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Charles University

Charles University Center of Advanced Materials (CUCAM)

Opening of the High-Resolution Electron Microscopy Laboratory
Jan 14th 2019

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Jul 2015 — Project call

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Jan 2017

Building the research team

Building the research infrastructure

Research activities

Jan 2019

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Call No. 02_15_003 (ERDF)
To support excellent research teams.

Project outputs:

1. Building the excellent research team
2. Upgrade of the research center infrastructure
3. Strengthening the international collaboration

Conditions:

1. More than ½ of center members must be new
2. Research is headed by established foreign professor
3. Team size is limited but not budget

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→ Excellent conditions – "a dream call"

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Building the research infrastructure

Research activities

Jan 2019

- Starting date planned for Aug 1st 2016
- Call results not announced yet

Starting in ca 75% operation capacity

- Hiring only 2/3 of new researchers
- Buying just the essential equipment
- Two labs reconstructed

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Materials synthesis lab

Materials characterization and catalysis lab

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- Building the research infrastructure
- Research activities

Jan 2019

100+ % operation

Total project allocaction	215 214 556,41 Kč
Including investments	97 004 359,05 Kč

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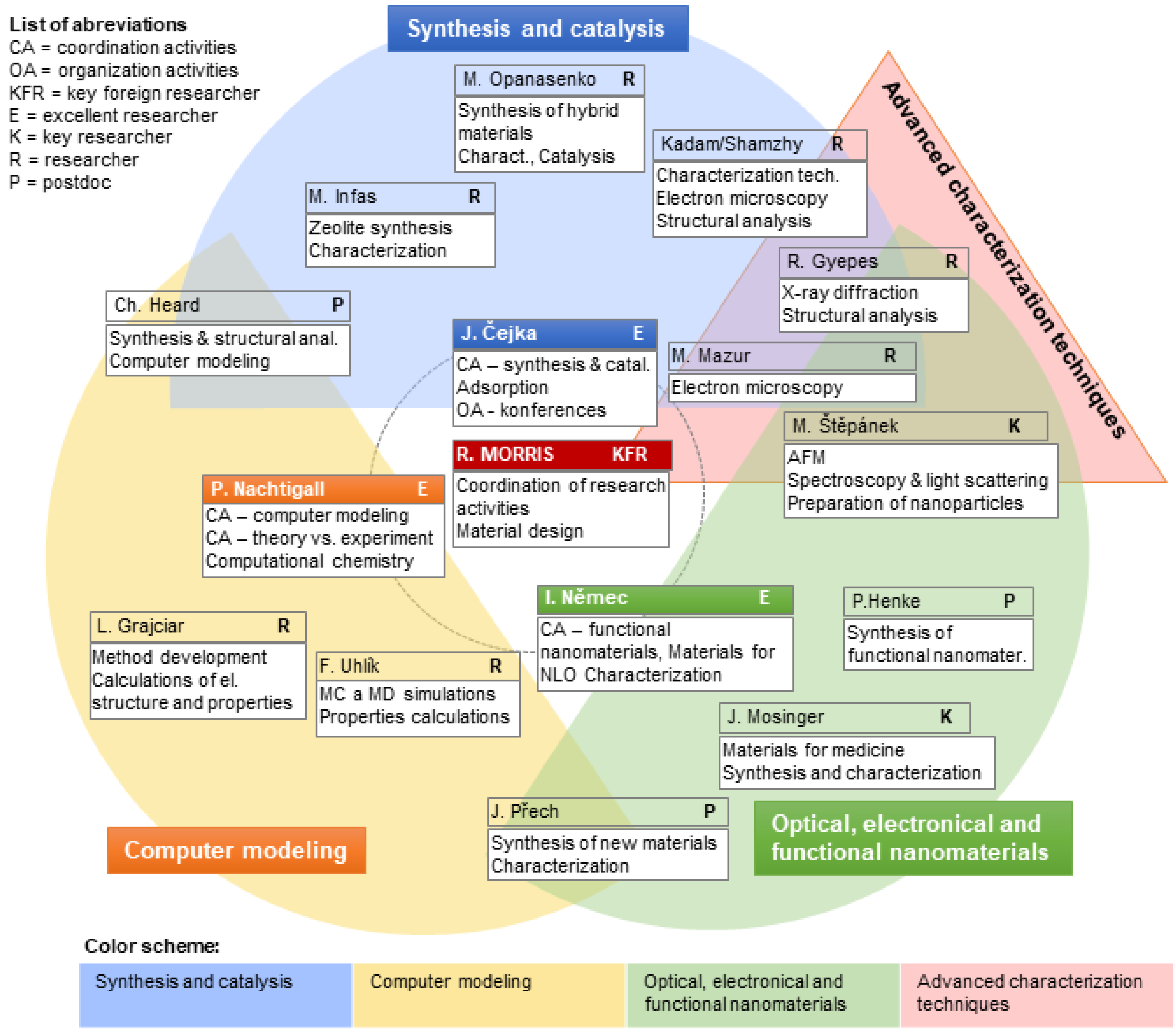

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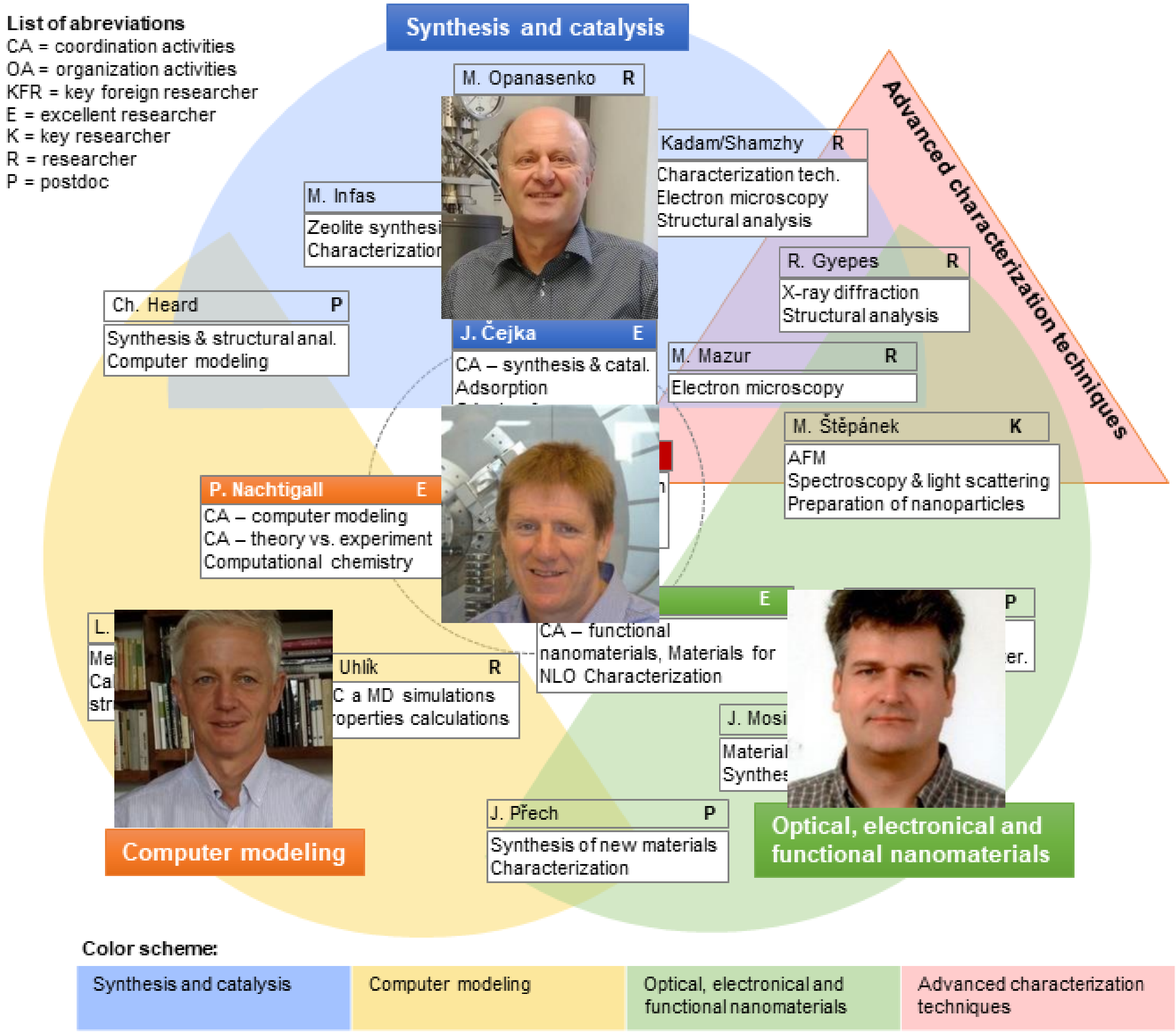
<u>CUAM structure:</u>

KFR	Prof. Russell E. Morris
ER's	Prof. Jiří Čejka
	Prof. Ivan Němec
	Prof. Petr Nachtigall

6 foreigners *vs.* 10 Czechs/Slovakes
8 below 35 years *vs.* 8 above 35



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Building the research team
100%

