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et d'Ingénierie
Supramoléculaires

Grenoble, 11 June 2018

Stimulus responsive and self-assembled materials

Luisa De Cola

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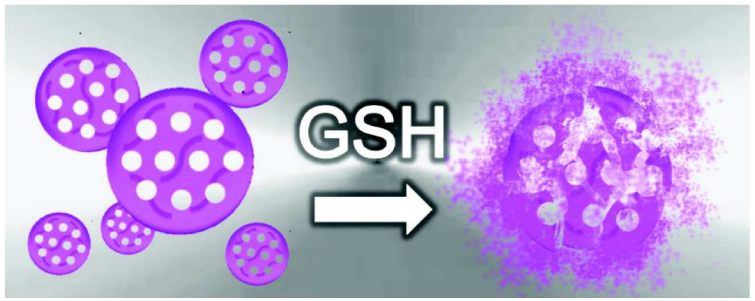
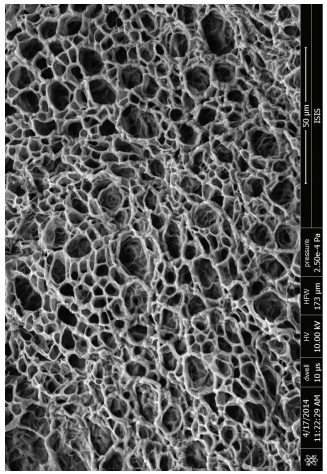
Outline

Breakable Nanocontainers

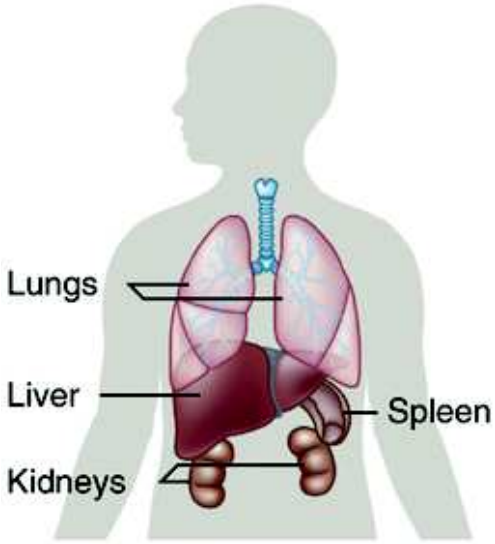


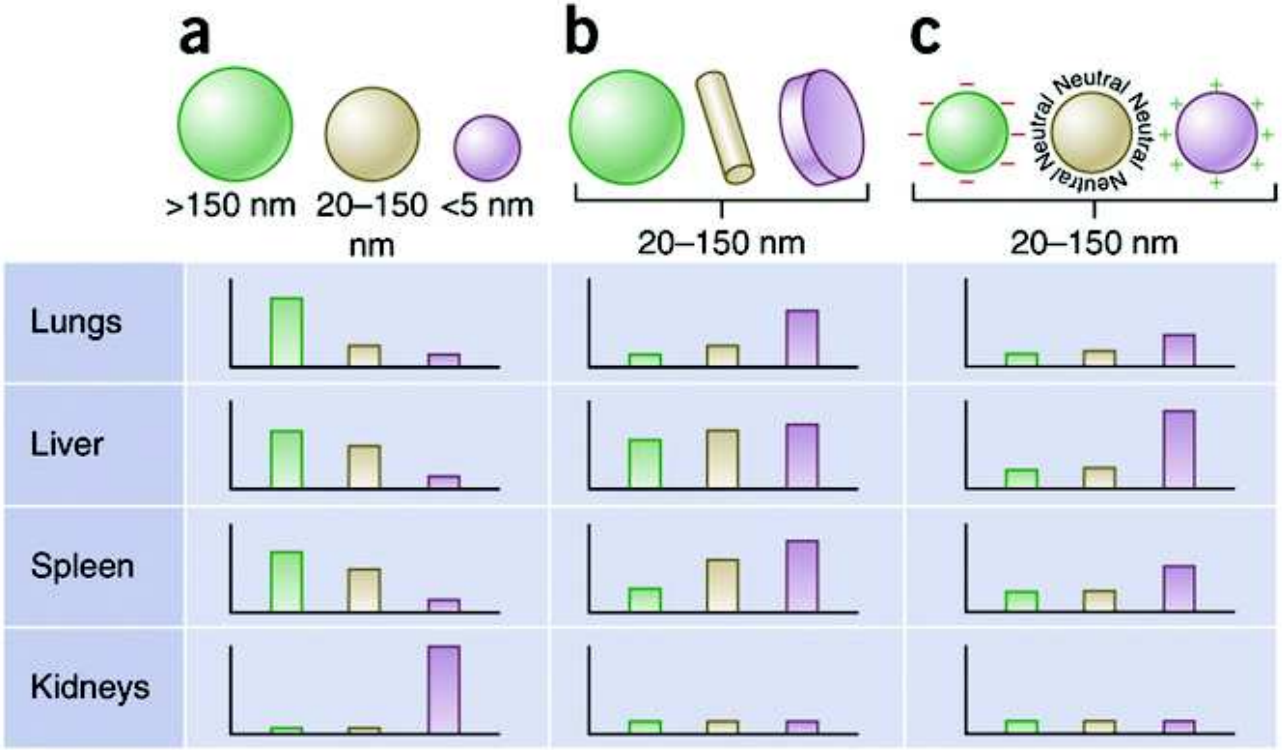
Hybrid hydrogels



Assembling virus like particles

Nano is important in medicine





M. Ferrari et al. *Nature Biotech*. **2015**, *33*, 941-951

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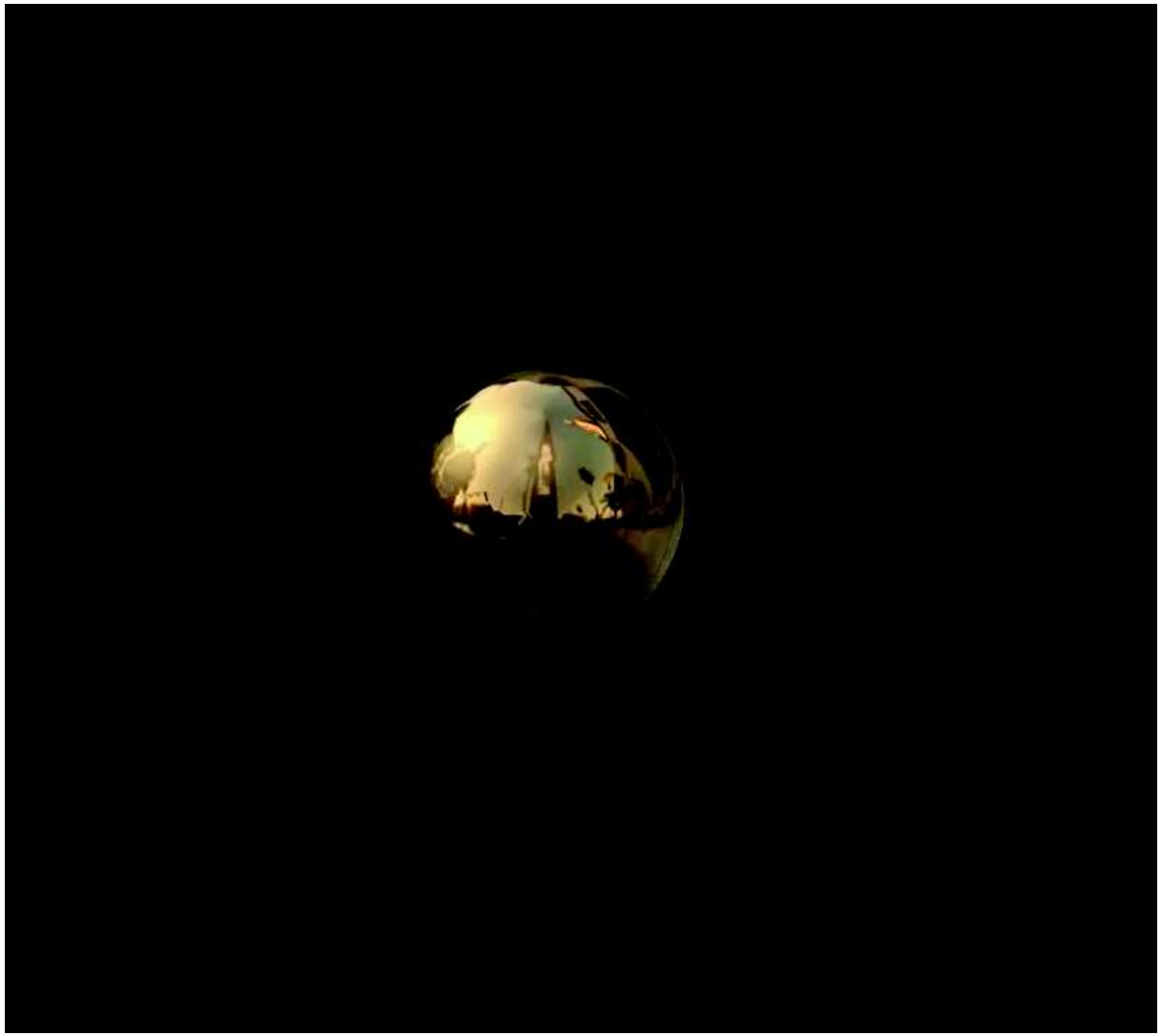
What can we do to eliminate the nanoparticle?

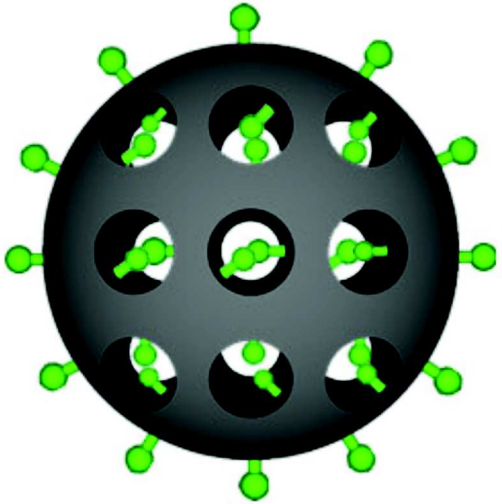


Mesoporous Silica Nanoparticles (MSNs)

A promising platform for controlled drug delivery

Controlable sizes and shapes

Easy functionalization of pores and/or particle surface



Tunable pore sizes

Possible loading of molecules

Biocompatibility

Incomplete elimination and bioaccumulation issues hinder a faster clinical translation as nano-medical tools

M. Vallet-Regí, J. Zink, T. Bein, G. Stucky, R. Ryoo R, C. Hyeon, C. Brinker, K. Cai, U. Wiesner, …

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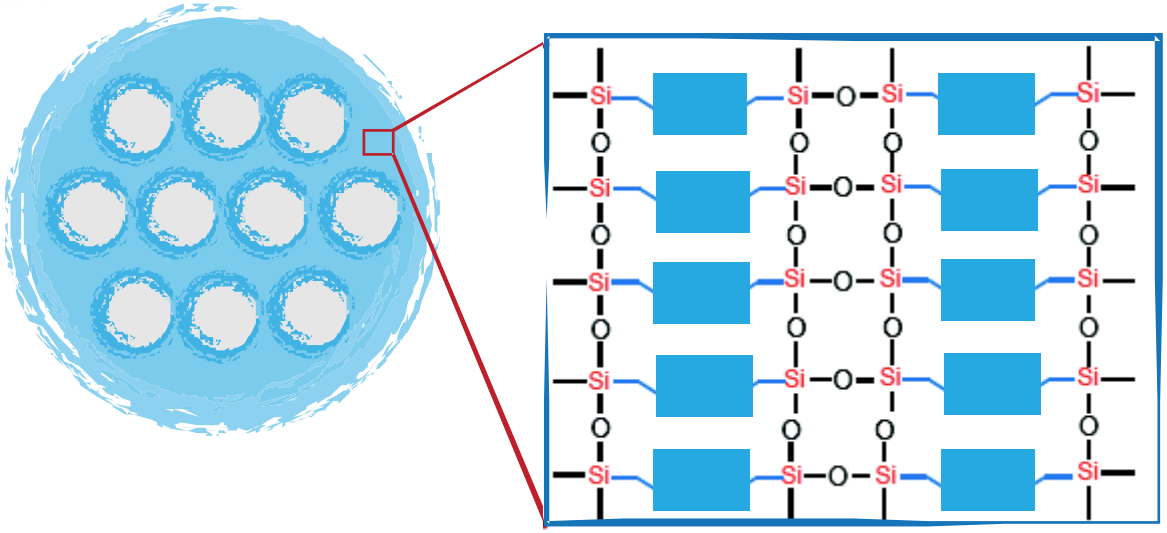
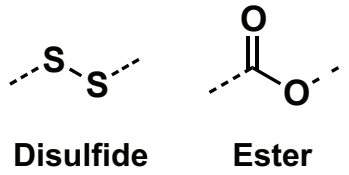
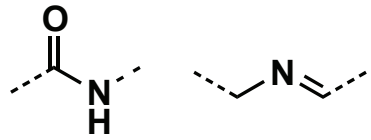
Breakable porous silica





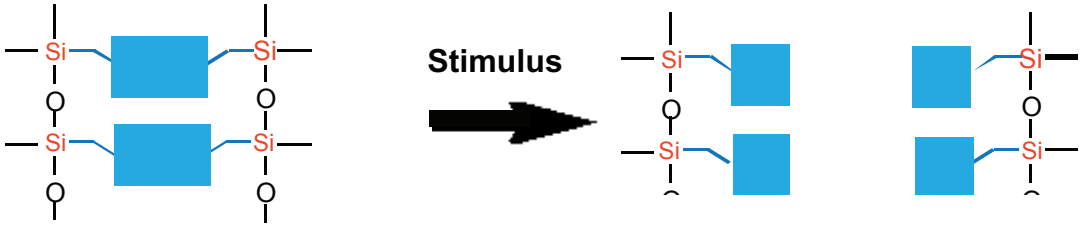
Disulfide Ester



Amide Imine

For peptides (enzymatically cleavable)

Chem. Eu. J. **2016**, *22*, 3697



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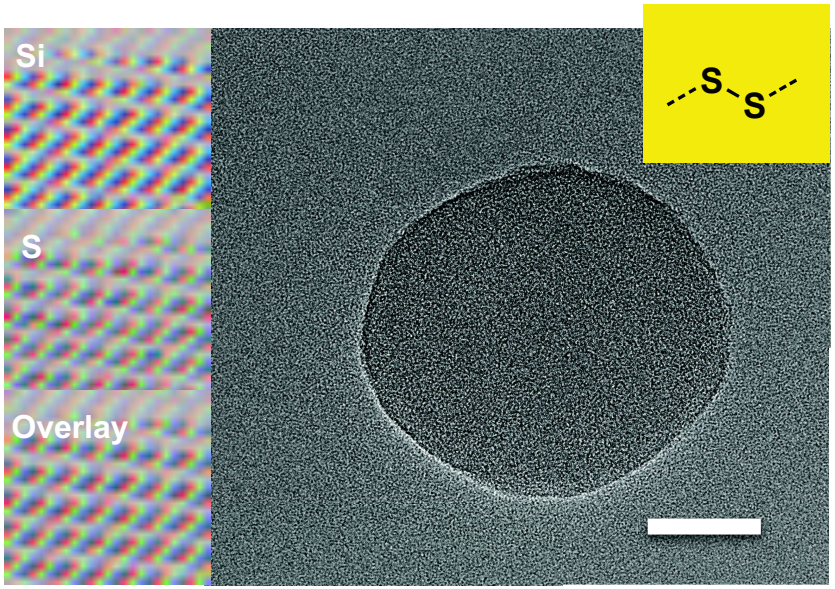
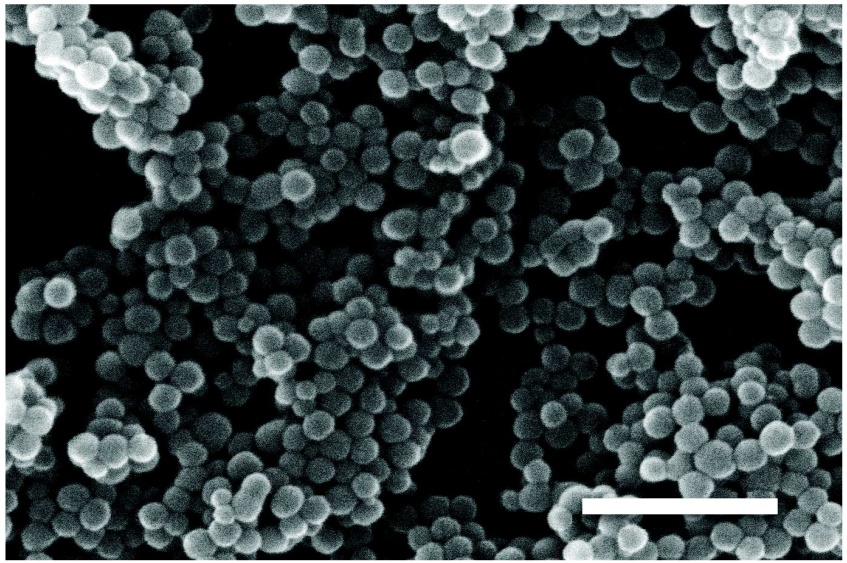
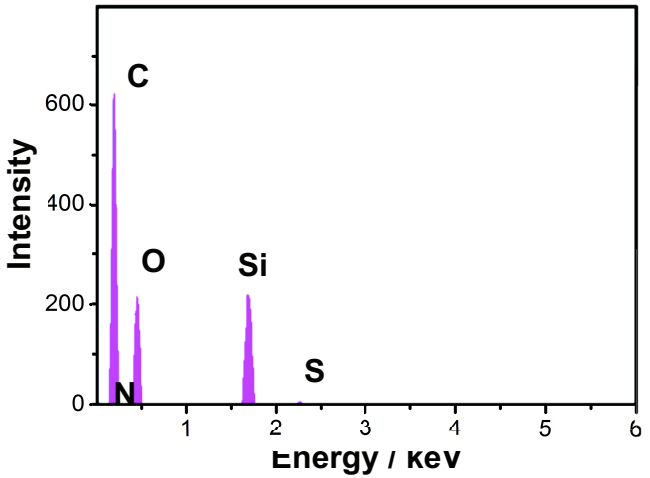
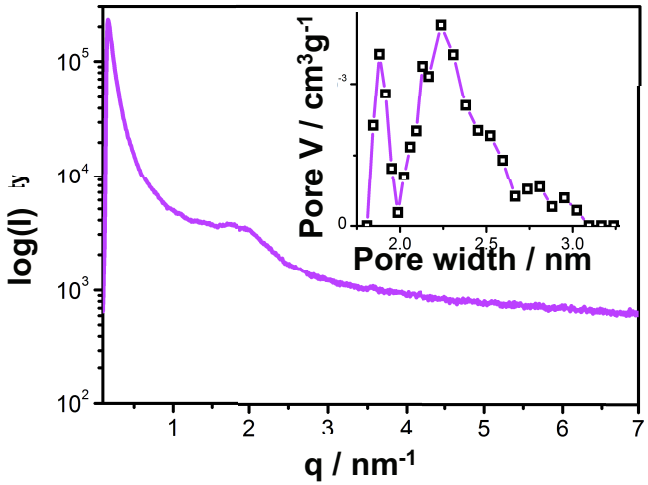
Big particles and characterization



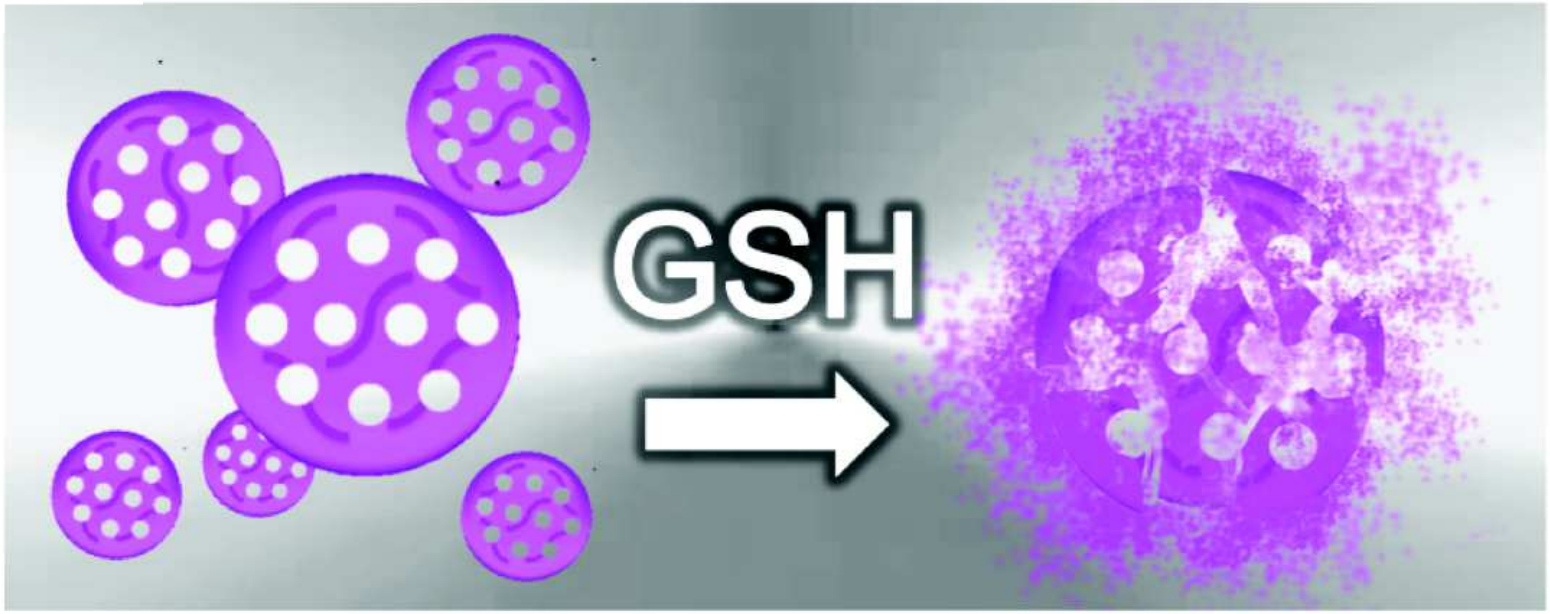


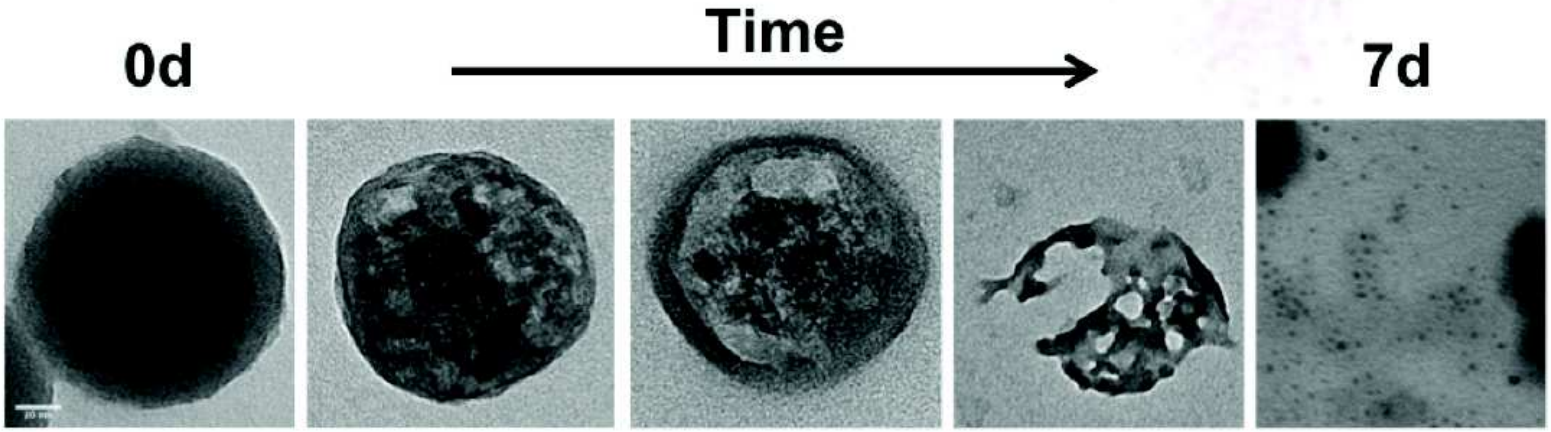




Nanoscale, **2016**, *8*, 7240-7247

Breakable porous containers





L. Maggini et al. *Nanoscale,* **2016**, *8*, 7240-7247

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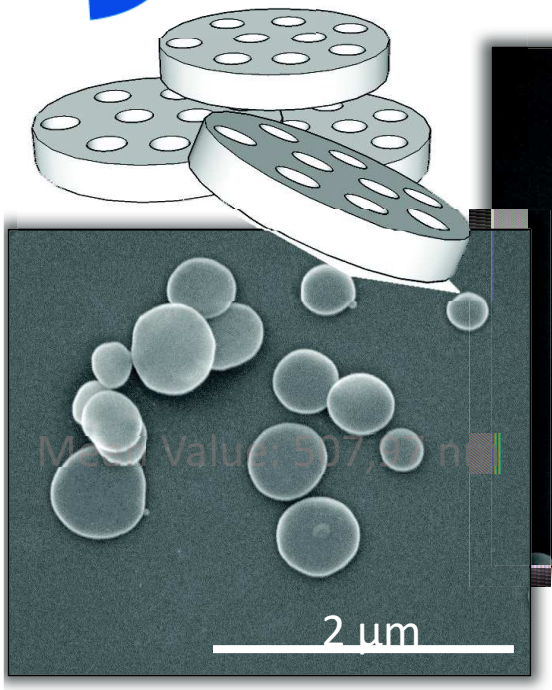
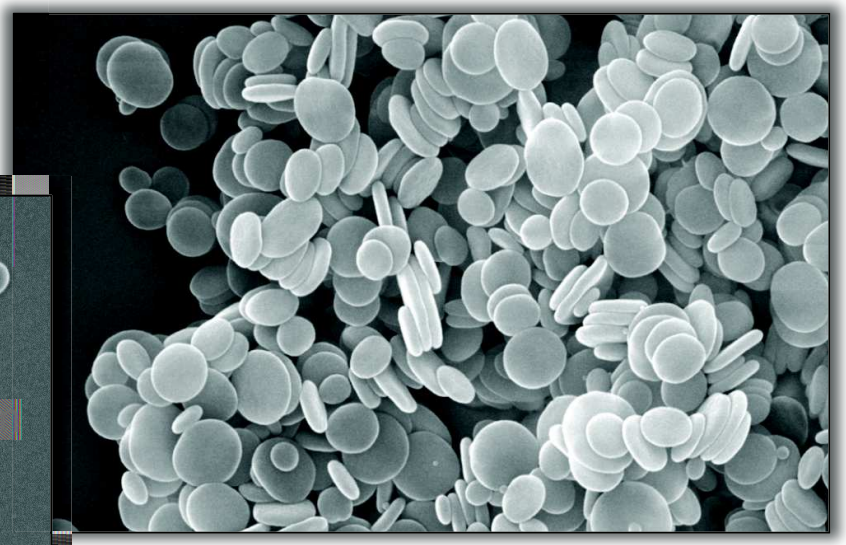
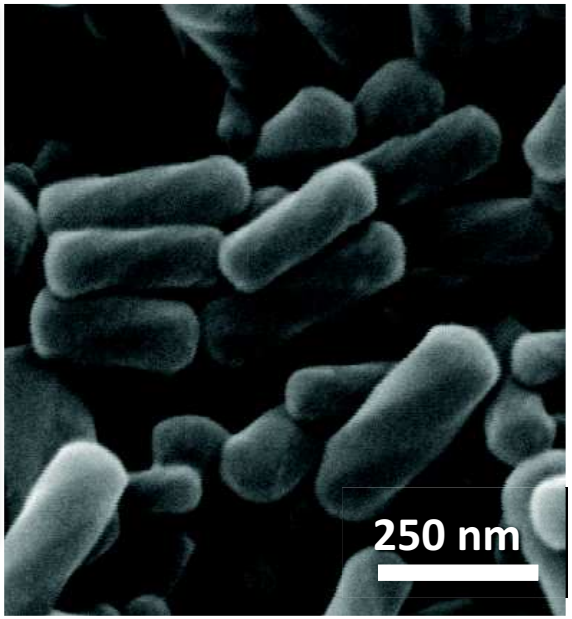
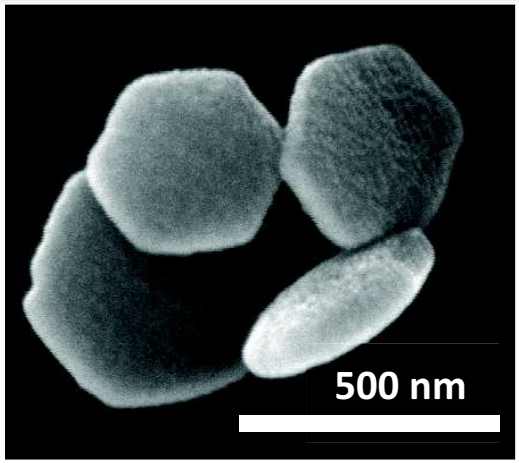
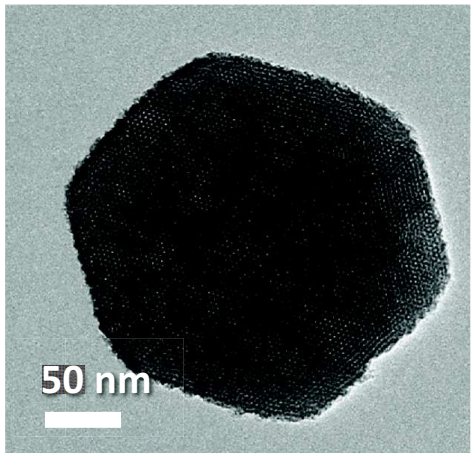
Changing morphology

