Multifunctional nanolayers, self-healing and slow release coatings against biocorrosion and biofouling

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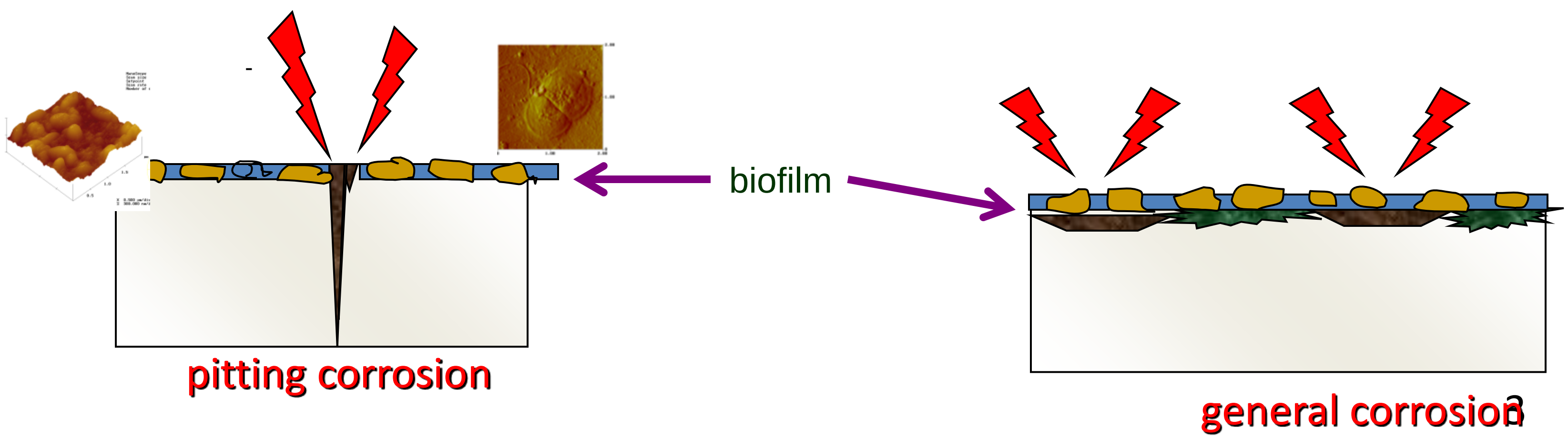
Outline of the presentation

- **Corrosion and microbiologically influenced corrosion** (inhibition by inhibitors, biocides)
- **Inhibition of corrosion and biofouling by multifunctional:**
  - **molecular layers:** Langmuir-Blodgett films, self assembled layers;
  - **smart coatings:**
    - self-healing coatings
    - slow-release coatings

Corrosion, microbiologically influenced corrosion

- widespread problem, energy/efficiency loss
- undesired accumulation of **deposits (organic, inorganic, microbia**l), initialization of localized corrosion
- **micro/macrofouling .**

Resolution: **dissolved inhibitors, coatings**



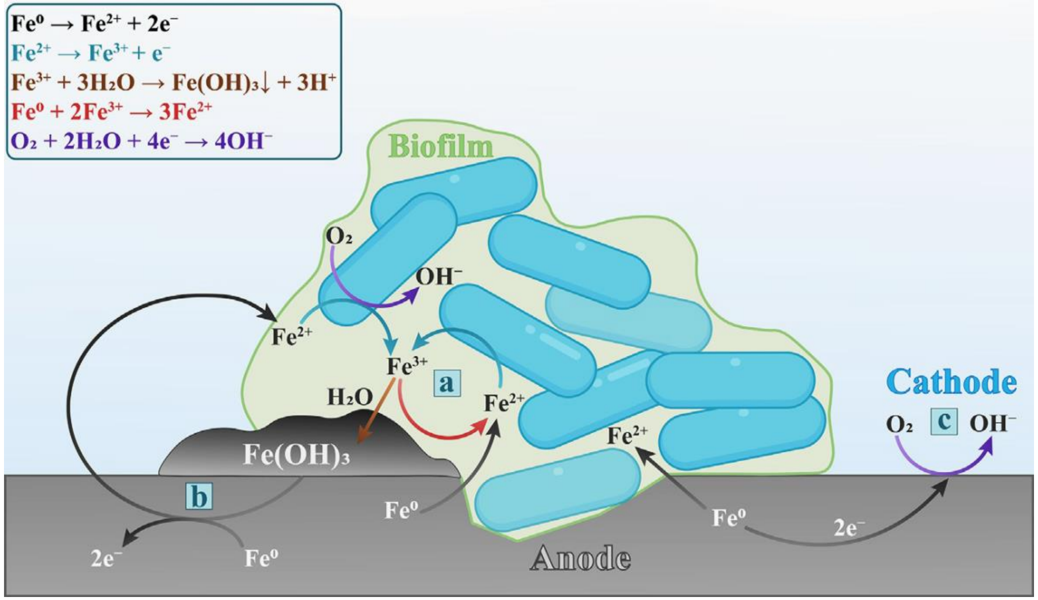
Microbiological corrosion

- Corrosion relevant microorganisms (bacteria, fungi) can deteriorate solid surfaces by:
  - **their presence,**
  - **aggressive metabolites,**
  - **biofilm formation (niche for corrosive microbes) grown on solids influences the metal surface**:
    - alters the transport of chemicals from or toward the metal surface.
    - forms a diffusion barrier for certain chemical species;
    - protective (passivating) film can be removed by biofilms,
    - oxidation-reduction conditions change,
    - alters the structure of an inorganic passive layer, releases/removes the metal ions from the surface

Why are these microbes corrosive?

- **Aggressive metabolites of corrosion relevant microbes (**bacteria (autotroph, heterotroph, acid producer, slime former etc)), fungi, algae, cianobacter, protozoa
- **Stimulated corrosion** → localized
- **Biofilm** → patchy, heterogenic, source of non-specific affects
  - **Microbial cell clusters,** discrete aggregates of densely packed cells
  - **Interstitial voids** without any biomass
- **Diverse chemistry within biofilms**
- **Uneven distribution of oxygen near to the surface**

MIC Mechanisms by which Fe^{2+} -oxidizing microbes accelerate Fe0 corrosion



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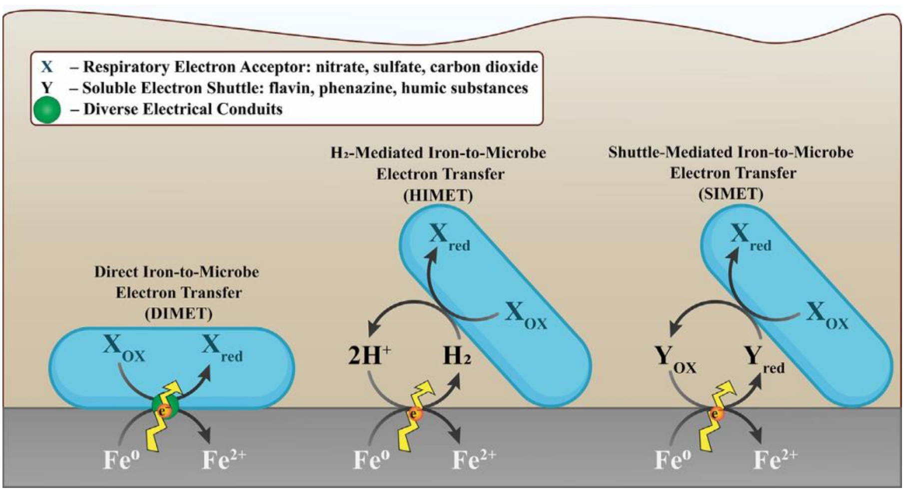
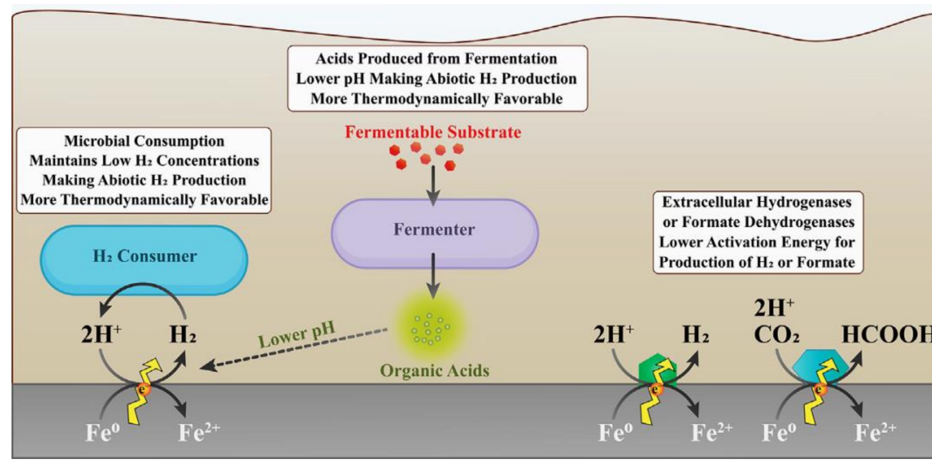
(a) Fe2+ derived from abiotic Fe0 oxidation is microbially oxidized to Fe3+, with the consumption of O2 and the formation of poorly soluble Fe(III) oxide minerals such as Fe(OH)3; (b) Abiotic oxidation of Fe0 coupled with the reduction of Fe(OH)3 generating Fe2+; (c) Creation of an oxygen concentration cell with electrons derived from Fe0 oxidation in an O2-poor anodic region transferred to a region where O2 is available.

Anaerobic iron corrosion mechanisms



Mechanisms for microbial Fe0 oxidation under anaerobic conditions.



Microbial mechanisms for enhancing Fe0 corrosion with H_2 or formate serving as an electron shuttle. Microbial production of organic acids may decrease the pH near the Fe0 surface. Extracellular hydrogenases or formate dehydrogenases can lower the activation energy for the production of H_2 or formate.

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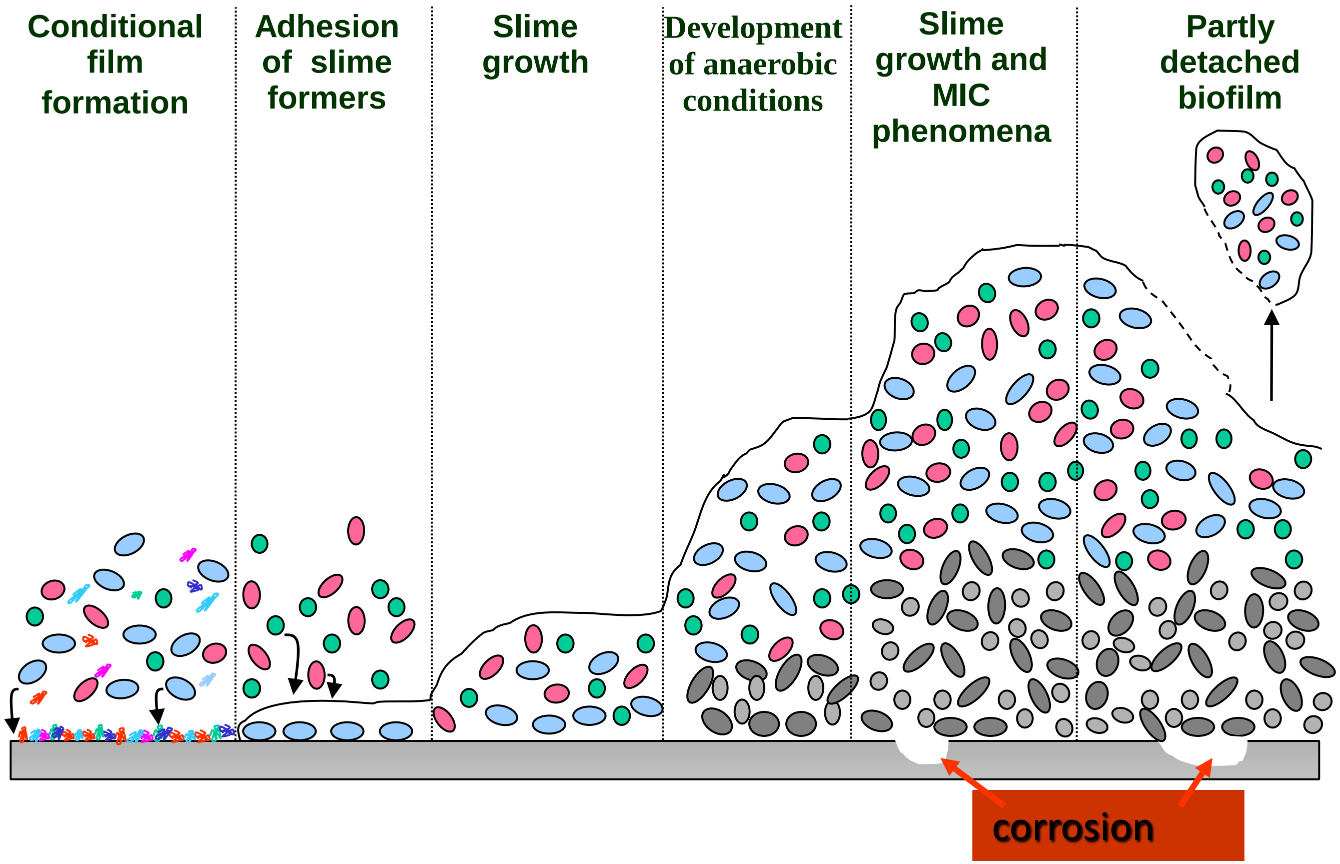
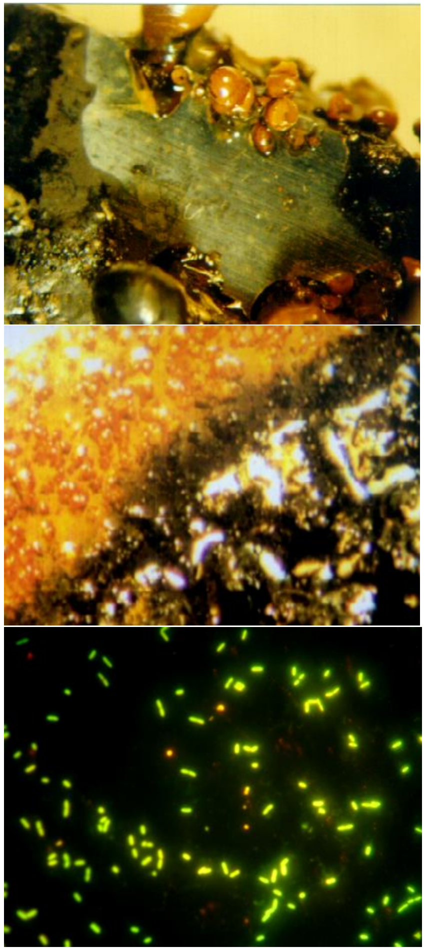
Biofilm formation