Building the research infrastructure
94%

Research activities

Large number of public procurements

Building the lab for HRTEM

Examples of equipment purchased:
- X-ray powder diffractometer
- Raman spectrometer
- GC MS and GC machines
- Adsorption measurement devices
- Quantum yield spectrometer
- FTIR spectrometer
- Computer cluster
- Ultramicroton
- Sputter coater
- Autoclaves, owens
- ....
- HRTEM

Already almost finished: 94%

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Feb 2017:	First procurement for HRTEM announced
Feb 2018:	Third procurement successfully finished
Jun 2018:	UK-JEOL contract signed

Jan 2019

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Building the research team

Building the research infrastructure

Research activities

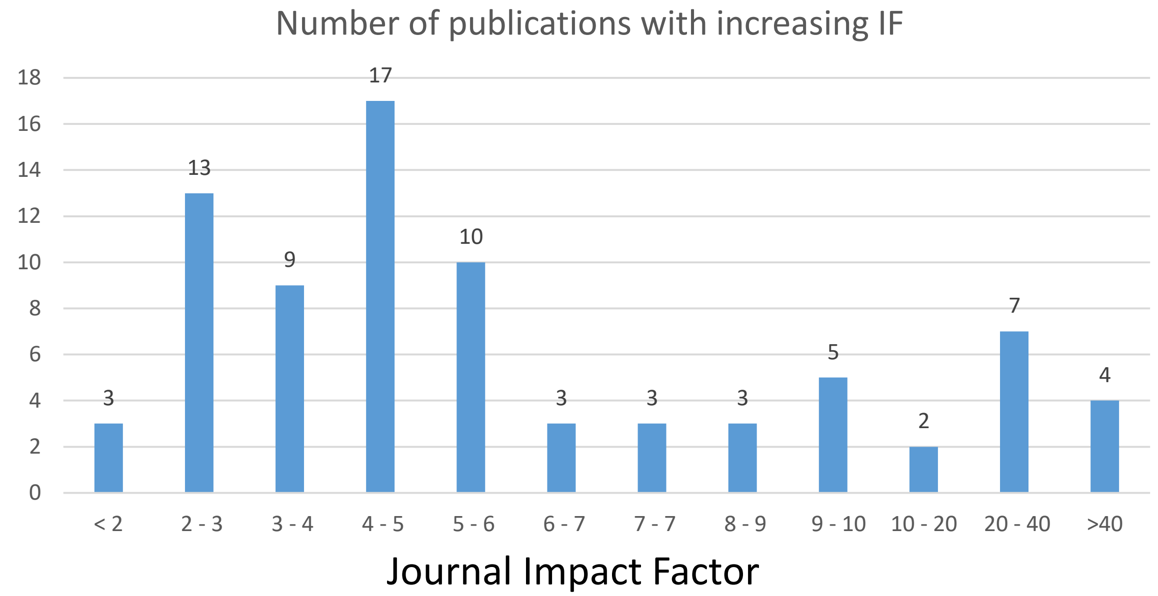
Jan 2019

Research output

In 24 month of full operation + 5 months reduced operation

79 papers, including Nature Chemistry (3), Chem. Soc. Rev. (4), Advanced Materials (4), Angewandte Chemie (2), J. Mater. Chem. A (4)

37 out of 79 at IF > 5; ~50% within international collaboration



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Where we are in Jan 2019:

Project outputs:

1. Building the excellent research team
   DONE.
2. Upgrade of the research center infrastructure
   94% finished (few items planned for later stage)
3. Strengthening the international collaboration
   Twinning H2020 – application filed Nov 2018
   H2020-ICT-2018-2020 – application filed Nov 2018
   BTHA – running, 2017-2020

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Charles University Centre for Advanced Materials

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Advanced Materials

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- *mastering the design, research and development of new and improved materials will remain key for achieving the goals of the European Innovation Policy, in agreement with the European Strategy for a smart, sustainable and inclusive growth (EUROPE 2020).*

- *All major economies have initiatives in Advanced Materials*

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Mission

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- **Our mission** is to develop a leading Centre of Excellence in Advanced Materials located at Charles University (CU) in Prague, covering the Design, Synthesis and Application.

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Vision

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Our vision is that creative chemistry completed in CUCAM will be the wellspring from which the next generation of advances in materials technology design will flow.

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3Ps strategy

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The strategy by which we will achieve this vision is based on developing the three Ps

- **Prague**
  - the environment and facilities – making these world-class
- **People**
  - The young talent in the centre is extremely important – our priority is to develop their careers so they are competitive with anyone in Europe
- **Promise**
  - The potential for top quality science is very much at the forefront of why this group of scientists make an exciting centre

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Success

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- For a successful Centre the output of the work should be more than the sum of the individual researchers.
- An internationally recognised and trusted centre of scientific excellence
- A Centre that has impact!

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HRTEM facility

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- Investment in **Prague**
  - Purchase of new instrumentation
- Investment in **People**
  - Dr Michal Mazur
    - Research training trips to the UK and Sweden

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Science

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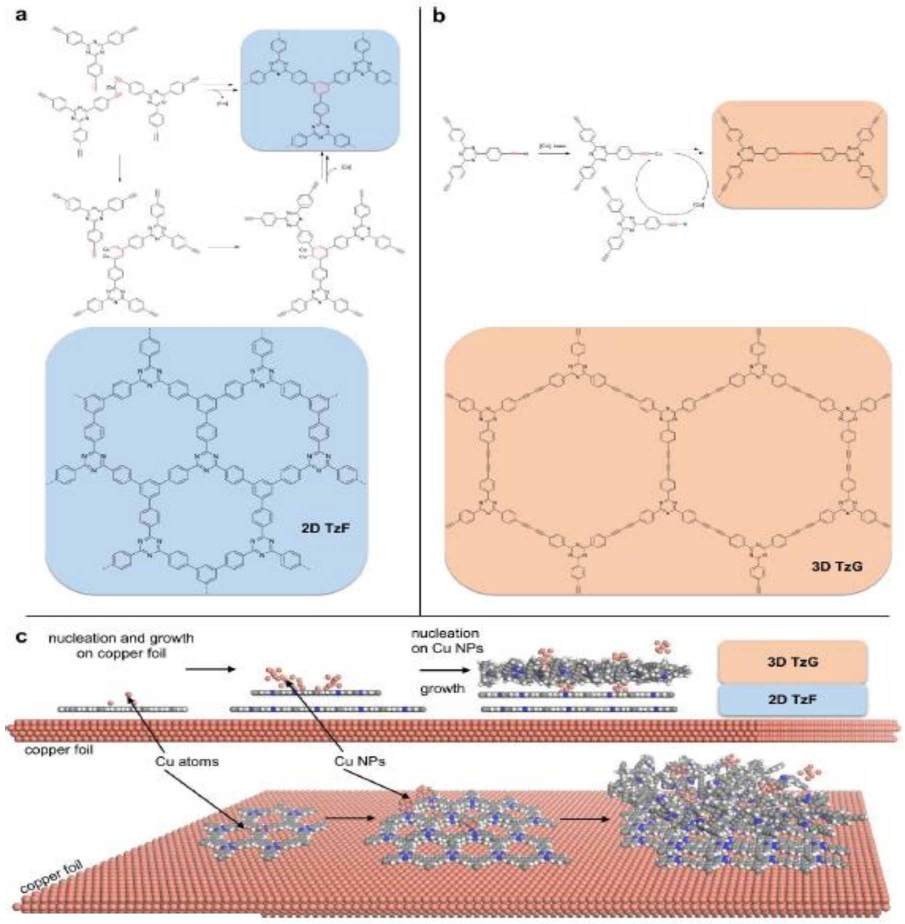
Theme 1. ADOR routes to 'unfeasible' inorganic and hybrid catalysts

Theme 2. Electronic and Optical Materials
Novel modular materials for nonlinear optics.
Design and Synthesis of free-standing 2D organic materials.

Theme 3. Multifunctional delivery of multifunctional therapeutic agents
Design and synthesis of materials for storage of multiple therapeutic agents.
Formation of modular mixed-matrix composite materials.