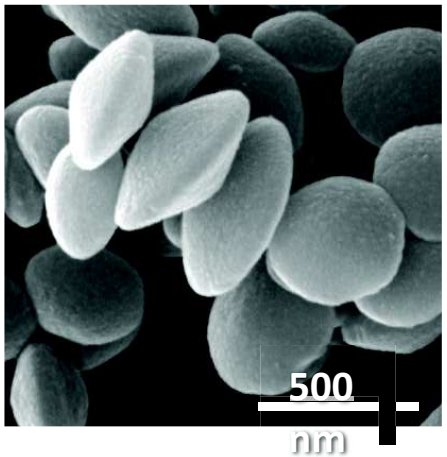


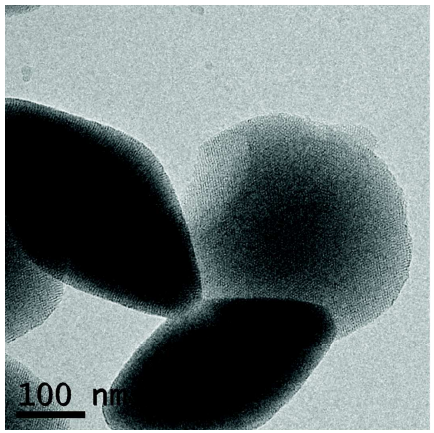












Leana Travaglini and LDC *Chem. Mat*. In press

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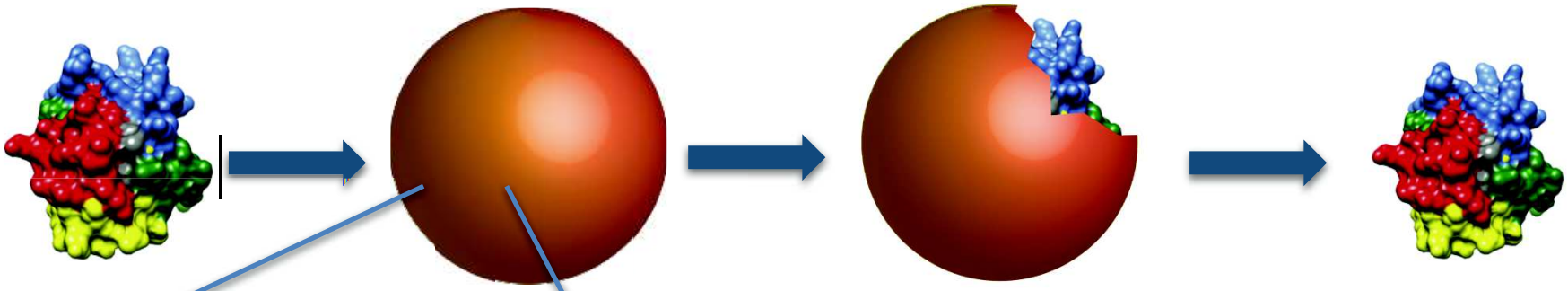
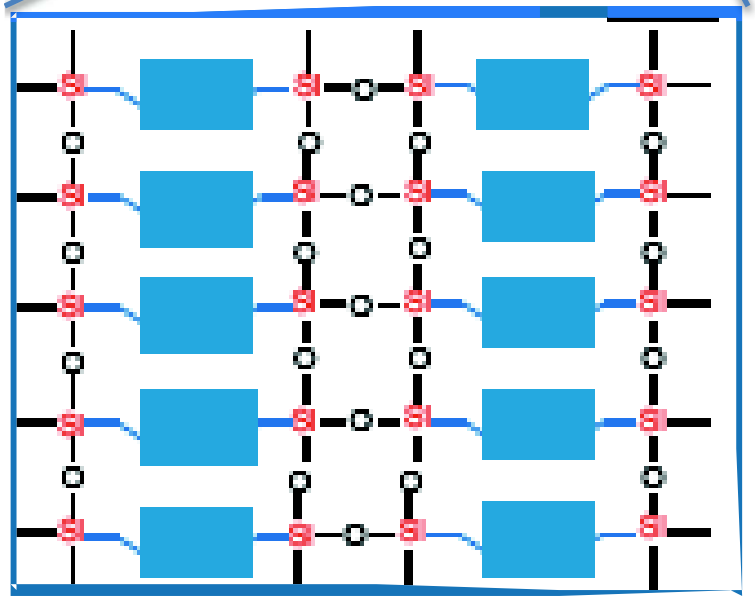
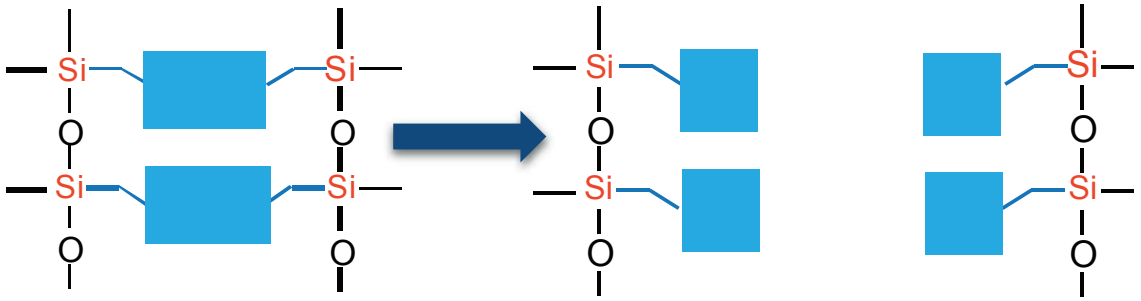
Breakable Shell







R-S-S-R → R-SH HS-R

And many others…

PCT Int. Appl. (2015), WO 2015189402 A1 20151217

E.A. Prasetyanto, A. Bertucci, D. Septiadi, R. Corradini, P. Castro-Hartmann, L. De Cola *Angew. Chem. Int. Ed.* **2016**, 55, 3323–3327

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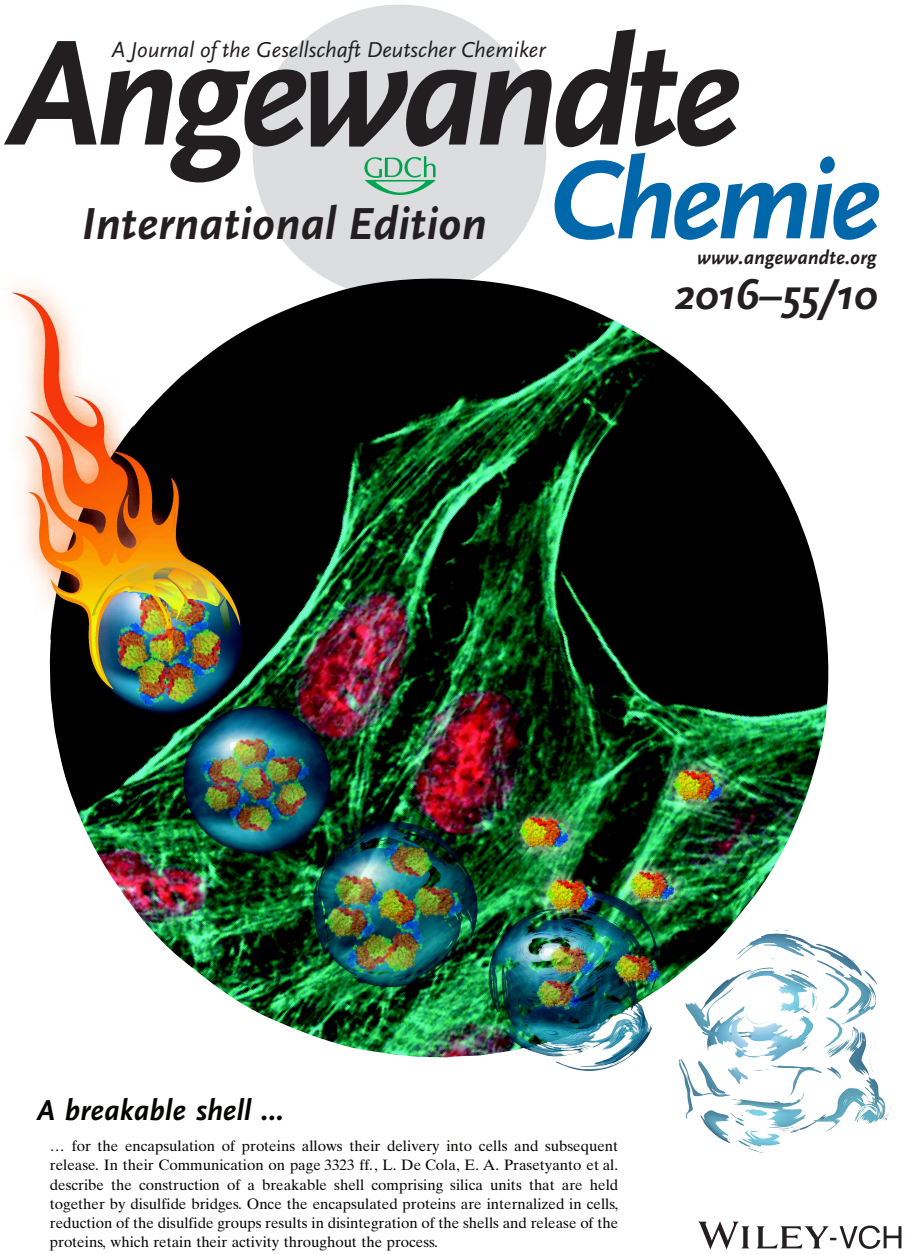
Protein and enzyme delivery

Eko Prasetyanto

Angew. Chem. Int. Ed.
2016, 55, 3323–3327



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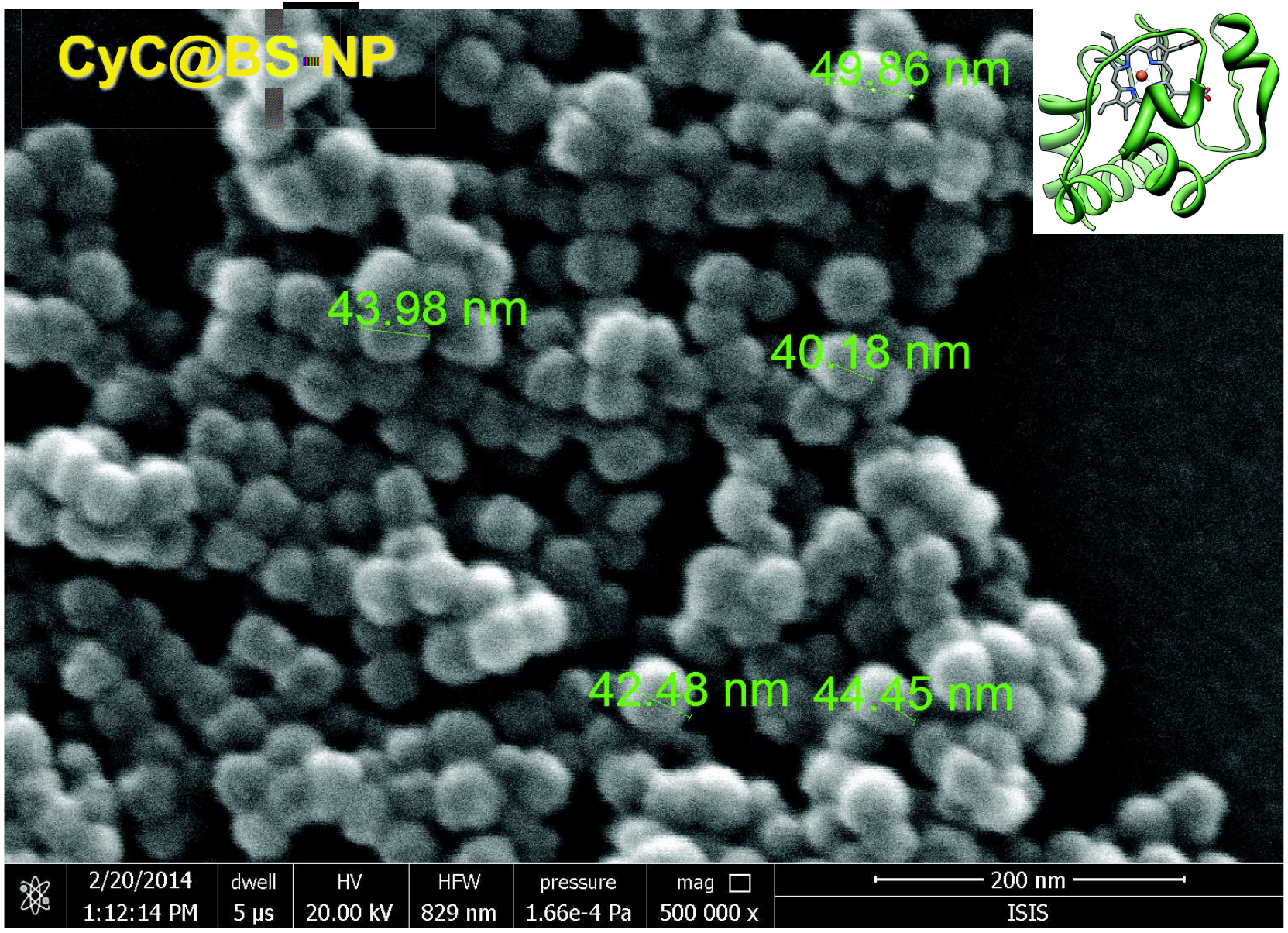
Breakable Shell



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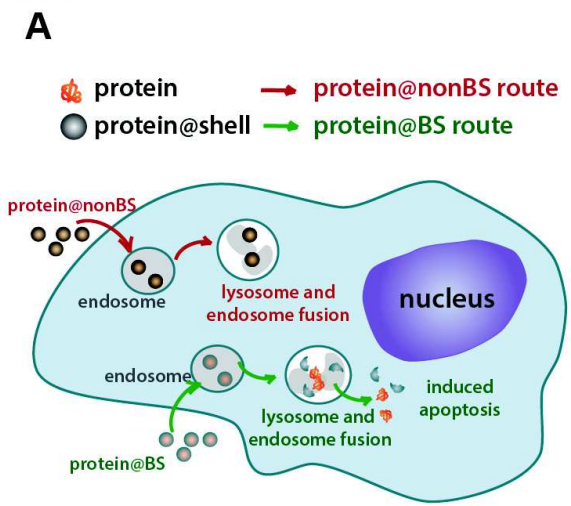
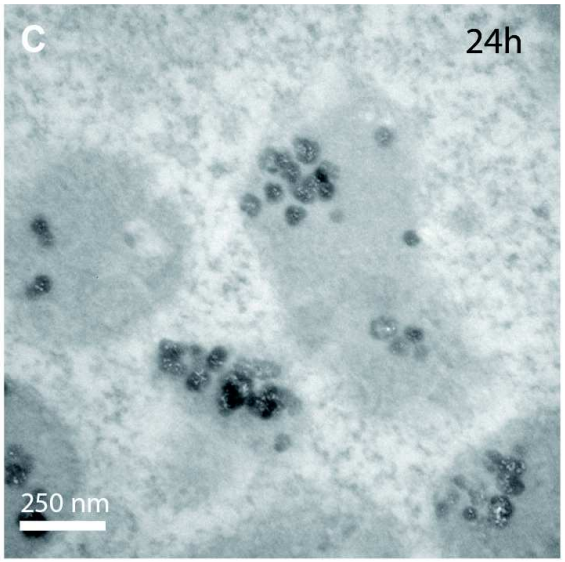
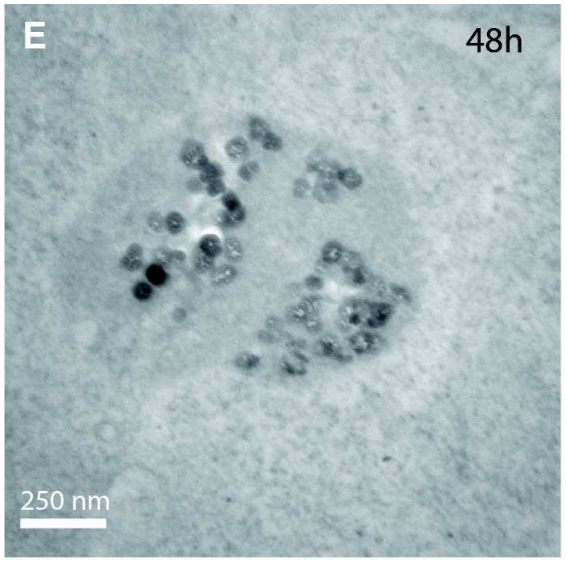
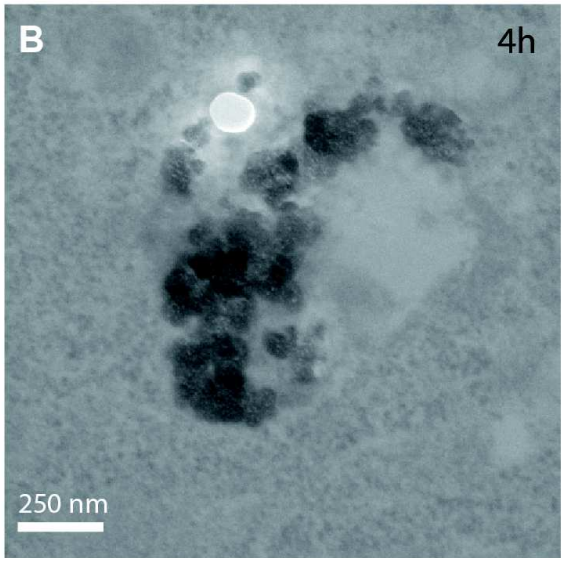
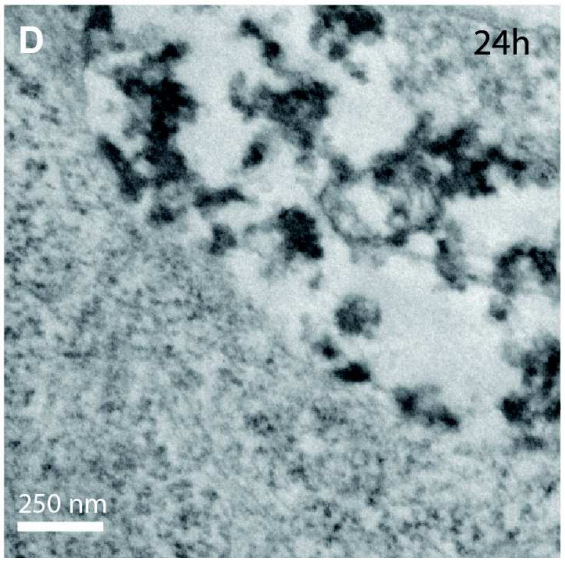
In Vitro Breakability

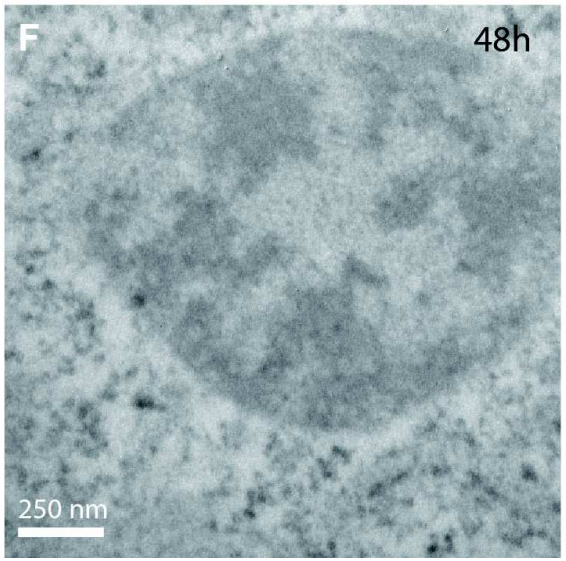






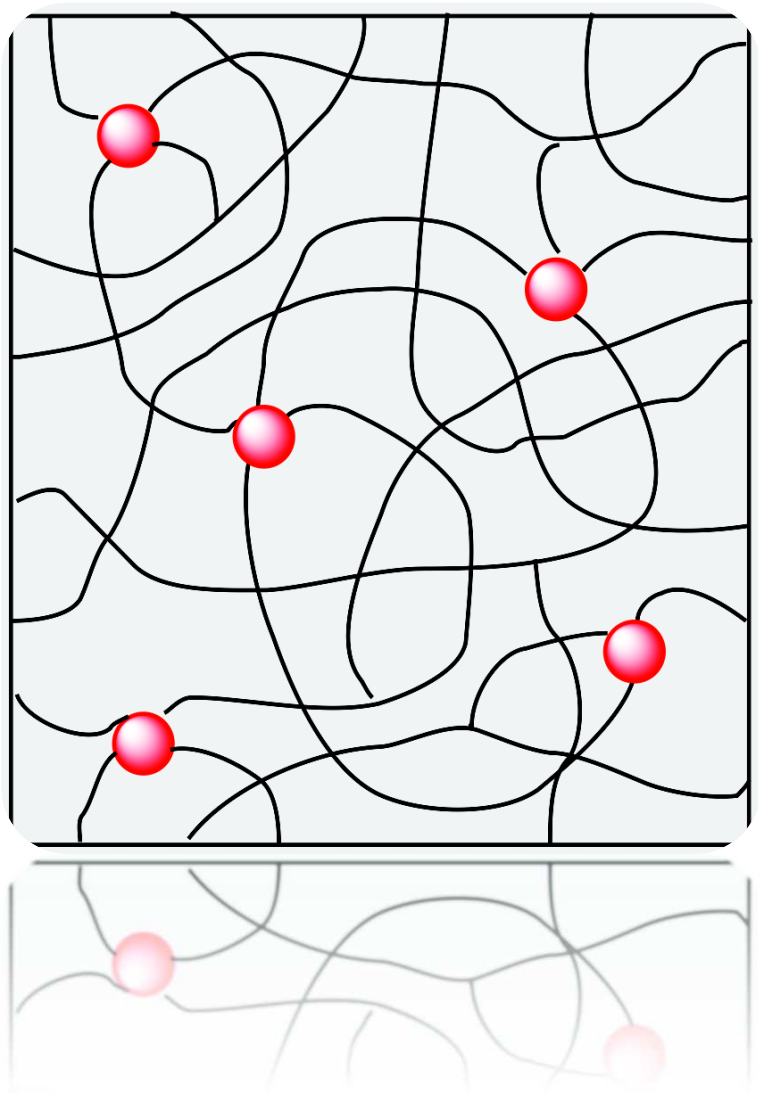






Angew. Chem. Int. Ed. **2016**, 55, 3323–3327

Nanoparticles as components for hydrogels



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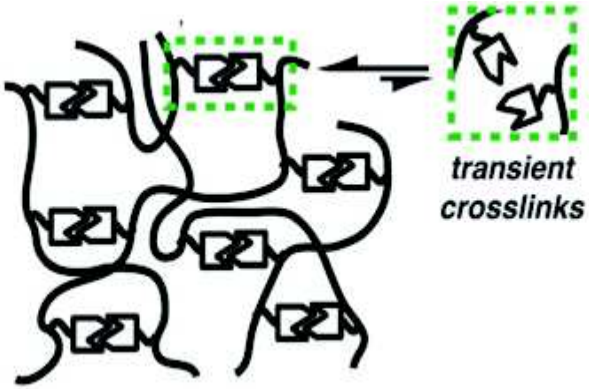
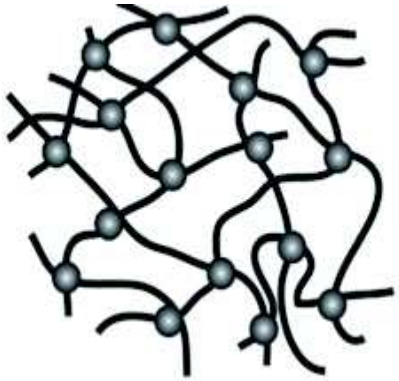
Hydrogels

Physical cross-linking



Chemical cross-linking



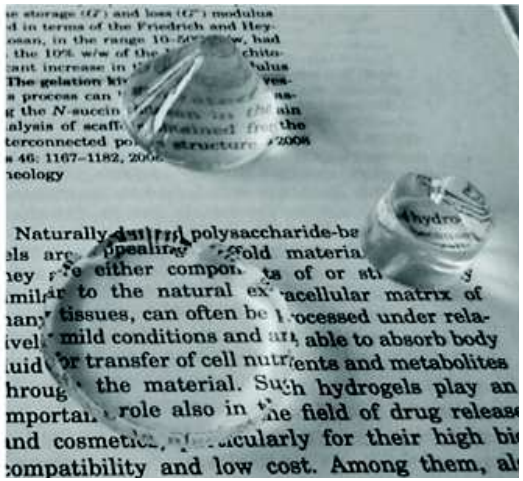
3D networks composed of cross-linked hydrophilic polymer chains

from *Chem. Soc. Rev.*, **2012**, *41*, 6195

hydrophilic character

biocompatibility

any shape and size



adapted from *Nature*, **2010**, *463*, 339-343
and *Carbohydrate Polymers*, **2015**, *125*, 103-112

