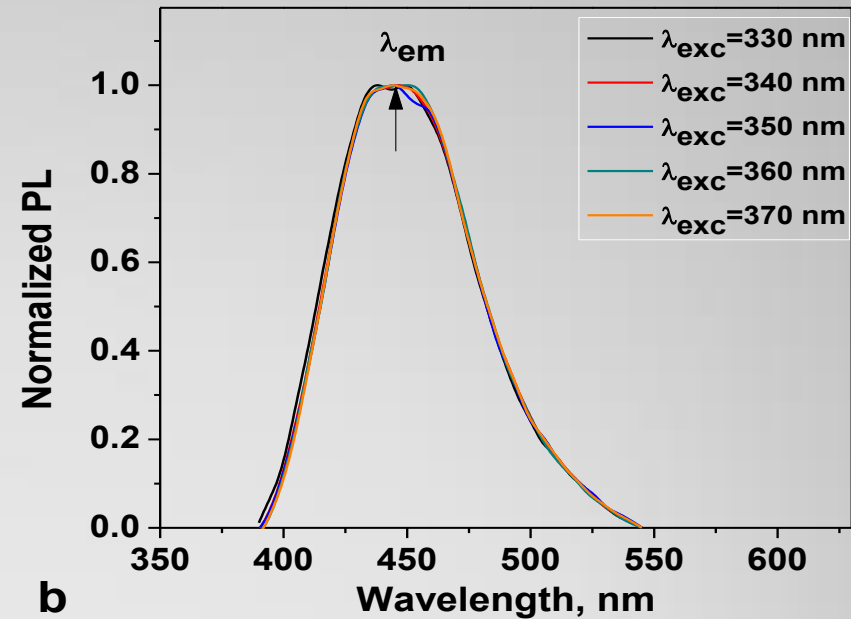
PL spectra of water extracted CDs(a) and CDs (b) obtained after removing of solid support in SiO₂(11)-NH₂@CDs and SiO₂(11)@CDs (inset). The excitation wavelength of 340 nm