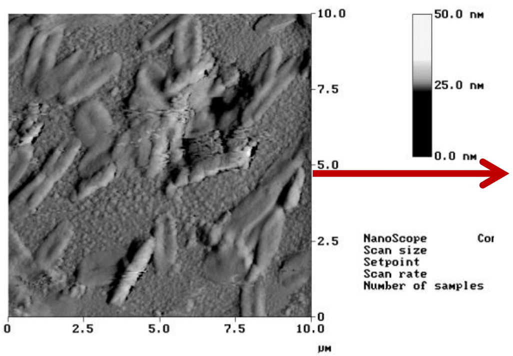


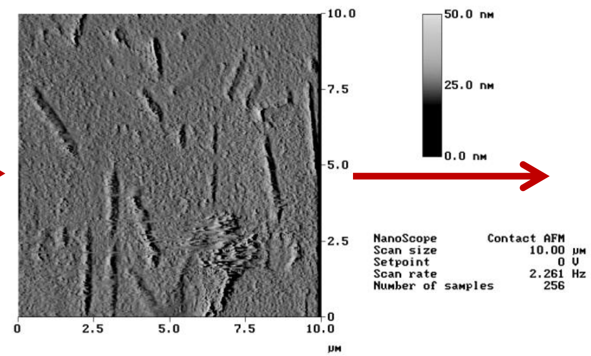
Microorganisms on surfaces

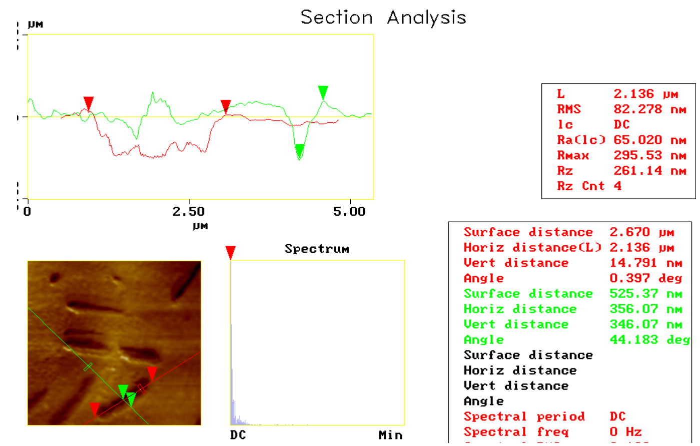
Acidproducer microorganisms on mild steel:

etched metal surface; beneath the microbes:

350 nm deep grooves in 1 day!

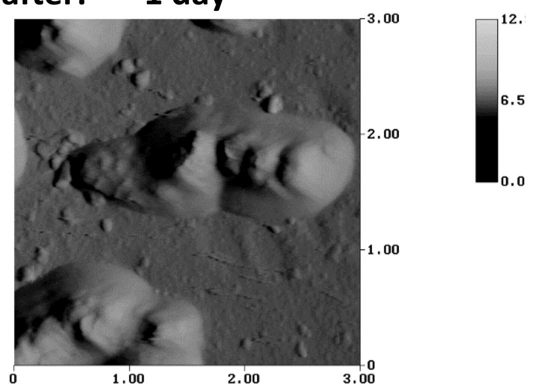




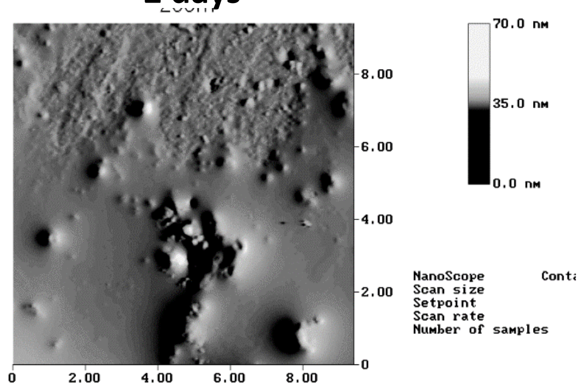


Time-dependent biofilm formation on pyrite by *Leptospirillum ferrooxidans*

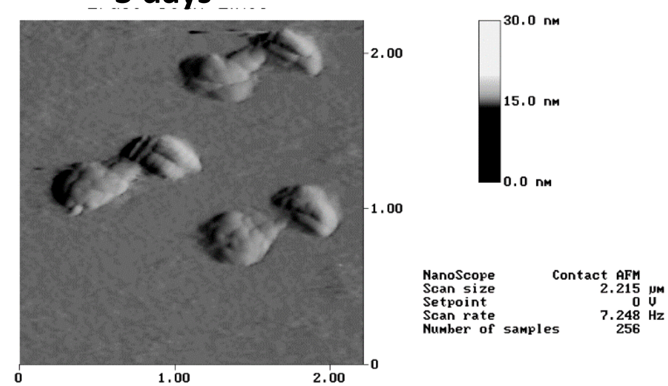
after: 1 day



2 days



3 days



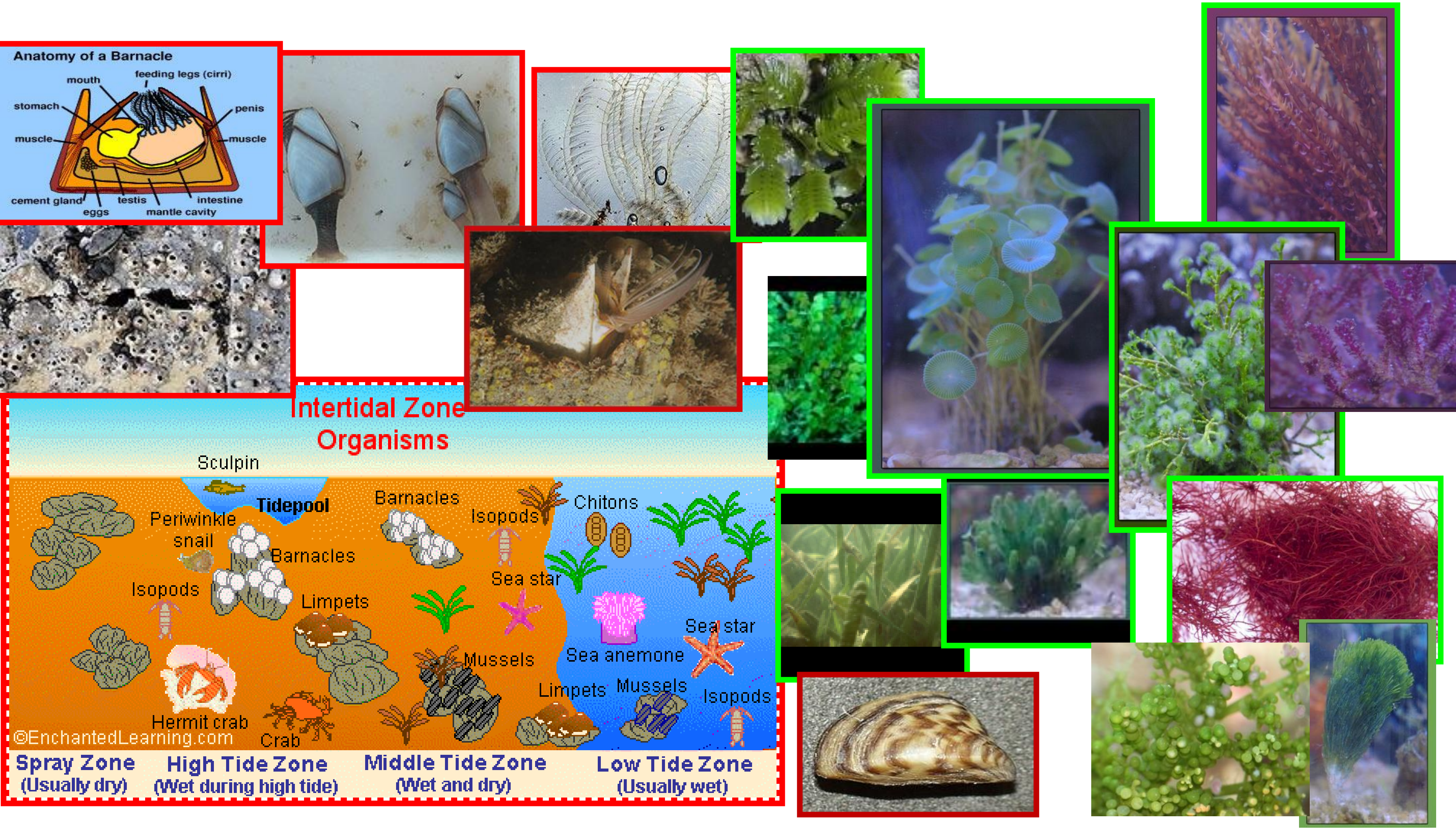
Microorganisms, biofilm, biofouling

- **Biofouling is everywhere, grown on biofilms**:

  ship, semi-submersible, fixed offshore structure, emergency fire pump suctions, fixed platform access ladders, heat exchangers, water-cooling pipes, propellers and ballast water tanks, power stations , offshore structures




Macroorganisms in biofouling (bivalves: e. g. clams, oysters, mussels, barnacles, mollusk, larvae, algae, hydroids, crabs, worms, shrimps)



Inhibition of microbial activity by dissolved chemicals

- **Chemicals** (single/mixture)
  - ♣ ♣ **kill microorganisms**
  - ♣ ♣ **inhibit the microbial growth and the biofilm formation**
  - ♣♣ **broad spectrum** (bactericid, fungicid, algicid)
  - ♣ ♣ **optimal dose**
  - ♣ ♣ **most appropriate for the system**

Inhibition of microbially influenced corrosion by coating

- **Inhibition of growth, proliferation, colonization of micro- and macro-organisms**

For preventing and controlling biocorrosion:

- **traditional coatings incorporated with** biocides (polyurethane, fluorinated compounds, epoxy resins, polyimides, silicone, coal-tar epoxy, and polyvinyl chloride;
- **antibacterial polymers containing quaternary ammonium compounds**: synergistic effect between the positive QUATS ions and halide anions
- **conductive polymers:** polypyrrole, polyaniline, polythiophene: antibacterial ability against Gram-negative and Gram-positive *bacteria*; poly(*N*-methylaniline: against SRB; Ag nanoparticles -coupled bithiophene hybrid films against algae, etc.

Coating types, deposition techniques

- **Chemical** (**Langmuir-Blodgett molecular and self-assembled layer deposition**, sol-gel technique, plating);
- **Physical** (sputtering, bonding, condensation)
- **Mechanical** (spraying, dip and spin-coating, painting);
- **Some others**: plasma-enhanced and metal-organic chemical vapor deposition, atomic layer deposition, ion implantation, surface activated bonding, physical vacuum deposition, radio frequency magnetron sputtering, etc.

Corrosion inhibitive and repellent NANOLAYERS on solid surfaces

The advantages of nanolayers:

- Active molecules are used in smaller quantity,
- Lower costs,
- Reduced environmental impact.

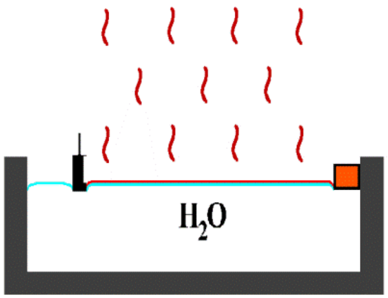
LB film: **fundamental research**

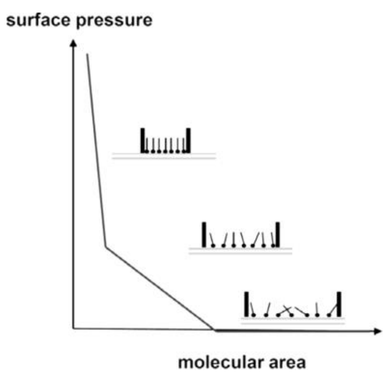
SAM layer: **applied research**

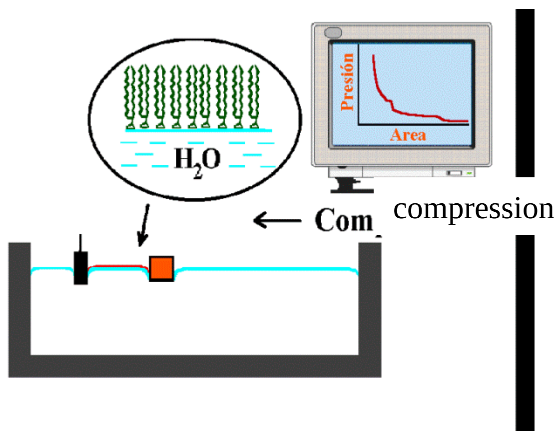
Langmuir-Blodgett film deposition

LB layer:

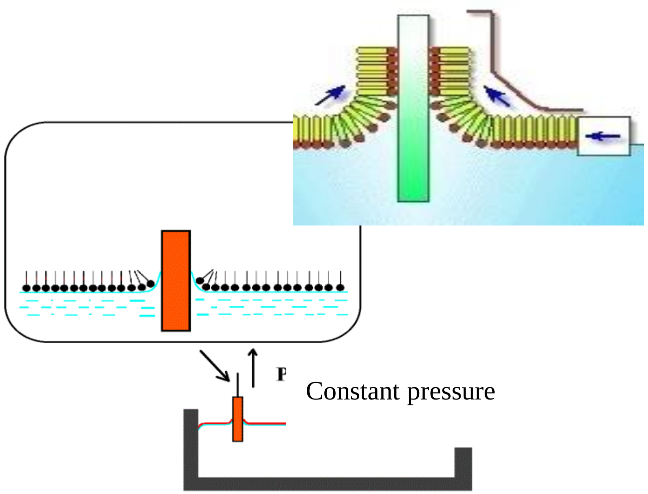
- stabile
- keeps the ordered structure
- film structure is influenced by the pH, t°, salt content of the subphase and by the transfer conditions