O. Wichterle, D. Lím, *Nature,* **1960**, *185*, 117
F. Lim, A. M. Sun, *Science,* **1980**, *210*, 908

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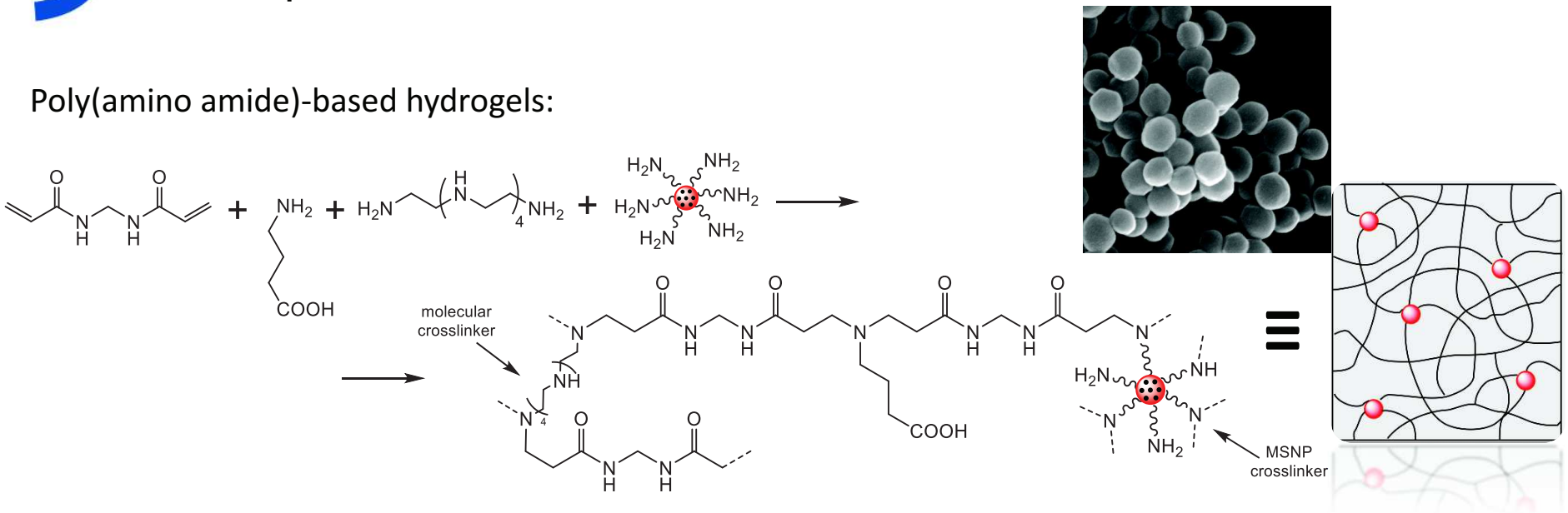
Synthesis of MSN-hydrogels

Poly(amino amide)-based hydrogels:



Reaction that proceed in water

At body temperature

Very fast and quantitative

No catalyst or external factors

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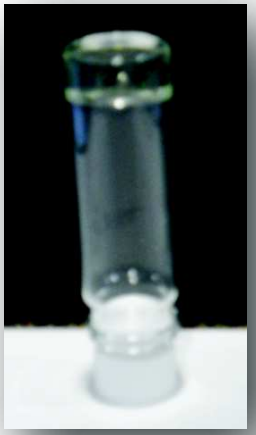
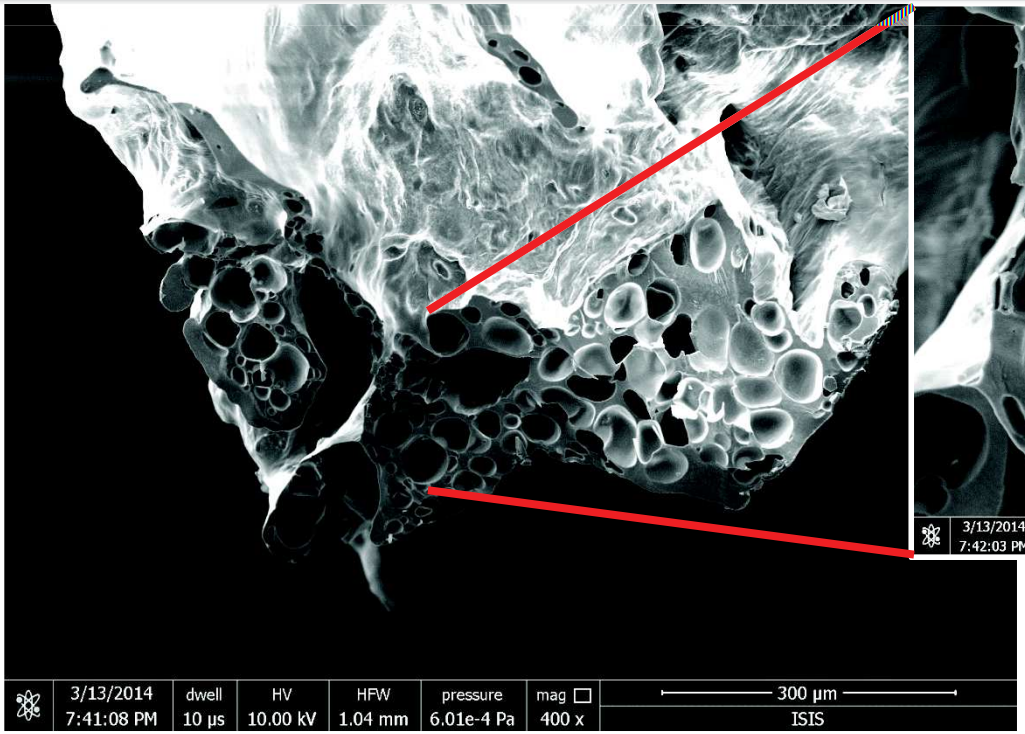
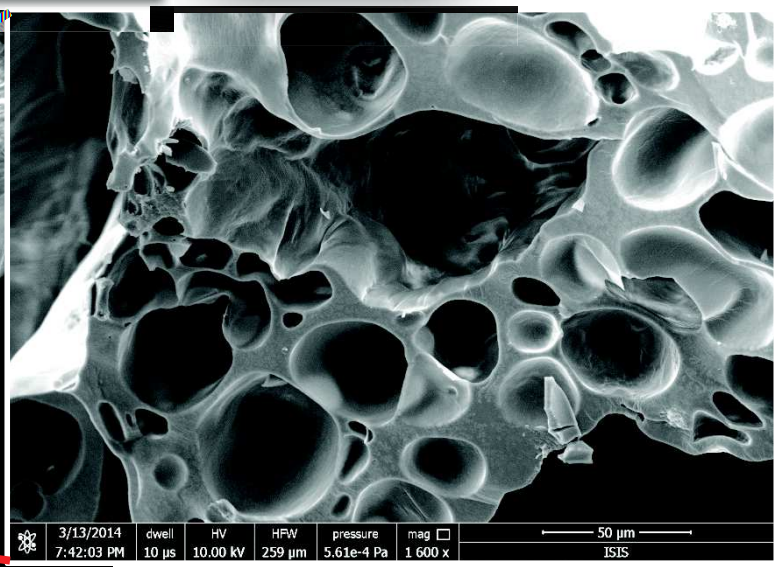
MSN-hydrogel

- ✓ Reversible swelling/collapsing behavior
- ✓ % H_2O = 93% (weighting method)
- ✓ Self-healing ability even after 15 days
- ✓ Very elastic thanks to the presence of MSNs



inverse test tube method





Size of the pores ranging from 30 to 80 µm

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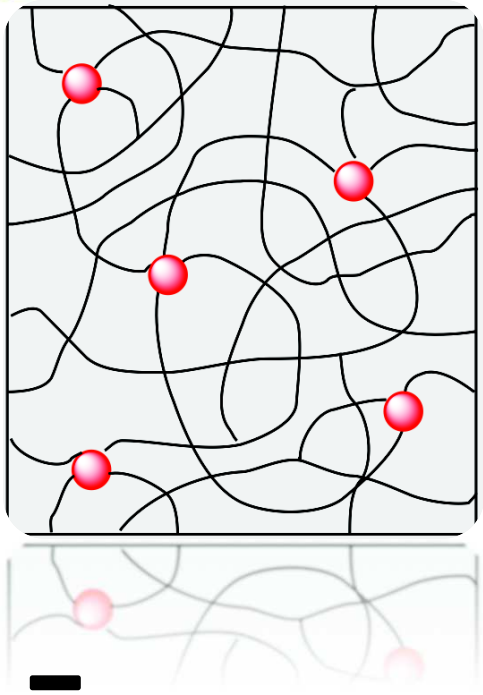
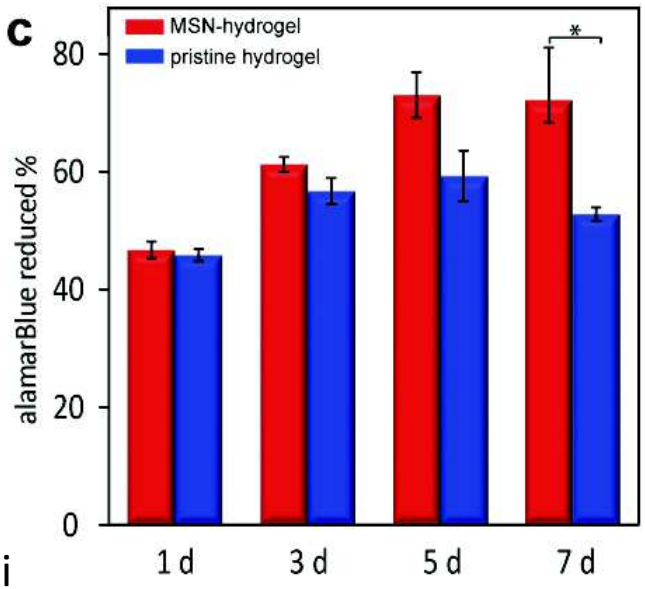
MSN-hydrogel

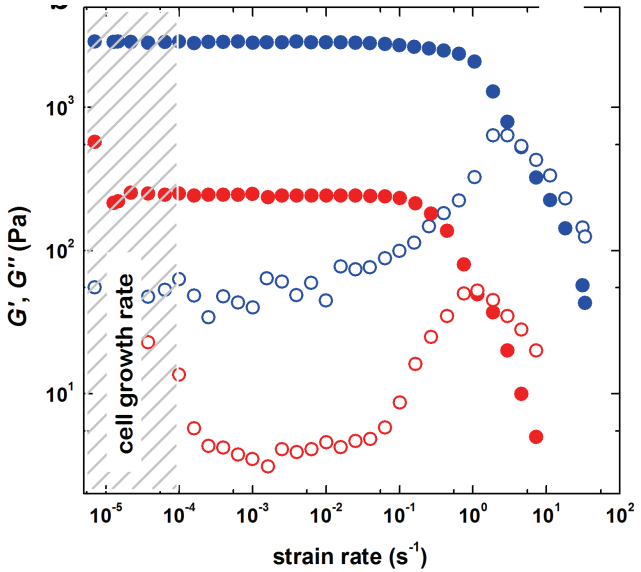


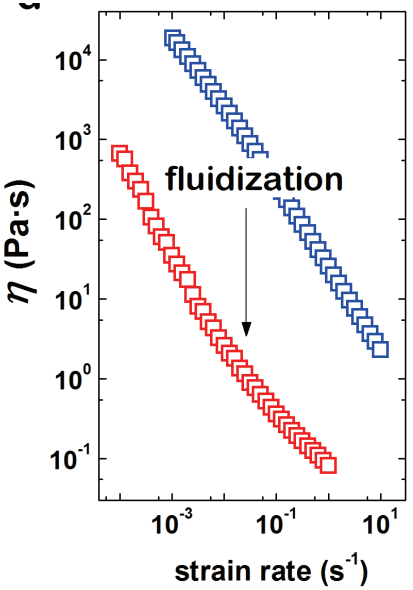
Giuseppe Alonci



MSN-hydrogel is more deformable and fluid than pristine hydrogel

Rheologycal characterization performed by I. Lopez-Montero





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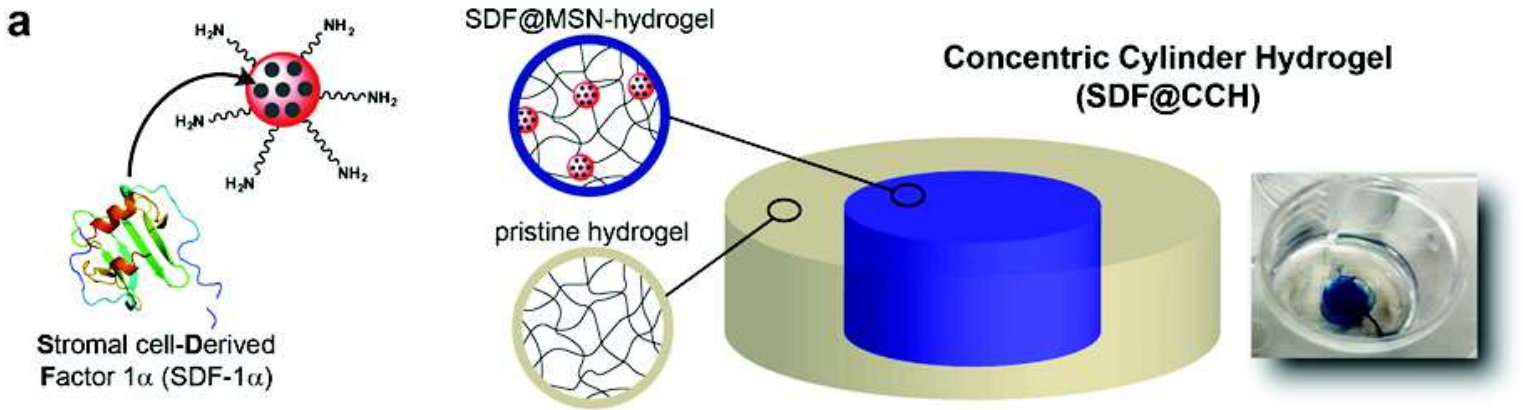
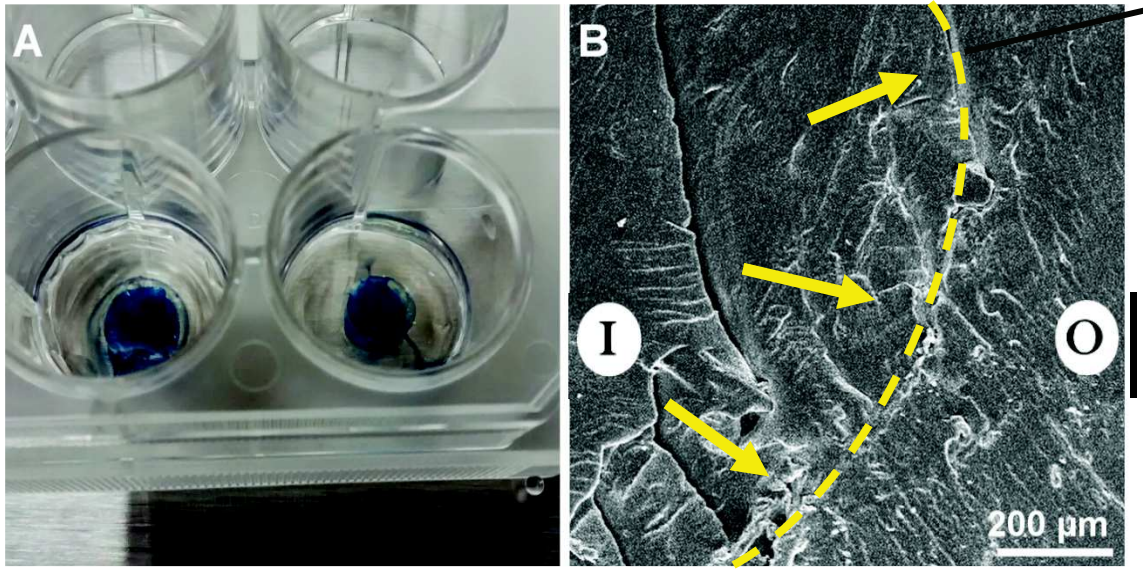
Concentric Cylinders Hydrogel (CCH)





Interface
between
inner core (I)
and
outer layer (O)

F. Fiorini in collaboration with Dr. E. Tasciotti Methodist Hospital Houston

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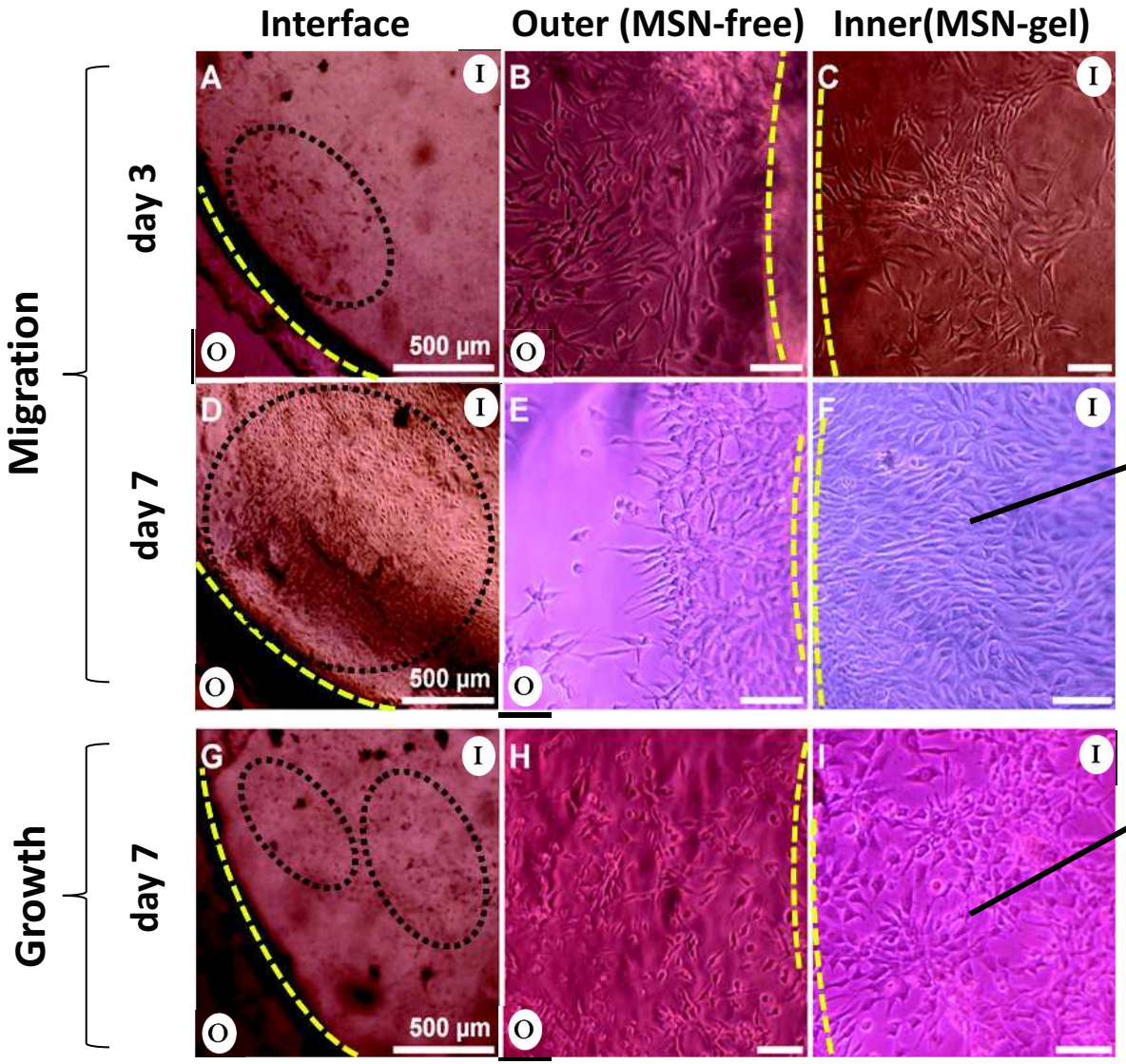
Cells migration in CCH



Aligned towards the Inner core releasing SDF-1α

Colony formation

Scale bar is 50 µm where non marked.

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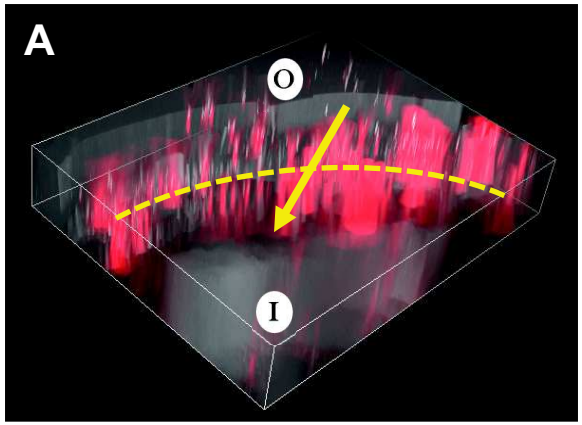
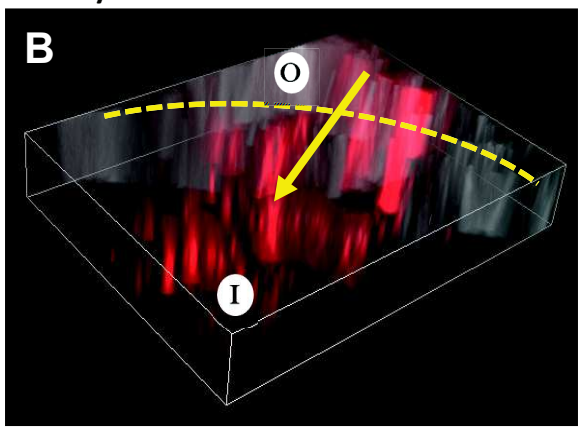
Cells migration in CCH

day 2

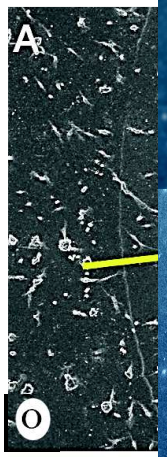


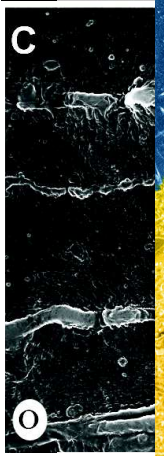
day 5



Width: 700 µm; Height: 525µm; Depth: 102µm

SEM im





F. Fiorini et al. *Small,* **2016**, *12*, 488

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Injectable through a syringe

In collaboration with Silvana Perretta group IRCAD, Strasbourg

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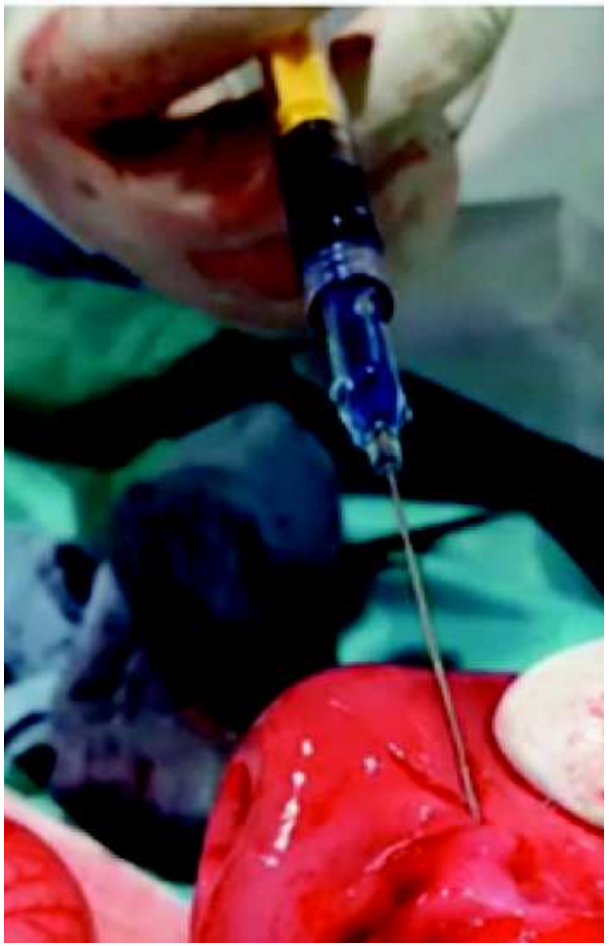
Hydrogel in tissue



Injection of the pre-hydrogel solution between the mucous and the serous membrane of an explanted pig's stomach.

Federica Fiorini and Pietro Riva

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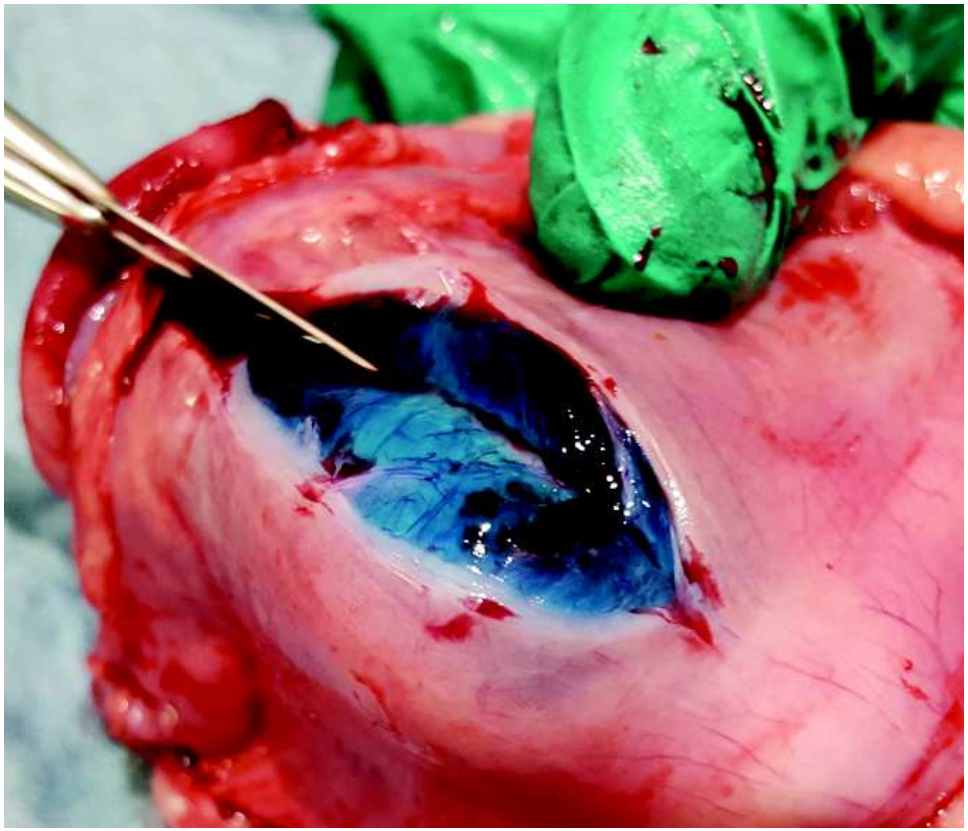
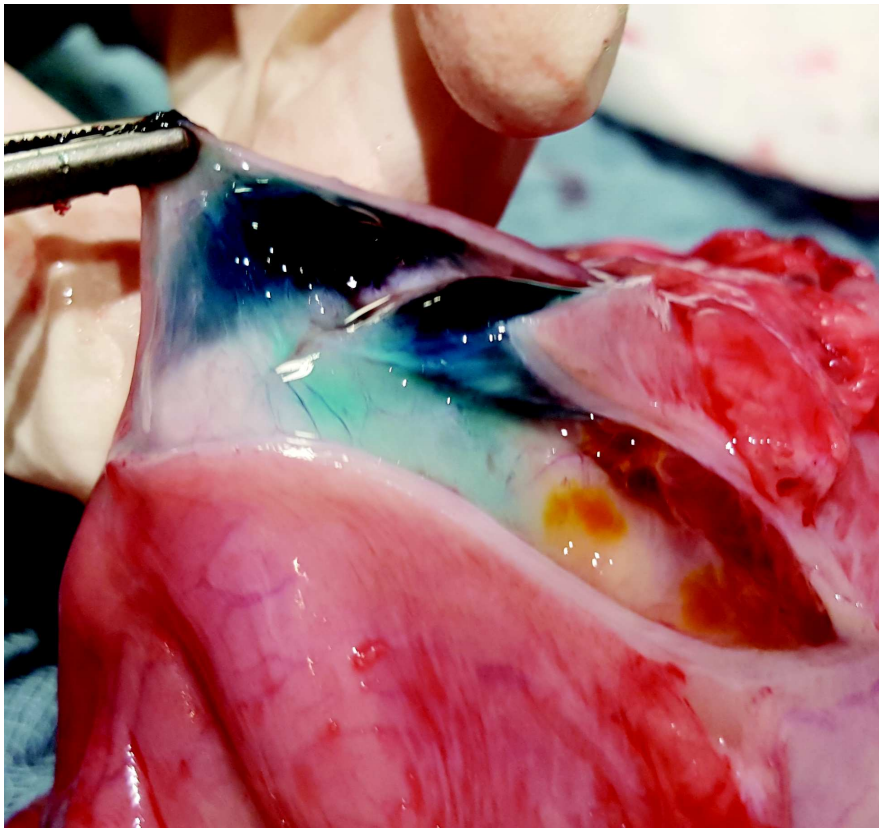
Supramoléculaires