Photoluminescence



Excitation-dependent

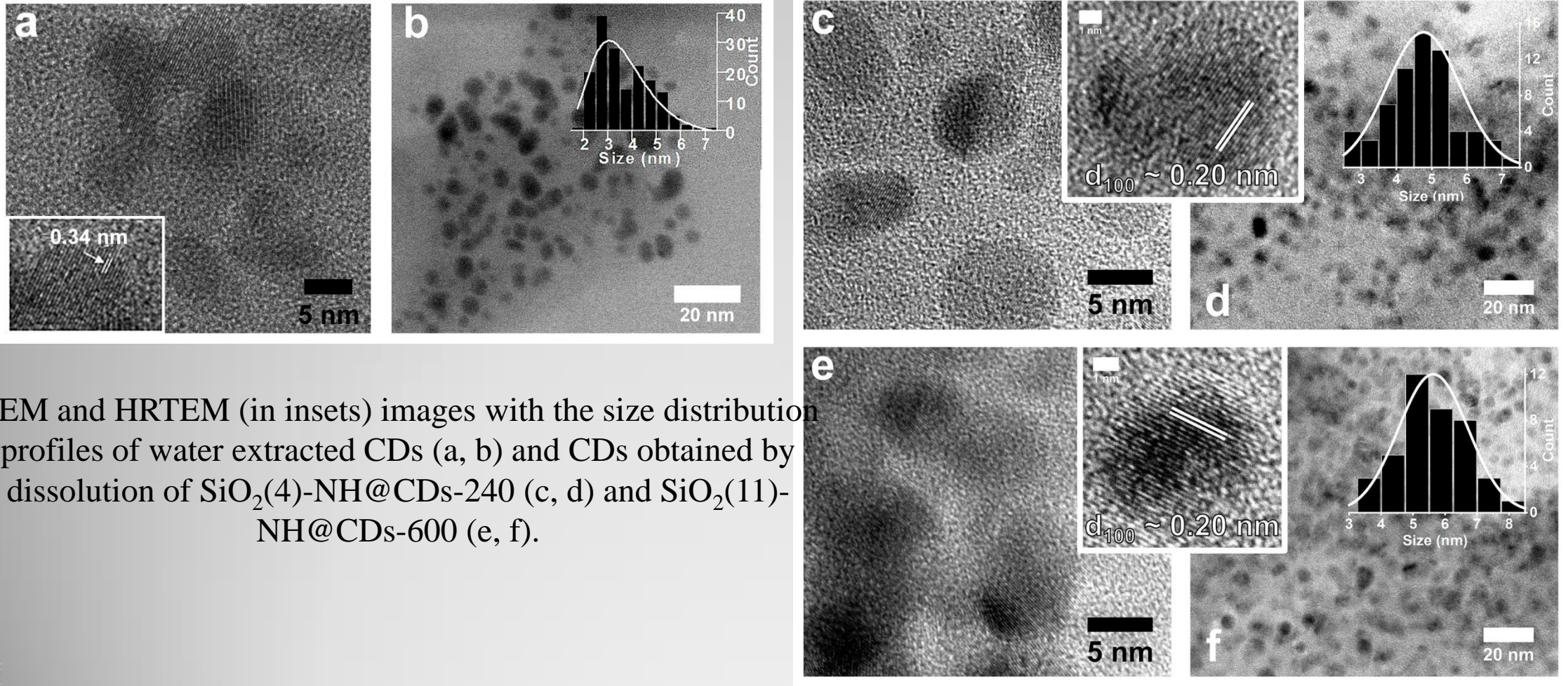


Excitation-independent

Normalized PL spectra of non-fixed CDs from SiO₂(11)@CDs-480 (a) and CDs after removing SiO₂(4)-NH₂@CDs-240 (b)



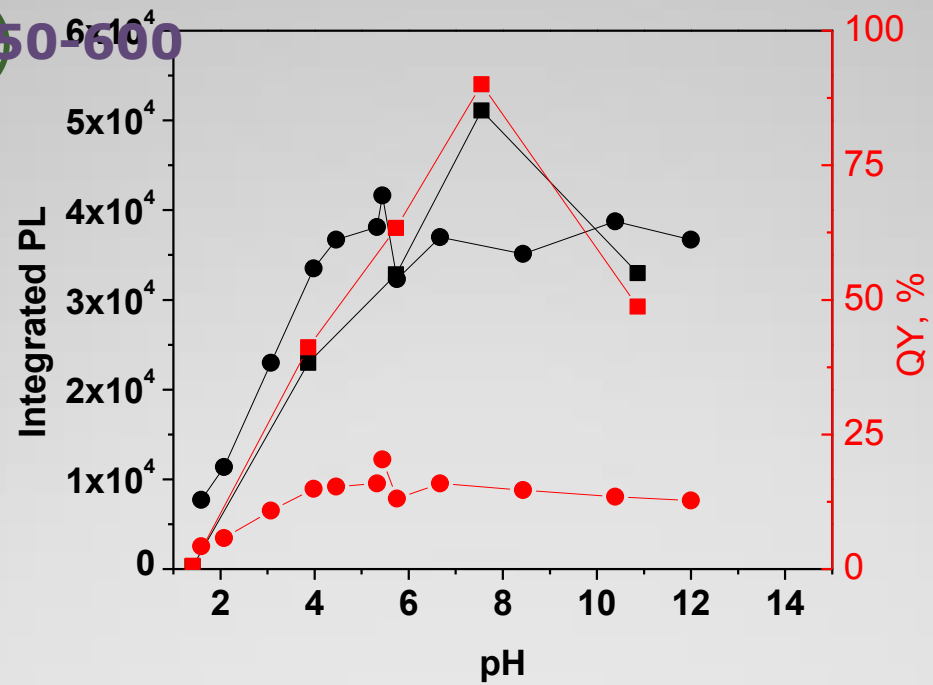
Particle size. TEM



TEM and HRTEM (in insets) images with the size distribution profiles of water extracted CDs (a, b) and CDs obtained by dissolution of SiO₂(4)-NH@CDs-240 (c, d) and SiO₂(11)-NH@CDs-600 (e, f).