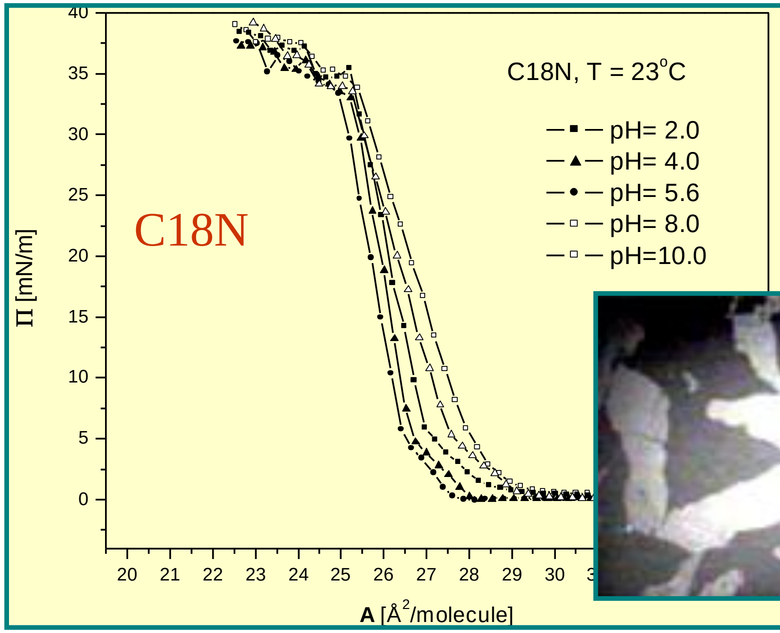


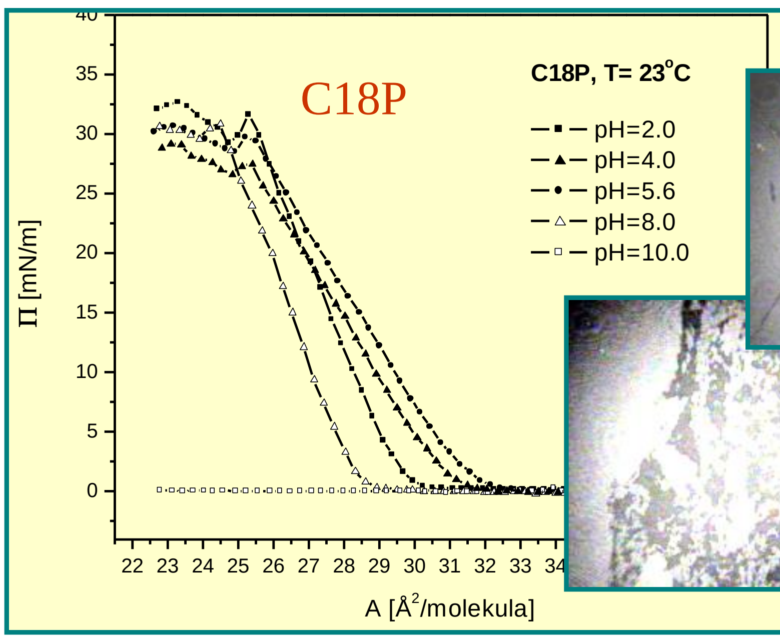
deposition of mono- and multimolecular layers

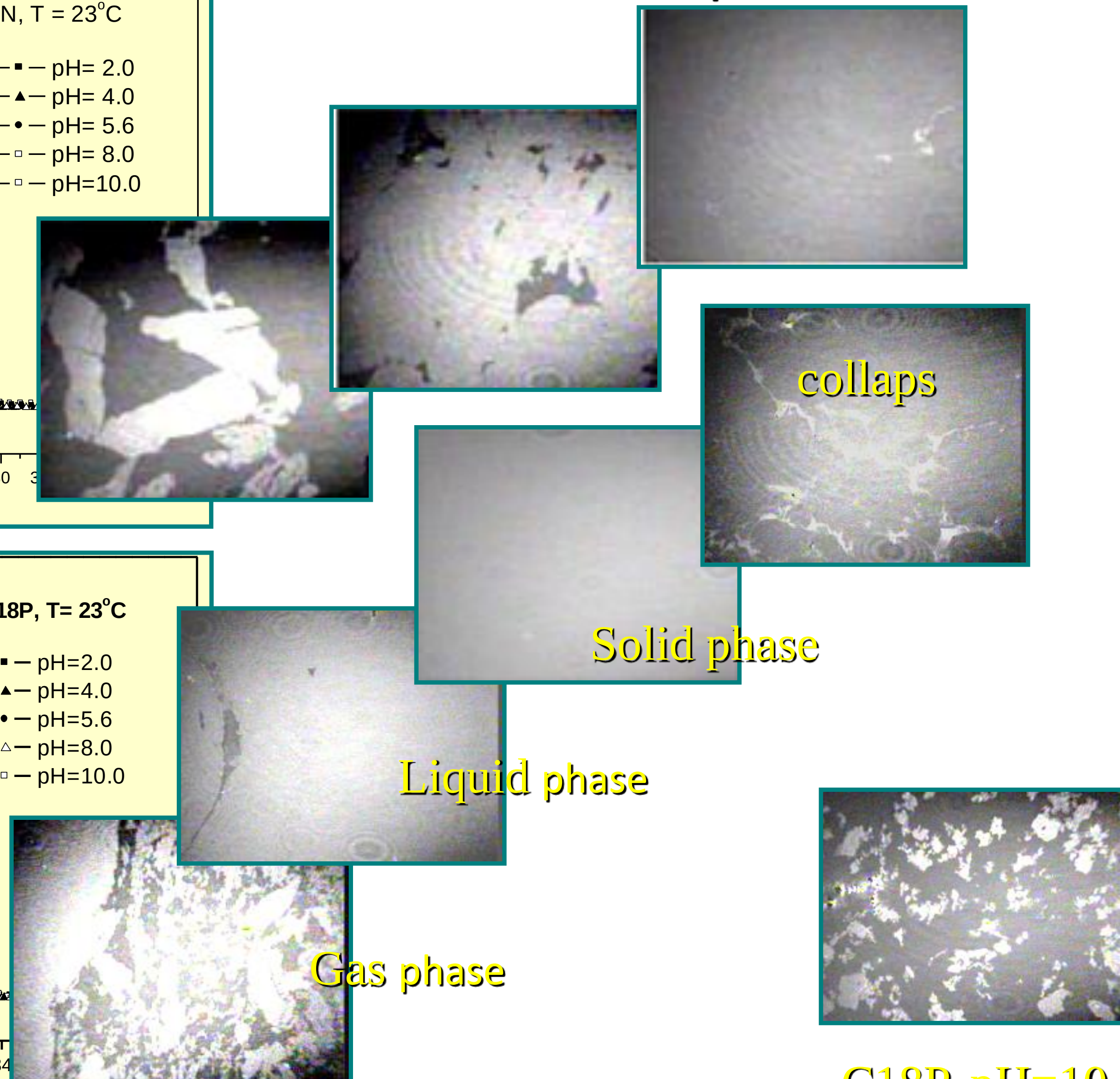


Isotherms and Brewster Angle Microscopic Images

Influence of the pH





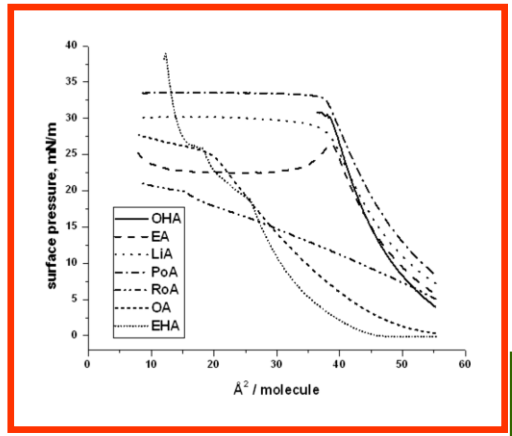


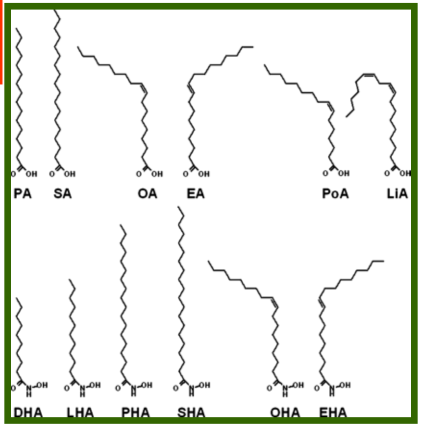
C18P, pH=10

Influence of the molecular structure and the deposition conditions

Deposition is influenced by:

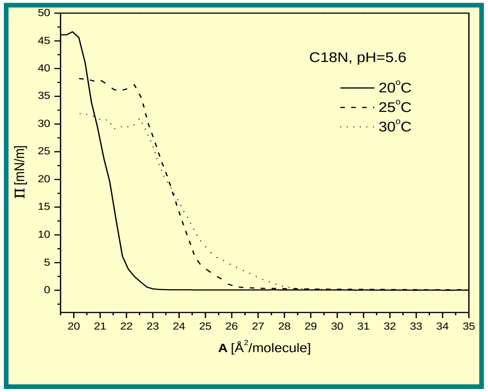
Molecular structure:



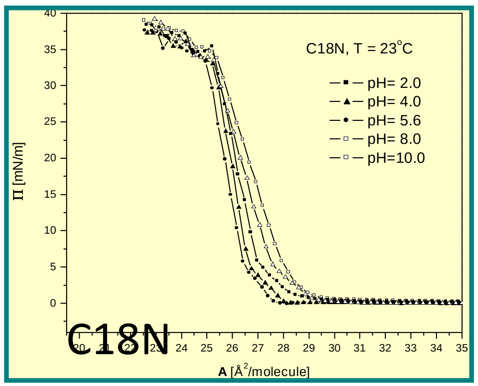


Temperature:

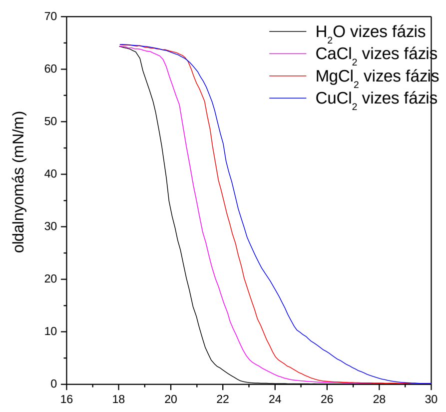
C18N



pH:

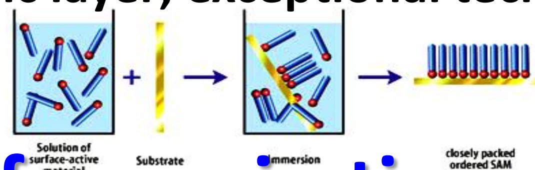


Cations in the subphase:



Self assembling

Protective ultra-thin organic layer; exceptional technological importance in the nanotechnology.



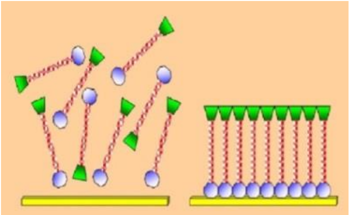
Spontaneous self-organization of molecules (or particles) into well-ordered structure, due to:

♣ **physical and chemical interaction between substrates and amphiphiles**

- **physisorption: $\Delta H < 10$ kcal/mol**
- **chemisorption: $\Delta H > 10$ kcal/mol**

♣ **intermolecular interaction among the amphiphiles**

- **hydrogen bond ; • donor/acceptor interaction ;**
- **covalent bond**



SAM deposition

Organic solvent: disfavored, dissociation is hindered

Aqueous solution: low solubility, layer and rust formation goes parallel, water molecules could be involved into the nanolayer

Surface coverage:

- low: amphiphiles lie parallel to the metal surface
- high: hydrophobic moieties perpendicular to the solid or tilted, all-trans configuration (max. van der Waals interaction)

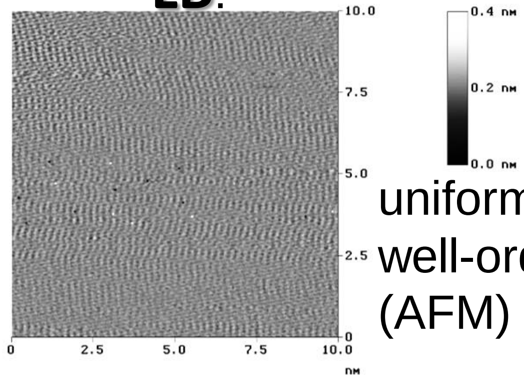
Role of the oxide layer:

active, oxide-free metal surface (alkane thiols)

oxide/hydroxide layer on metal surfaces (head groups: phosphono, carboxylic, amino)

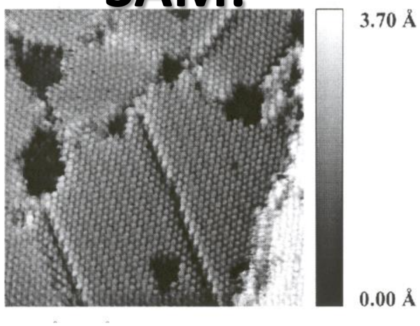
Comparison of LB and SAM layer structure

LB:

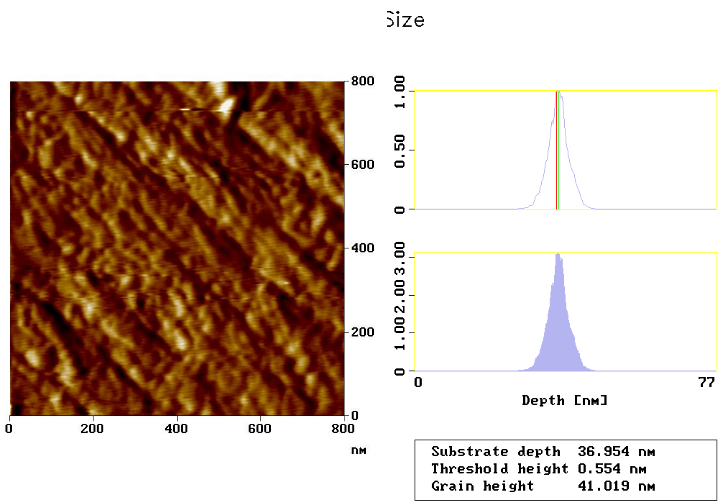


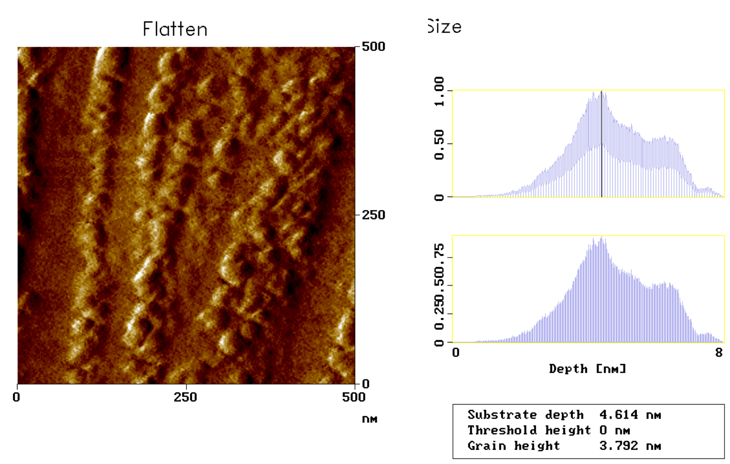
uniform, well-ordered (AFM)

SAM:



defects, vacancies, holes (STM)





Grain size distribution

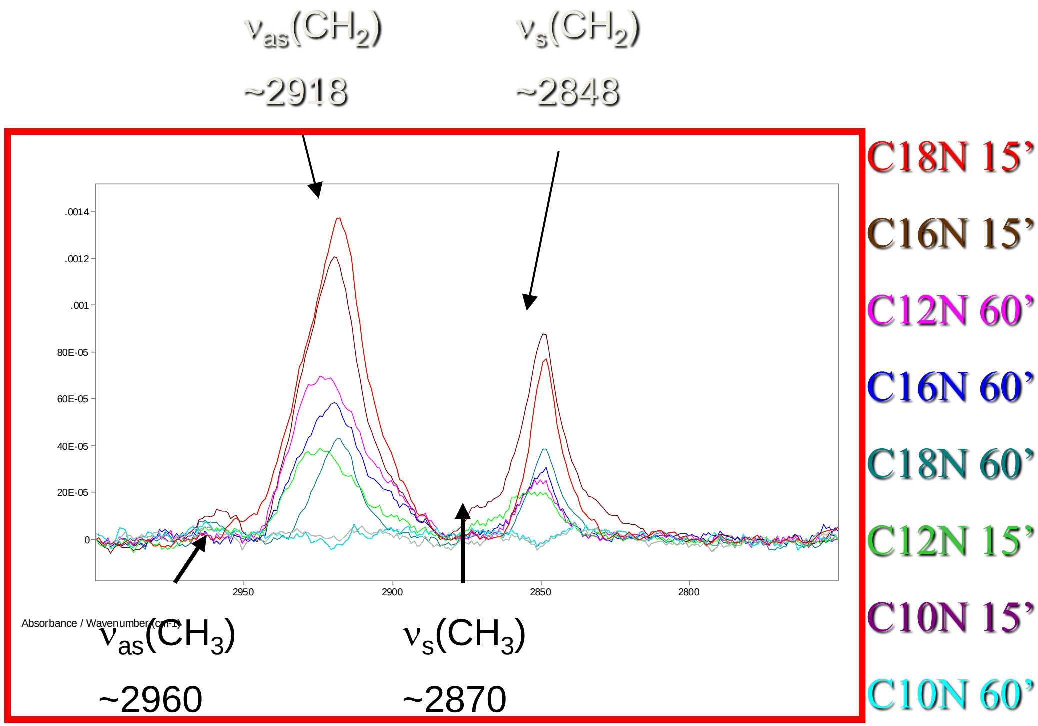
Comparison of LB and SAM layers' wettability

contact angle [o]	copper	copper+C16N LB		copper+C18N LB		copper+C18P LB
		1 layer	5 layers	1 layer	5 layers	1 layer
advancing	78	121	125	128	129	124
recending	31	92	96	95	102	88
surface energy [ergscm^{-2}]	63.49	27.55	25.03	25.66	21.28	30.08

contact angle [o]	iron	iron+C16N SAM	iron+C18N SAM	iron+C18P SAM
advancing recending	120 68	123 72	126 95	68 32
surface energy [ergscm^{-2}]	62.99	40.02	25.66	42.39

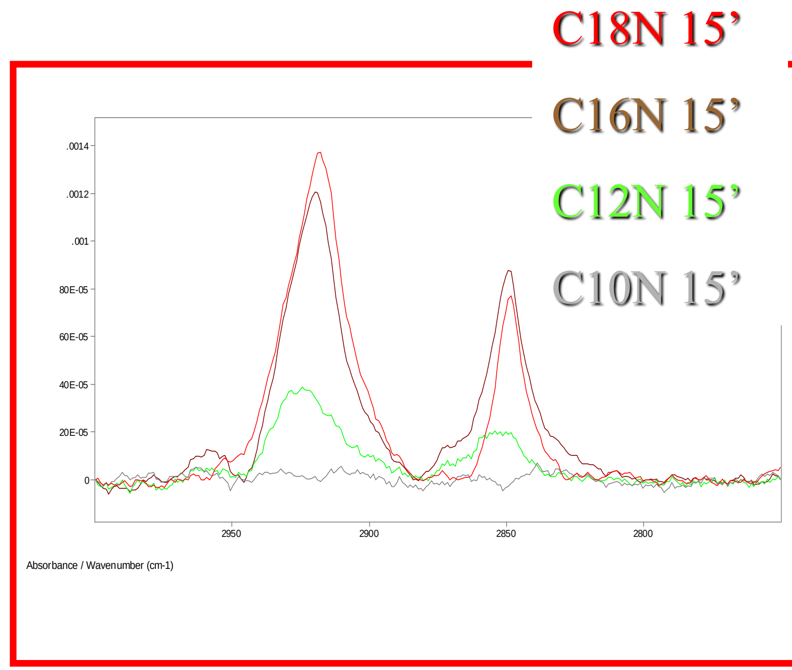
SAM layers on copper surface

IRRAS (0° polarization)

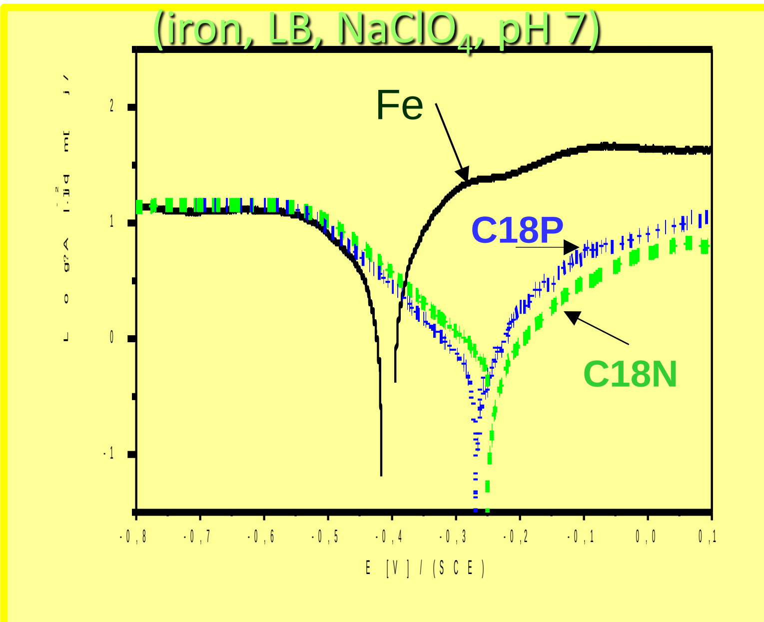


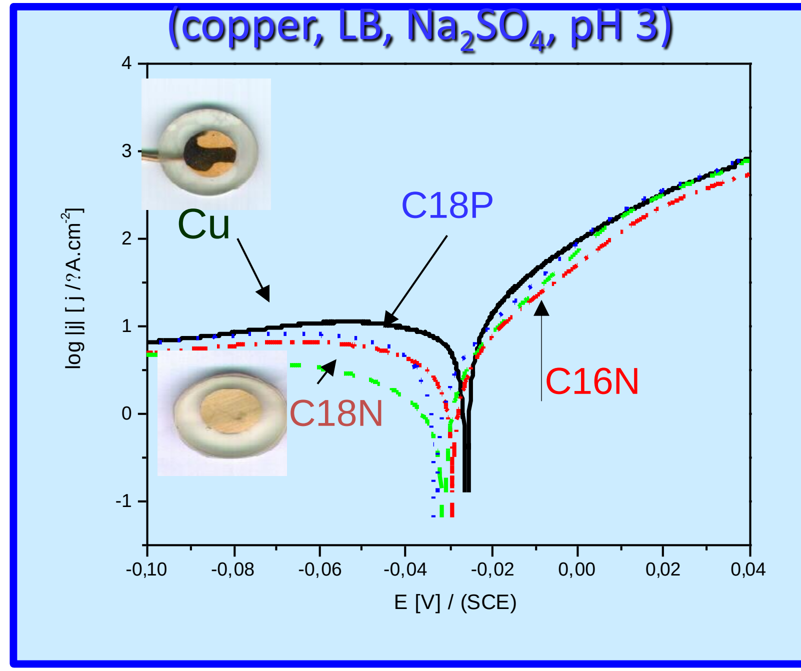
Influence of the deposition time

Influence of the alkyl chain length



Anticorrosion efficiency of LB films



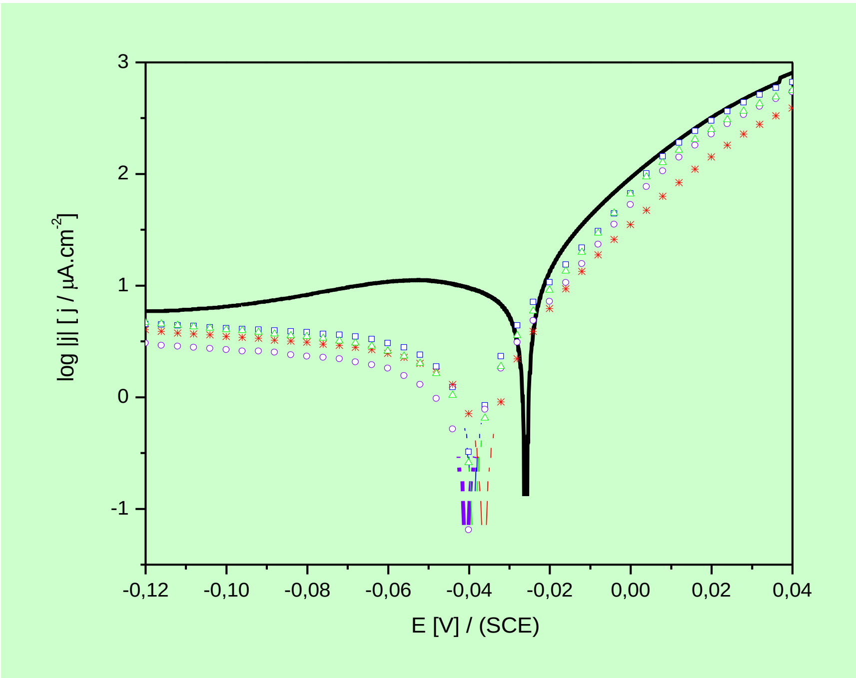


iron electrode	E_{korr} [mV]	j_{korr} [µA.cm^{-2}]	η [%]
blank	-412	2.36	-
C18P	-268	0.13	94
C18N	-251	0.09	96

copper	E_{korr} / [mV]	j_{korr} / [µA.cm^{-2}]	η [%]
blank	- 26	0,91	-
C18P	-33	0,47	48
C16N	-29	0,35	62
C18N	-31	0,25	73

Anticorrosion efficiency of SAM films

copper; polarisation experiments, general corrosion, (Na_2SO_4, pH 3)