Hydrogel in tissue

After about 5 minutes the tissue was cut opened, to expose the different layers. The hydrogel was perfectly gelified and integrated with all the anatomic structures





In collaboration with IRCAD and IHU, Dr. Silvana Perretta

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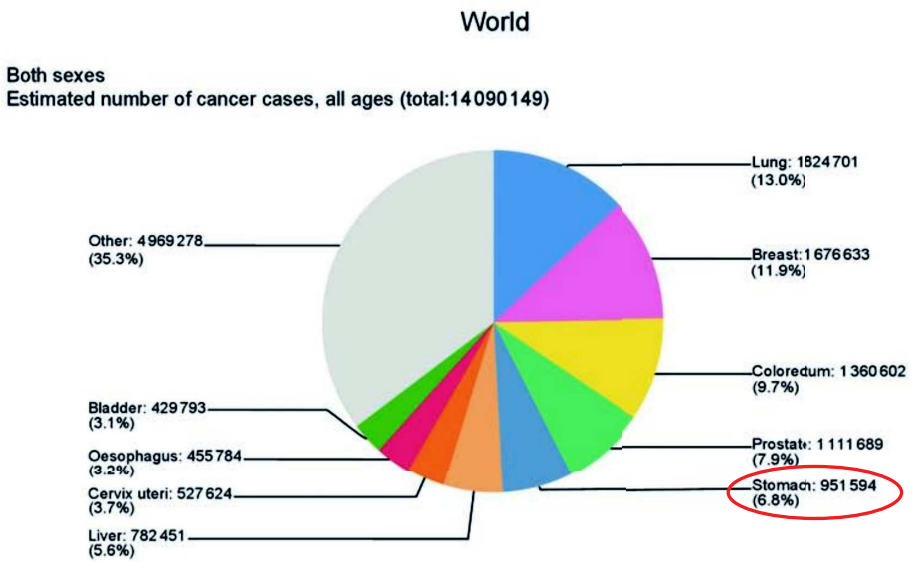
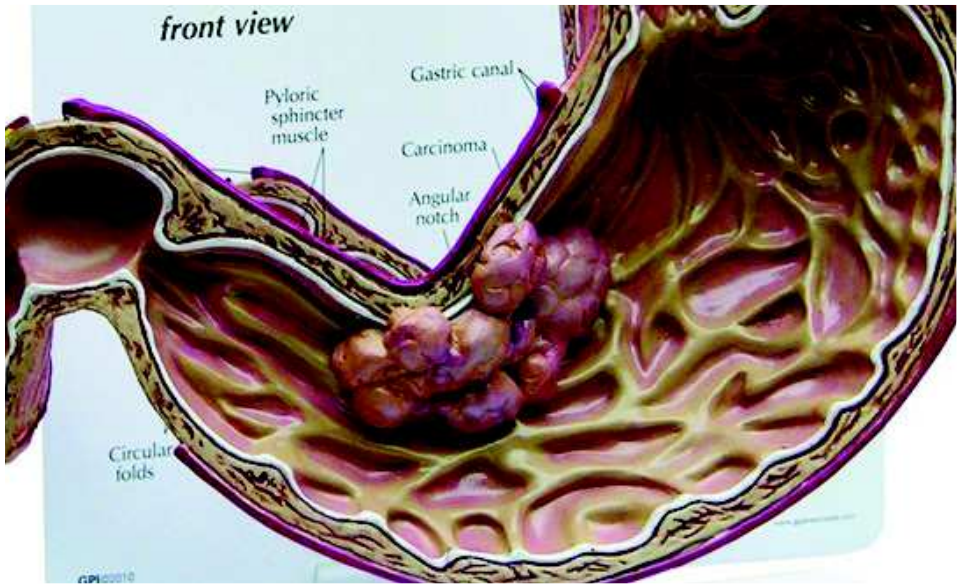
Hydrogels for Tumor Surgery





Canadian Scientific Journal 2 (2014)
Comparative survival analysis of patients with stomach cancer after combined surgery

Stomach cancer is the **5th** most common in the world
and the **3rd** for mortality

IARC, World Cancer Report 2014

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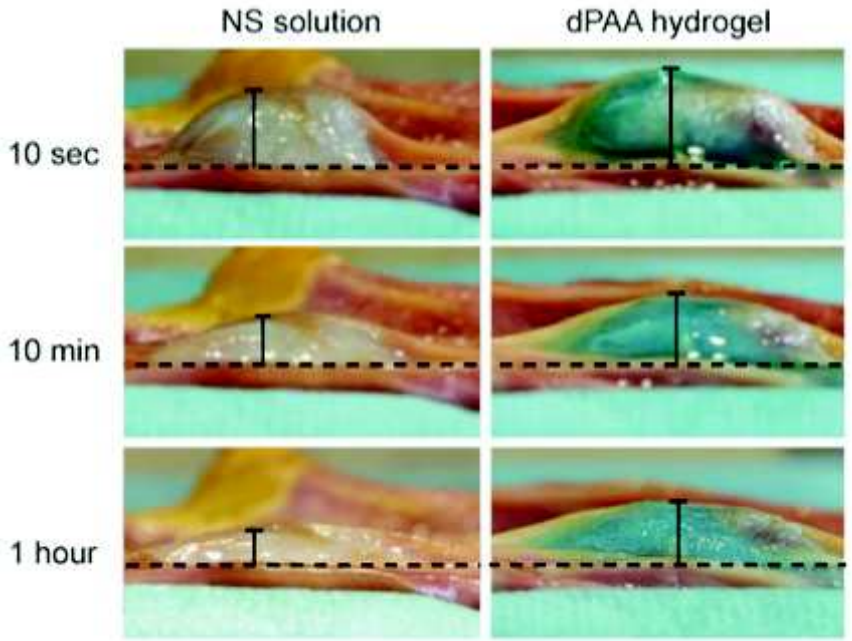
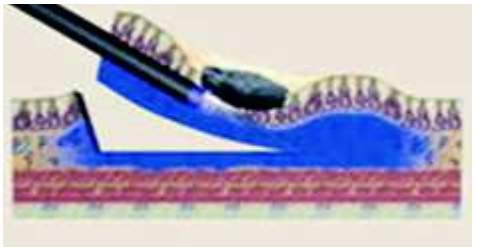
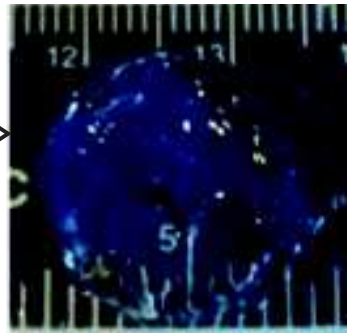
In vivo applications



Degradable hydrogels able to release drugs

Biodegradation
and release





Endoscopic
Submucosal
Dissection

Submucosal fluid cushion (SFC)

- ✓ Formation of a solid SFC
- ✓ Significant mucosal lifting
- ✓ No change in shape and consistency
- ✓ Long-lasting and reliable SFC

L. De Cola, G. Alonci, F. Fiorini, P. Riva, S. Perretta Int Patent filed January 2017
Manuscript submitted

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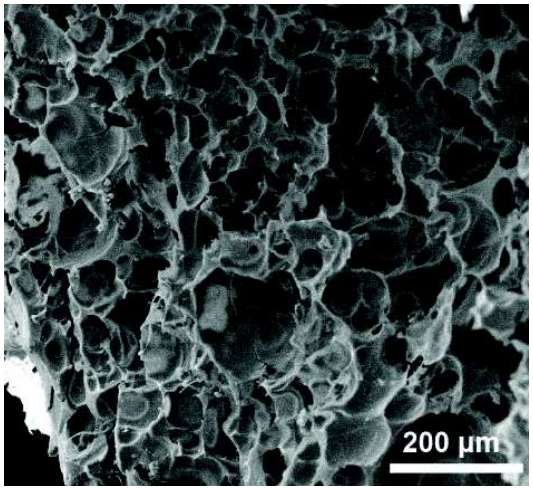
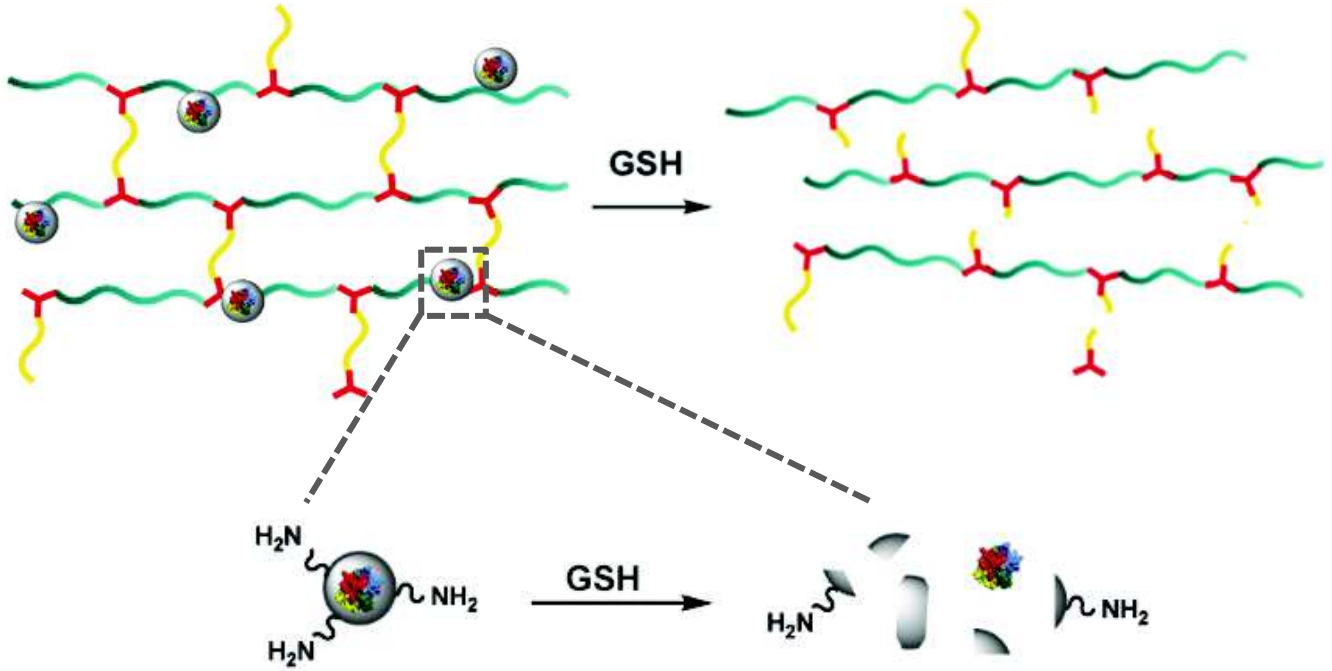
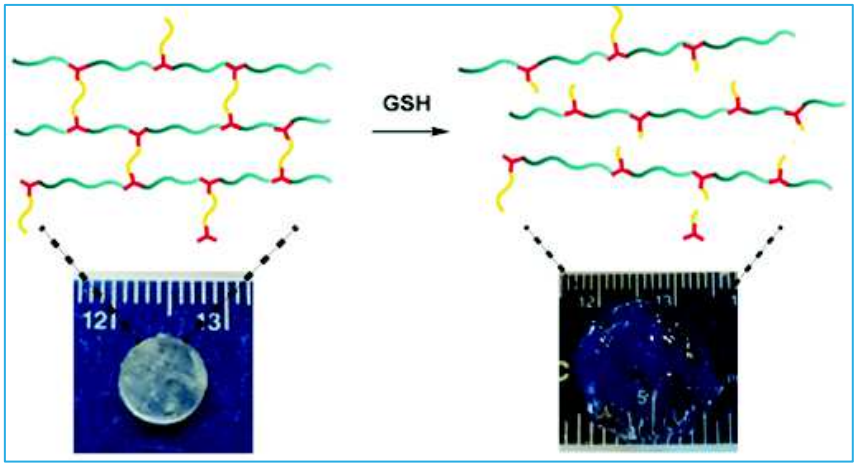
Degradable hybrid hydrogels







Angew. Chem. Int. Ed. **2016**, *55*, 3323

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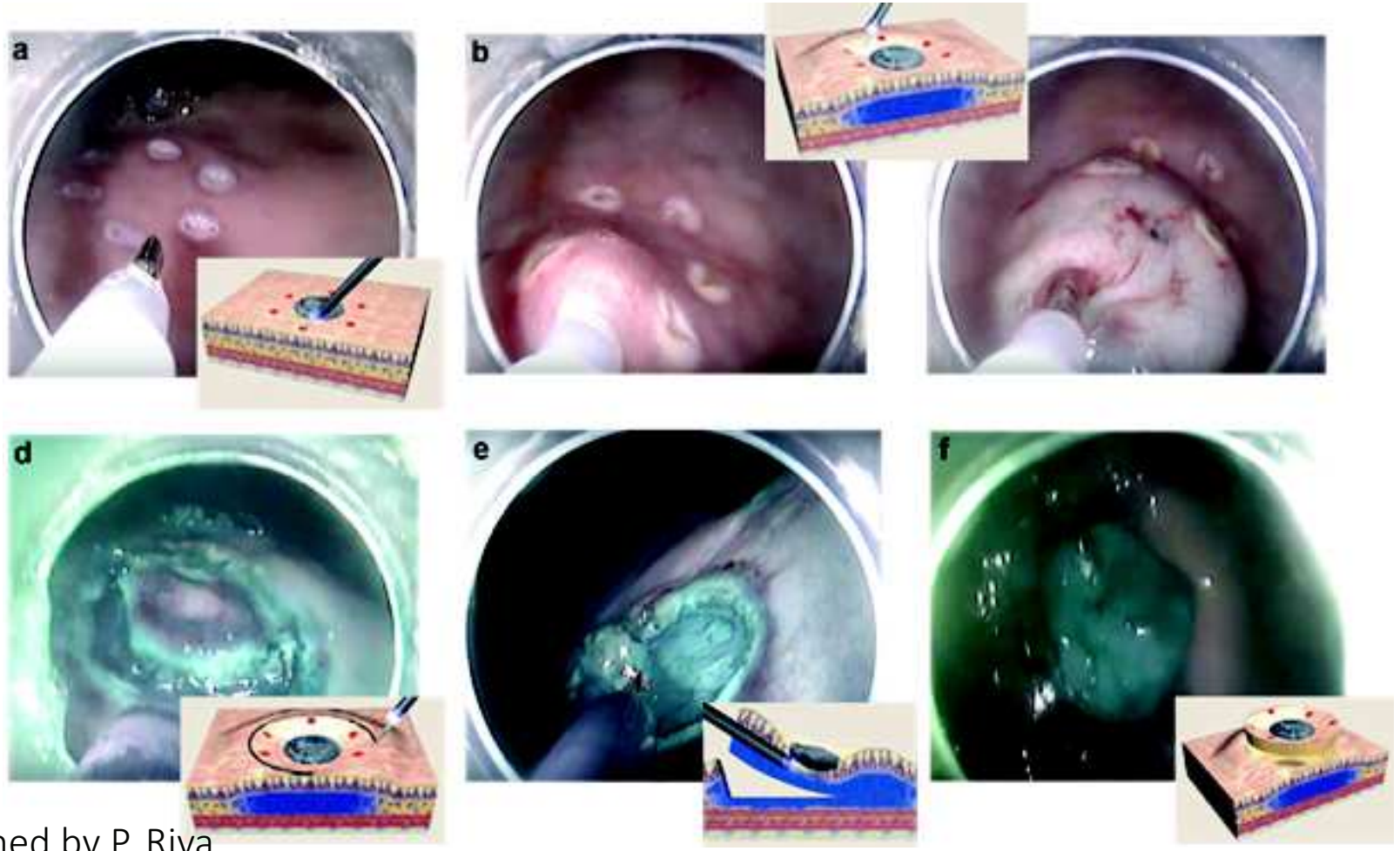
Endoscopic submucosal dissection *in vivo*

- ✓ Clear and stable mucosal elevation
- ✓ Use of electrocautery tools
- ✓ Protecting layer of hydrogel under the resected mucosa: potential release of antibiotics



ESD performed by P. Riva

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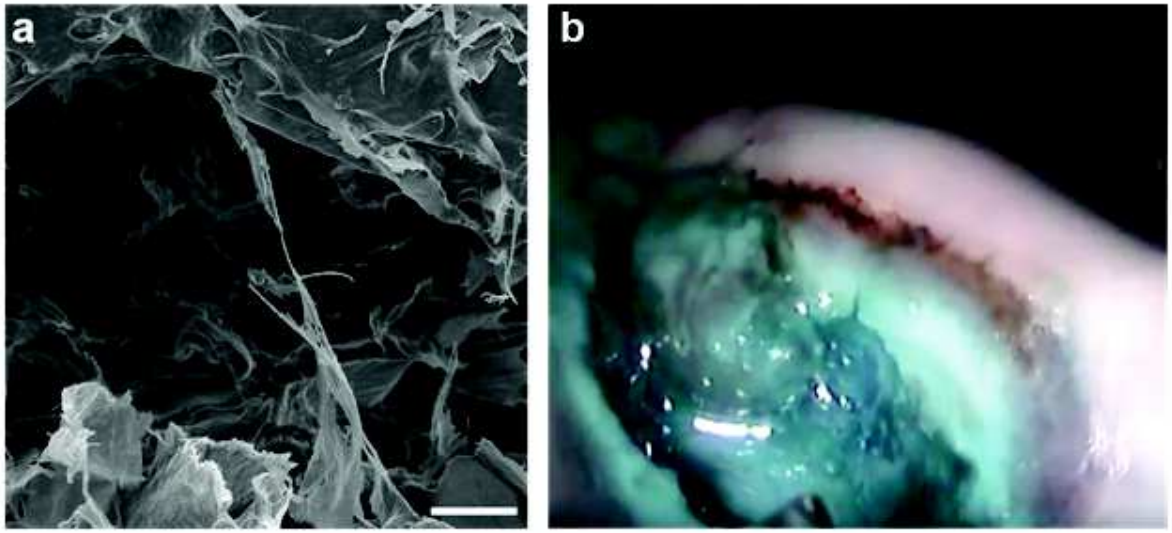
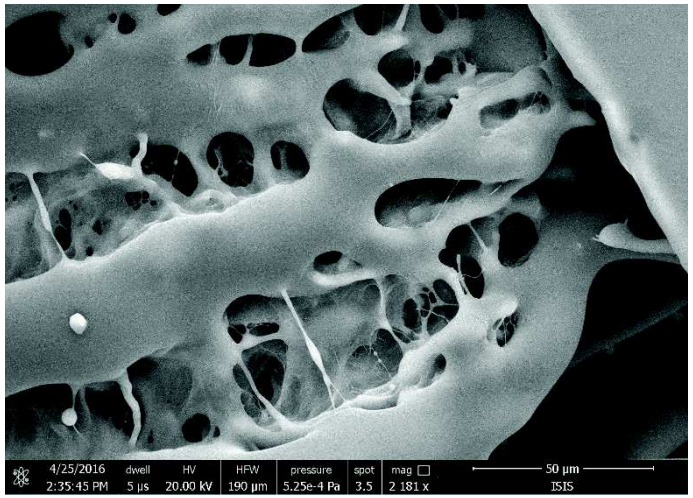
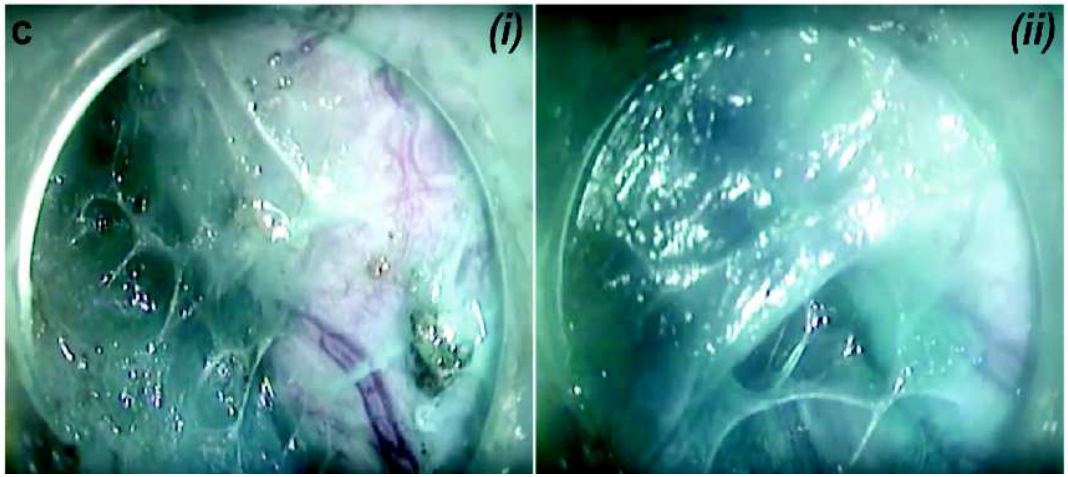
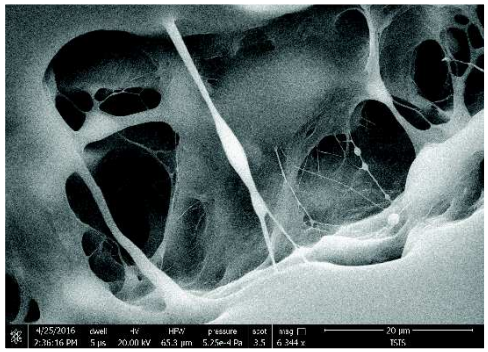
Injectability and gelation time *in vivo*



Approx. 3 min







L. De Cola, F. Fiorini, P. Riva, S. Perretta
Int Patent filed January 2017

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Self-assembled systems

Self-assembled structures for self-assembling proteins…

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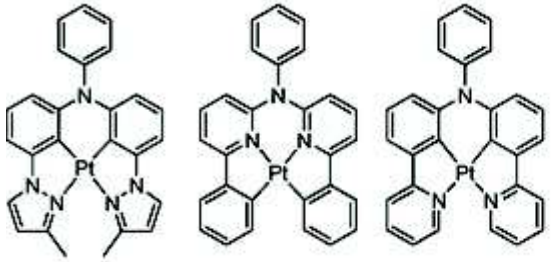
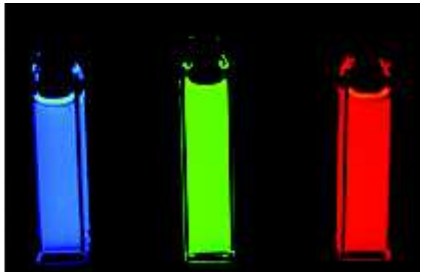
Why platinum(II) complexes?

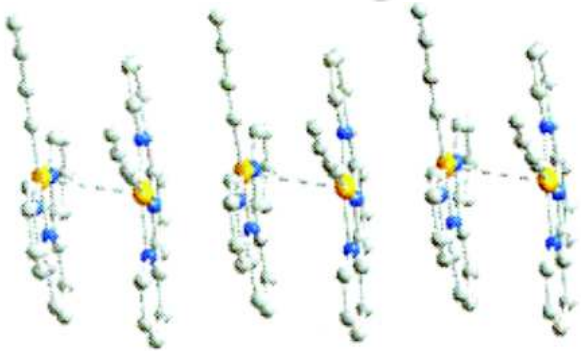
- High emission





S. Huo *Inorg. Chem.*, 2010, **49**, 5107

- Tunable emission color
- Chemical and photochemical stability
- High tendency to stack



VWW Yam *J. Am. Chem. Soc.*, 2002, **124**, 6506

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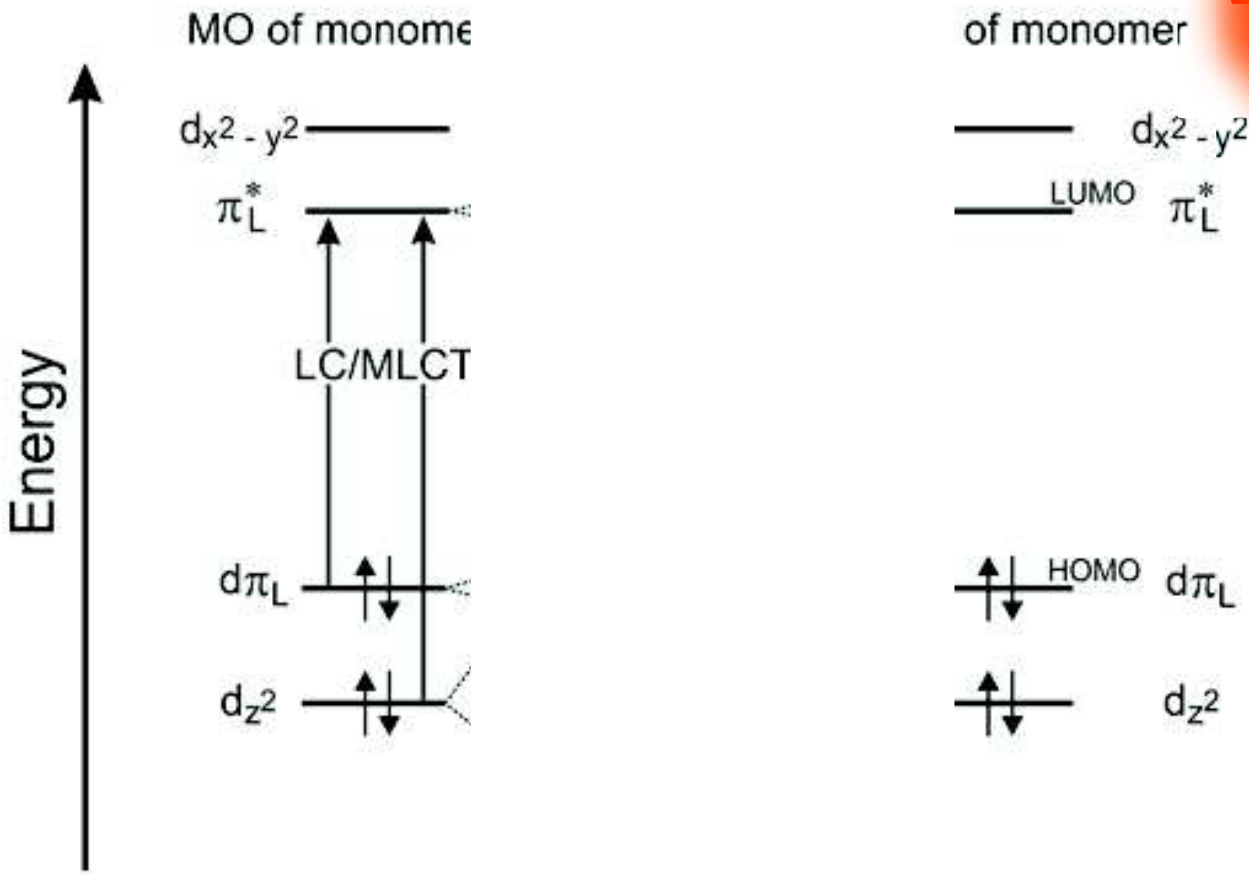
Platinum complexes

Pt(II)