Quantum yield

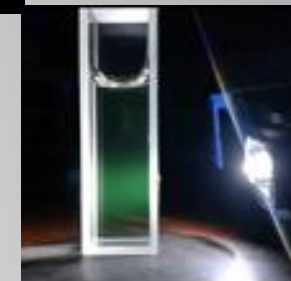
SiO₂(11)-EDA@CDs-150-600



Samples	QY, %
SiO ₂ (11)-EDA@CDs-150-600	90
SiO ₂ (11)-EDA@CDs-180-600	18
SiO ₂ (6)-NH ₂ @CDs - 60	6.28
SiO ₂ (6)-NH ₂ @CDs - 180	3.61



Daylight



White flashlight



365 nm UV-lamp

SiO₂(11)-EDA@CDs-180-600



Realization

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Excitation-Independent Blue-Emitting Carbon Dots from Mesoporous Aminosilica Nanoreactor for Bioanalytical Application

Albina Mikhraliieva, Vladimir Zaitsev*, Yutao Xing, Horácio Coelho-Júnior, and Rubem Luis Sommer

Cite this: *ACS Appl. Nano Mater.* 2020, 3, 4, 3652–3664

Publication Date: March 16, 2020

<https://doi.org/10.1021/acsanm.0c00363>

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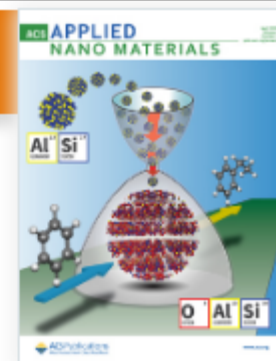
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Benefit of porous silica nanoreactor in preparation of fluorescence carbon dots from citric acid

Albina Mikhraliieva¹ , Vladimir Zaitsev¹ , Ricardo Q Aucélio¹, Henrique B da Motta¹ and Michael Nazarkovsky¹

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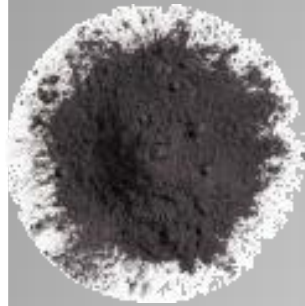
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Top-down

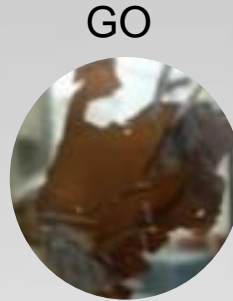


Preparation of SiO₂-GOQDs



Graphite

1. H₂SO₄/H₃PO₄ (180 ml:20 ml)
2. KMnO₄ (9 g)
3. 50 °C, 12 h
4. H₂O₂ (30%, 20 ml)
5. Washing with H₂O, HCl, EtOH
6. Vacuum-dry