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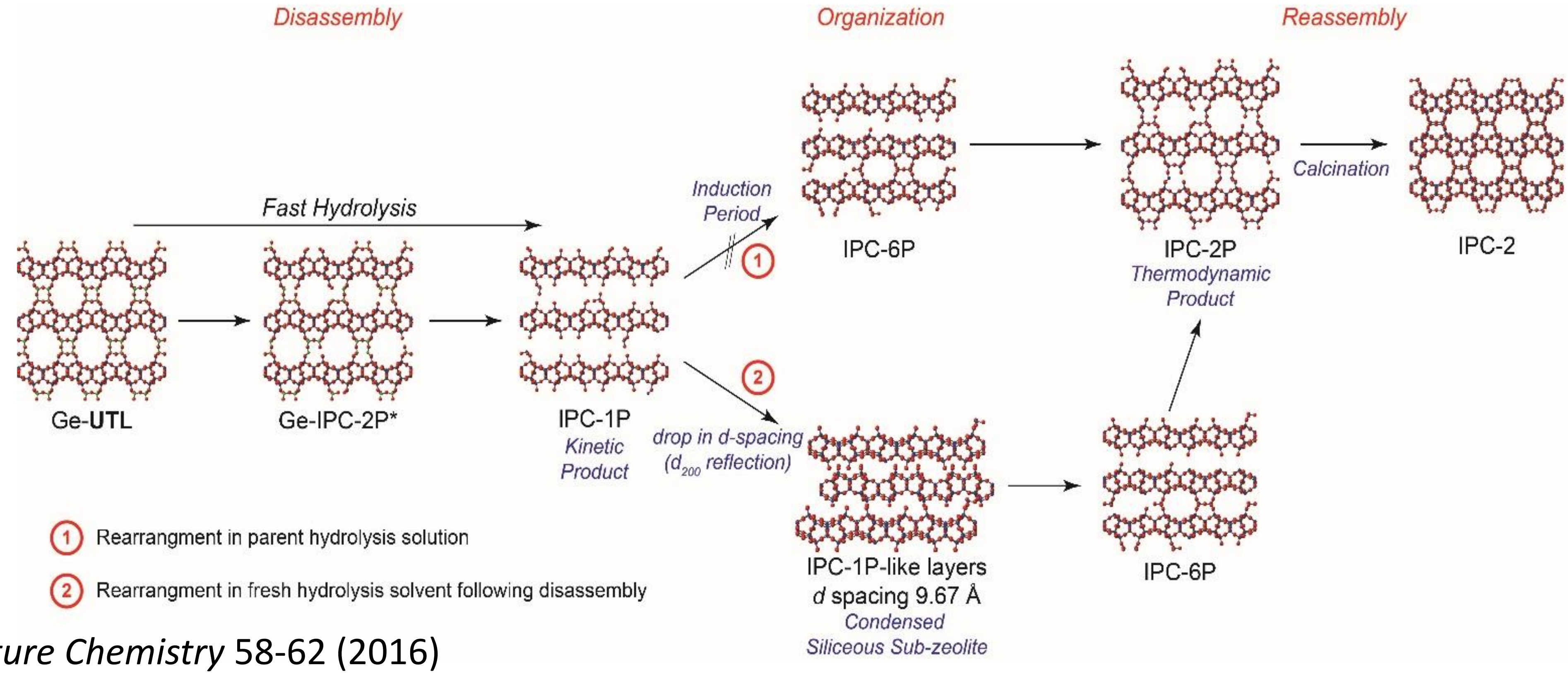
ADOR process



Mazur et al *Nature Chemistry* 58-62 (2016)
Mazur and coworkers *Nature Protocols* In press (2019)
Mazur and coworkers *J. Am. Chem. Soc* Submitted (2019)

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Polymer nanoparticles

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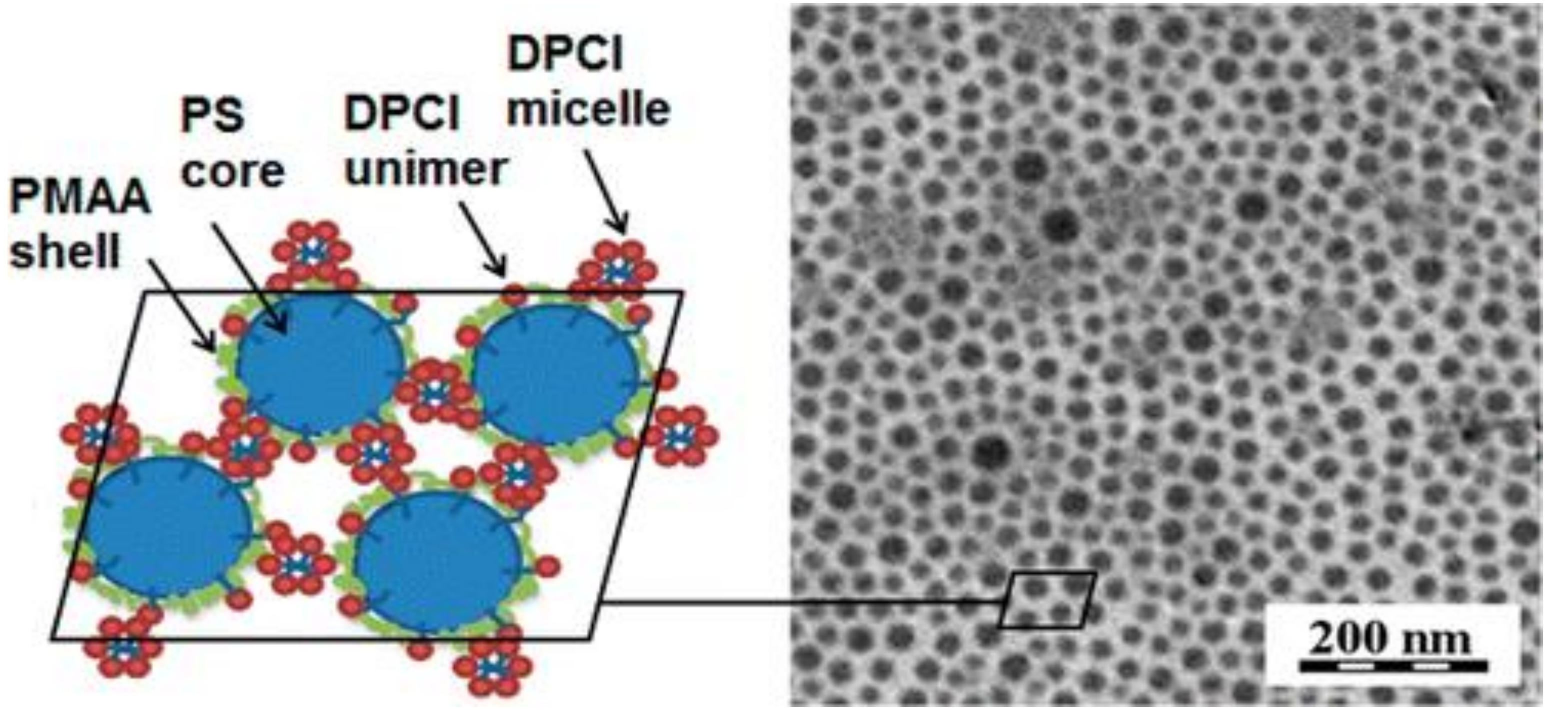

Stepanek and coworkers *Soft Matter* 7578-7585 (2018)
Stepanek, Mosinger and coworkers *ACS Appl. Mater. Interface* 36229-36238 (2017)

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Werner von Siemens prize 2017 for the most significant result in basic research

Discovery of a new method for zeolite synthesis and its application in catalysis

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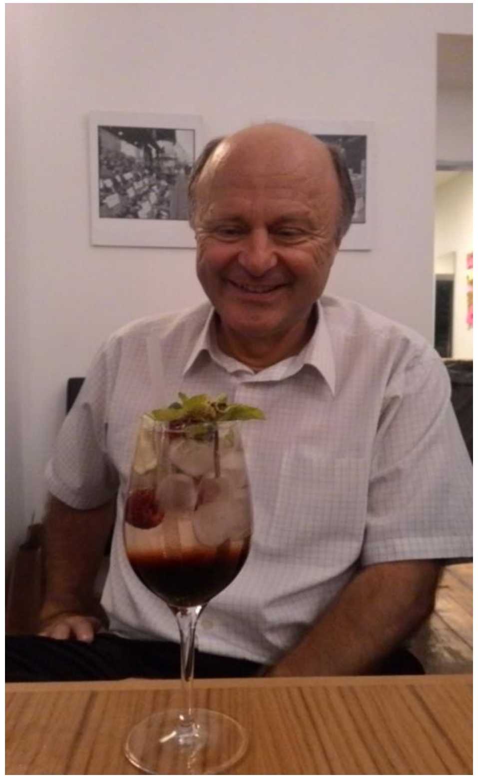
Jiři Čejka awarded €1.9 M ExPro GAČR Grant

Only 5 Expro Grants was given in Czech Republic in chemistry

“ADORable catalysts”

Project based on the design, preparation and application in selective catalysis of novel zeolites synthesised by unconventional approach



GAČR
CZECH SCIENCE FOUNDATION

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Electron Microscopy Laboratory at the Faculty of Science