Pt(II)



changes in the
excited state nature

R. Eisenberg, V. Yam,
G. Williams, ME Thompson
C.-M. Che, M. Kato,
D. Bruce, F. Castellano

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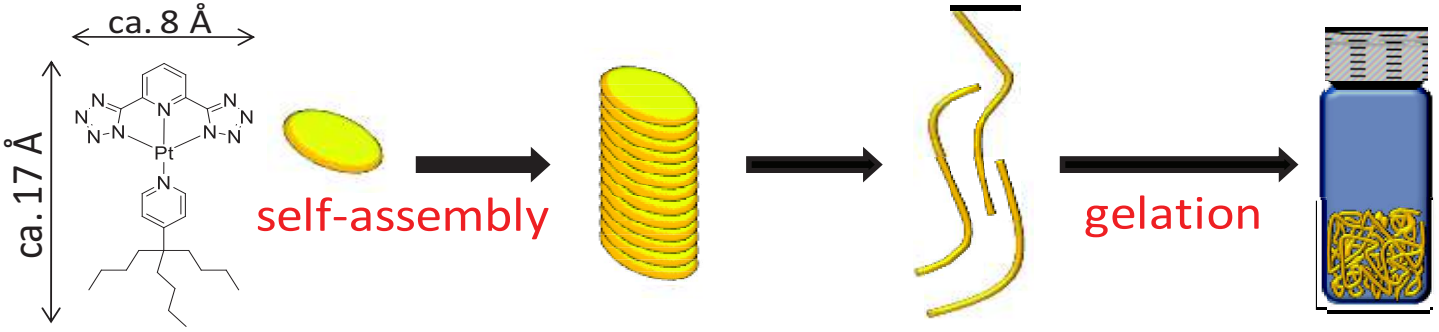
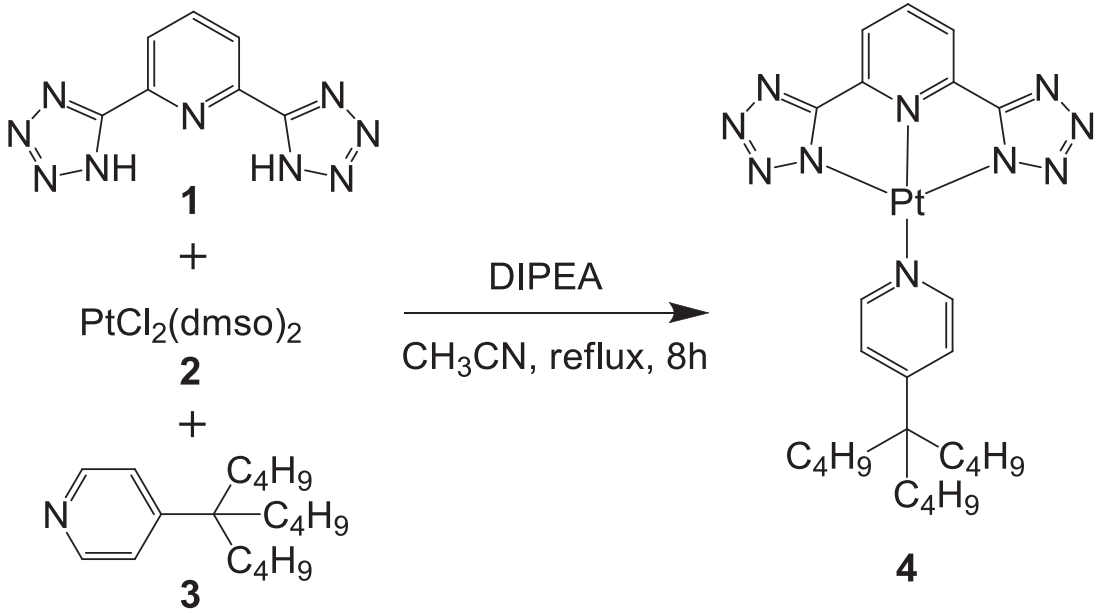
Highly Luminescent Pt(II) Complexes in the solid state





C. A. Strassert, et al. *Angew. Chem. Int. Ed.*, **2011**, *50*, 946 - 950

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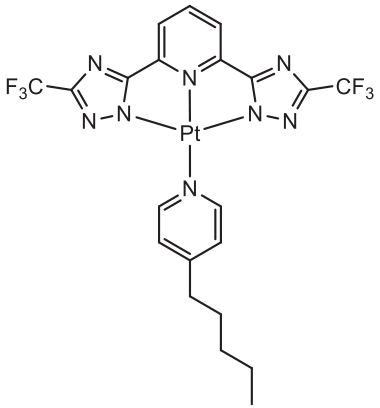
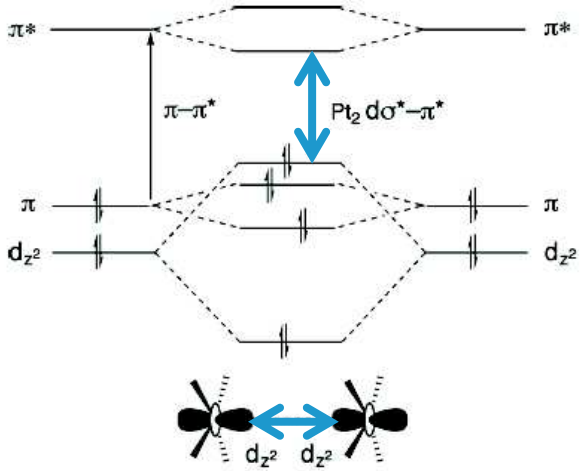
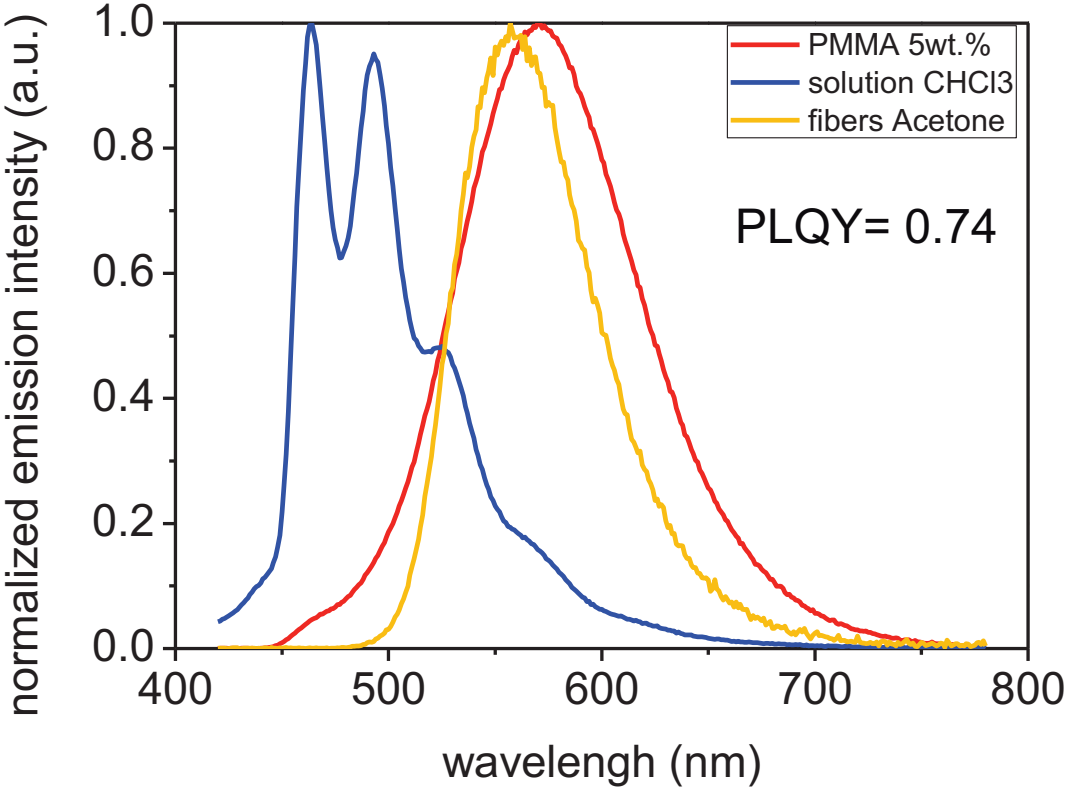
Photophysical characterization of the aggregates







Control of the degree of Pt···Pt

Chem. Commun., **2014**, *50*, 6461

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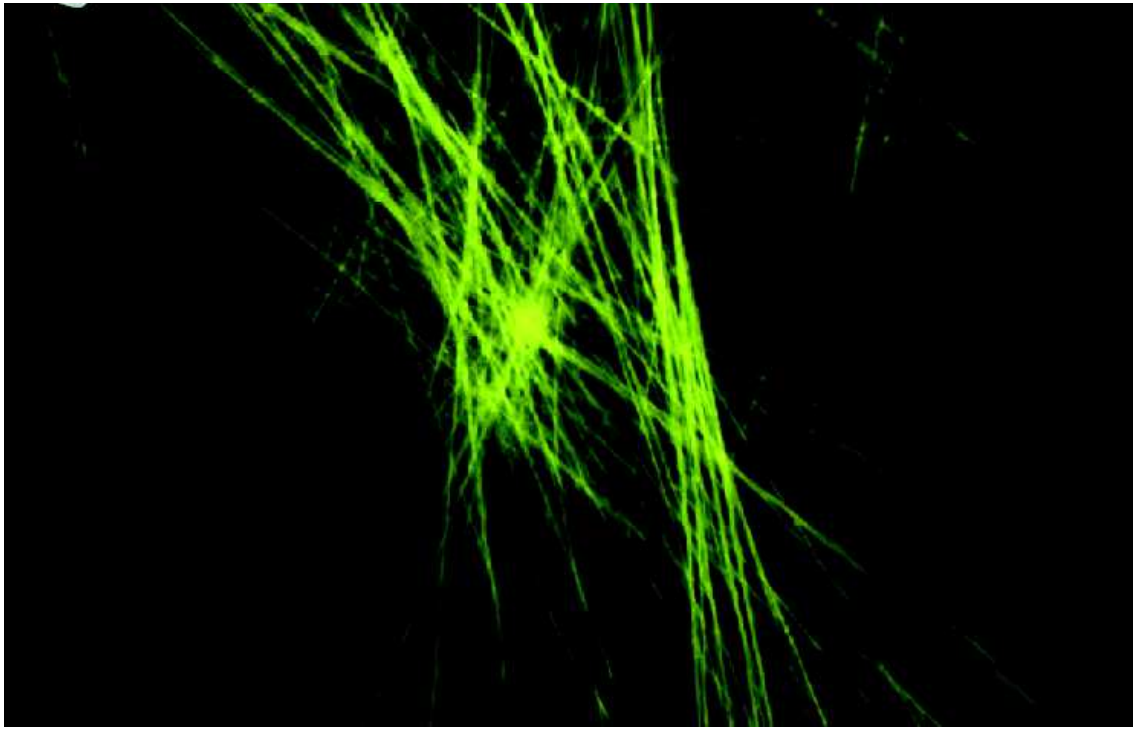
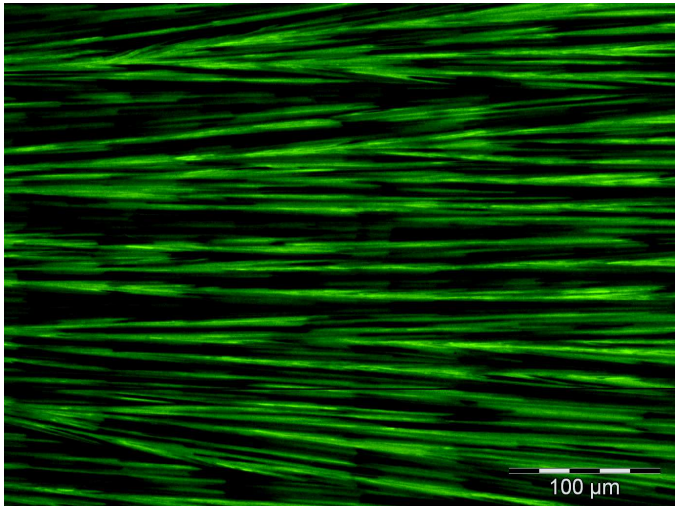
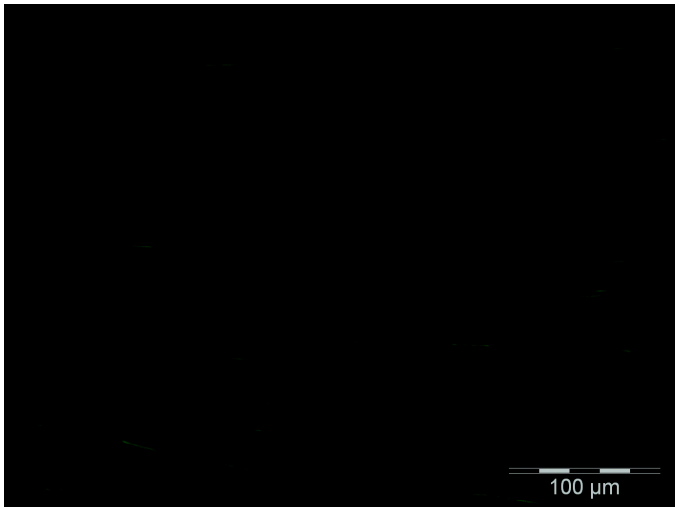
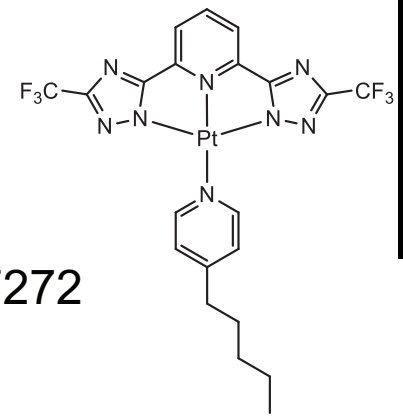
Polarized emission









M. Mauro, L. De Cola et al.
Chem. Commun. **2014**, *50,* 7269-7272

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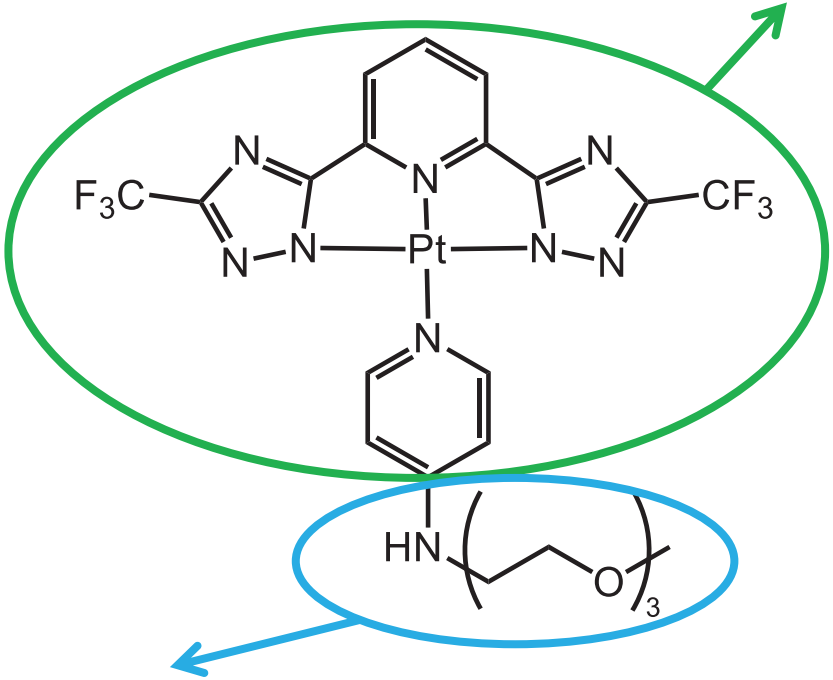
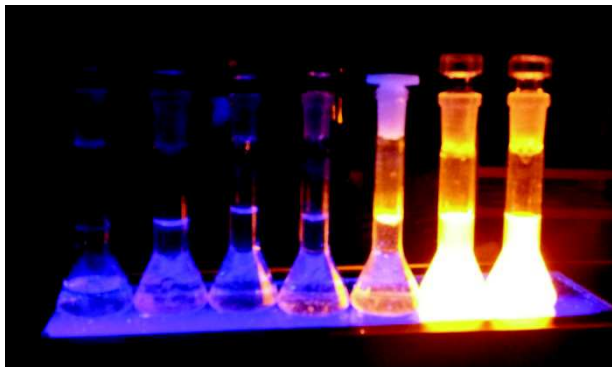
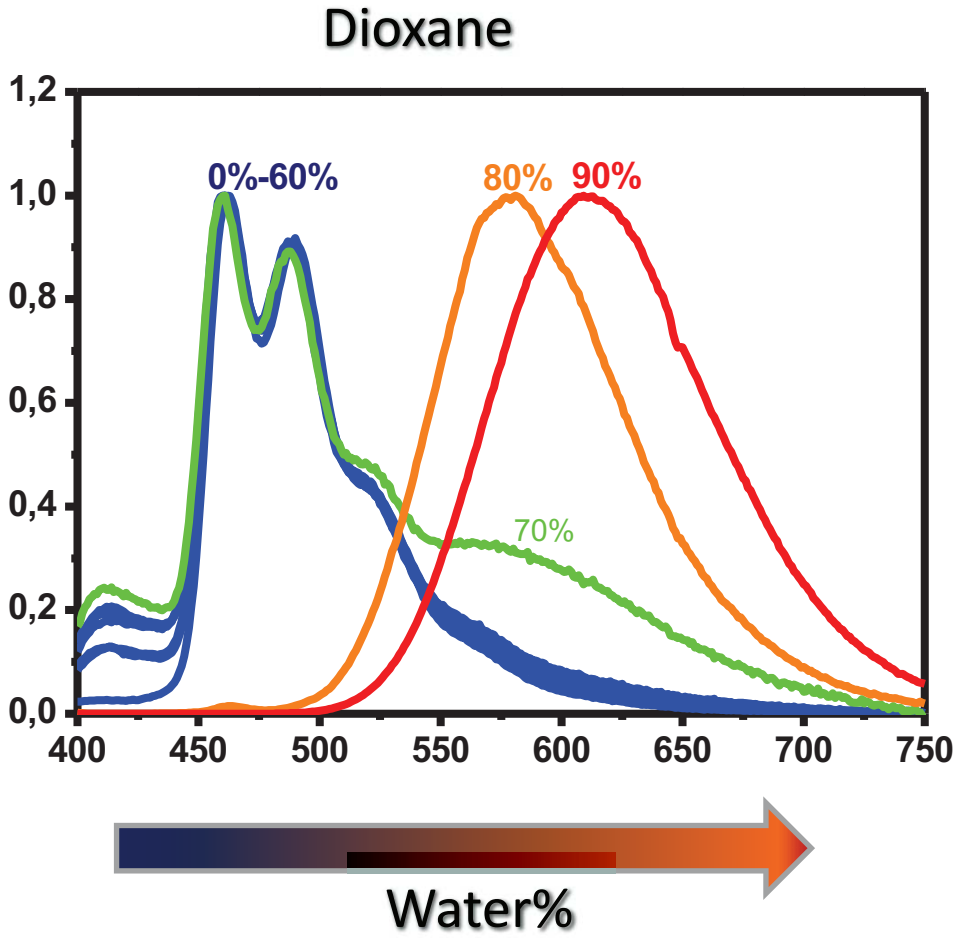
Changing aggregation with solvent

hydrophobic



hydrophilic





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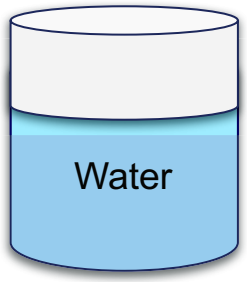
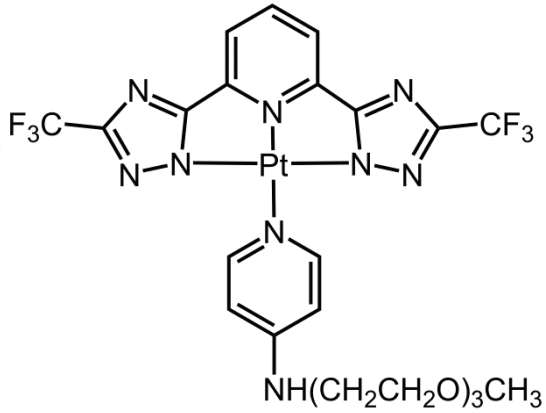
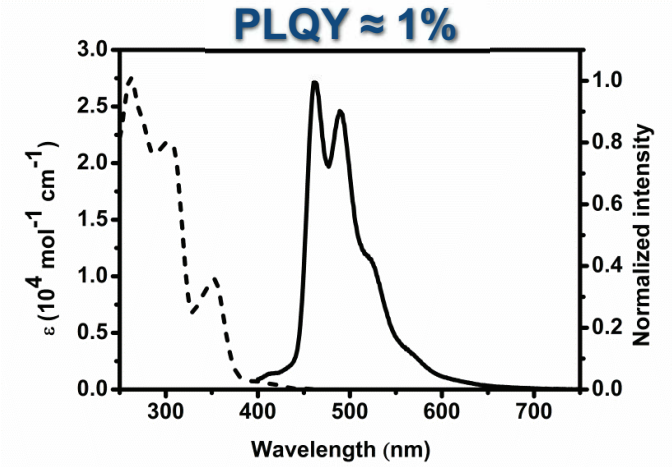
Self-assembly of Pt(II) complexes

dioxane solution of molecularly dissolved complex **(M)**





PLQY ≈ 1%



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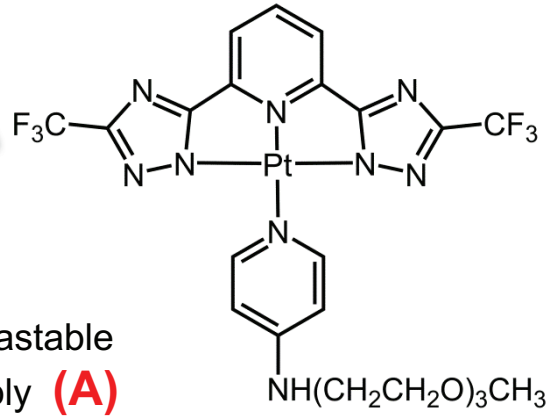
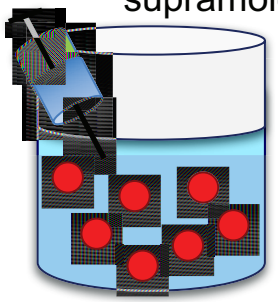
Self-assembly of Pt(II) complexes

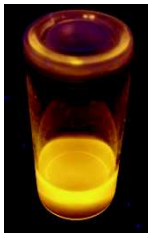
dioxane solution of molecularly dissolved complex **(M)**

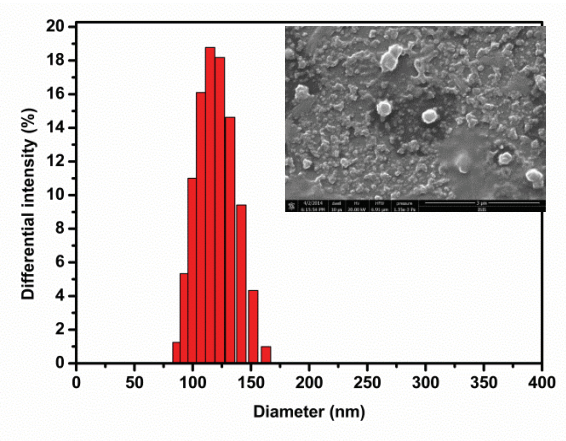


Kinetically-trapped metastable supramolecular assembly **(A)**

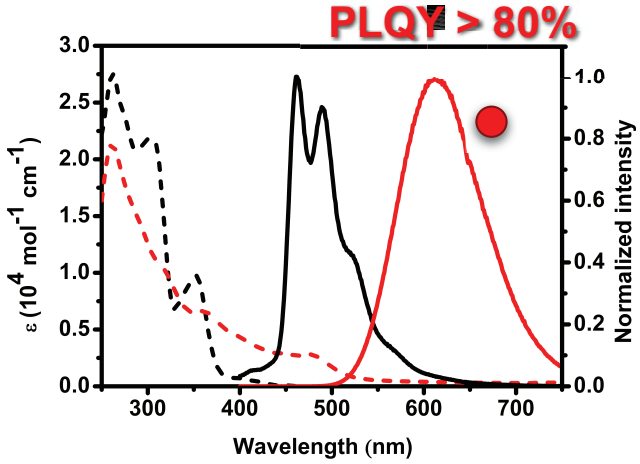
0D







PLQY > 80%



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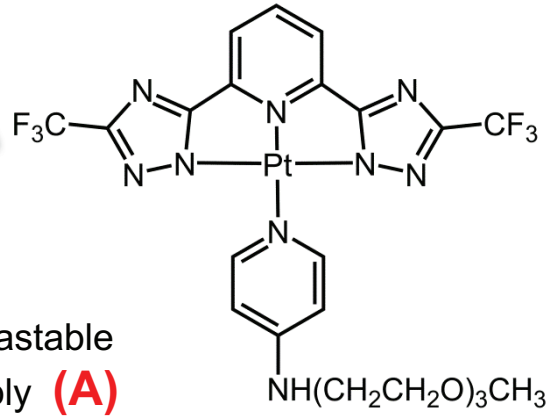
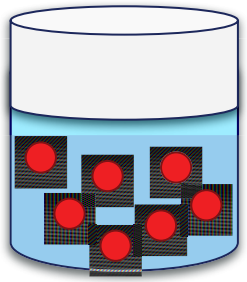
Self-assembly of Pt(II) complexes

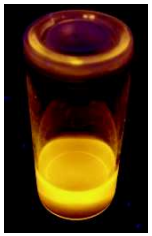
dioxane solution of molecularly dissolved complex **(M)**



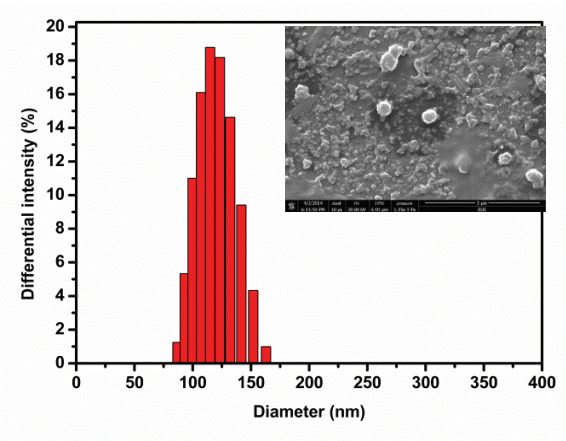
Kinetically-trapped metastable supramolecular assembly **(A)**

0D

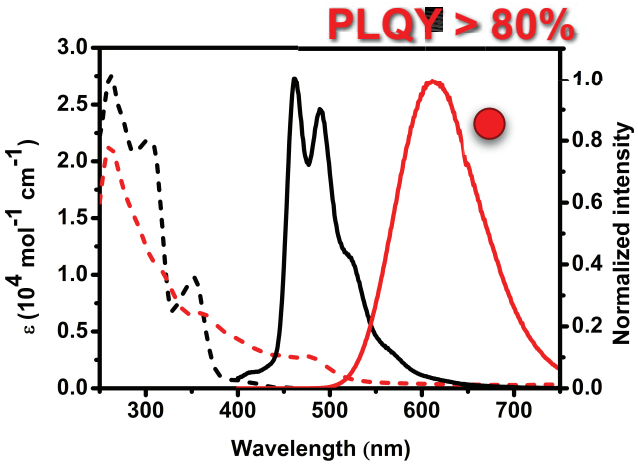




aging



PLQY > 80%



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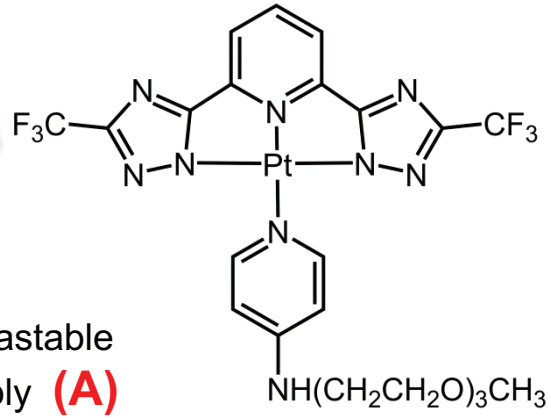
Self-assembly of Pt(II) complexes

dioxane solution of molecularly dissolved complex **(M)**

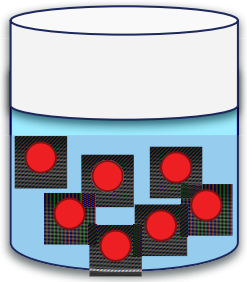


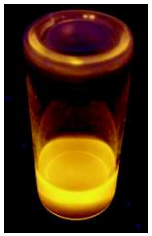
Dioxane:water mixture, time

Kinetically-trapped metastable supramolecular assembly **(A)**

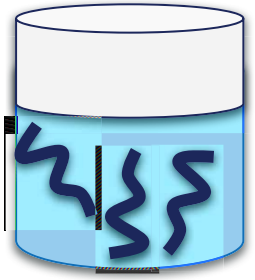
Thermodynamically stable supramolecular assembly **(C)**