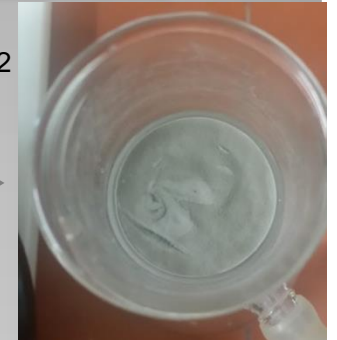
GO

Ultrason. 2h



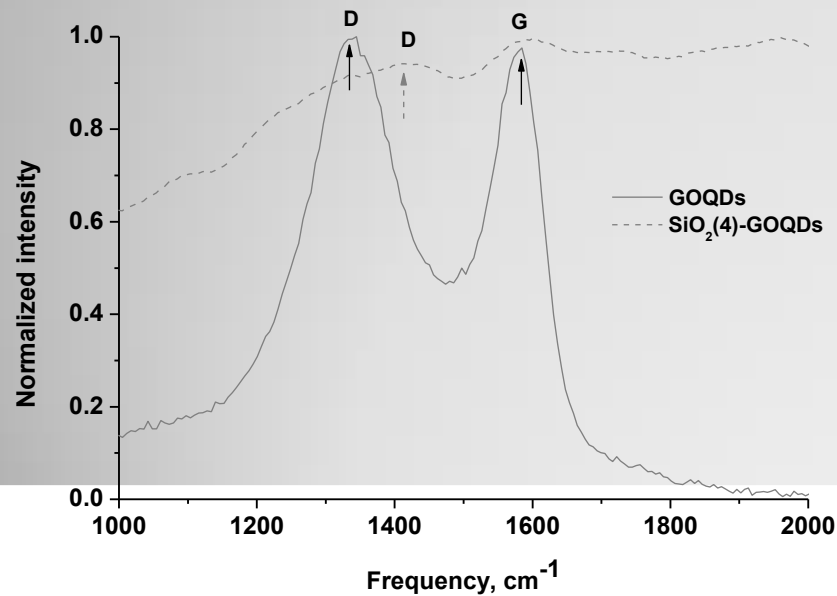
GOQDs

SiO₂(4)-NH₂
DCC
DMF

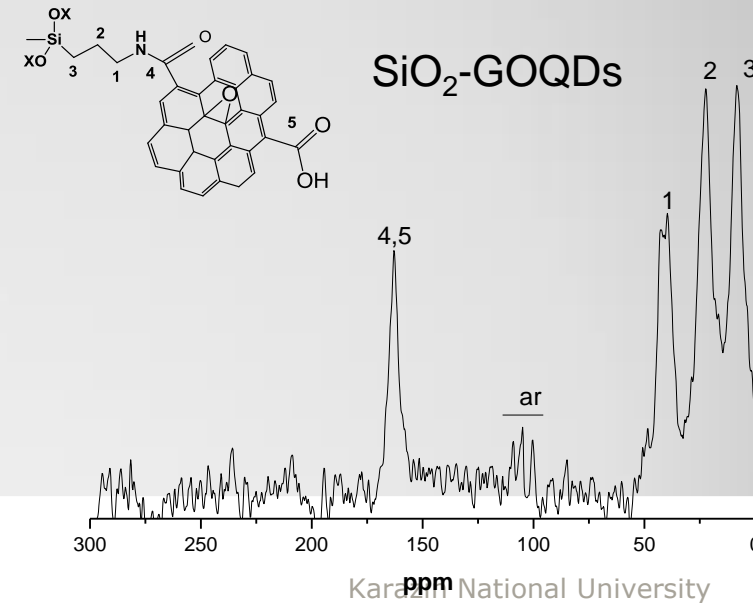


SiO₂-GOQDs

Raman spectra

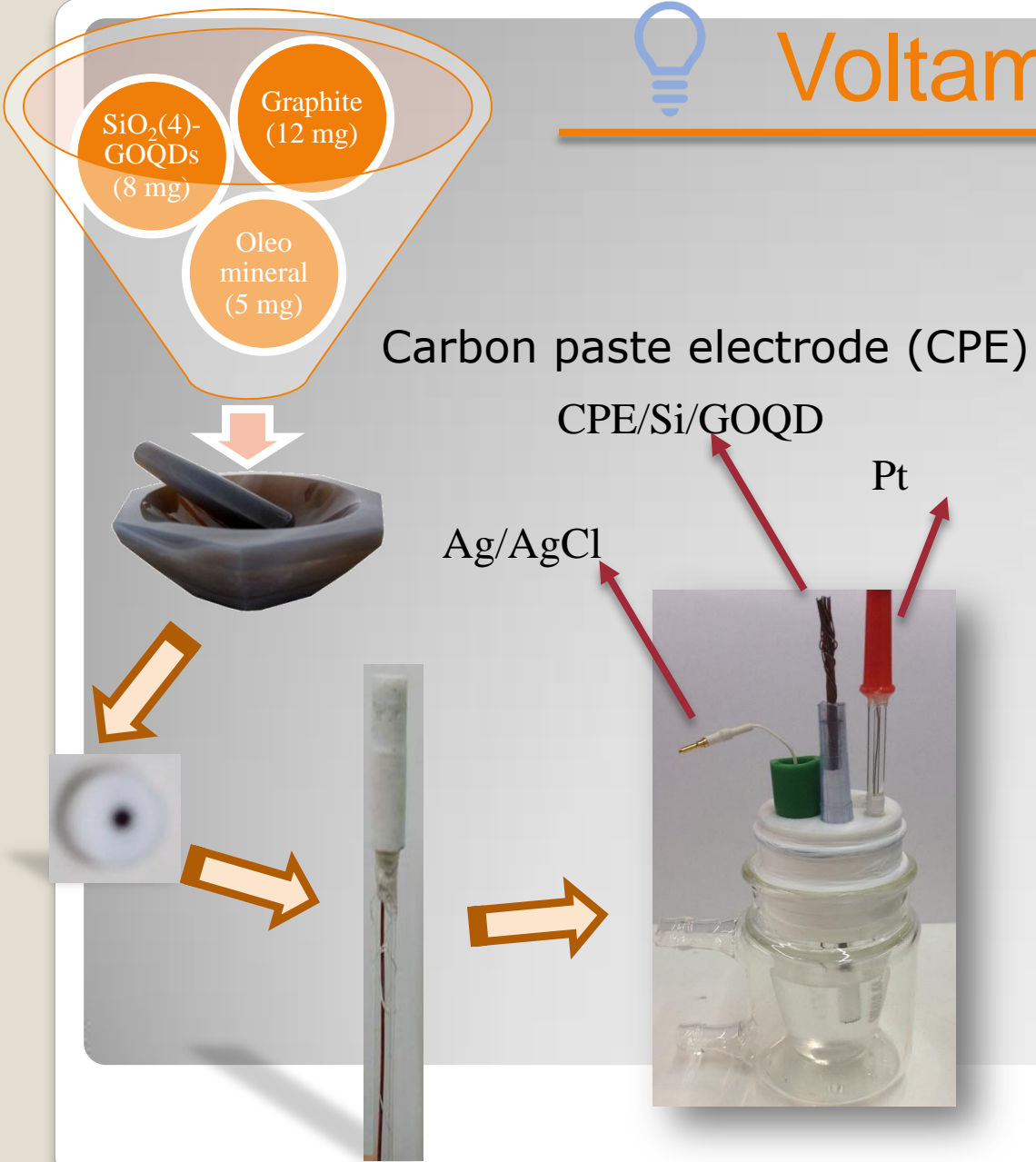


¹³C CP/MAS RMN, solid state



