ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2024022

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Co-investigator	Professor Barbara La Ferla, University of Milano Bicocca, ITALY
Co-investigator (*)	Professor Giancarlo Capitani, University of Milano-Bicocca, ITALY
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Experiment title	Carbon Quantum Dots from hydrothermal sustainable approaches: a TEM morphological insight.
MRF Instrument	**TEM JEOL**
Access Route	Direct Access
Science Areas	Chemistry
Sponsored Grant	Yes
Grant Title	Multifunctional Compounds for a Multitarget Approach Against Neurodegenerative Disorders (MULTIFUN)
Start Date	01/10/2023
Similar Submission?	-
Industrial Links	-

Days requested: 4
Previous GP Number: no
DOI: -
Sponsor: Other
Grant Number: PRIN n. 2022N9E847
Finish Date: 30/09/2025

Non-Technical Abstract

The present proposal will synergically integrate and enforce the previous studies of a wider research program aiming at the development of a sustainable synthesis strategy of Carbon Quantum Dots (CDs), which will be reliable and sustainable. We submit a request for an experimental campaign finalized at performing TEM analyses on a series of CDs samples prepared through the hydrothermal approach at different reaction parameters (temperature, time, carbon source). The main scope is to study the influence of the reaction conditions on the morphological aspects of the samples with particular care to the graphitic cores' growth (optimistically, it could be interesting, with an HR approach, to have an estimation of the lattice fringes) and the dimensional distribution and shape of the dots. We also aim to obtain information about the CDs formation process.

Publications

Vercelli B. et al. Nanomaterials 2023, 13, 1365; https://doi.org/10.3390/nano13101635.
Vercelli B. et al. Molecules 2023, 28(1), 72; https://doi.org/10.3390/molecules28010072.
Vercelli B. et al. Elec. Acta, 2021, 138557, https://doi.org/10.1016/j.electacta.2021.138557

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E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

Sample record sheet

Principal contact Professor Giancarlo Capitani, University of Milano-Bicocca, ITALY
MRF Instrument **TEM JEOL** **Days Requested:** 4
Special requirements:

SAMPLE

Material	Carbon Quantum Dots obtained at 160°C from citric acid and urea (carbon based graphitic structures	Carbon Quantum Dots obtained at 200°C from citric acid and urea (carbon based graphitic structures)	Carbon Quantum Dots obtained at 200°C from glucose and urea (carbon based graphitic structures)
Formula	-	-	-
Forms	Solid	Solid	
Volume	cc	cc	cc
Weight	5 mg	5 mg	5 mg
Container or substrate	vial	vial	vial
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

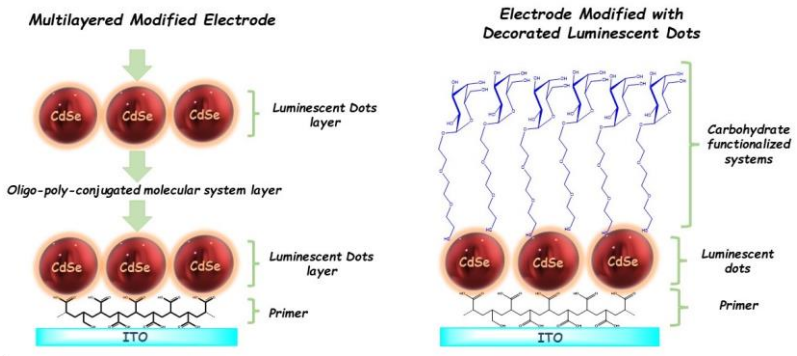
Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	No	No
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)

1. Background and Context

The group of Electrochemistry and Nanomaterials of the ICMATE-CNR in Milan has a long-time experience in the realization and characterization of self-assembled nano-systems, obtained by the alternation of semiconductor nanocrystals or noble metals clusters and oligo-polyconjugates molecular systems or carbohydrates functionalized systems (Scheme 1), for optoelectronic, photovoltaic and biomedical applications. This is a consolidated blue-sky research activity documented by a series of publications in high-impact ISI journals.

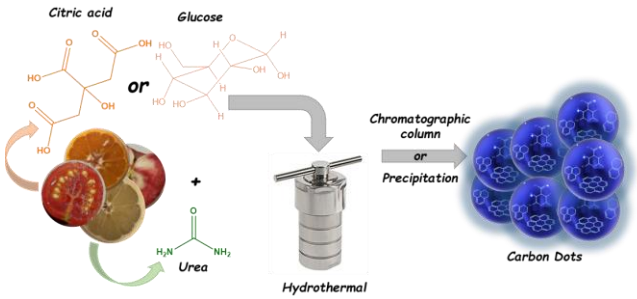


Scheme 1. – Self-assembled Nanosystems

Within this research program and considering the growing request for eco-sustainable, non-toxic nanomaterials, the research studies were recently devoted to the employment of carbon quantum dots (CDs) as a “green” and cheap alternative to noble metal and calcogenide-based dots.