0D

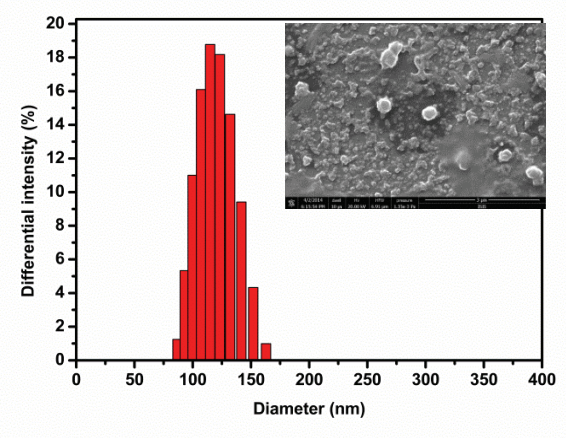




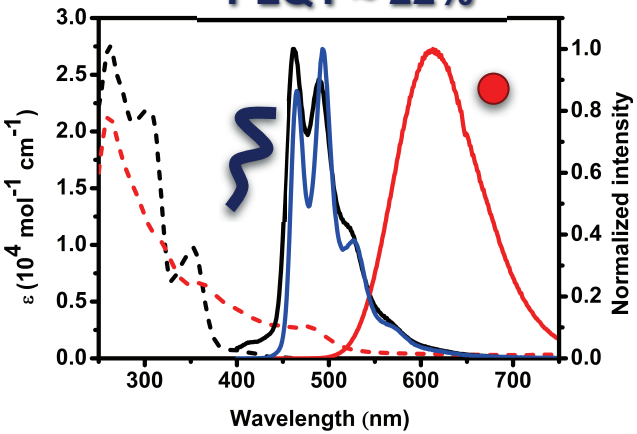
aging

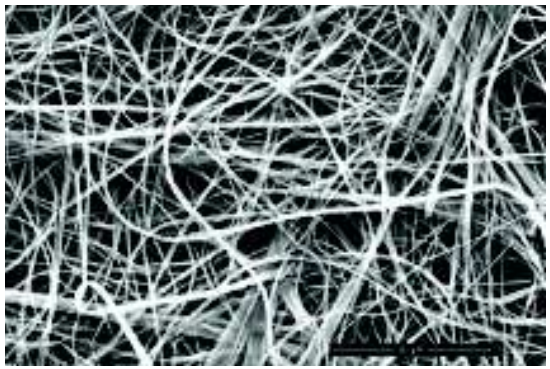


1D



PLQY ≈ 22%





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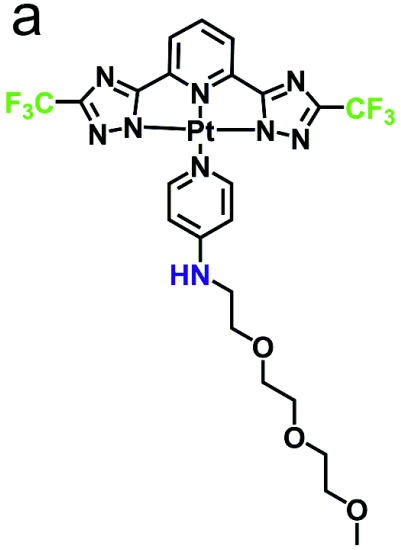
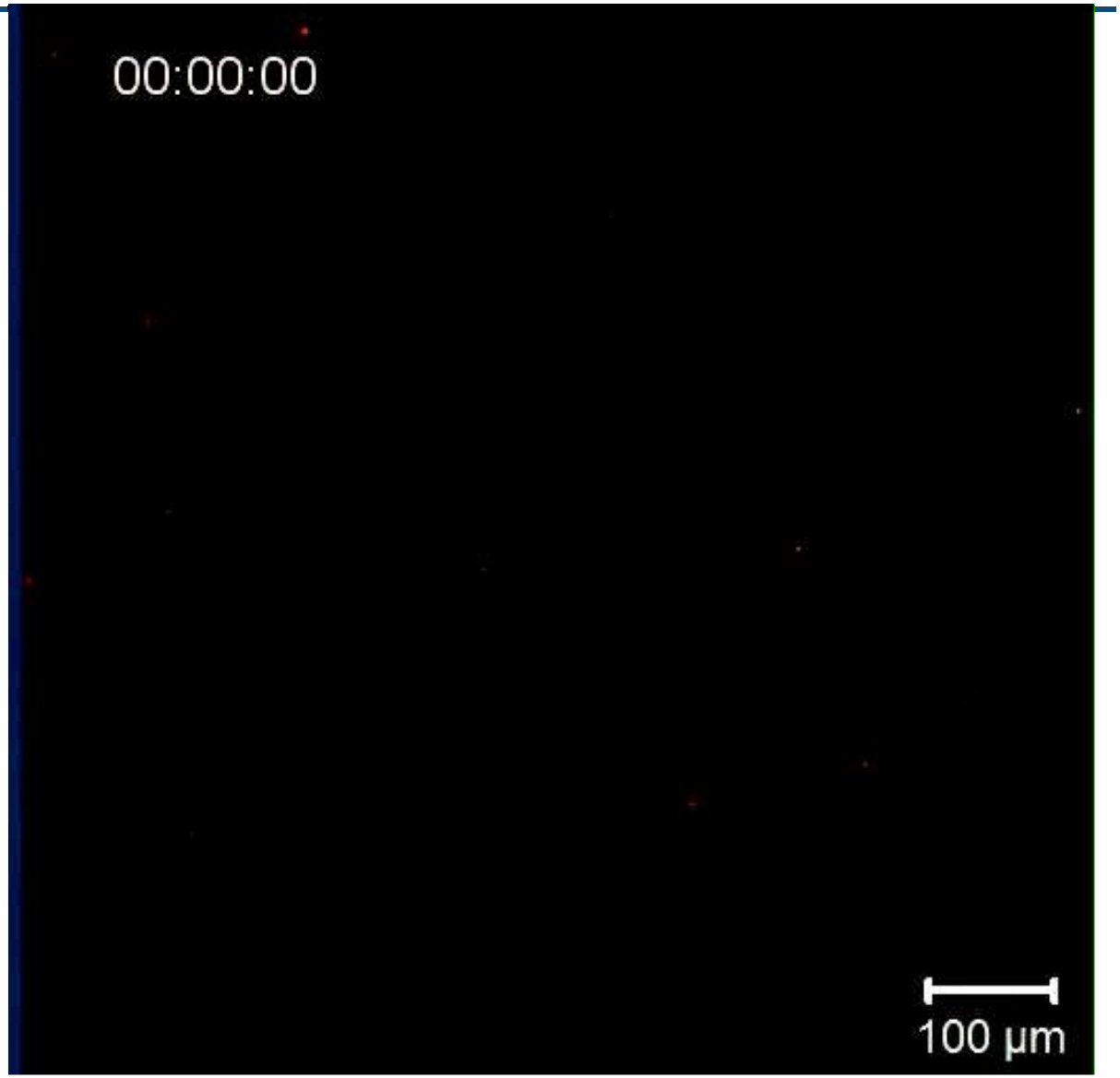
83% H_2O/17% Dioxane

a



1



A. Aliprandi, M. Mauro, L. De Cola *Nature Chemistry* **2016** *, 8,* 10

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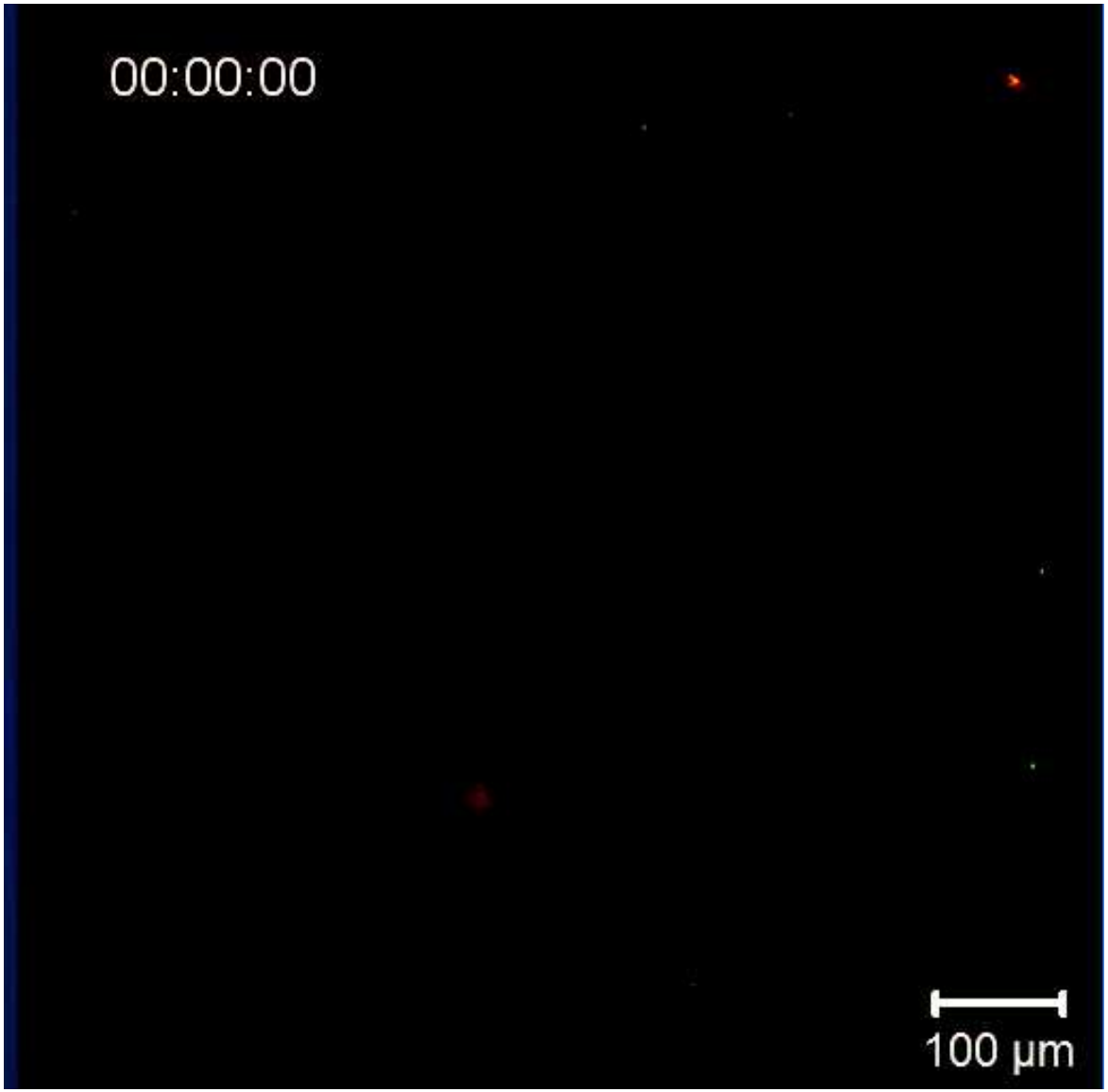
75% H_2O/25% Dioxane



A. Aliprandi, M. Mauro, L. De Cola *Nature Chemistry* **2016** *, 8,* 10

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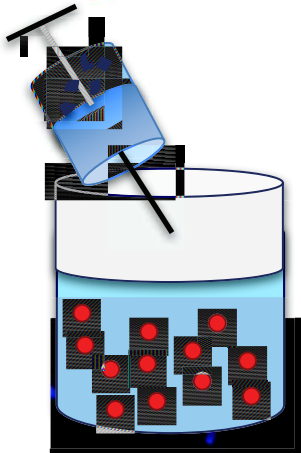
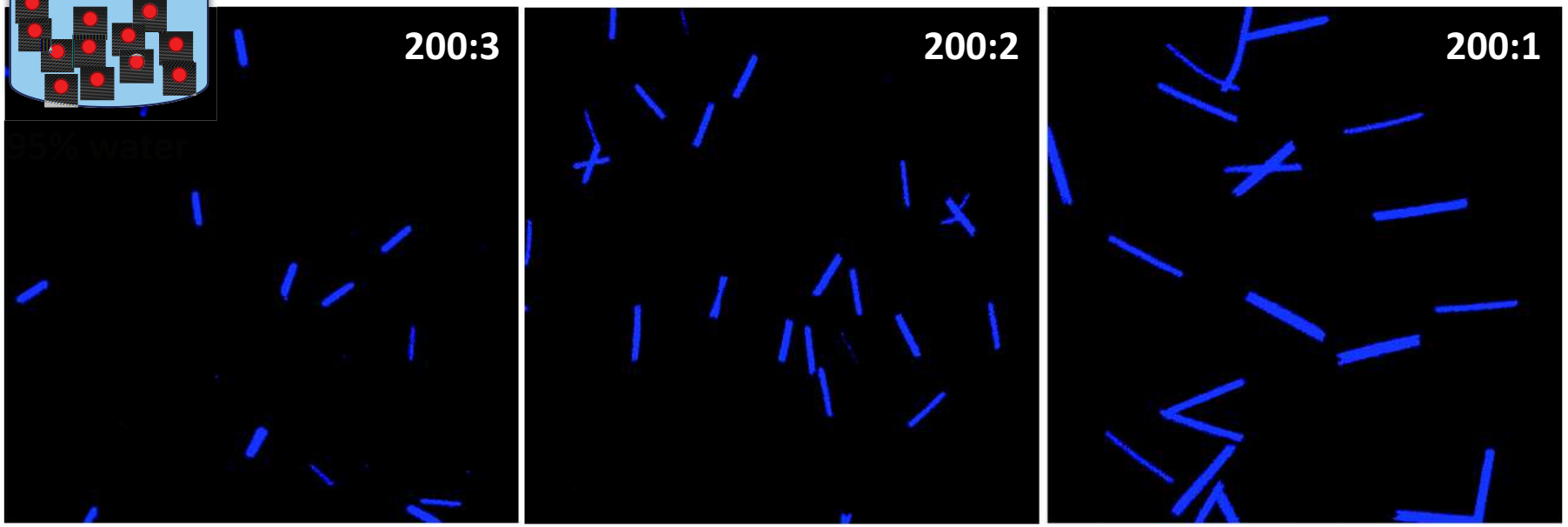
Uniform growth of the assemblies





6.19 ± 0.54 μm **8.42 ± 0.81 μm** **14.17 ± 0.86 μm**

A. Aliprandi, M. Mauro, L. De Cola *Nature Chemistry* **2016** *, 8,* 10


nature
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JANUARY 2016 VOL 8 NO 1
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Making light of
self-assembly pathways
MULTIVALENCY
Sugar-coated fullerenes fight infection
NICKEL CATALYSIS
Activating amides
SUPRAMOLECULAR GELS
Metal–organic cages branch out

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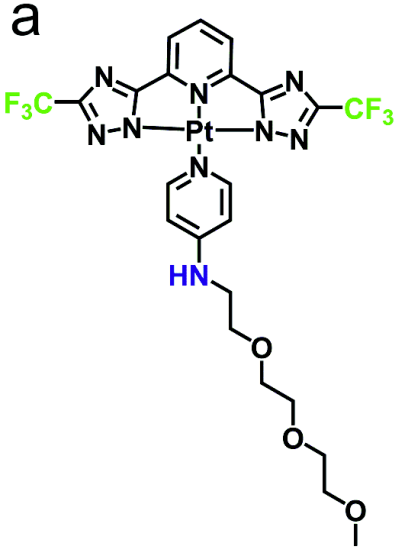
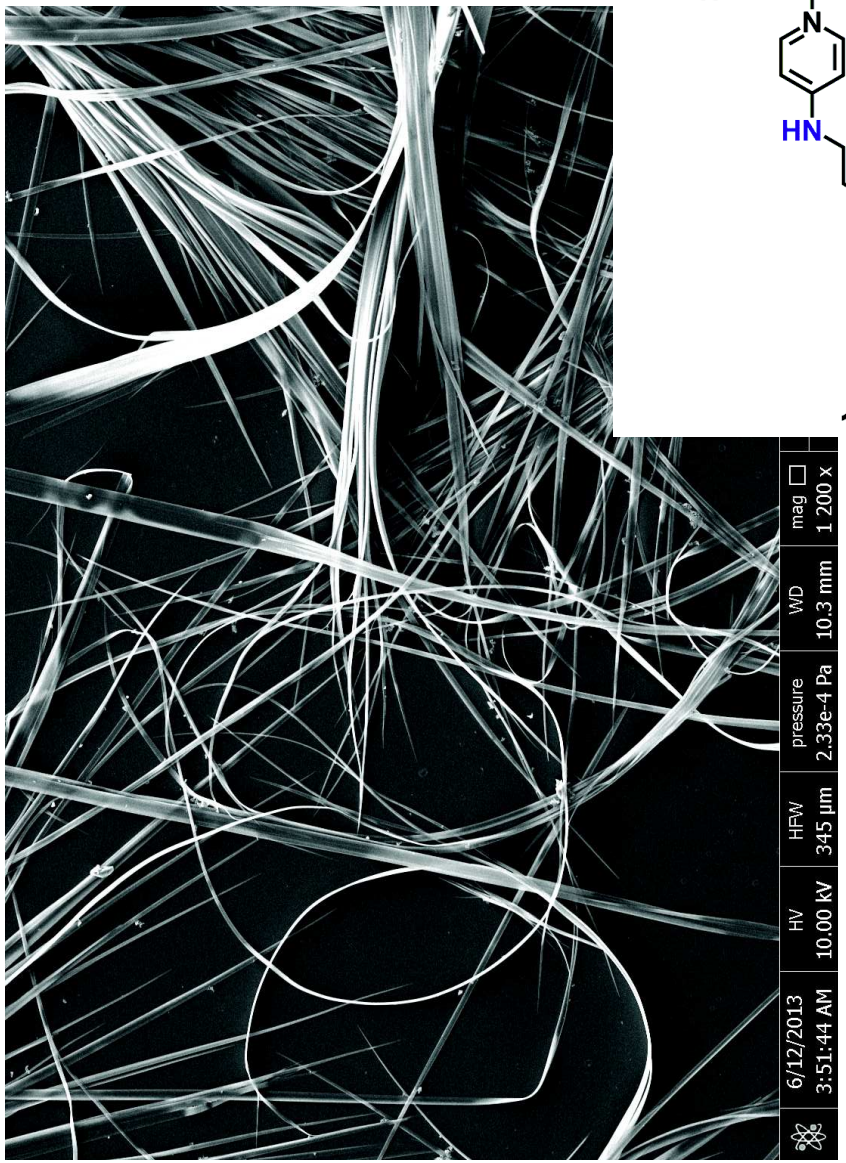
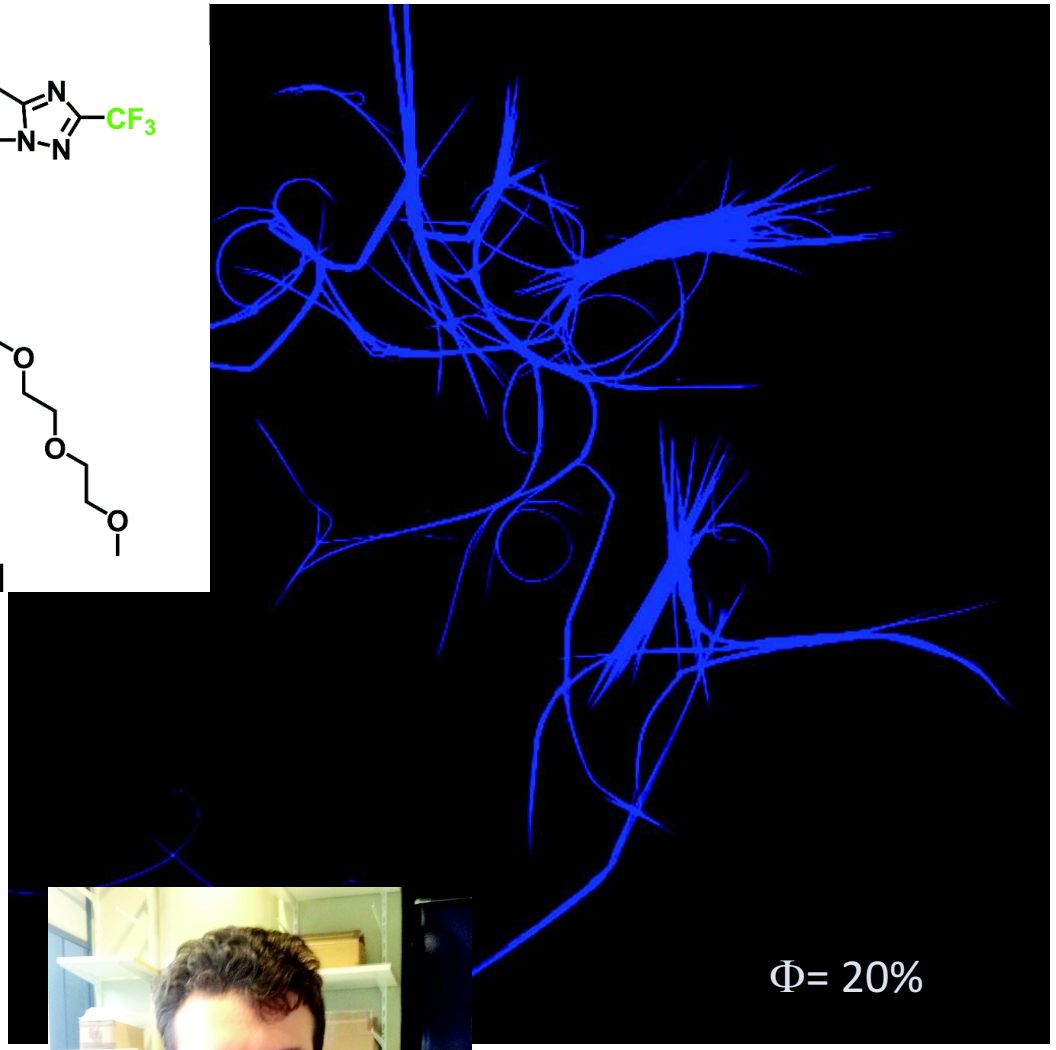
Morphology of the aggregates

a



1





Alessandro Aliprandi

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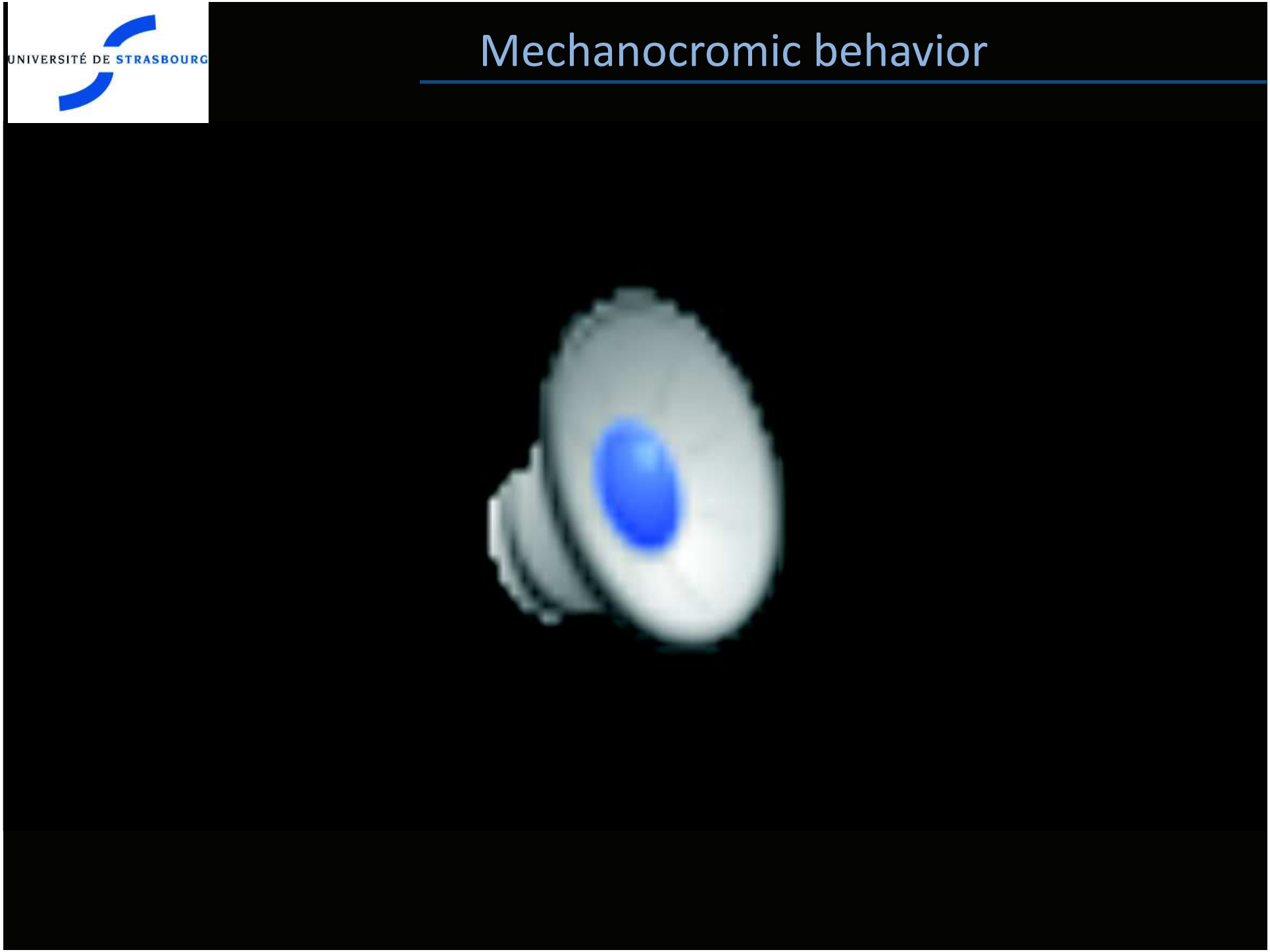
Mechanocromic behavior



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Reversibility of the mechanocromism

Adv. Funct. Mater. **2016**, *26*, 5271

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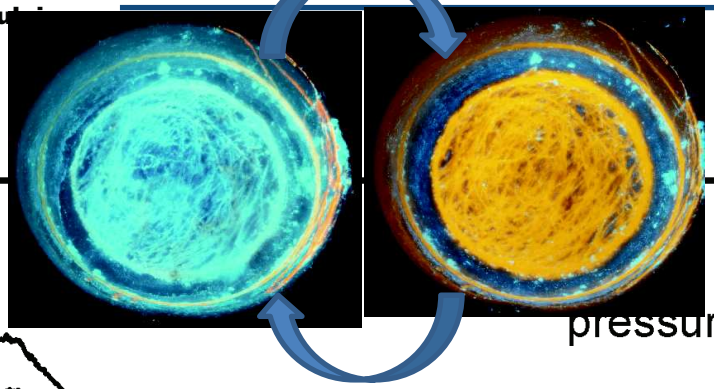
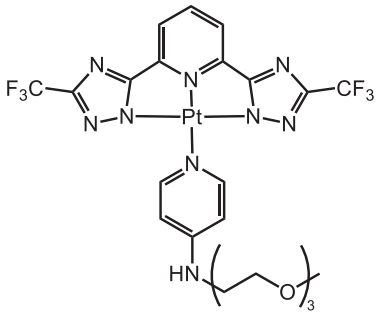
Supramolécu

Pressure dependent emission





In collaboration with Prof. M. Kappes, Dr. S. Lebedkin, KIT, Germany

What happen at the nanoscale?