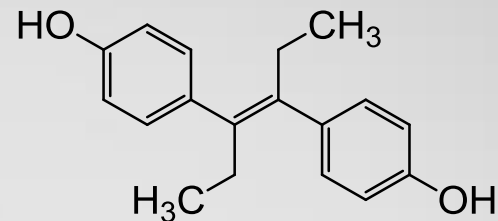


Voltammetry - SiO₂-GOQDs

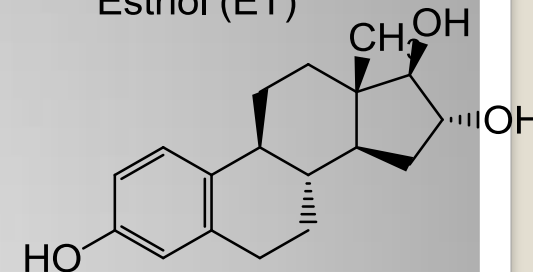


Analytes

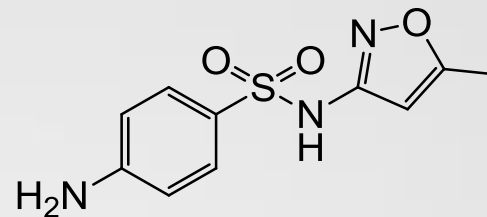
Dietilestilbestrol (DES)



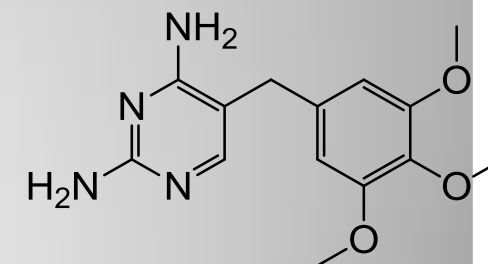
Estriol (ET)



Sulfametoxazol (SMZ)



Trimetoprim (TMP)