— copper

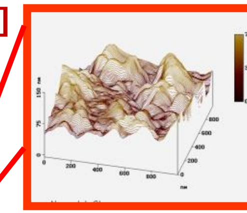
–•– C18N-SAM,

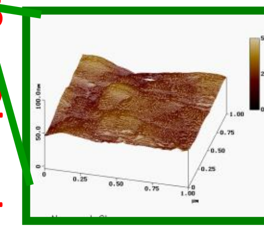
–□– C16N-SAM

–△– C12N-SAM

–✱– C10N-SAM

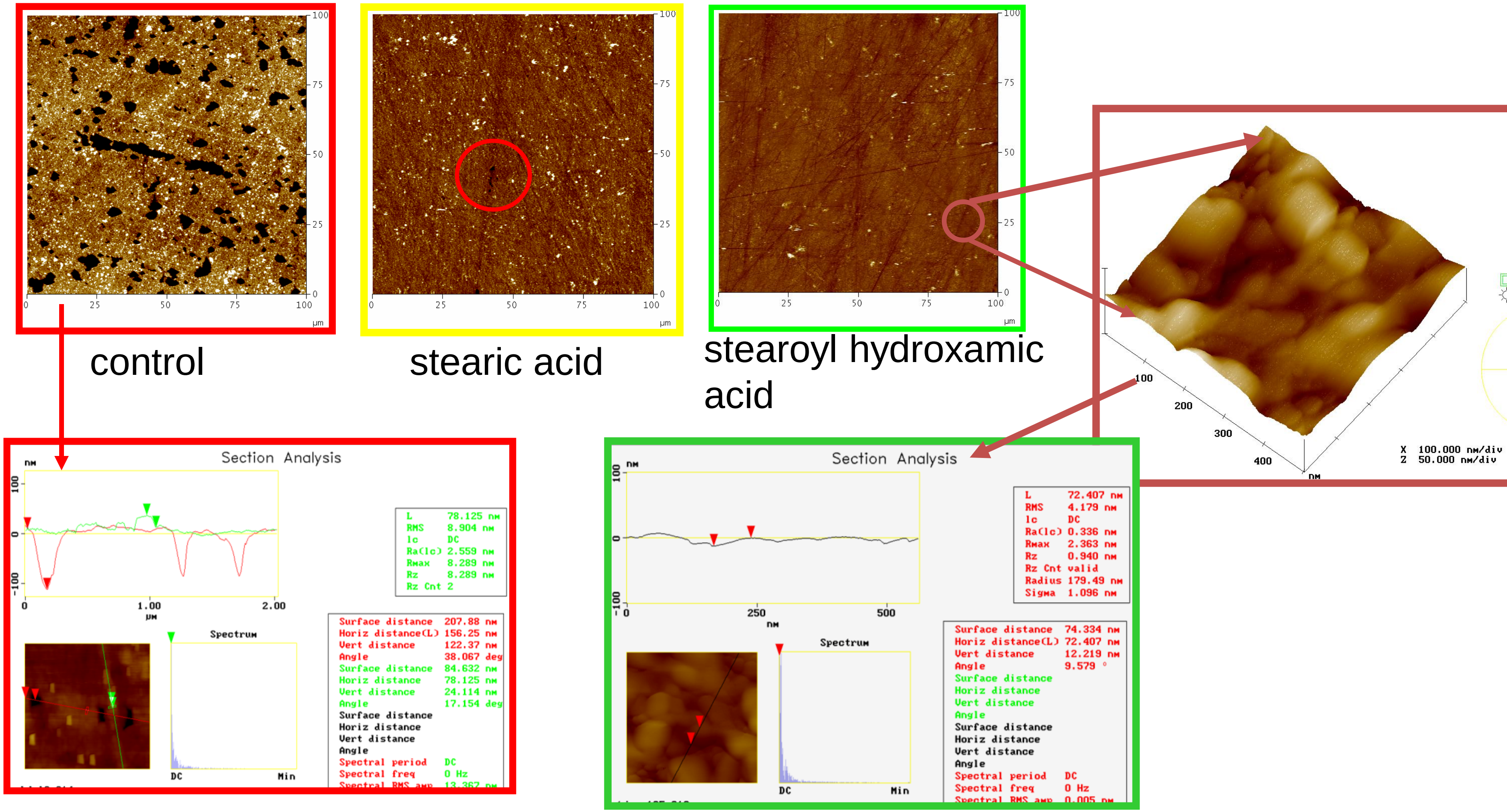
copper	E_{korr} / [mV]	j_{korr} / [µA.cm^{-2}]	η [%]
blank	- 26	0,91	-
C18N SAM	-40	0,14	85
C16N SAM	-39	0,16	82
C12N SAM	-38	0,17	81
C10N SAM	-36	0,13	76





Increase in the alkyl chain length in the SAM layer: anticorrosion efficacy increases

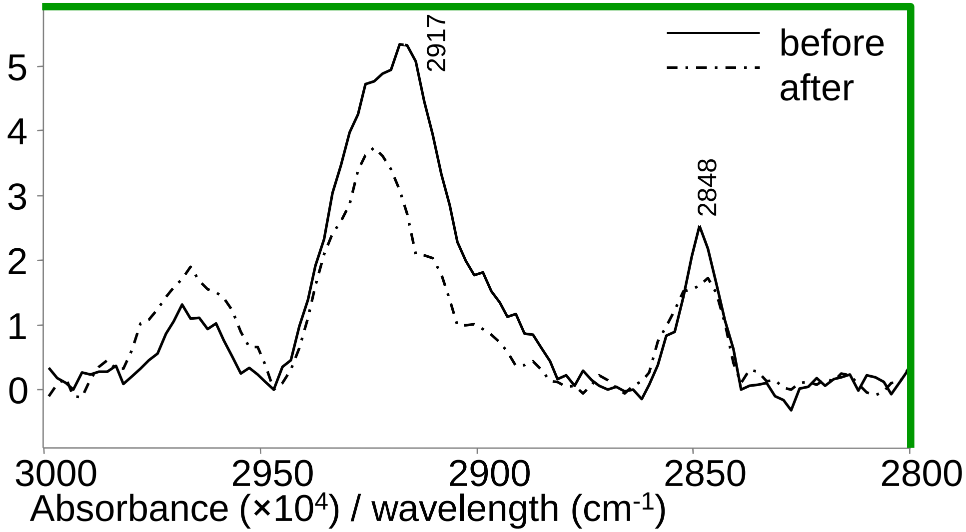
Influence of the amphipile structure in SAM layers on pitting corrosion of copper



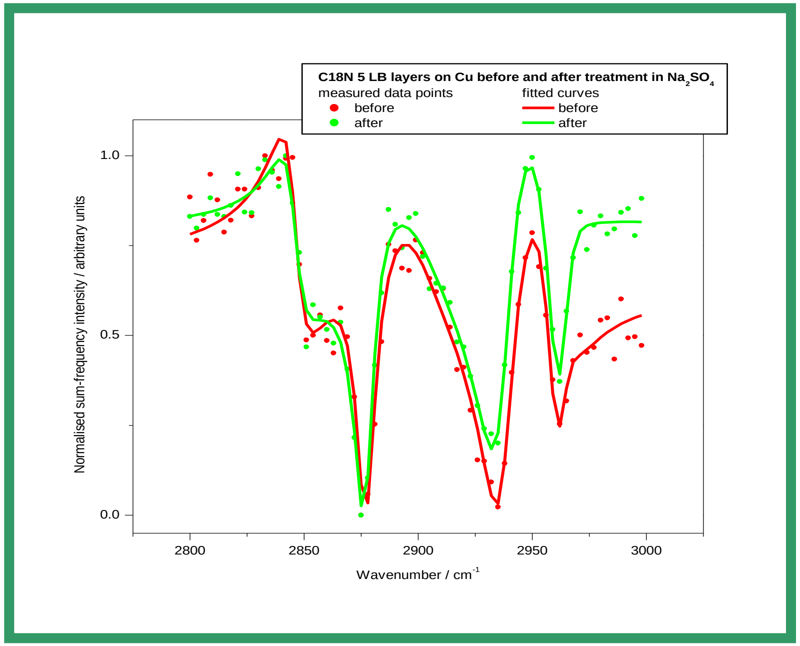
Stability of SAM nanolayers in water

Results of RAIRS and SFG measurements

After the corrosion experiments the nanolayers kept the well-defined ordered structure on the metal surfaces



RAIRS spectra of C18N SAM nanolayers on stainless steel 304-en

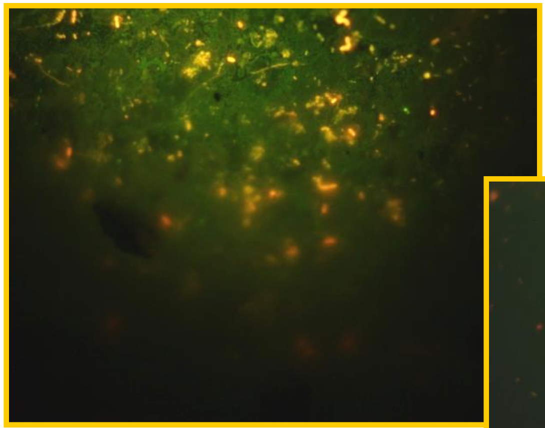


SFG spectra of C18N LB layer on copper

Microbes in biofilms, influence of the structure of the amphiphiles

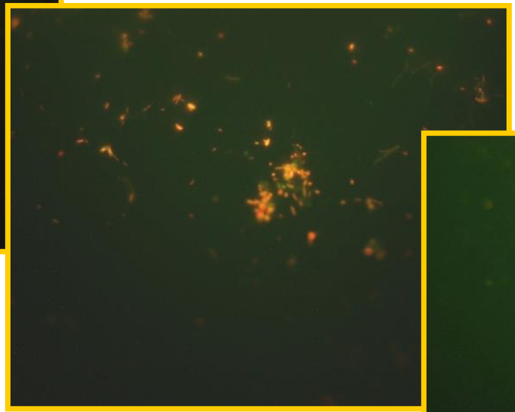
(cooling water, 5 days, SAM)