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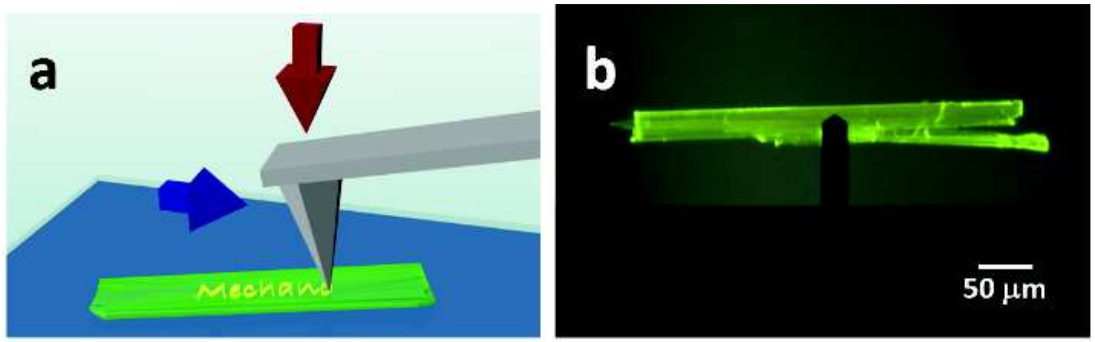
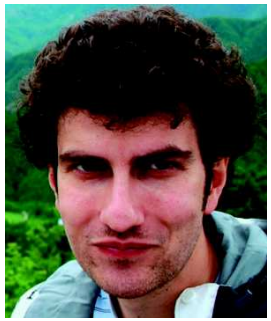
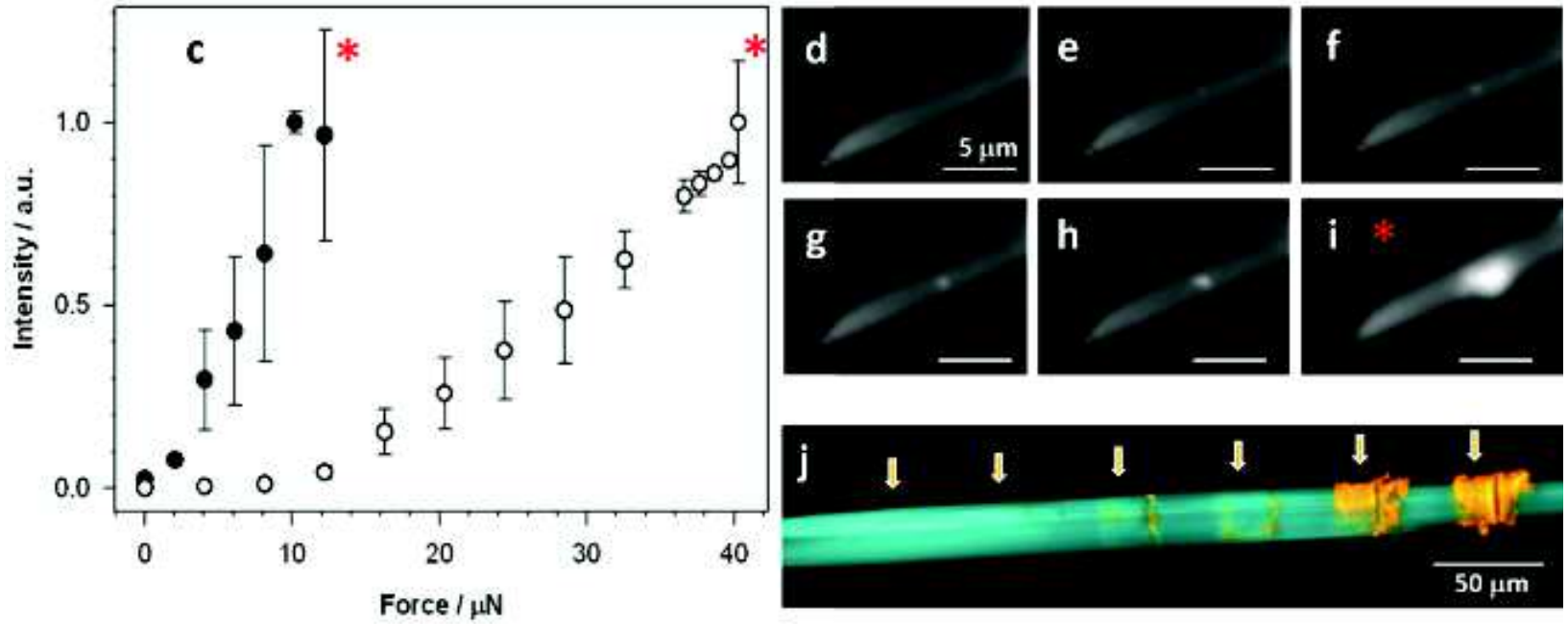
Writing on self-assembled ribbons





Dr. Damiano Genovese (KIT)

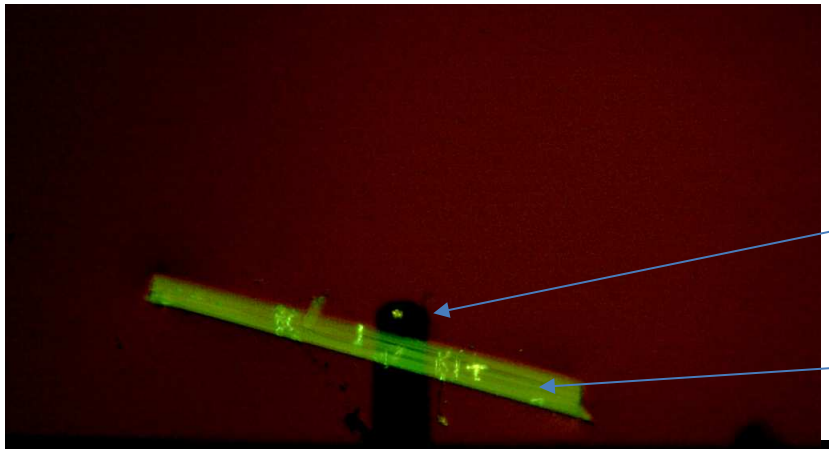
Dr. Michael Hirtz, Karlsruhe Institute of Technology (KIT)



D. Genovese, M. Hirtz et al. *Adv. Funct. Mater.* **2016**, *26*, 5271–5278

Writing at Nanoscale

We use an AFM tip to write information in the nanoscale on a micrometer sized mechanochromic support

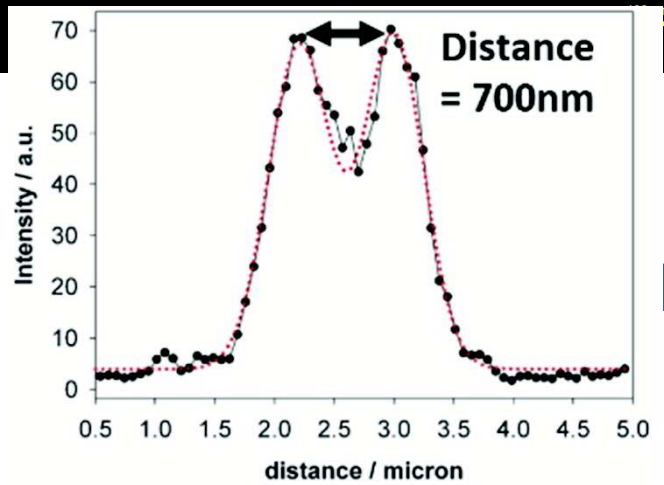


Michael Hirsch and Damiano Genovese

Cantilever and tip

Written ribbon

the tip is kept under constant force, in contact mode



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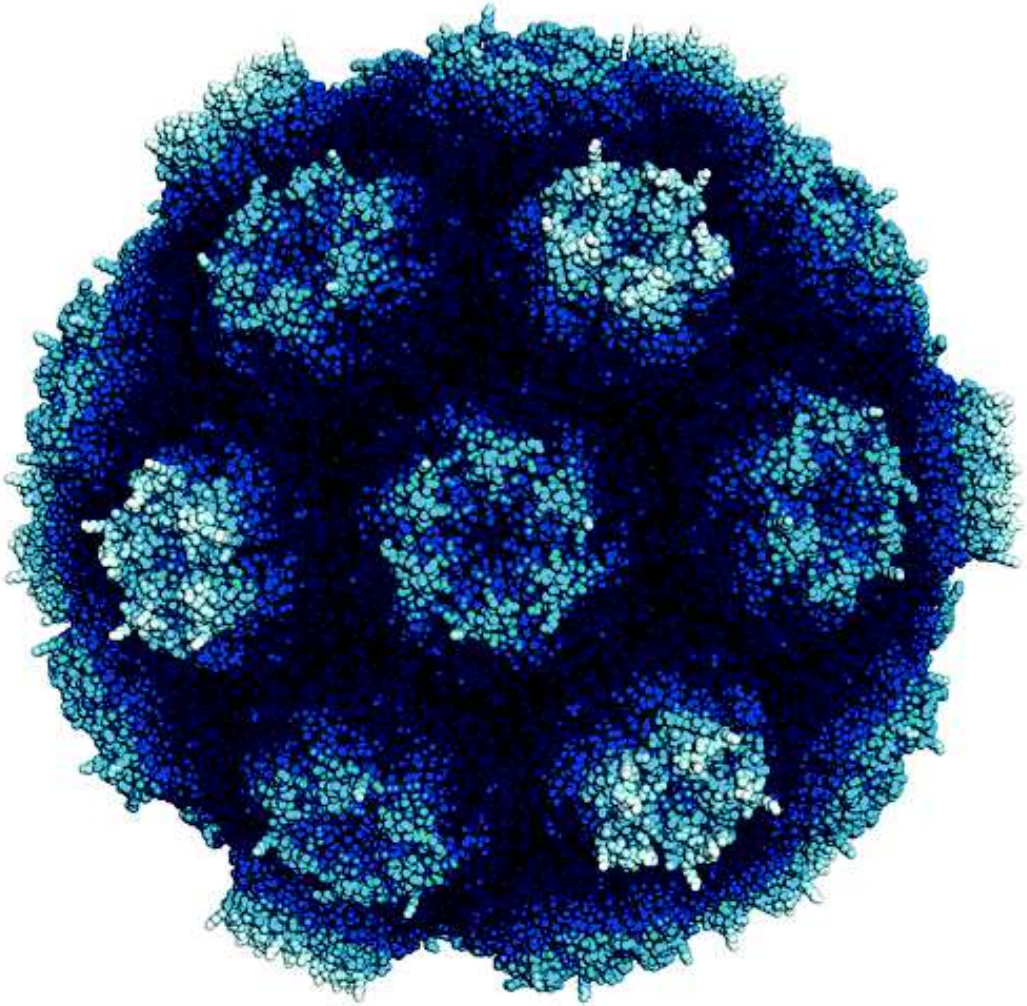

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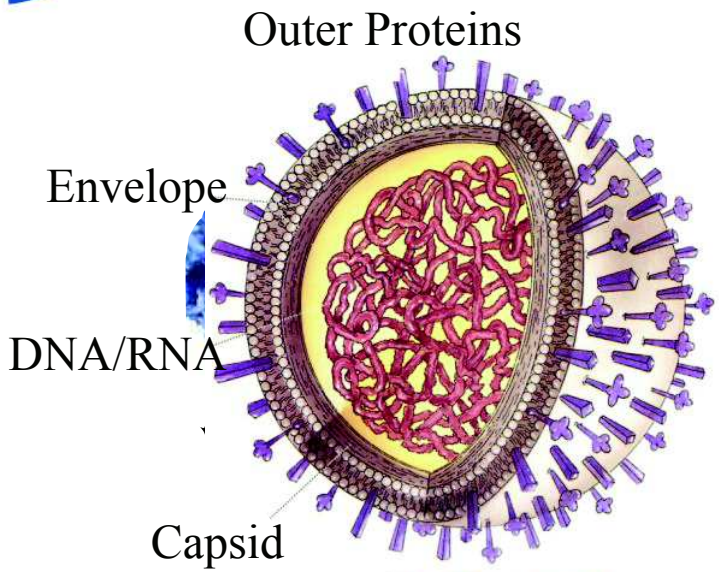
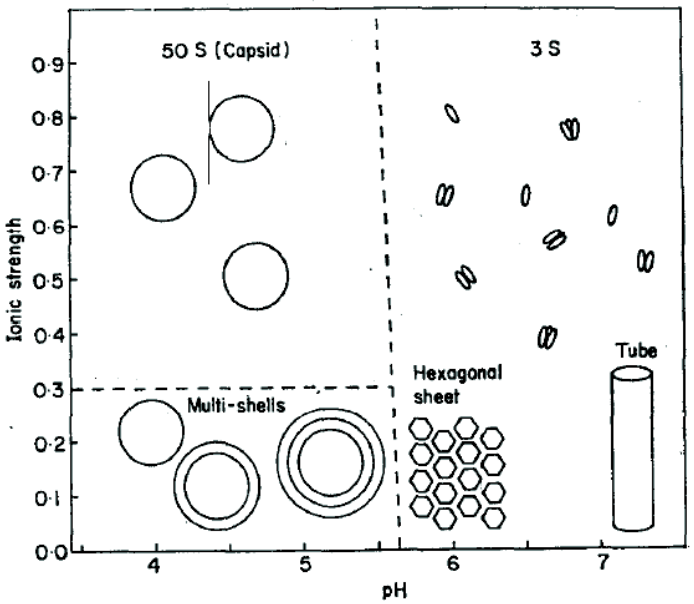
Virus: an introduction

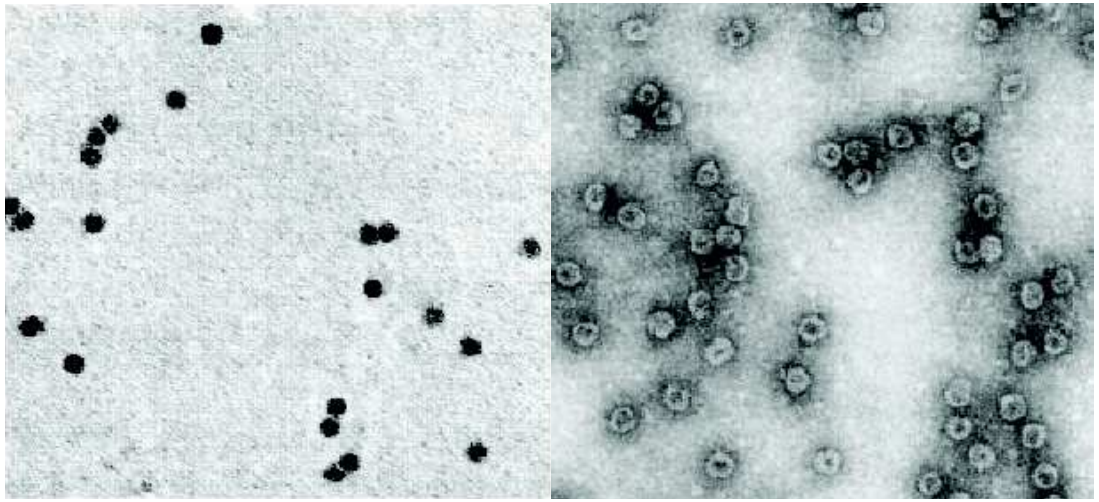


Shape control by pH and I



Adolph, K. W.; Butler, P. J. G.,
J. Mol. Biol. **1974,** *88*, 327-341.

Mineralization *via* encapsulation



Douglas, T.; Young, M., *Nature* **1998,** *393*, 152-155.

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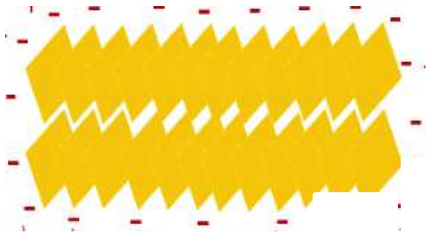
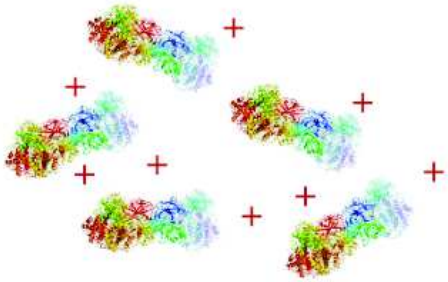
Reassembling of Proteins…

Can we induce a change in the shape with a self-assembled template?
Can we create highly luminescent virus like particles by self-assembly?
What will be the biological implication of the new "virus"?

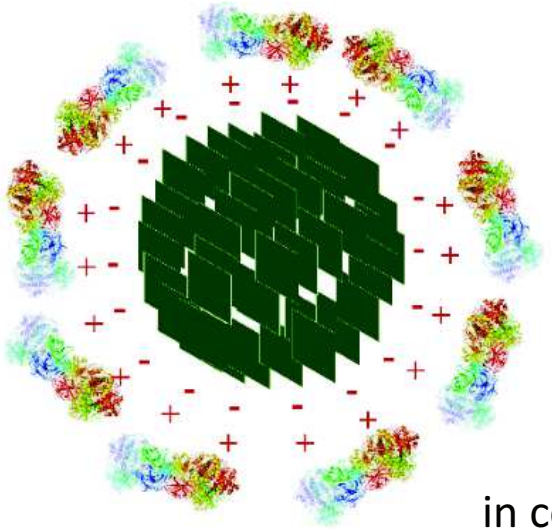


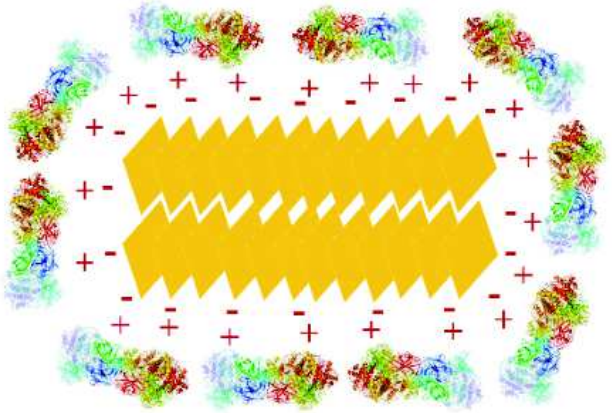
+



Self-assembled Pt(II) complexes

Coat proteins



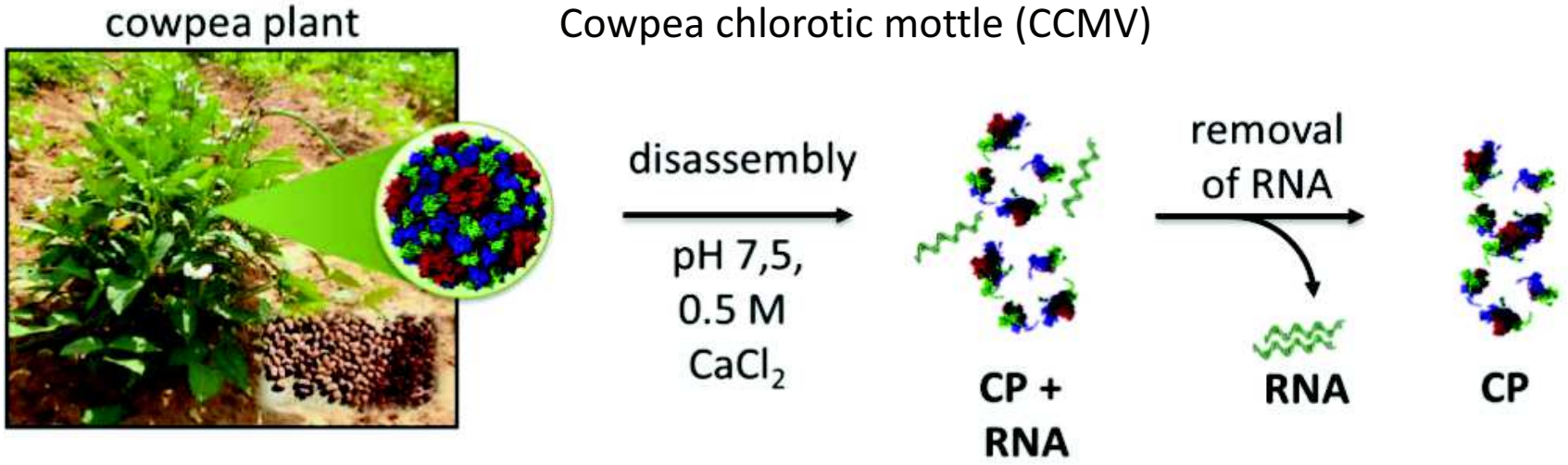


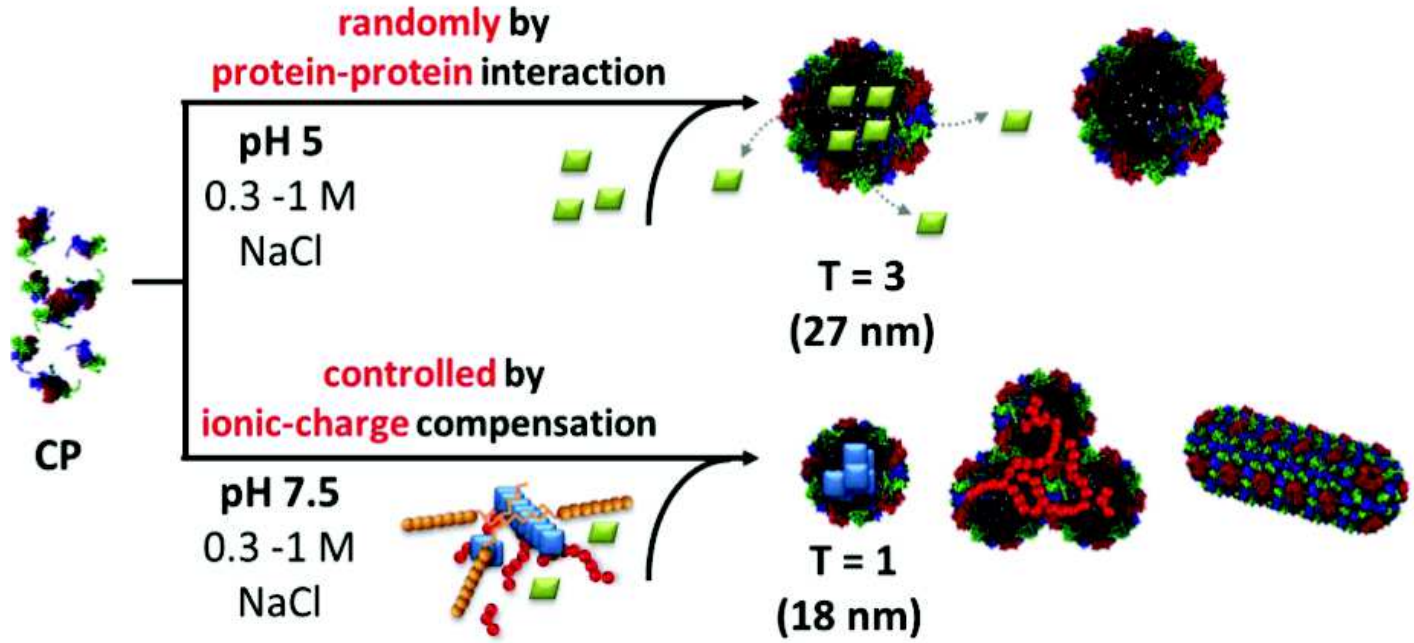
in collaboration with J. Cornelissen, University of Twente

Reassembling of Proteins - Concept

Disassembly of native CCMV into RNA and CPs

Cowpea chlorotic mottle (CCMV)





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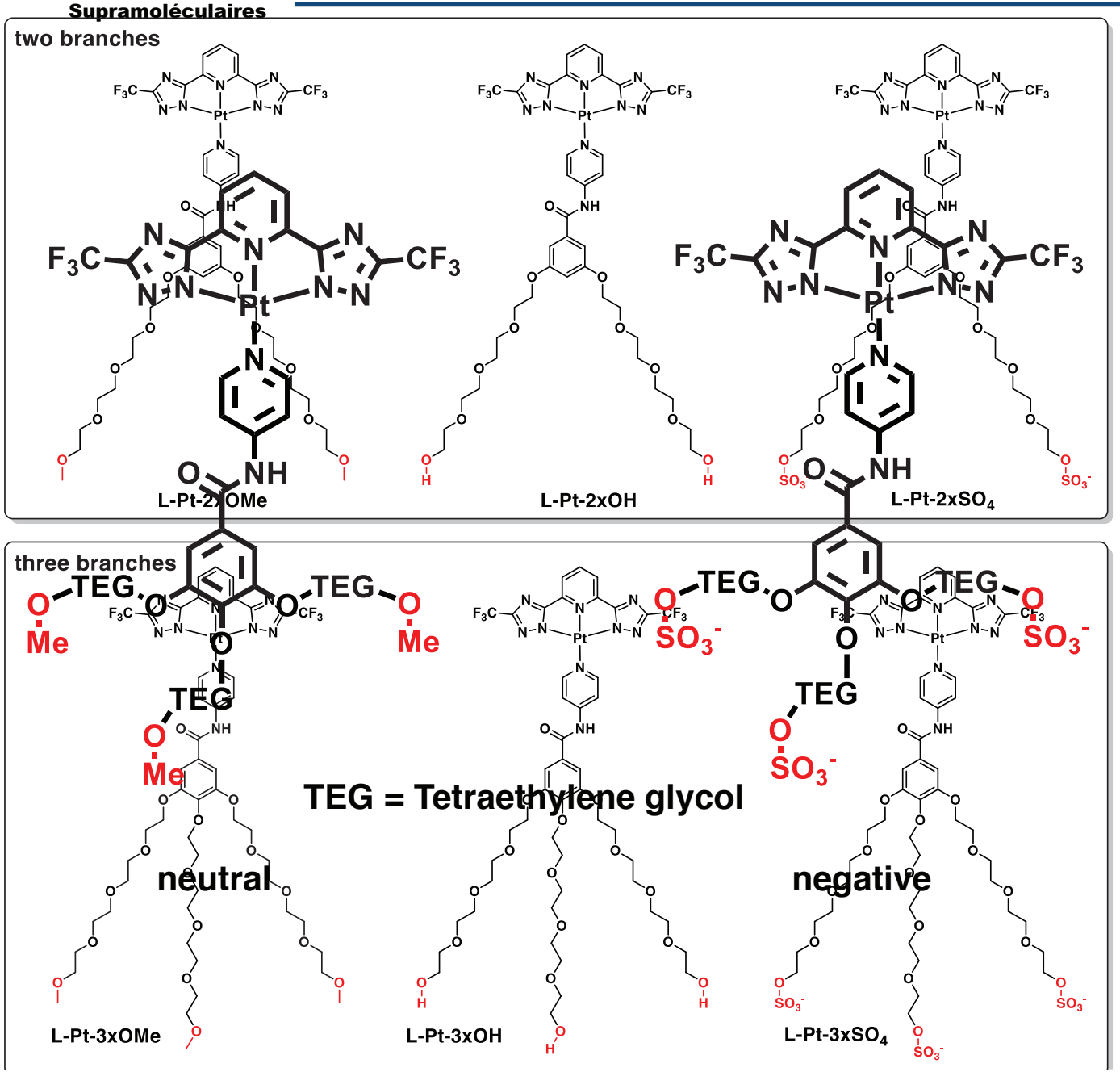
Pt(II) amphiphiles