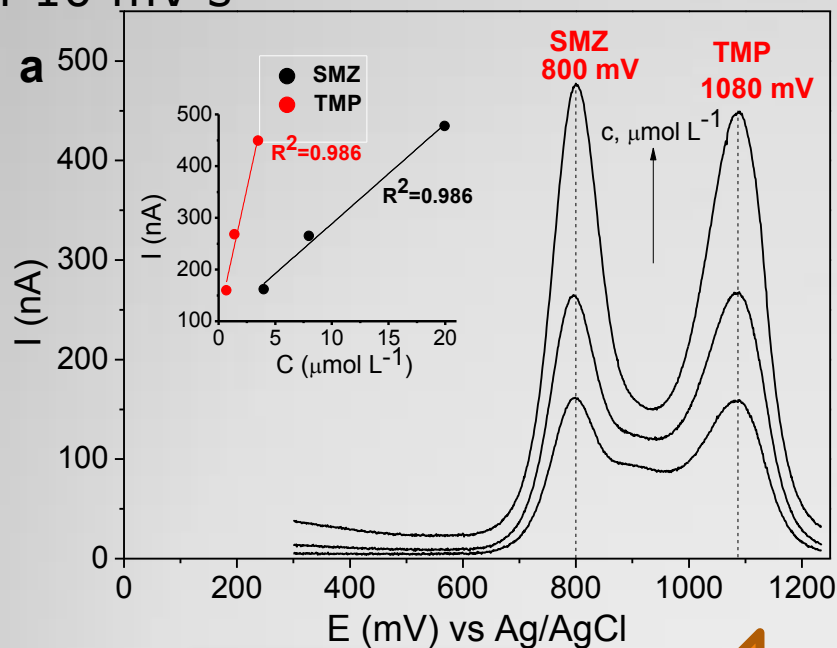




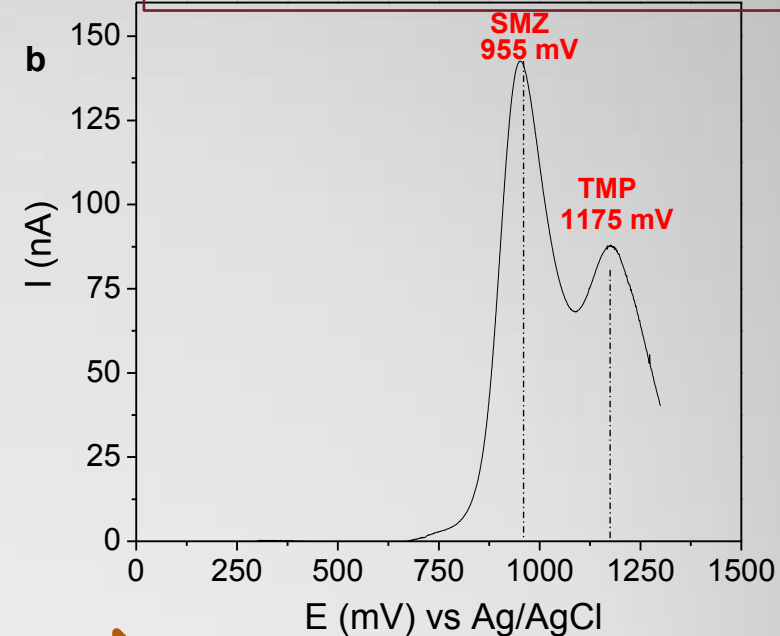
Detection of antibiotics

Differential pulse voltammograms (DPV)

pulse amplitude of 50 mV,
a pulse time of 50 ms,
a step potential of 1 mV,
a scan rate of 10 mV s⁻¹



SiO₂-GOQDs



Graphite

4.0, 8.0 e 20 $\mu\text{mol L}^{-1}$ de SMZ

4.0 $\mu\text{mol L}^{-1}$ de SMZ

Analytos: $c(\text{SMZ}):c(\text{TMP})=5:1$



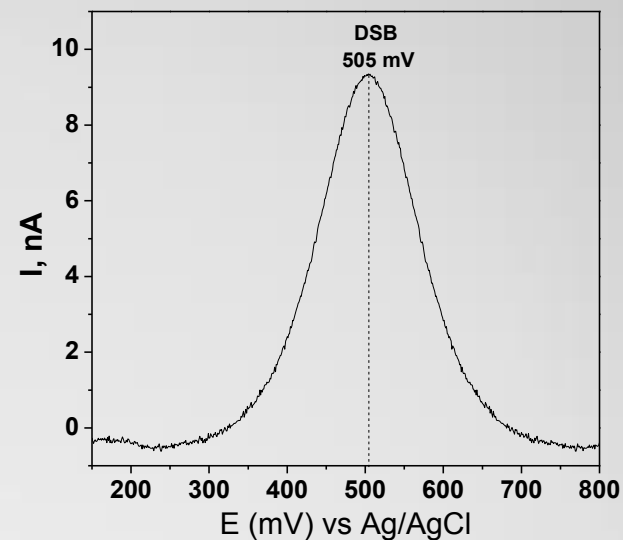
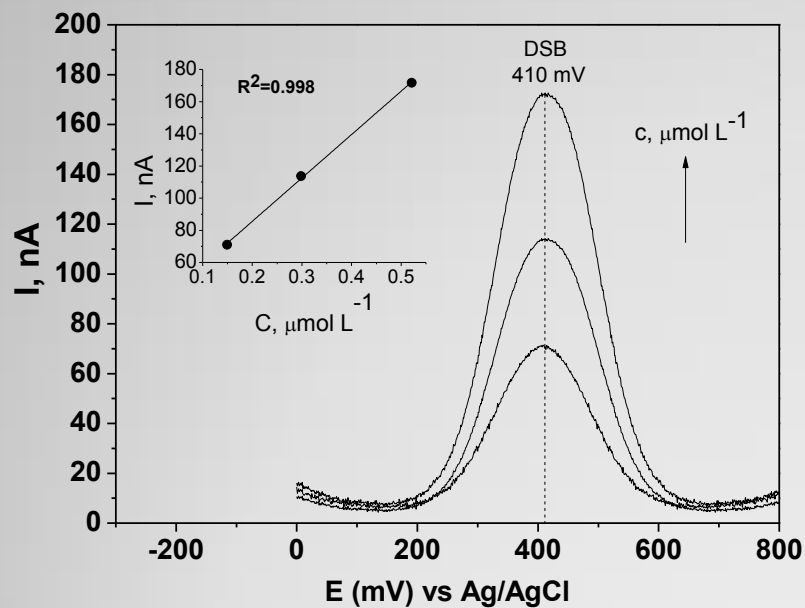
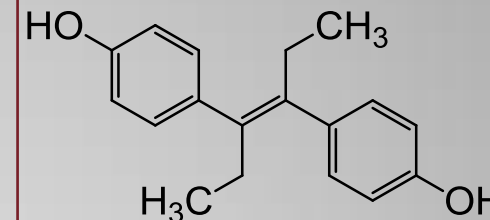
Detection of hormones