control



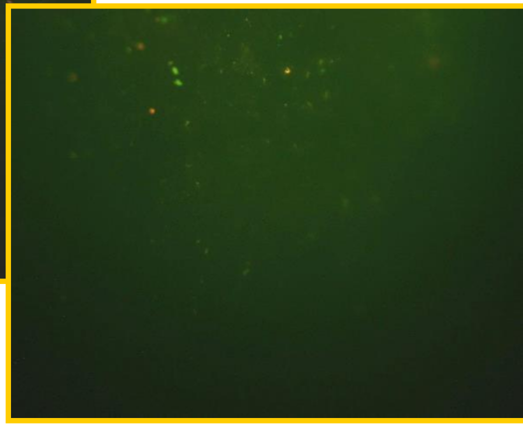
oleoyl hydroxamic acid

-CH=CH_in the side chain



stearic acid

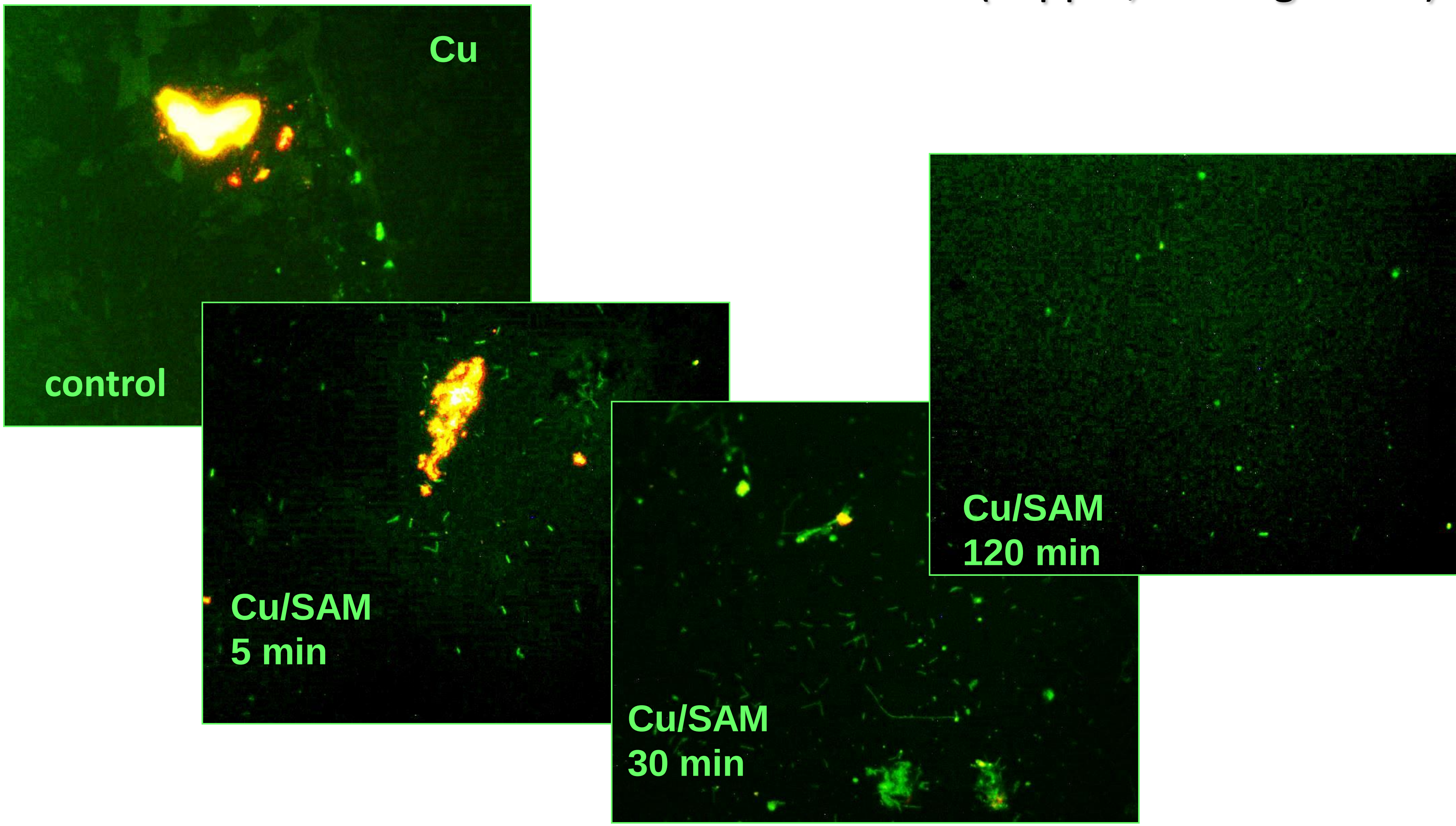
-COOHl head group



stearoyl hydroxamic acid

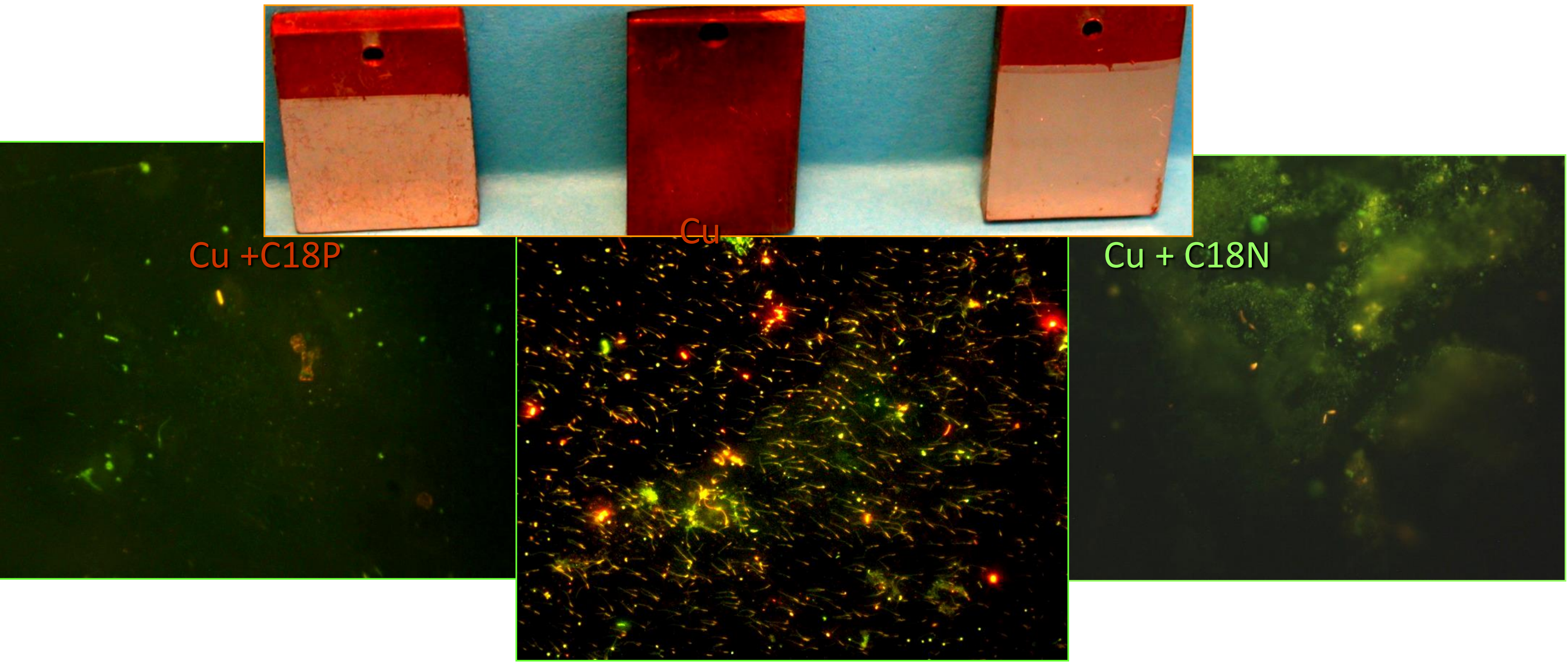
-NH(CO) head group

Influence of the SAM layer deposition time on the biofilm formation after one week (copper, cooling water)

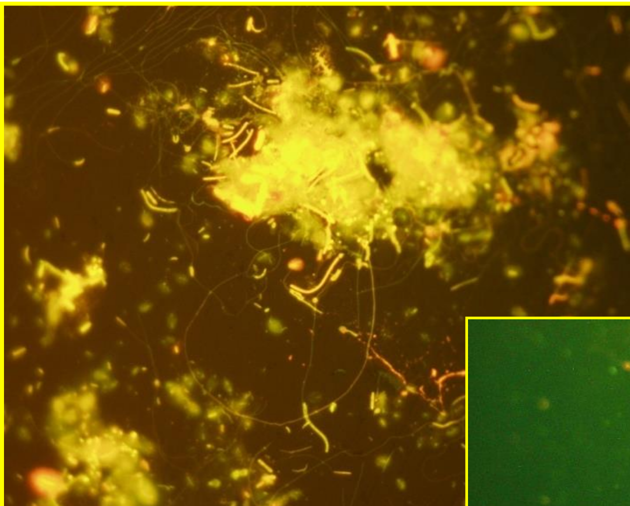


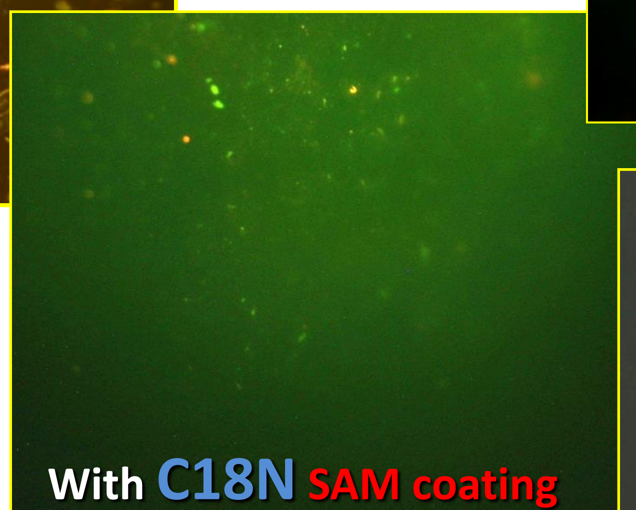
Influence of LB monolayers on corrosion and on bacterial adhesion

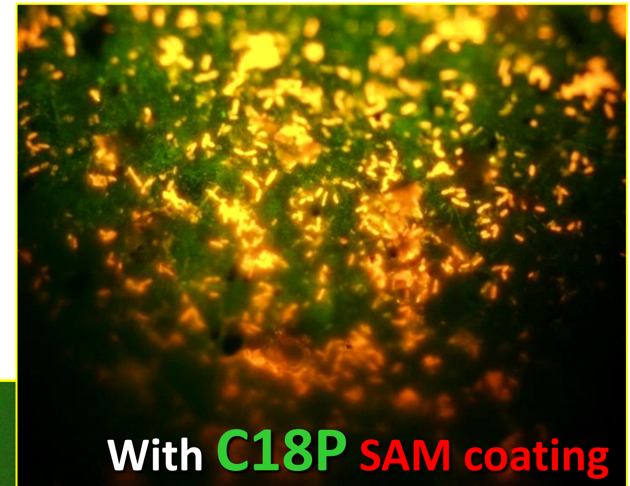
(copper, cooling water, 5 days)

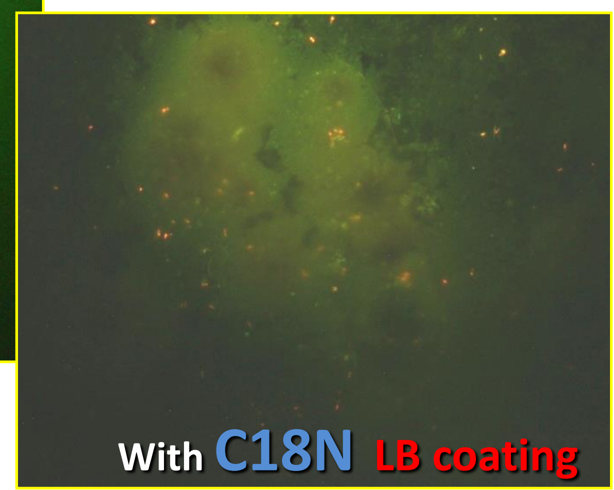


Iron in cooling water, 5 days

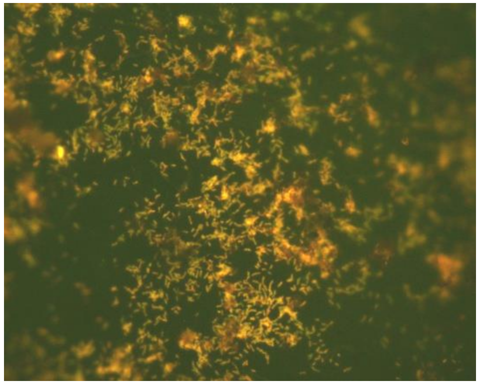

Without coating



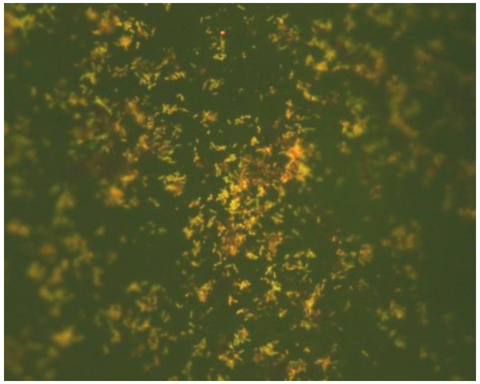




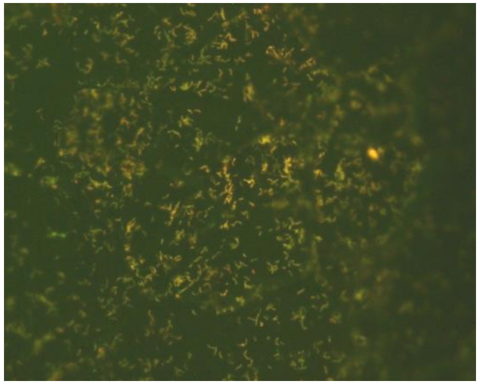
Biofilm of anaerobic sulfate reducers on iron: Influence of self assembling coating time



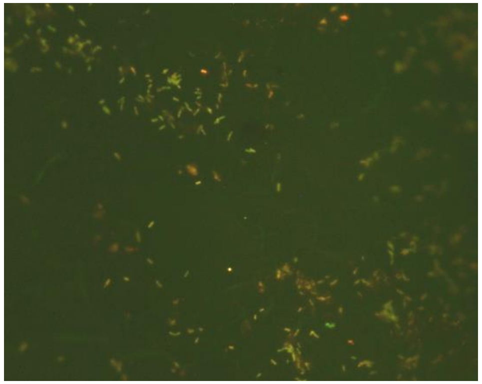
Untreated



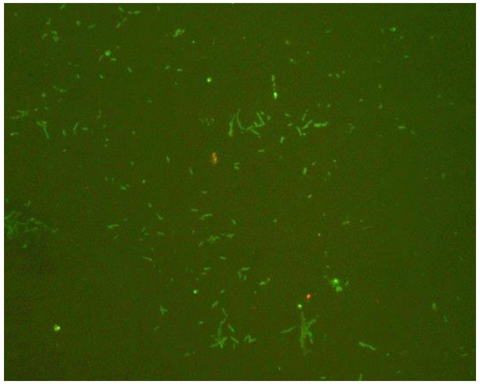
after 5 min



after 15min



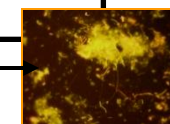
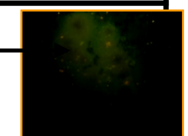
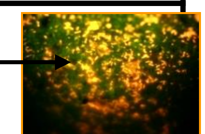
after 30min

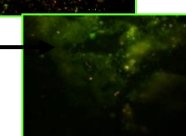


after 60min

Correlation: surface energy and microbial adhesion

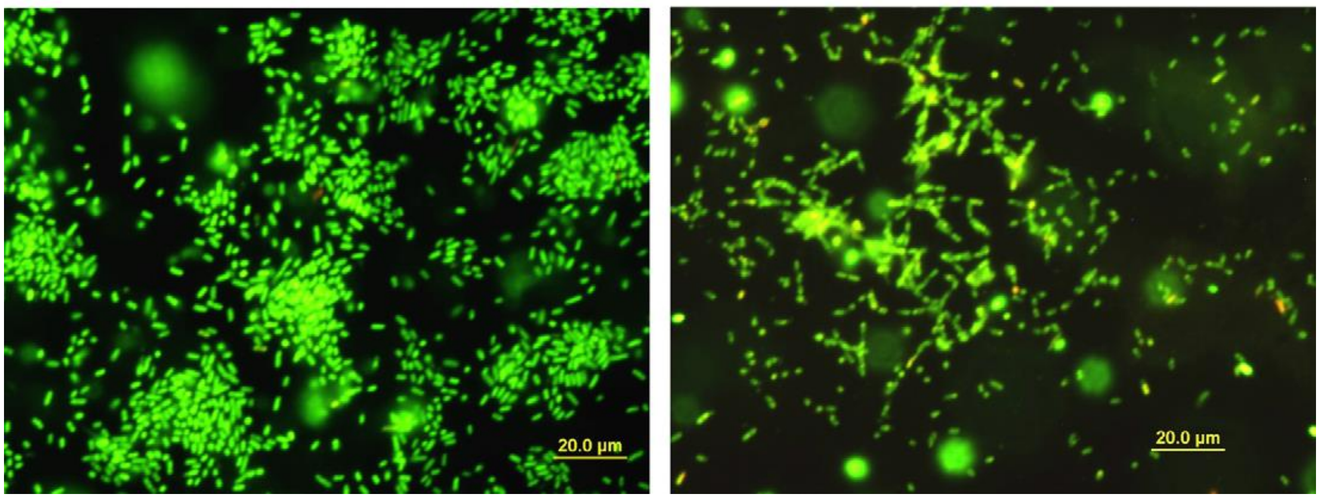
	surface energy [ergscm^{-2}]	microorganisms in biofilms [cellcm^{-2}]
iron	62.99	5.2x10^{5}
+C18N LB monolayer	25.06	3.6x10^{3}
+C18P LB monolayer	42.39	1.6x10^{5}
copper	56.67	1.2x10^{5}
+C18N LB monolayer	25.66	6.8x10^{2}
+C18N LB multilayer	21.28	1.7x10^{2}



Influence of the coating wettability on bacterial adhesion

CH_3 NH_2



bacteria are organized in **clusters** **tree-like** biofilm morphology

NH_2 :hydrophilic *N*-(6-aminohexyl)aminopropyltrimethoxysilane
CH_3: hydrophobic hexadecyltrichlorosilane

Ploux et al.;Colloids and Surfaces B: Biointerfaces 57 (2007) 174–181

Summary on nanolyers that influence the corrosion and biofilm formation, biocorrosion

The corrosion and microbial adhesion/MIC inhibition efficiency by LB and SAM nanolayers of phosphonic and hydroxamic acid amphiphiles depends on the: ⁎ Type of coated metals;

⁎ **Wettability** influences corrosion inhibition and microbial adhesion

⁎ P-LB/SAM on iron : corrosion is controlled; microorganisms invade the surface, colonies are adhered, but the biofilm adheres loosely

⁎ N-LB/SAM on copper: corrosion is controlled; only few cells adhere onto the coated metal surface

♣ **Correlation** between the surface energy and the number of the adhered microorganisms: nanolayer-coated metal of low surface energy: the number of microbes decreases with order of magnitudes, i.e.