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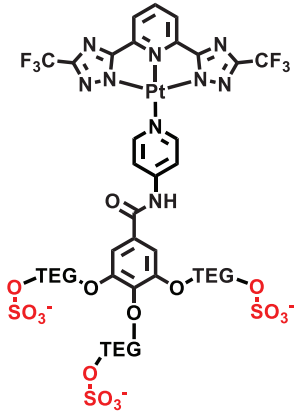
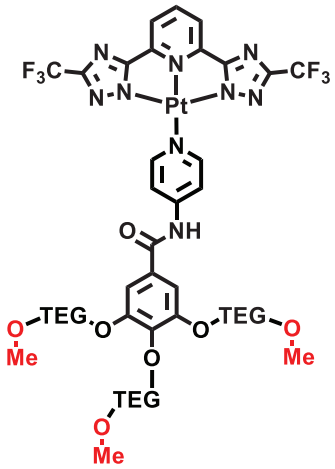
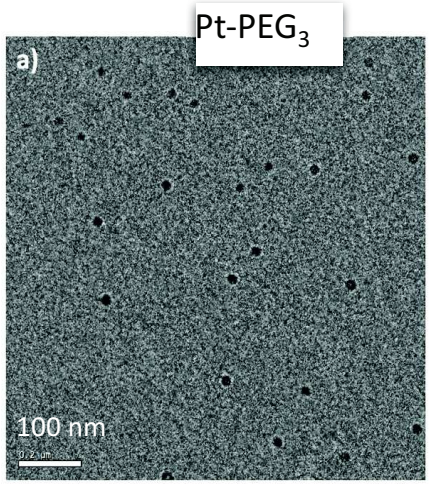
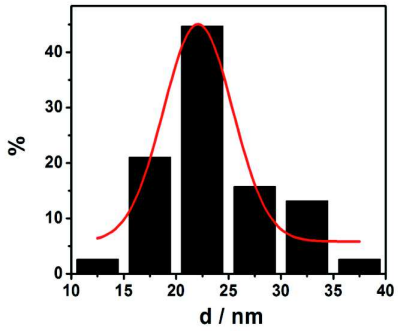
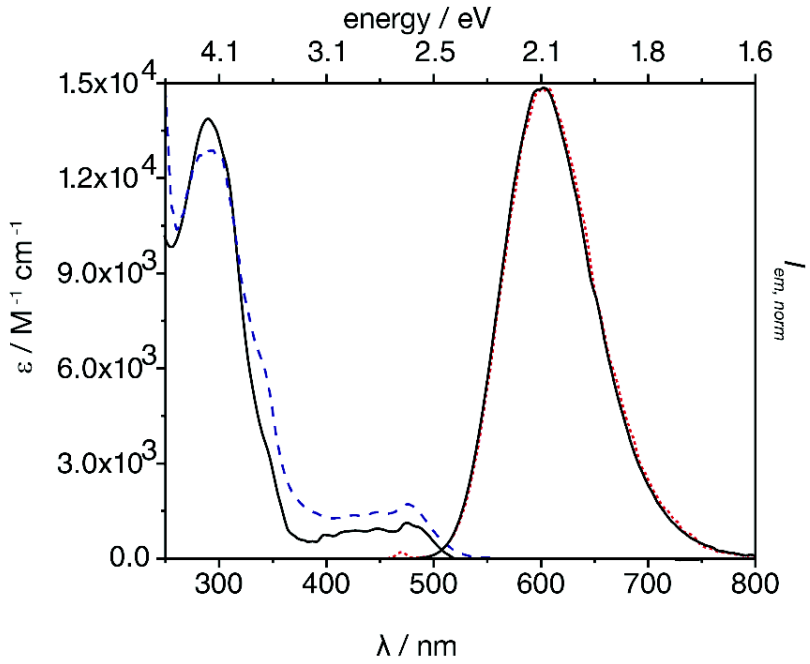
Photophysical properties of the complexes











Water/Buffer solution 10^{-5} M

^{3}MMLCT based emission

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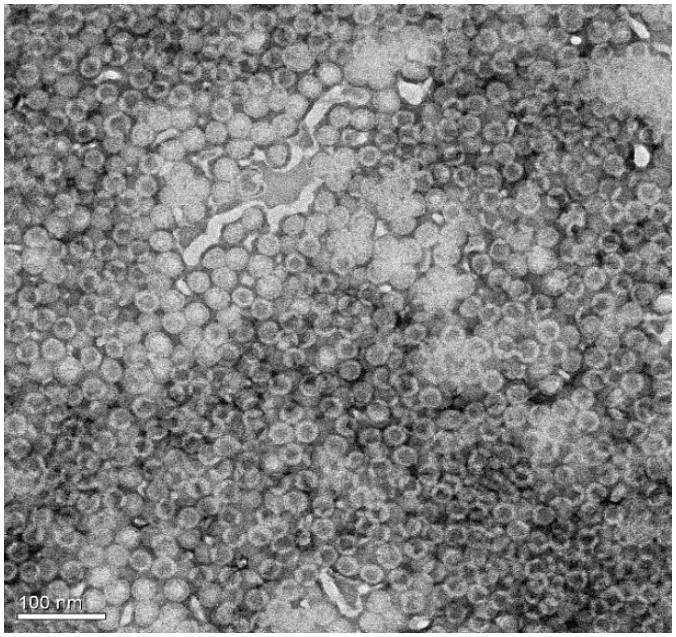
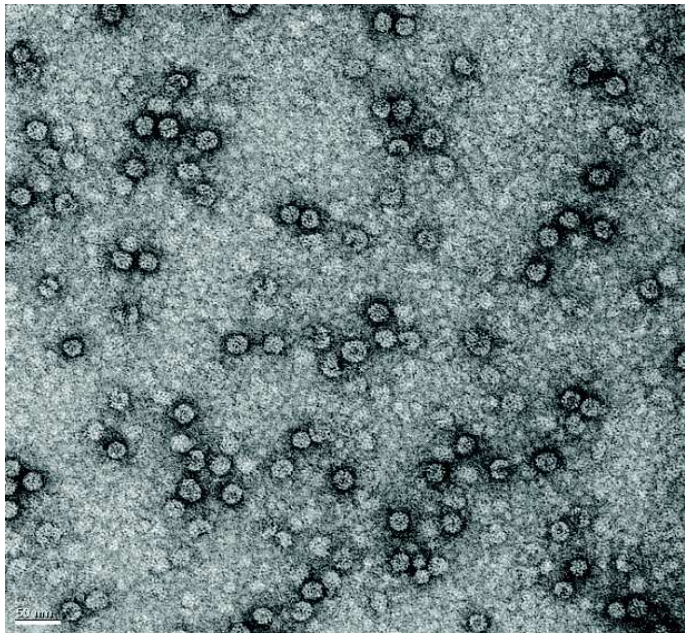
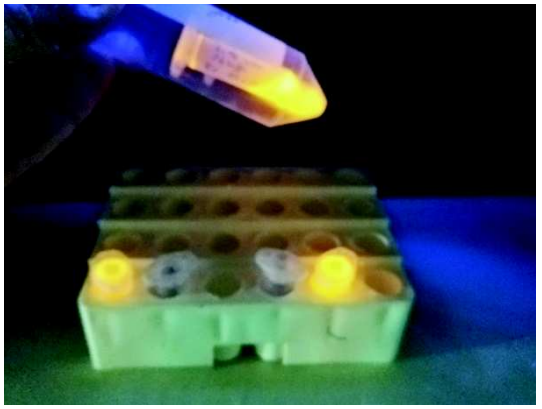
Virus-Like Particles Formation

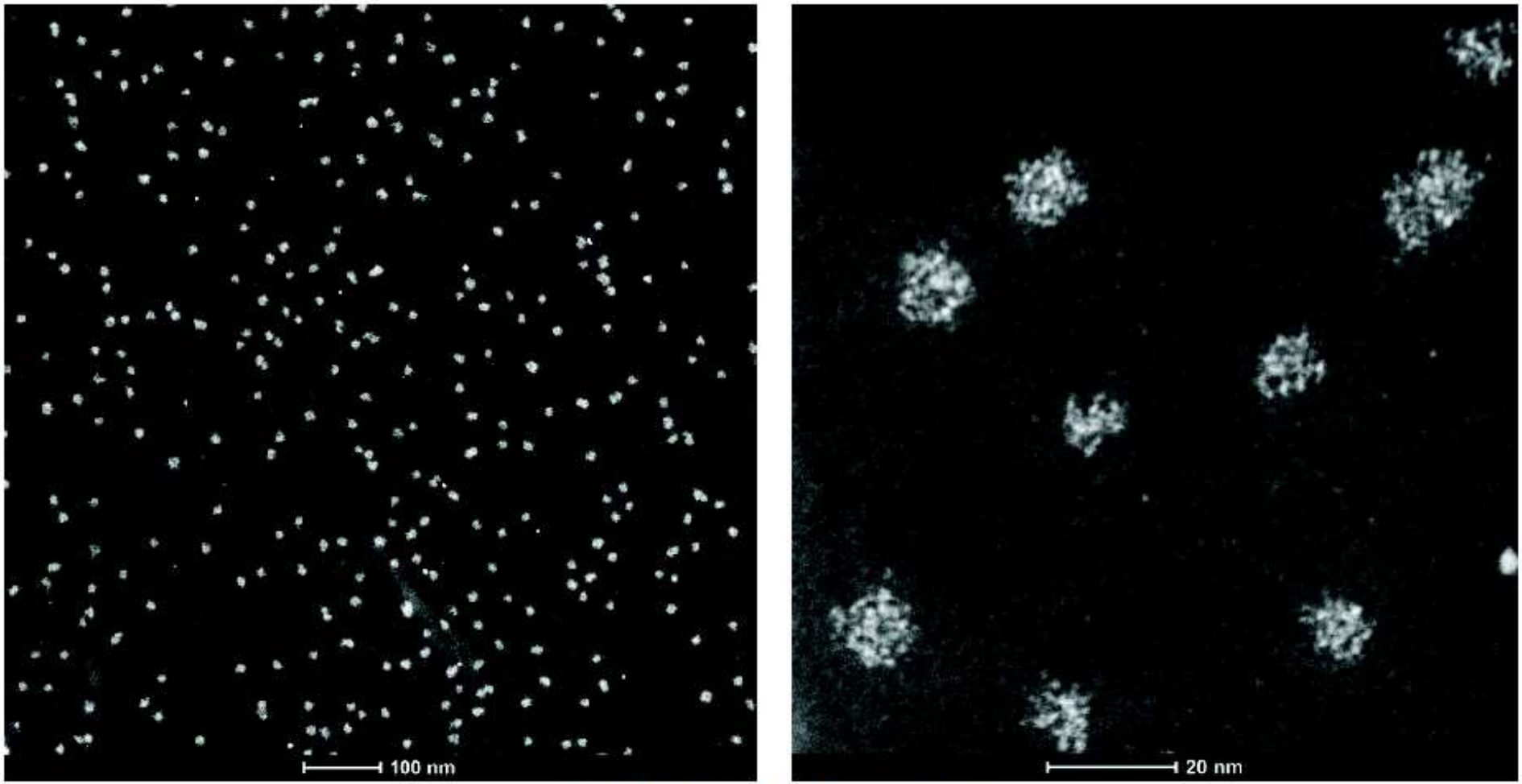






S. Sinn et al.
J. Am. Chem. Soc. **2018**, *140*, 2355-2362

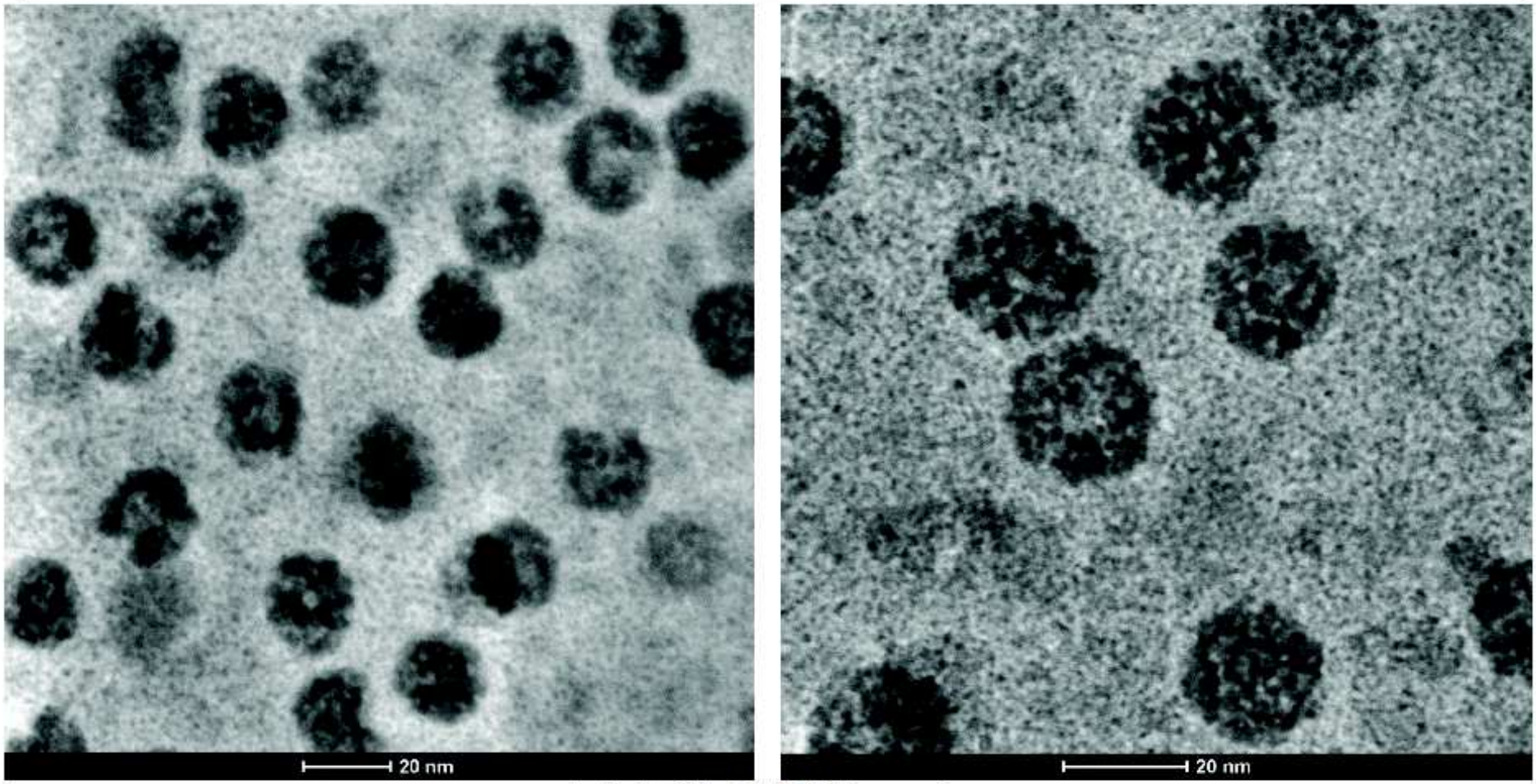
Capsid-Pt-Cp-C2-1 unstained



HAADF-STEM imaging

Dr. C. Kuebel

Capsid-Pt-Cp-C2-1 stained



HAADF-STEM imaging

Dr. C. Kuebel

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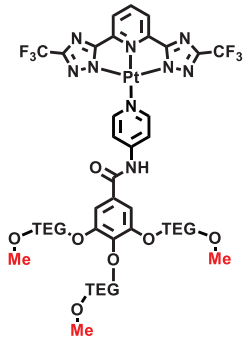
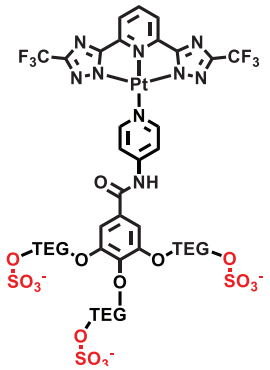
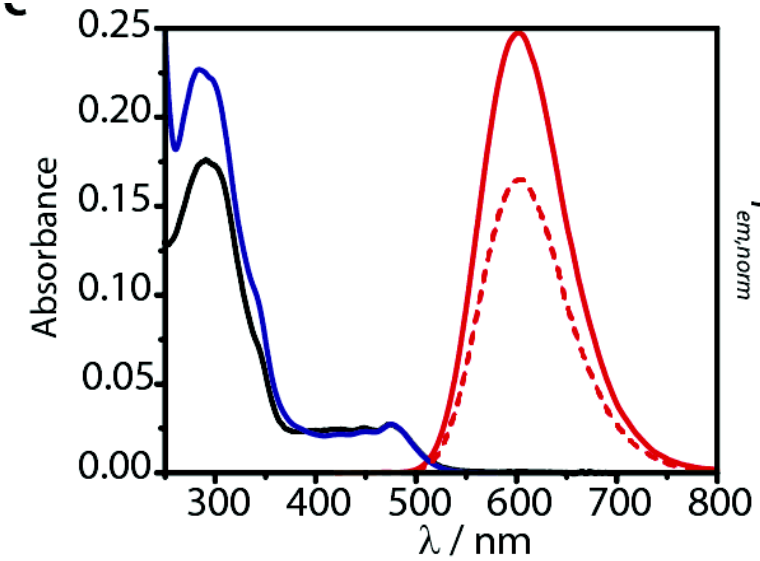
Photophysics of the VLP

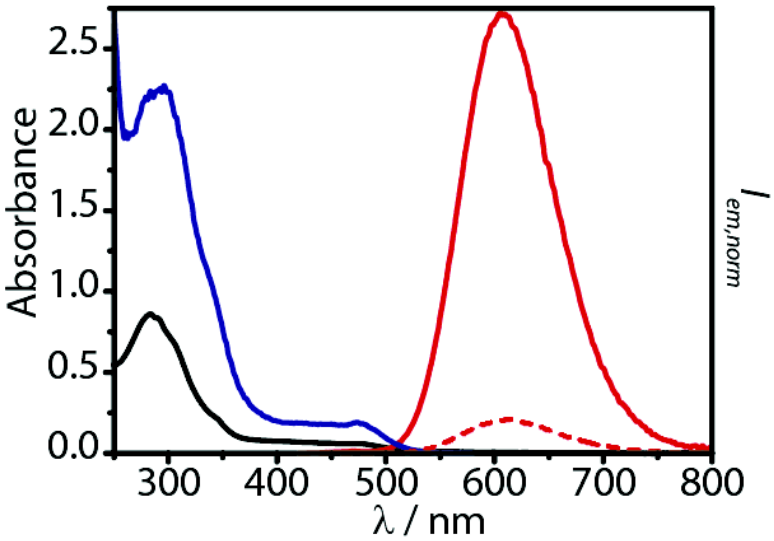
VLP@neutral





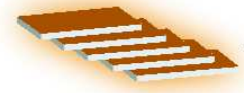
VLP@negative

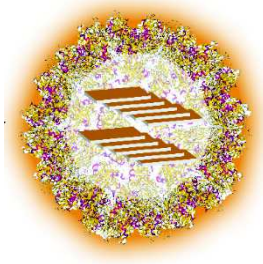




$\phi_{complex}$ = **36%**

ϕ_{VLP} = **55%**

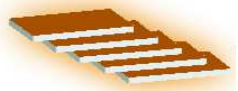


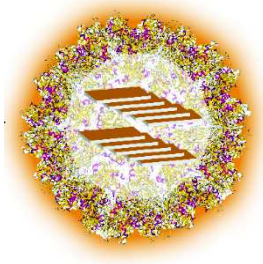


EF = **1.5**

$\phi_{complex}$ = **4%**

ϕ_{VLP} = **52%**





EF = **13**

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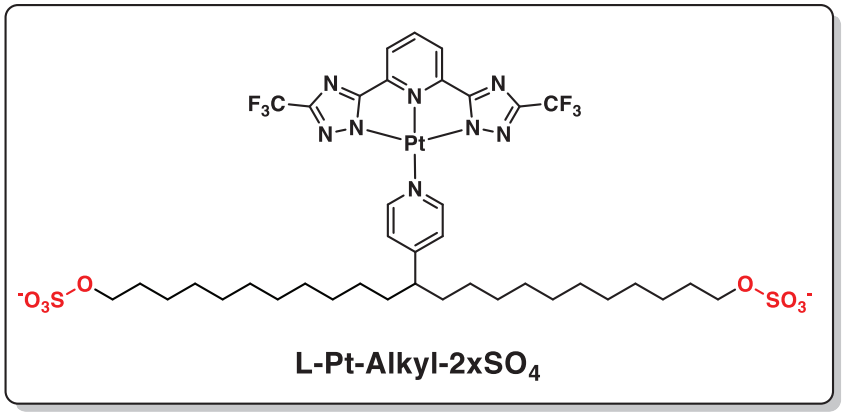
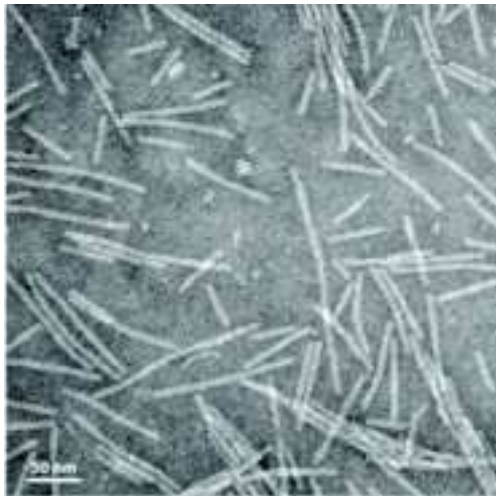
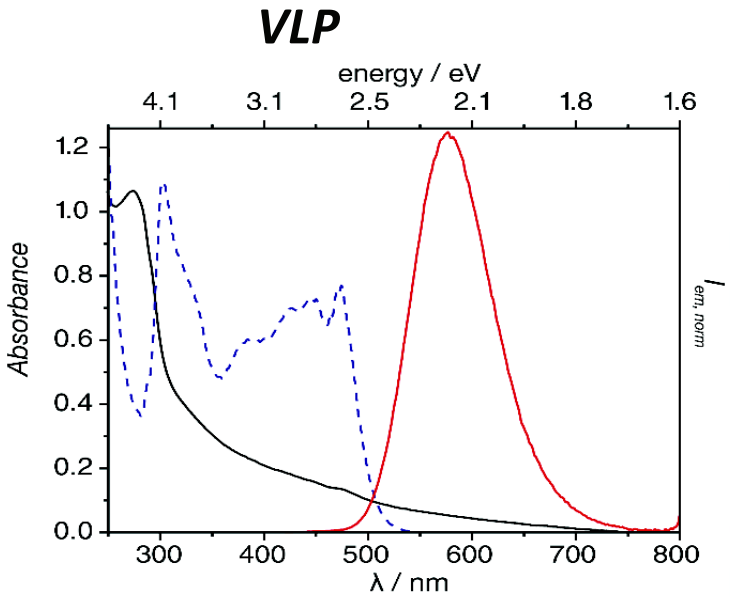
Shape Change Induced by the template

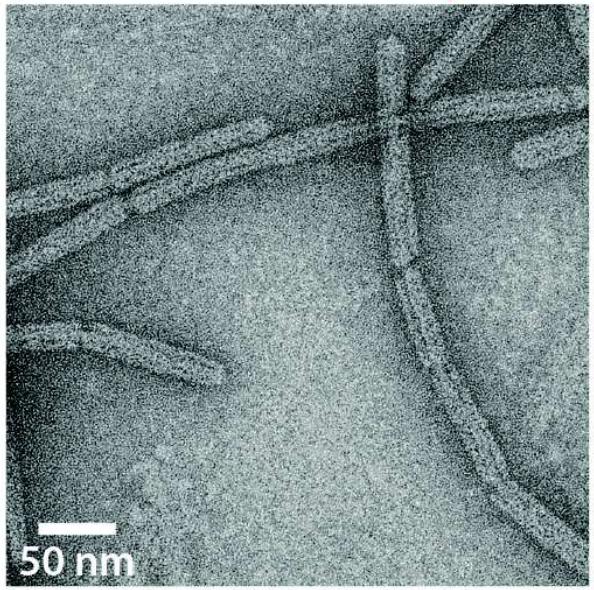




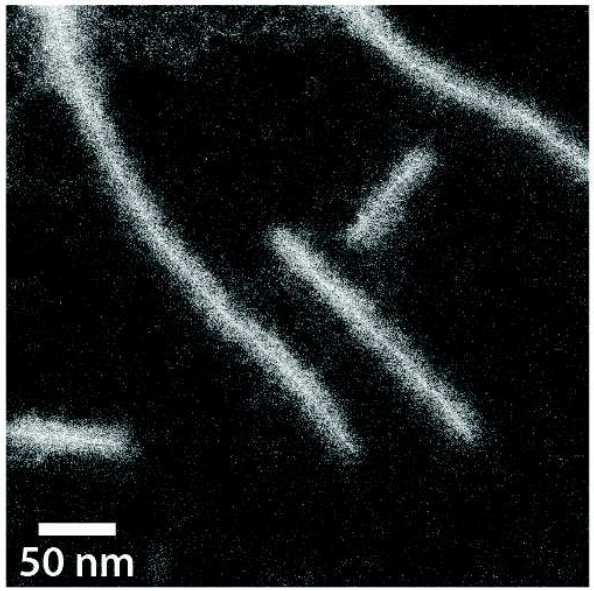


b

VLP⊃2xUSO4



d



S. Sinn et al.
J. Am. Chem. Soc. **2018**, *140*, 2355-2362

ϕ_{VLP} = **23%**

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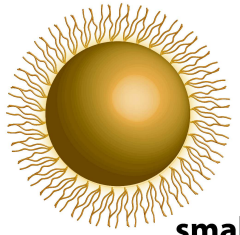
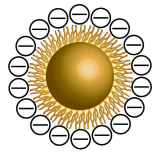
Schematic pathways

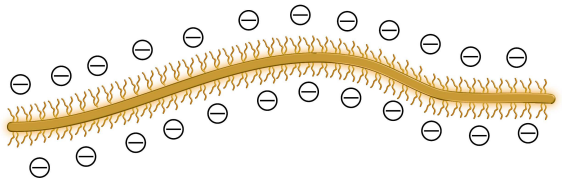
self assembly

Pt-3xTOMe **Pt-3xTSO$_4$** **Pt-2xUSO$_4$**

luminescent Pt(II) assemblies







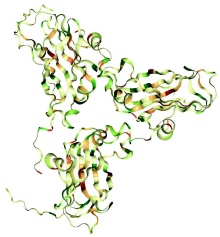
small assemblies

large assemblies

self assembly with CCMV coat proteins

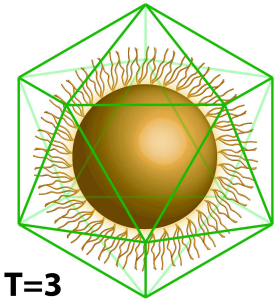
electric neutral cargo

negatively charged cargo



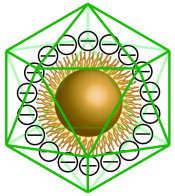
luminescent virus-like particles

protein shell



T=3

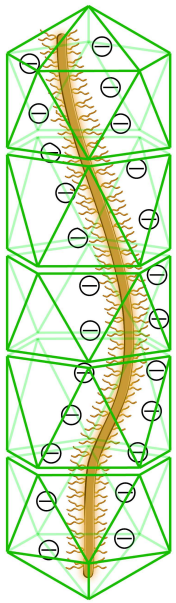
VLP⊃3xTOMe



T=1

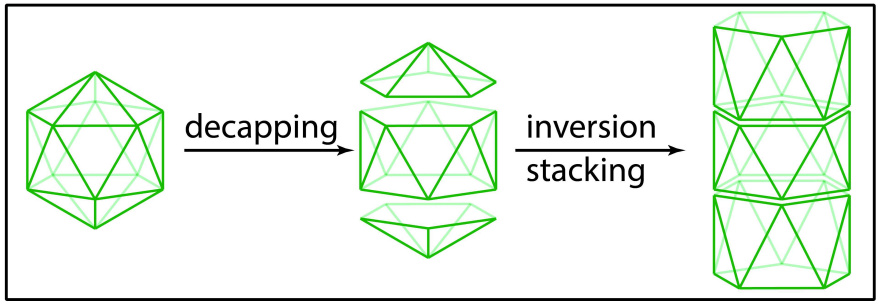
VLP⊃3xTSO$_4$

icosehdral protein shell



tubular protein shell

VLP⊃2xUSO$_4$



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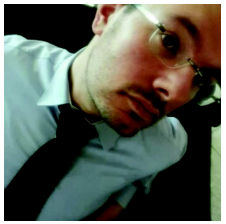
Who is in the lab doing the work....


Dr. Leana Travaglini


Dr. Frank Biedermann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Dr. Charles Lochenie


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Etienne Piantanida


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Prof. T. Baumert, Dr. E. Robinet

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