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Electron Microscopy

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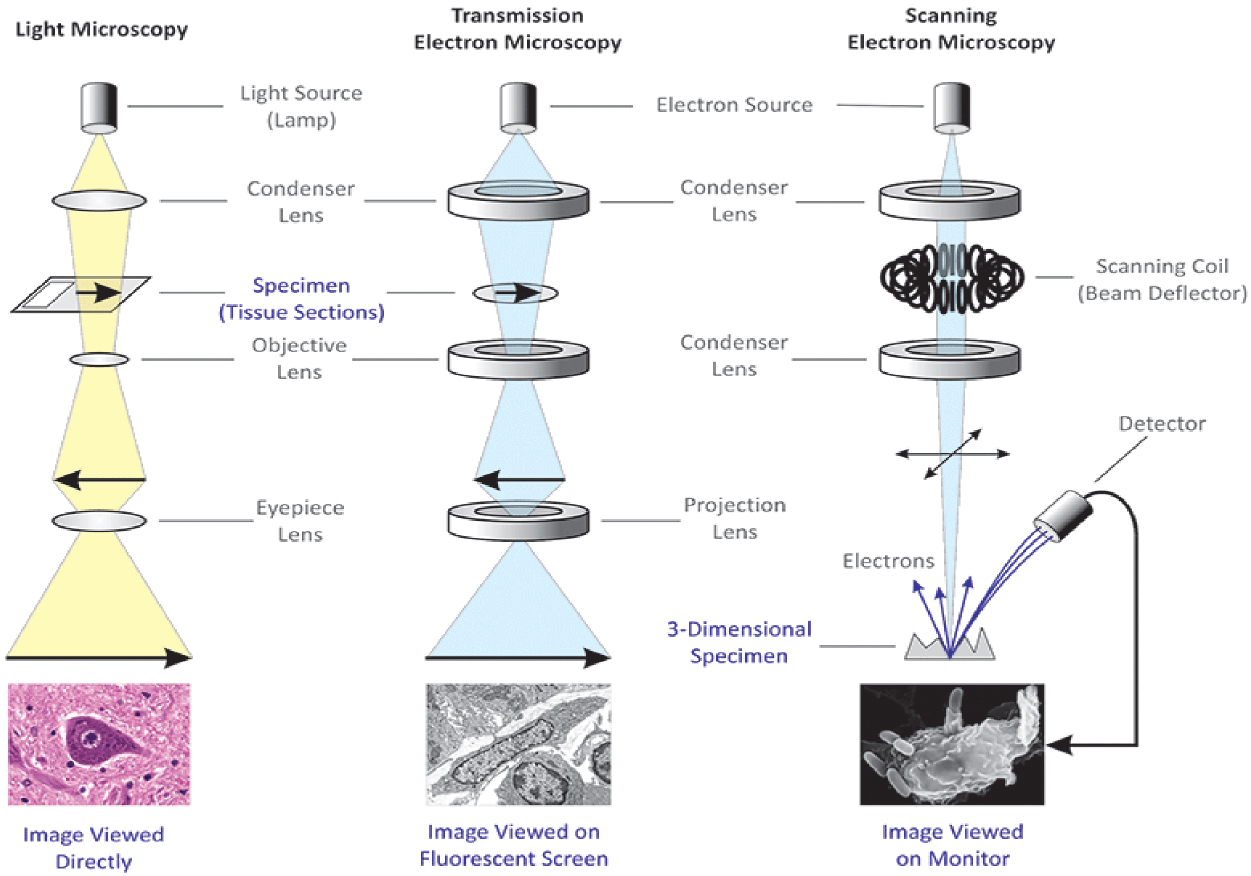

microbiologyinfo.com

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Key steps in Electron Microscopy science

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1924: French physicist **Louis de Broglie** (1892–1987) realizes that electron beams have a wavelike nature similar to light. Five years later, he wins the Nobel Prize in Physics for this work.

1931: German scientists **Max Knoll** (1897–1969) and his pupil **Ernst Ruska** (1906–1988) build the first experimental TEM in Berlin.

1933: **Ernst Ruska** builds first electron microscope that is more powerful than an optical microscope.

1941: **Manfred Von Ardenne** and **Bodo von Borries** patent - electron scanning microscope (SEM).

1981: **Binnig and Rohrer -** detailed images of atoms on the surface of a crystal of gold.

1986: **Binnig and Rohrer** share the Nobel Prize in Physics with the original pioneer of electron microscopes, **Ernst Ruska**.

2017: **Dubochet, Frank, and Henderson** helped to develop cryo-electron microscopy. The 2017 Nobel Prize in Chemistry has been awarded for work that helps researchers see what biomolecules look like.

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https://www.explainthatstuff.com/electronmicroscopes.html

Transmission Electron Microscopy

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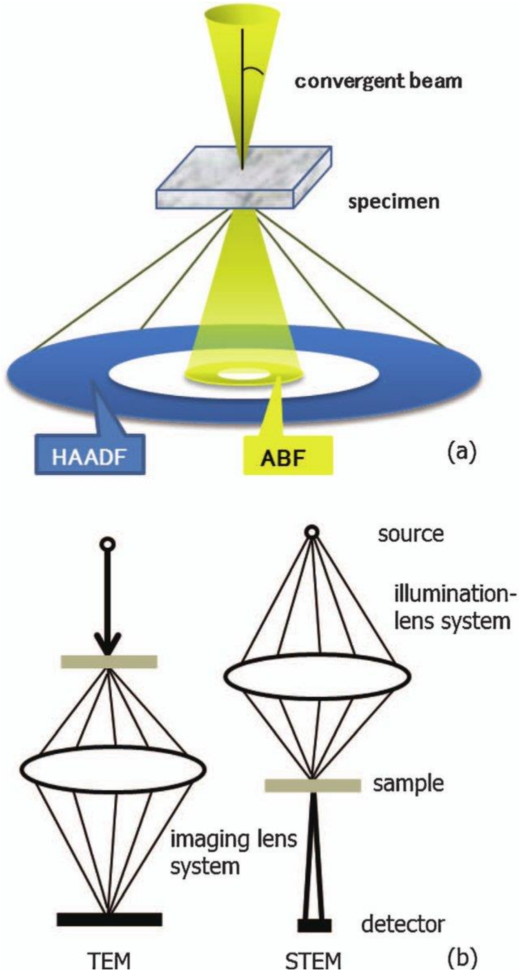

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JEOL JEM NEOARM-200F

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Microscope is equipped with:

- Schottky-type Field Emission Gun (30-200 kV voltage)
- Condenser Lens with Cs aberration correction
- CMOS camera (4096 x 4096 pixels, up to 200 fps redout)
- Specimen tilting stage (+/- 35^{o})
- STEM image acquisition unit
- Phase plate
- Cryo holder for low-temperature measurements
- EDS detector for elementary analysis (Be to U)

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Microscope is able to image:

- Structure of zeolites and zeolitic materials
- Structures in atomic resolution
- Metal-organic frameworks (MOFs) structures
- Carbon nanomaterials
- Polymer nanoparticles
- Self-assembly supremolecular structures (micells, vesicles)

Resolutions:

- HAADF Resolution 0.10nm
- TEM point resolution 0.23nm

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Equipment

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Ultramicrotome Leica EM UC7

Automatic Plunge Freezer Leica EM GP2

High Vacuum Coater Leica EM ACE600

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Zeolites

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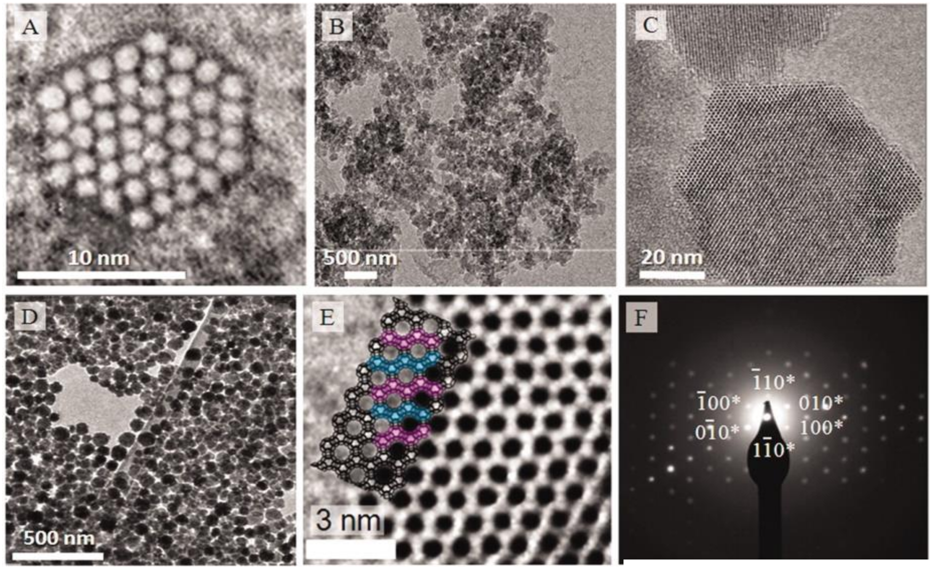
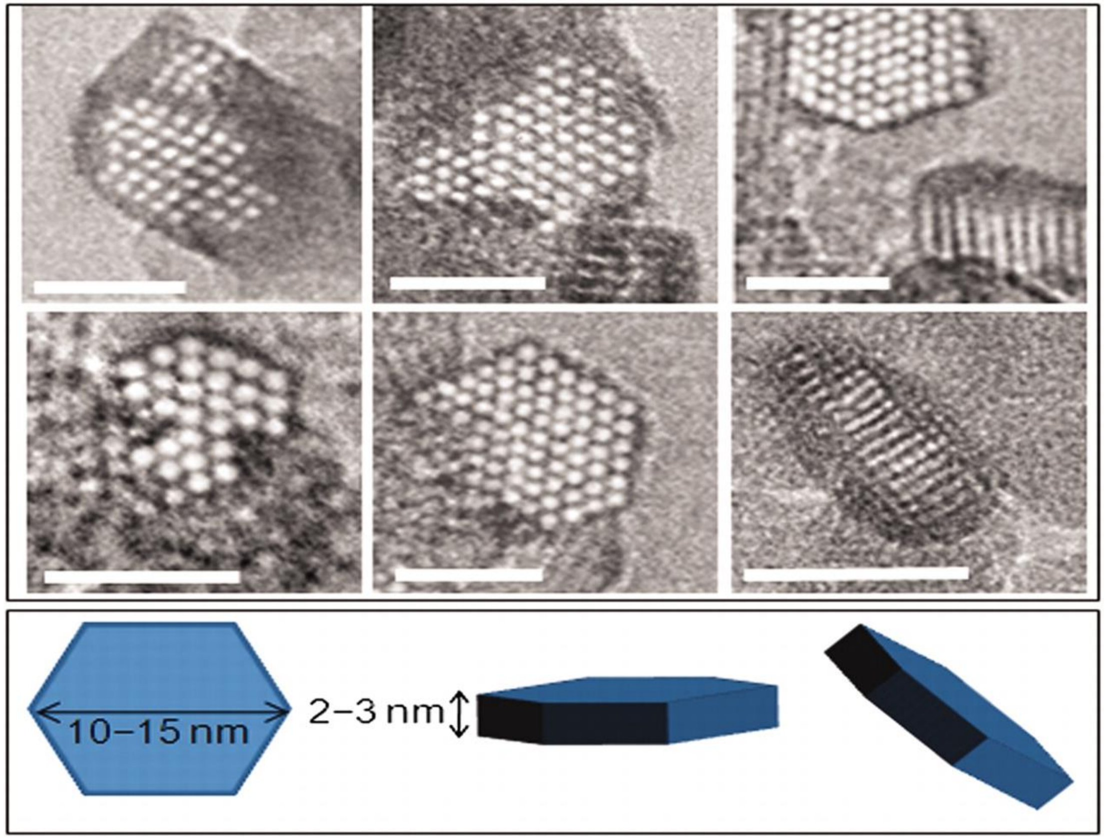
Porous aluminosilicates