Differential pulse voltammograms (DPV)

pulse amplitude of 50 mV,
a pulse time of 50 ms,
a step potential of 1 mV,
a scan rate of 10 mV s⁻¹

Dietilestilbestrol (DES)

Analito:



SiO₂-GOQDs



Graphite

0.15, 0.30 and 0.52 μmol L⁻¹

0.15 μmol L⁻¹



Publication

Issue 52, 2020, Issue in Progress

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RSC Advances

Graphene oxide quantum dots immobilized on mesoporous silica: preparation, characterization and electroanalytical application†

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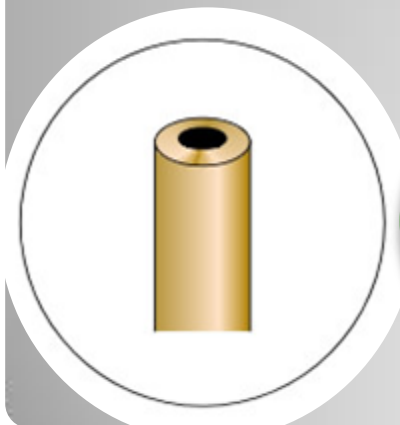
[Albina Mikhralieva](#), ^a [Vladimir Zaitsev](#), ^{*ab} [Oleg Tkachenko](#), ^{cd} [Michael Nazarkovsky](#), ^a

[Yutao Xing](#) ^e and [Edilson V. Benvenuti](#) ^c

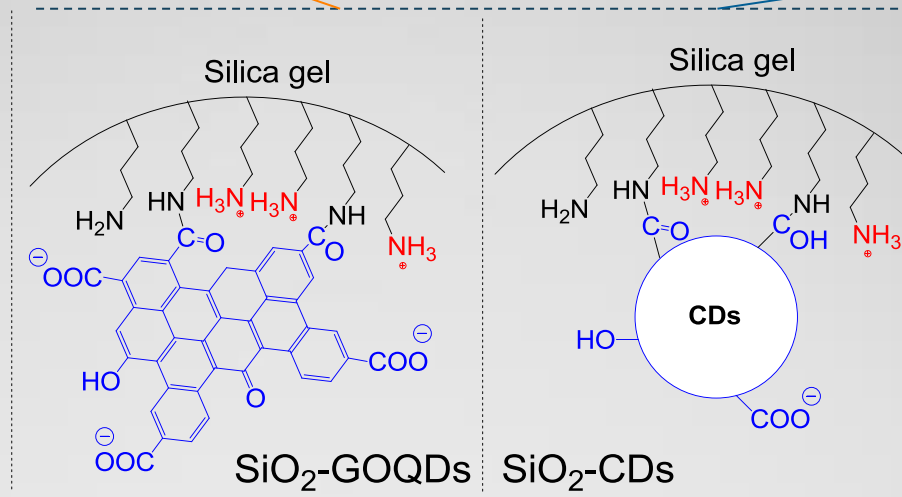
Materials and application



Adsorbents for solid-phase extraction



Modified carbon paste electrode



Antibacterial and antiviral activity



Acknowledgment



Thank you!