♣Low surface energy ⟶ low number of adhered microbes

History of the antifouling coatings

---1500BC-300BC	--- 400 - 1860	1860 - 2003	2003 - 2011	2005 -
Pitch, copper plating	Wax, tar, asphalt, train oil, rosine, Sulphur, arsine	Copper sheathing and heavy metal based coatings	Organotin, copper; Copper sheathing and heavy metal based coatings	Self-polishing coatings with biocides; Copper sheathing and heavy metal based coatings

Toxicity of noble metals to microorganisms :
Ag>Hg>Cu>Cd>Cr>Pb>Co>Au>Zn>Fe>Mn>Mo

TBT:banned, replaced with copper-based anti-fouling coatings.
Copper: banned from 2018 because of their detrimental impact on the environment.

Alternatives to biocide-based and antifouling coatings

- **Electric field:** direct electrontransfer between the solid surface and the microbial cell causing the electrochemical oxidation of the intracellular substances (ferrocen deriv., conductive paints)

- **Microbicidal polymers:** highly active, good environmental compatibility

- **Nanoparticles, nanocomposites with antibacterial activity:**
  - **Nanostructured metals on support (e.g. SiO2)** ($MeSiO_2$, Me: **Ag**, Cu) sol-gel technique

  - Metallic silver, polymer-silver nanocomposites nanosized Fe_2O_3

Biocide in the antifouling paints

- the biocide must be mobile, migrates to the coating surface and across the cell membrane to destroy microorganisms,
- loss of biocides by aqueous extraction and dissolution,
- increased concentration level is necessary,
- to reduce the amount of the biocides:

encapsulation: smart coatings.

Smart, self healing and slow release coatings

For inhibition of corrosion and microbially influenced corrosion:

- **increased functionalities** by
  - **Encapsulation of additives** (e.g., inhibitors, antifouling chemicals);
  - **Increased superhydrophobic character**;
  - **Chemical modification of the organic matrices.**

Why are „smart” these coatings?

- Self-healing coatings : **spontaneous or stimulated repair of damage under external stimuli** (mechanical impact, solvent, heat, light, etc.)

- Nano/microcontainers in a coating: **combination of different healing components in the same or different capsules;** optimized coating with different abilities; synergically increased self-healing activity (e.g., anticorrosion, antifouling, etc.)

Self-healing materials

- **Combination** of initiators, catalysts and co-monomers embedded in the same matrix.
- **Active material:** released from capsules and spheres.
- **Important:** compatibility of microcapsules with paint components; stability of the capsules in liquid paint and the duration for drying of the layer;
- **Two main types of carrier structures**:

  capsules and spheres

Capsules and spheres

- **Capsules**: spherical, hollow containers with an outer protective shell and inner core with active material
- **Spheres**: matrix structure with solid, usually homogeneous composition with dispersed active materials.

Based on diameter: nano- and microcapsules/spheres.

Release of the active substances

- **Matrix type spheres**: environment (temperature, pH, ionic strength) initiates water take-up, continuous leaching of the active materials by diffusion.

  Inhibitors and antifoulants leach from paints into an aqueous environment, repulsing or destroying organisms.

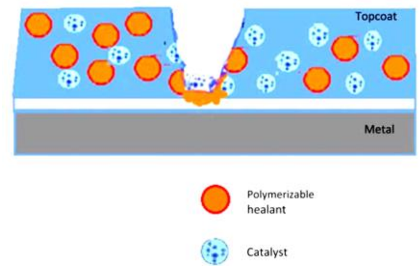
- **Core–shell capsules**, self-healing materials are released upon rupture of the shell capsule or by diffusion.

Types of smart antibacterial coatings

- **Coatings with bactericidal activity** (biocide, antifouling material, quat. ammonium compounds, etc.) **triggered by stimulus/stimuli**

- **“switching” or altering their antibacterial activity in response to stimuli** (“kill and release” coating)

External stimuli for healing processes

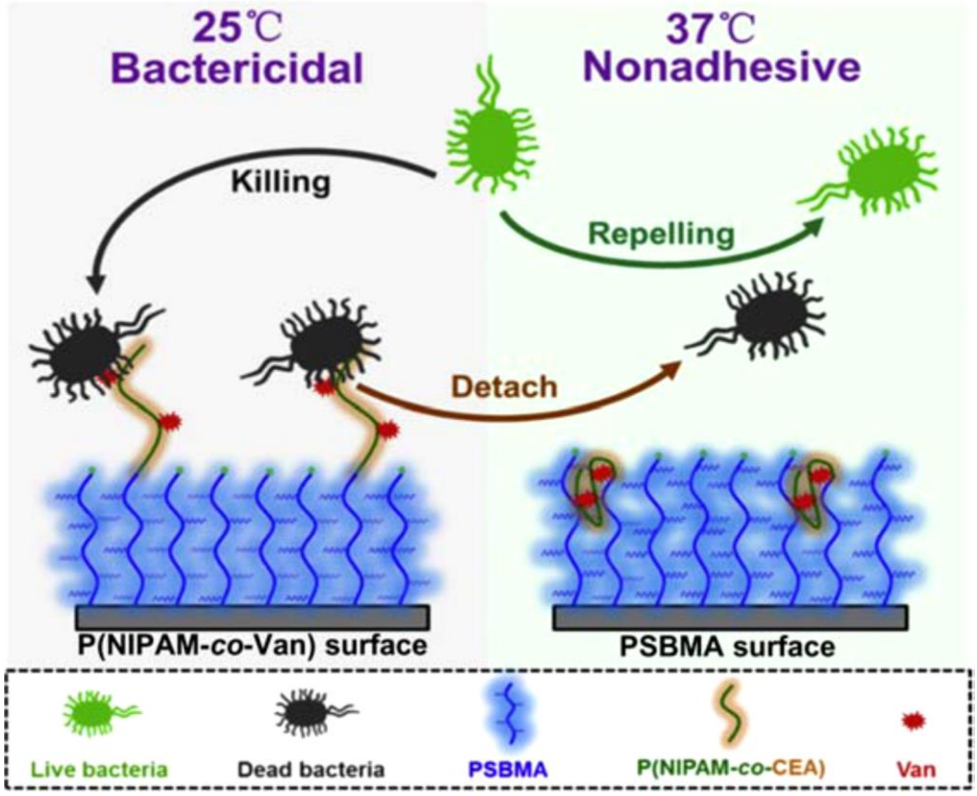


- **Mechanical influence:**
- **Temperature**:

open/close the capsules filled with active ingredients (shell of the capsules contains strong polyelectrolyte).

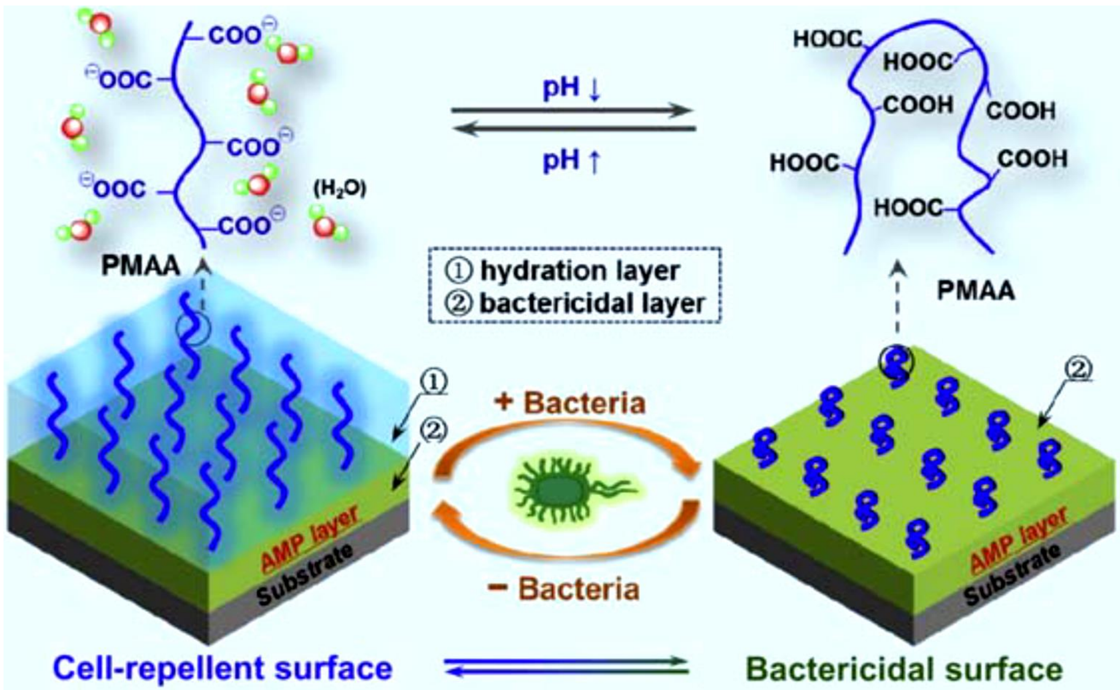
- **Changes in:**
  - **pH values**: capsules open or close depending on the pH values (shell contains polyelectrolyte, mesoporous nanoparticles);
  - **ionic strength, solvent; light, magnetic field, enzymatic degradation etc.**

Temperature-triggered switching between bactericidal and bacteria-repelling functionalities



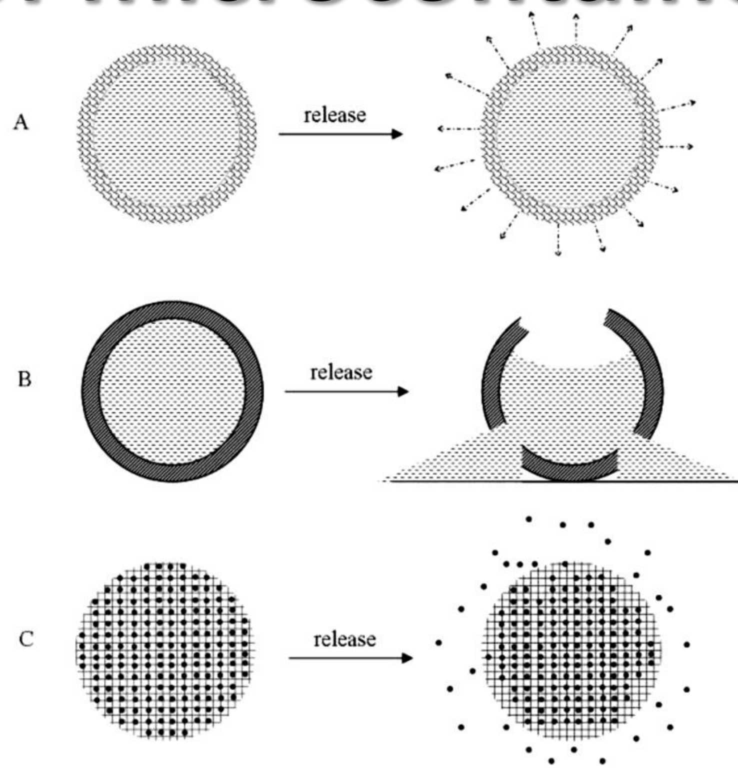
X. Wang, et al., ACS Appl. Mater. Interfaces 9 (46), **2017**, 40930_40939.

pH-triggered switching between a bacteria-repellent and bactericidal surface



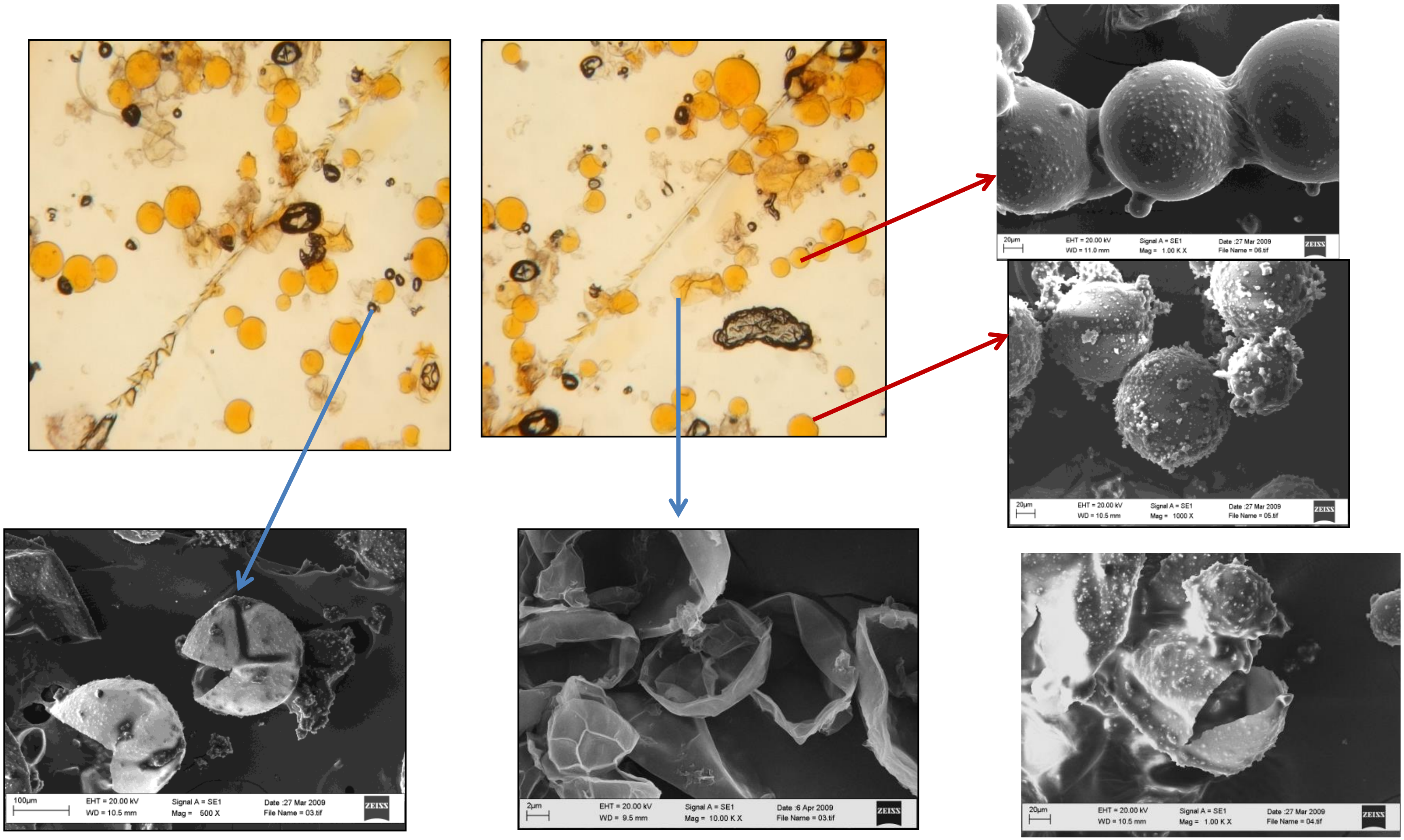
S. Yan, H., et al, ACS Appl. Mater. Interfaces 8 (37) (**2016**) 24471_24481.