Low electron-beam stability

Adsorbed water decreases stability

Low dose of electrons required





Mintova et al., Science, 335, 6064, pp. 70-73

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ADOR zeolites

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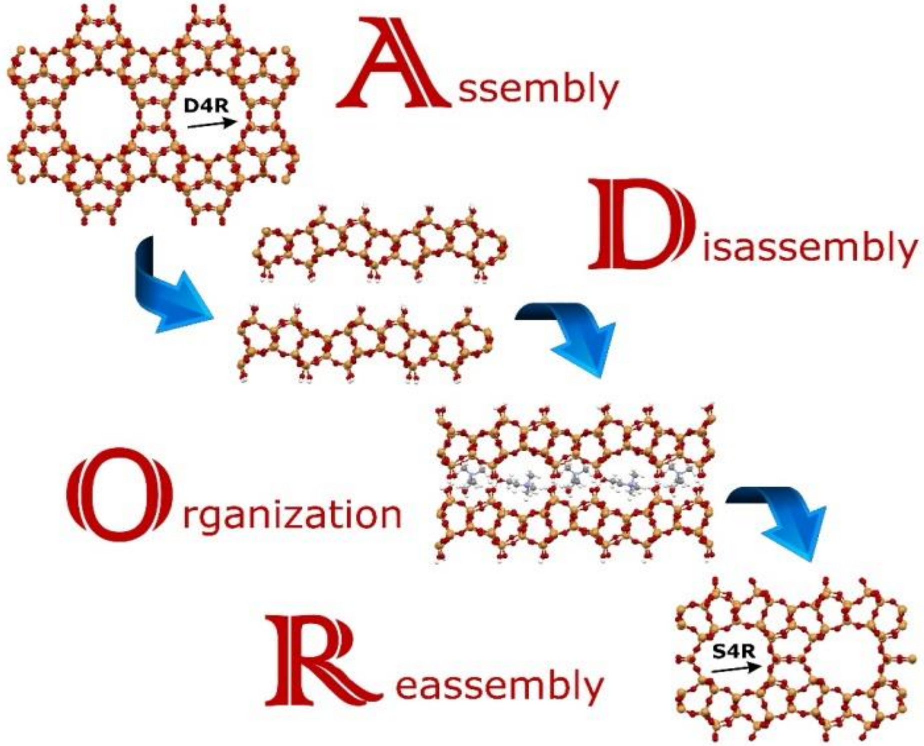

M. Mazur et al. J. Chem. Mater. A, 2018

The **ADOR** (**A**ssembly-**D**isassembly-**O**rganization-**R**eassembly) process involves the synthesis of 3D germanosilicate during first step. Then, selective disassembly of it to form a layered material followed by organization of layers and reconnection of them to get new zeolite.

The ADOR is a way for the preparation of layered zeolite precursors, that can be further modified to get the **related zeolitic architectures**.
12 new topologies were revealed so far.

Two recognised Czech Zeolites: PCR and *PCS

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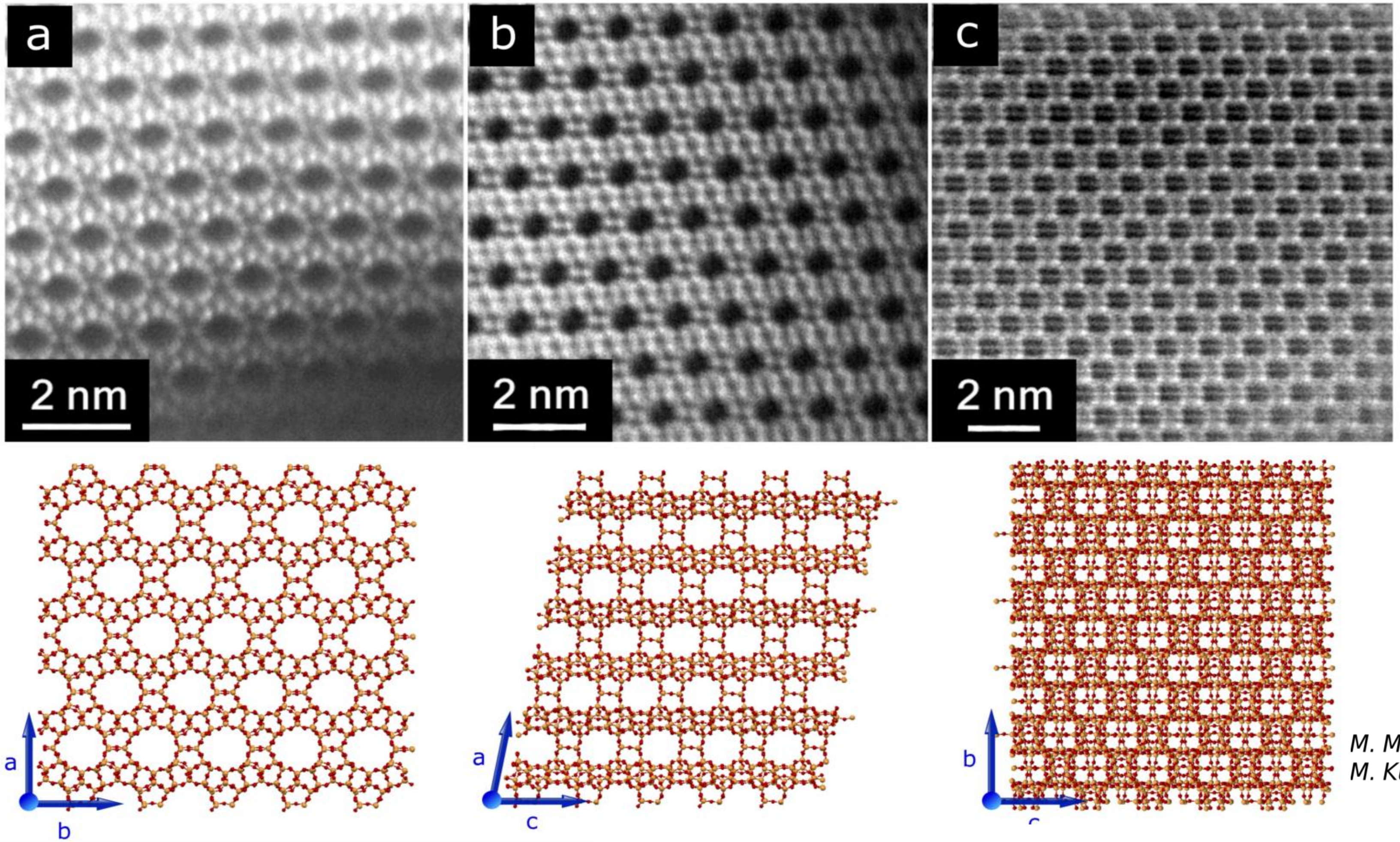
Imaging of ADOR zeolites



Atomic resolution images

Nanoscale measurements

M. Mazur, V. Kasneryk, J. Přech, F. Brivio, C. Ochoa-Hernández, A. Mayoral, M. Kubů and J. Čejka, Inorganic Chemistry Frontiers, 2018, 5, 2746-2755.