CDs are fluorescent carbon-based nanomaterials, which, since their discovery in 2004, gained grooving interest from the research community, because of their excellent fluorescence properties and surface rich in functionalities, which enables functionalization with a wide range of molecules, including receptors, bio-molecules, molecular semiconductors, etc. Furthermore, they are soluble in water, exhibit an extremely low toxicity, and an excellent biocompatibility useful for real-world biological applications. Their synthesis approaches are simple, sustainable, and could employ cheap and recyclable precursors derived from biomass and agro-industrial waste.

As a first approach, we focused on the development of a sustainable CDs synthesis strategy, which could be reliable and reproducible. We selected the hydrothermal approach owing to its feasibility for large-scale industrial applications, and we employed precursors that could be obtained from agro-industrial waste, like citric acid (CA) or glucose (Glu) as carbon sources and urea as both base and nitrogen sources (Scheme 2).



Scheme 2. – Scheme of hydrothermal preparation of Carbon Quantum Dots

We published two preliminary works dealing with the role played by the nitrogen centres and the thermal post-treatments, respectively, on CDs electrochemical and optical properties[1]. Then we studied the influence of the reaction parameters on CDs properties, and we recently published a work on the issues encountered in the synthesis/purification of red-emitting CDs[2]. The research

program was, also, the object of one Bachelor's and two Master thesis in chemistry at the University of Milano-Bicocca A further bachelor thesis program will begin next March. In 2020 the PI obtained the financial support of the CNR Short Term Mobility Program (STM 2020) to perform Raman and Photoluminescence (PL) emission studies on a series of CDs samples in the laboratories of the group of *Espectroscopía Molecular de Materiales para Electrónica Orgánica*, of the Universidad de Malaga (Spain). In 2023 the present research program obtained the financial support of the PRIN 2022 Project MULTIFUN. In light of what briefly described above, a morphological investigation through TEM is of crucial interest/importance, because it is expected to synergically support the above-mentioned Raman and PL-emission studies. We expect to obtain information on the influence of the reaction conditions on the formation of CDs graphitic cores and their dimensional distribution and shape. Furthermore, from the detection of eventual amorphous material around the CDs dots, we also expect to obtain information about the CDs formation process[3].

2. Proposed experiment

With the present proposal, we submit the request of a 4 days' experimental campaign finalized at performing TEM analyses on a series of CDs samples (optimistically 3/4) prepared through the hydrothermal approach at different reaction parameters (temperature, time, carbon source). The main scope is to study the influence of the reaction conditions on the morphological aspects of the samples with particular care to the graphitic cores' formation (optimistically it could be interesting, with an HR approach, to have an estimation of the lattice fringes) and the dimensional distribution and shape of the dots. We also aim to obtain information about the CDs formation process.

The results expected from the proposed experimental campaign are of paramount importance for the development of the research program in progress and briefly described at point 1, because they will support and enforce all the previously performed studies, using a facility (TEM instrument), which is not present in ICMATE-CNR laboratories. Furthermore, they will enrich a paper that is under preparation and will be part of both a talk that the PI is planning to present at the SCI2024 – XXVIII National Congress (Conference & Exhibition) and the work of thesis of a bachelor student in chemistry of the University of Milano-Bicocca.

3. Summary of previous experimental proposals or characterisation

The proposal in object is submitted to ISIS@MACH ITALIA for the first time.

4. Justification of experimental time requested

For the development of the present proposal, we selected the TEM JEOL instrument located at the IM@IT' Unit UniMIB, closer to the site of ICMATE-CNR in Milan, because it both meets the experimental requests and in case of analysis problems or specific sample preparations, it is possible to promptly intervene. We planned TEM analyses on 3/4 CDs samples for a total of 4 days, including eventual time waste related to possible issues in sample preparation, specific instrument settings, and unexpected problems during measurement execution and signal optimization/collection.

5. References

1. Vercelli B. et al. Elec. Acta, **2021**, 138557, https://doi.org/10.1016/j.electacta.2021.138557; Vercelli B. et al. Molecules **2023**, 28(1), 72; https://doi.org/10.3390/molecules28010072.

2. Vercelli B. et al. Nanomaterials **2023**, 13, 1365; https://doi.org/10.3390/nano13101635.

3. Wang X.Y. et al. ACS Sustainable Chem. Eng. **2018**, 6, 2, 1708–1716, https://doi.org/10.1021/acssuschemeng.7b02941;Guldi D. M. et al. J. Am. Chem. Soc. **2014**, 136, 49, 17308–17316, https://doi.org/10.1021/ja510183c.