Structure and release mechanism of microcontainers

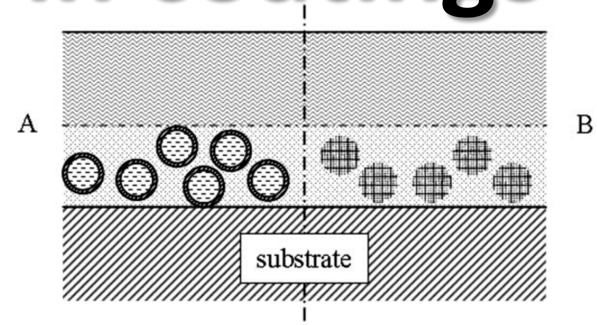


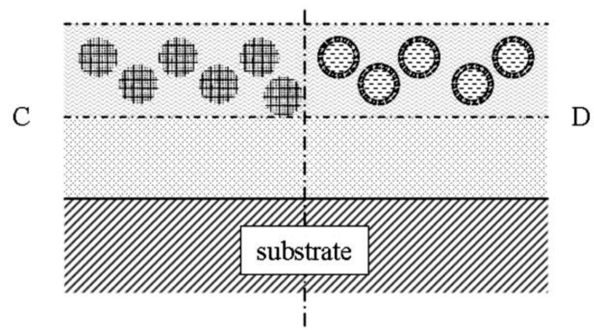
A) core–shell capsule with permeable shell, slow release by diffusion
B) core–shell capsules with dense shell, release at rupture of capsule wall;
C) solid matrix or porous sphere, slow-release by diffusion.

Microcapsules



Microcapsules and microspheres in coatings





A) Micro- or nanocarrier core–shell capsules with self-healing liquid
B) Slow-release spheres with homogenously dispersed inhibitor;
C) Antifouling carrier in the top coat: slow-release spheres
D) Core–shell capsules with permeable shell.

Corrosion under coating with inhibitor and microcapsules filled with inhibitor

commercial inhibitor in the coating

Inhibitor ‚A' involved in the coating

Inhibitor'A' in microcapsules

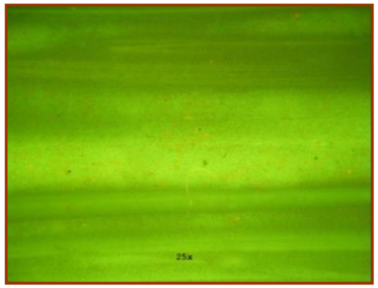
poly(ASSA)

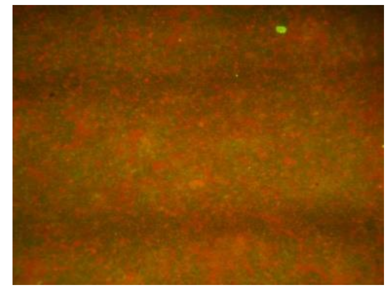
poly(uretane deriv.)

Microbial adhesion on coated smart surfaces (river water)

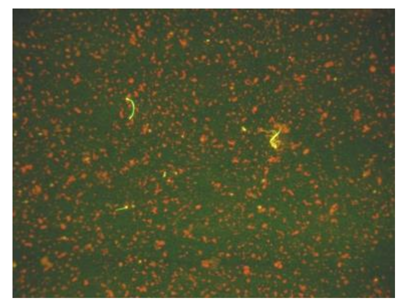
after: 1week 15 weeks

Coating without additive

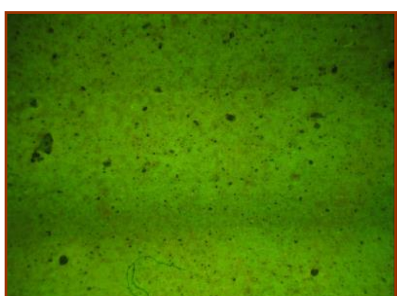


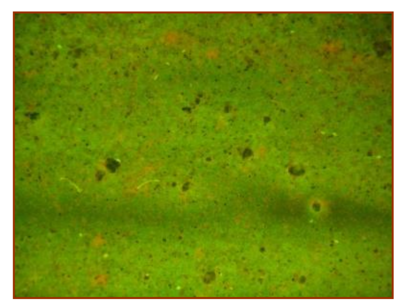


Coating with dissolved inhibitor



Coating with microcapsuled inhibitor

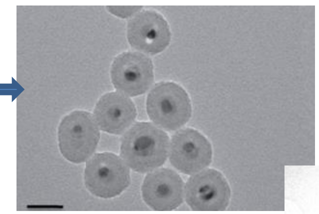




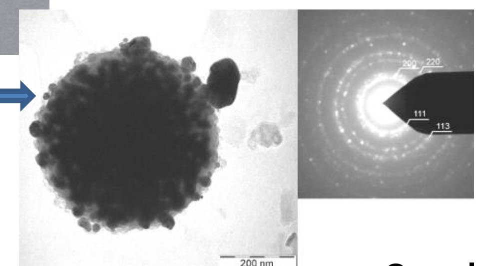
Types of silver nanoparticles

Ag in/on nanospheres:

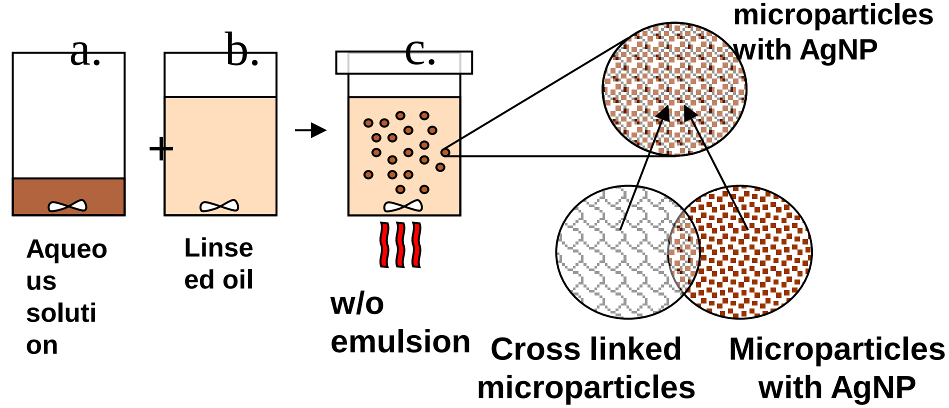
- in SIO_2 microspheres:



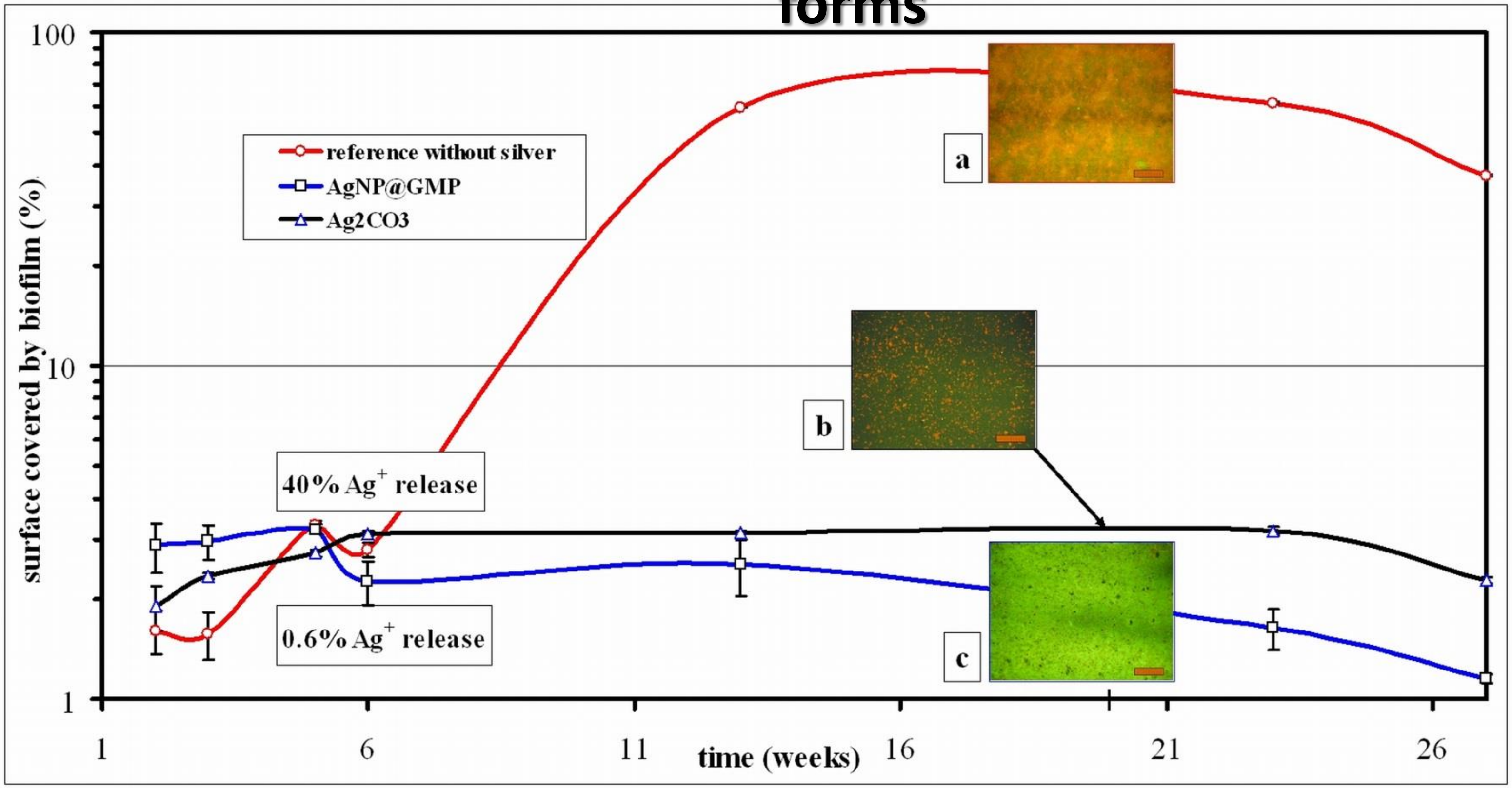
- on SiO_2 microspheres



- incorporated into biopolymer matrix



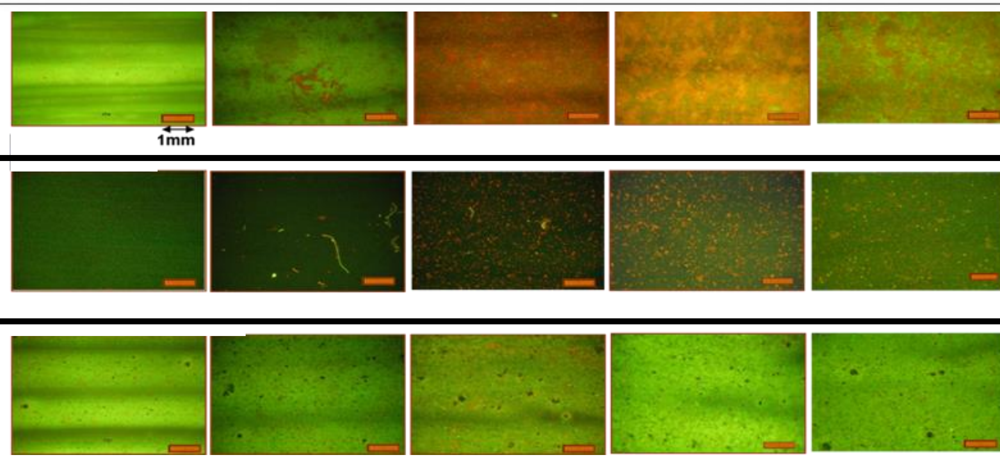
Antifouling efficacy of coatings with silver applied in different forms



Without silver

Ag_2CO_3

AgNP@GMP



Effect of the silver on the fouling

- **Silver nanoparticles** used either in combination with **inorganic** (SiO_2) or with **organic particles** (biocomposite) influenced the life cycle and adhesion of microorganisms.
- Algae growth: controlled **by slow release of silver** (Ag/SiO_2 NP).
- Encapsulated silver in coatings: **elongated antifouling efficiency**.

Summary

The anticorrosion and antifouling effects of nanolayers and smart coatings were demonstrated.

- **Molecular films on different metals:** actively inhibited both the corrosion and the microbial adhesion. The effectiveness depends on the molecular structure (alkyl, alkenyl chain, head groups) as well as on the deposition conditions (**LB film**: pH, ions, temperature; **SAM**:deposition time).
- **Smart coatings: self-healing activity**: core-shell structure, release of the active materials from the spheres : by external stimuli (mechanical impact, pH, ion strength, temperature). **Slow release**: continuous diffusion/dissolution of the active materials from the spheres. Effective inhibition of corrosion and biofilm/biofouling formation.

Colleagues involved into the work

T. Rigó (LB,SAM)

L. Románszki (LB,SAM)

L. Szabó (SEM,TEM)

É. Pfeifer (IR)

T. Keszthelyi (SFG)

T. Szabó (microcapsules)

E. Kálmán†

Thanks for attention

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