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Rotation Electron Diffraction

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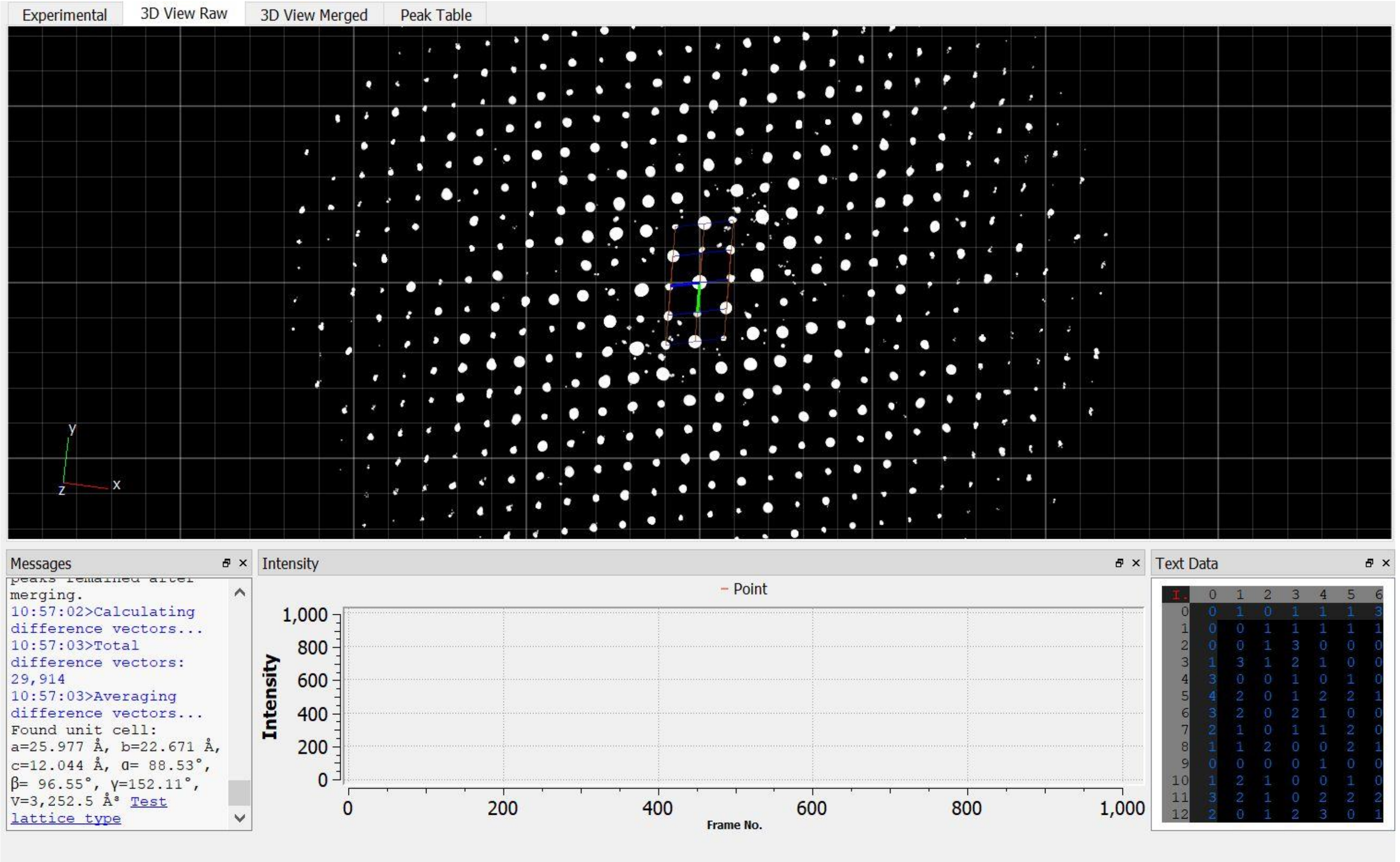

IPC-8 sample
(novel Czech zeolite)

J. Zhang et al. manuscript in preparation

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Rotation Electron Diffraction

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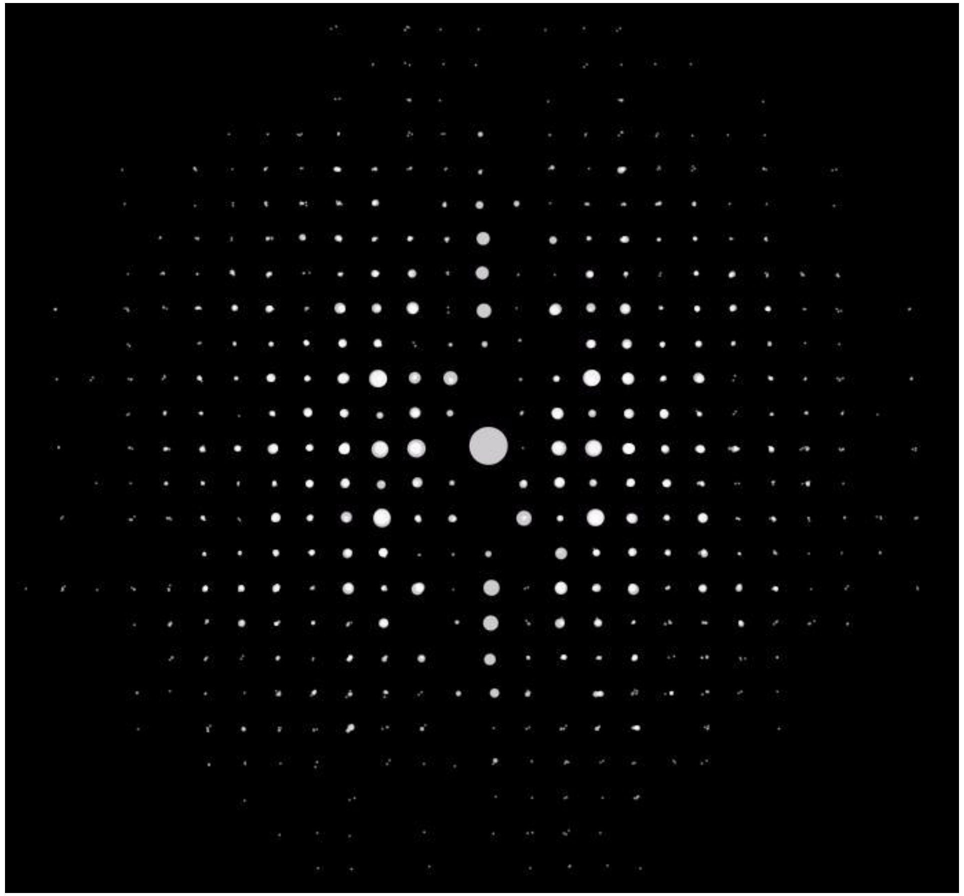

Kagomé geometry Cu-MOF

Method for solving the structure (describing atoms positions)

M. Infas H. Mohideen, et al. manuscript submitted

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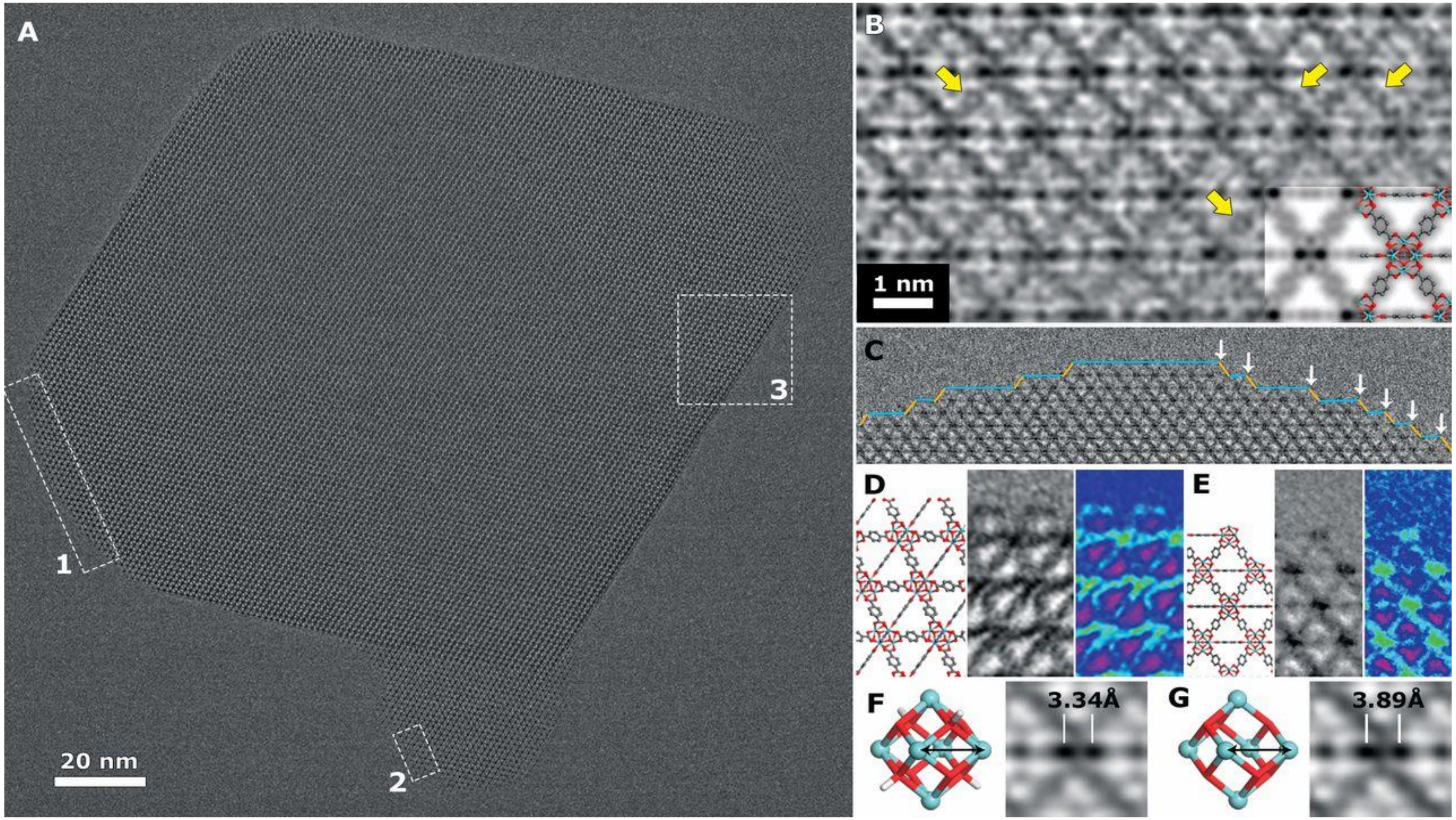
Beam-sensitive materials



MOF

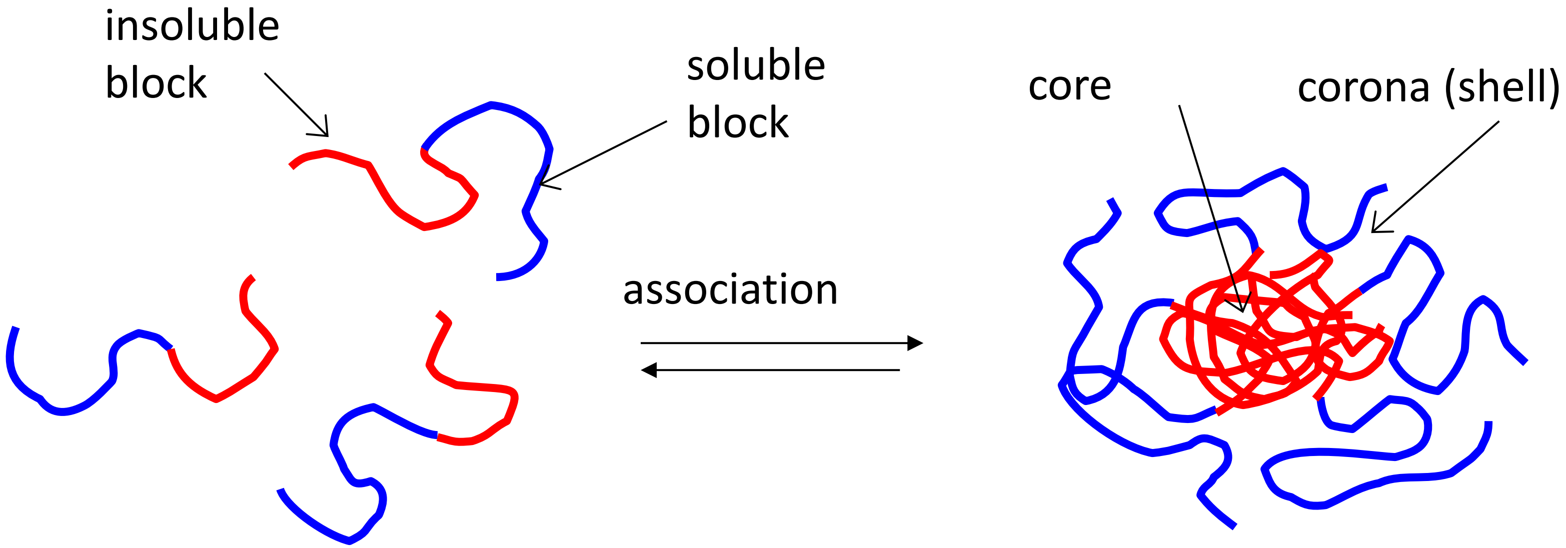
UiO-66 crystal

Measurements in cryo conditions makes sample more stable

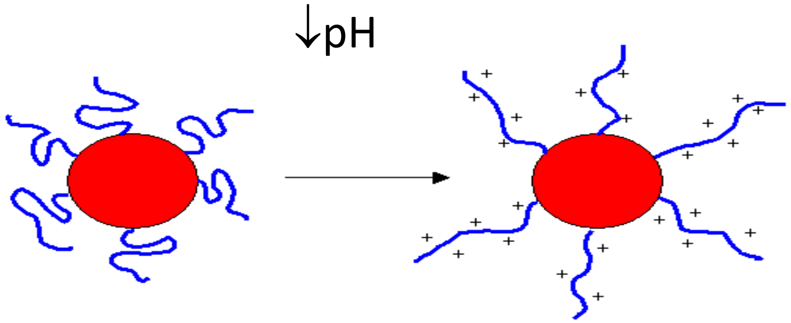
D. Zhang, Y. Zhu, L. Liu, X. Ying, C.-E. Hsiung, R. Sougrat, K. Li and Y. Han, ***Science****, 2018, DOI: 10.1126/science.aao0865*

Associating block copolymers

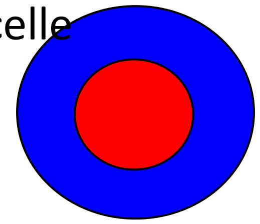
Block copolymers in selective solvents



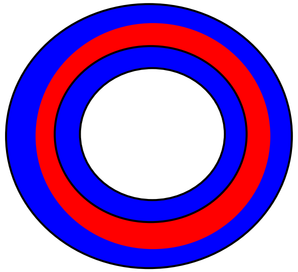
Responsivity to external stimuli such as pH for PE coronas:



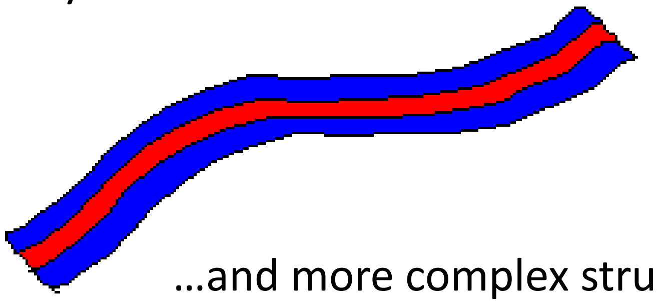
spherical micelle



vesicle



cylindrical micelle



…and more complex structures for multiblock and branched copolymers

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Electrostatic coassembly

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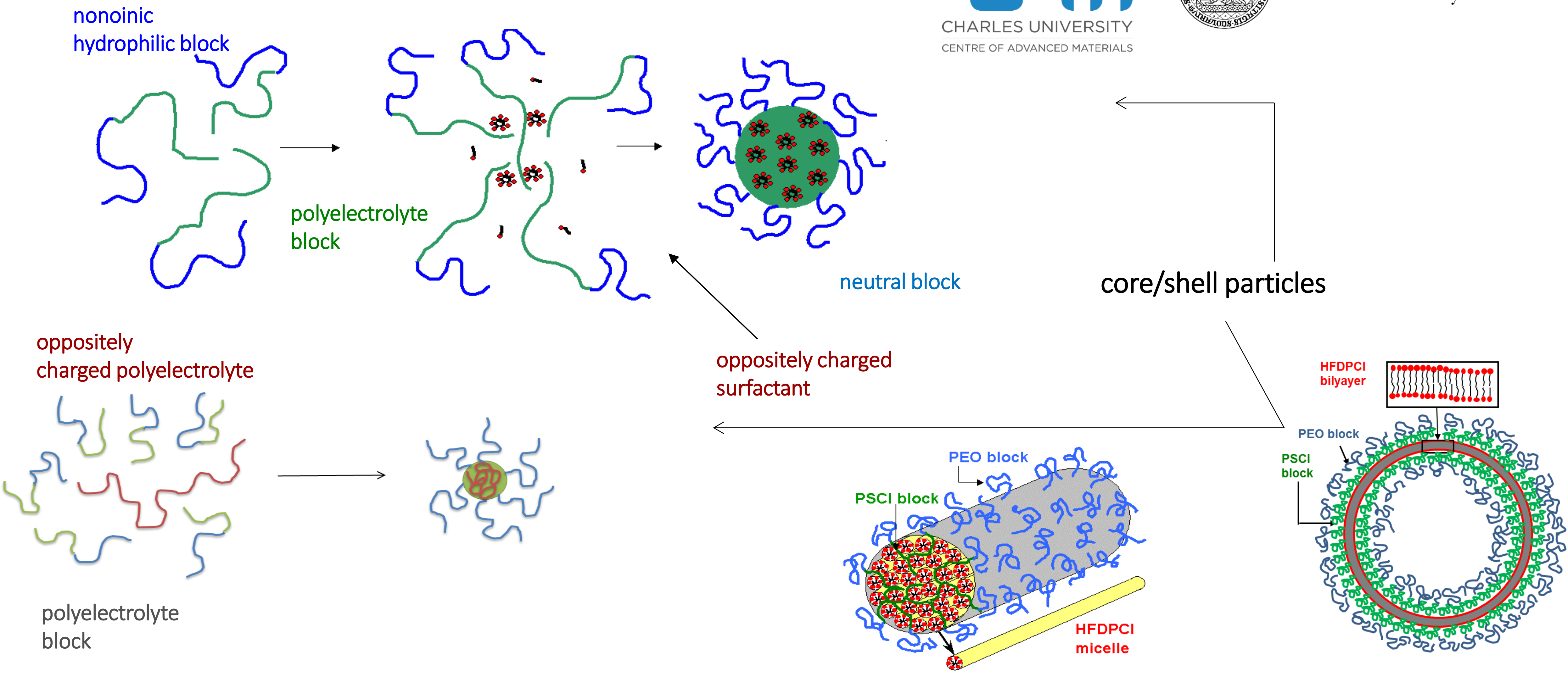

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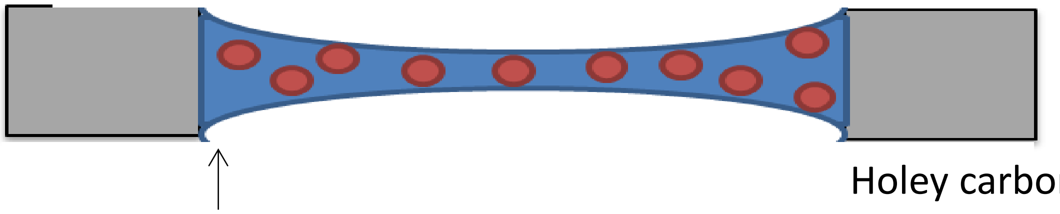
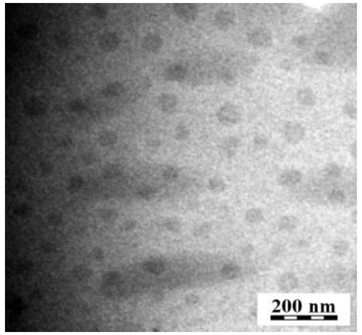
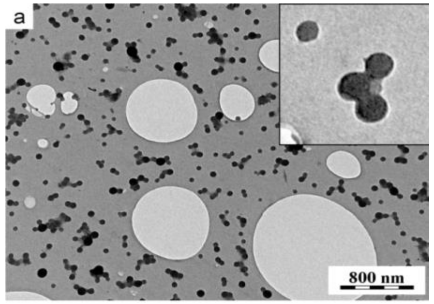
Cryo-TEM

Thin layer of NP aqueous solution trapped in holey carbon film is fast cooled (105 K/s) to form **vitreous ice**

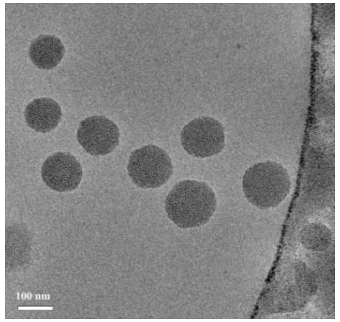


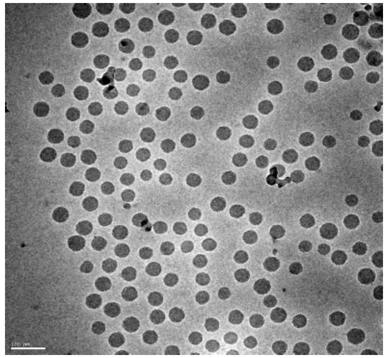
Dry sample TEM





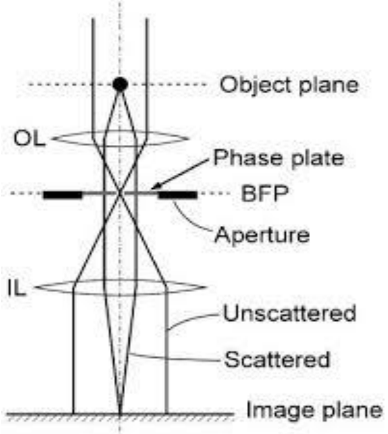
Cryo-TEM

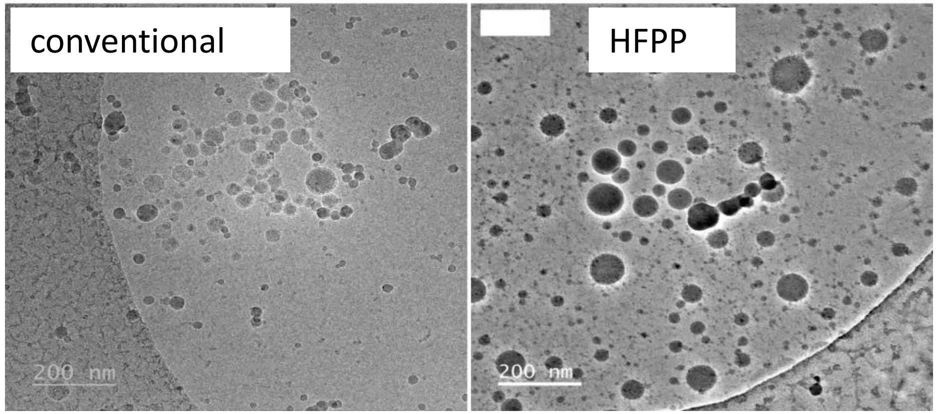




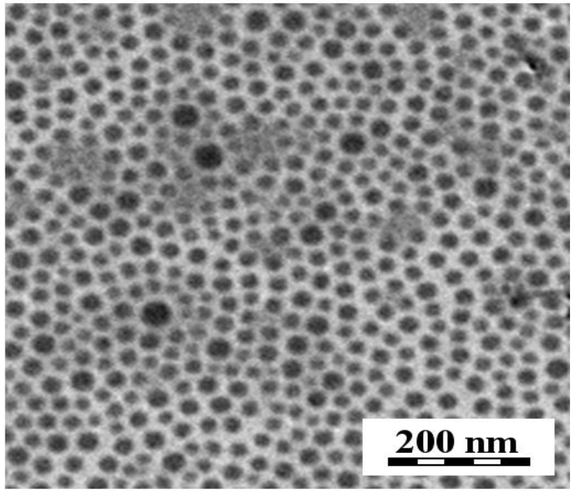
Hole-free phase plate

Contrast enhancement

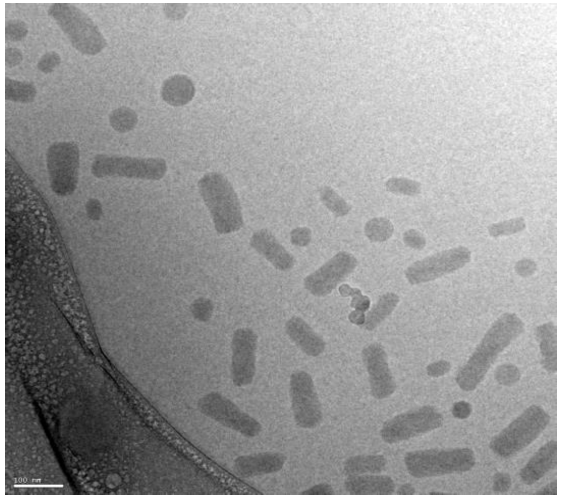




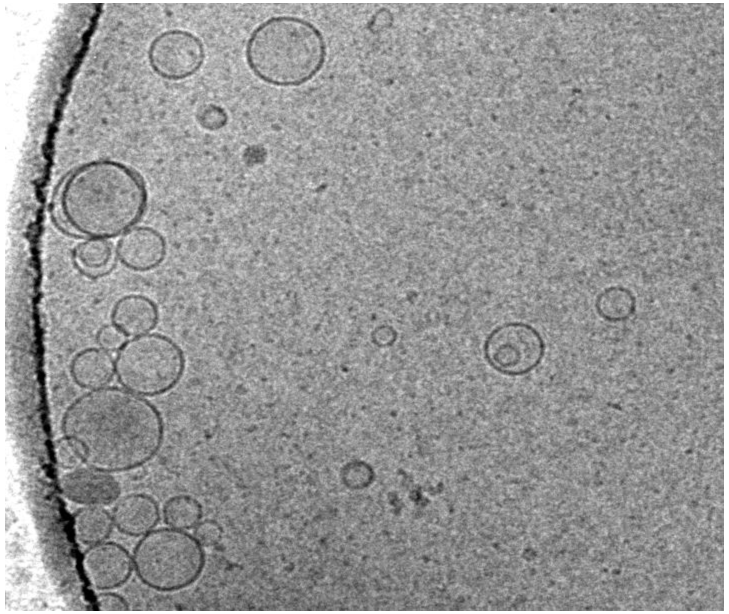
spherical micelles



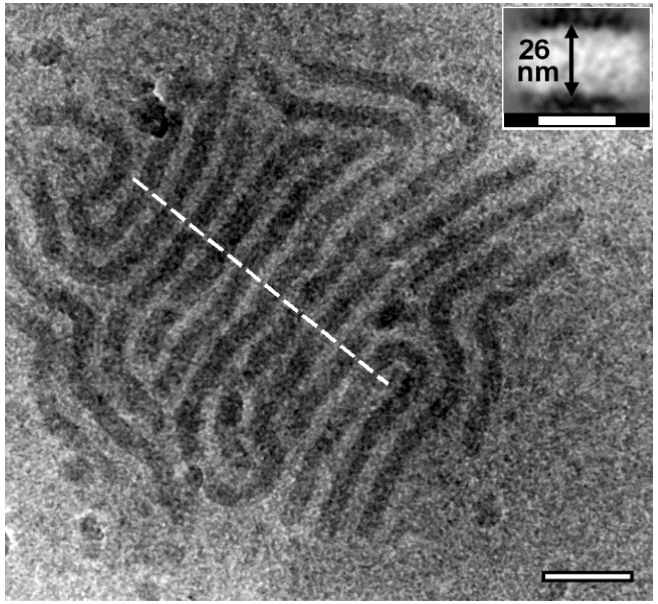
cylindrical micelles



vesicles



wormlike micelles



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