

Nanotechnology for the Consolidation and Cleaning of Wall Paintings

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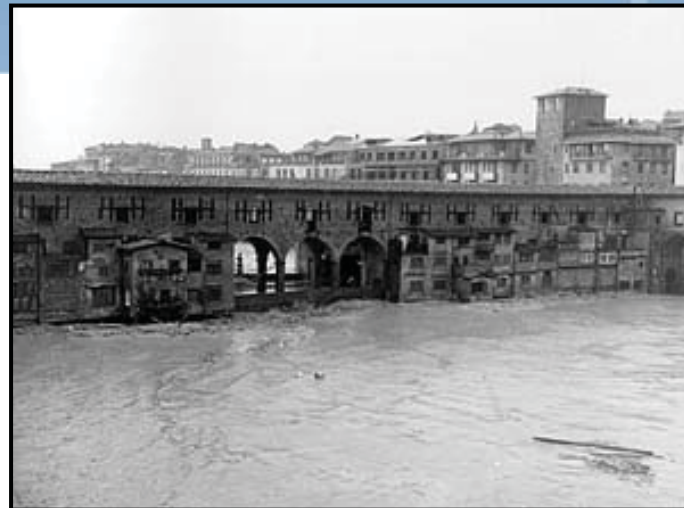


Giovanna
Poggi



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Florence, 1966

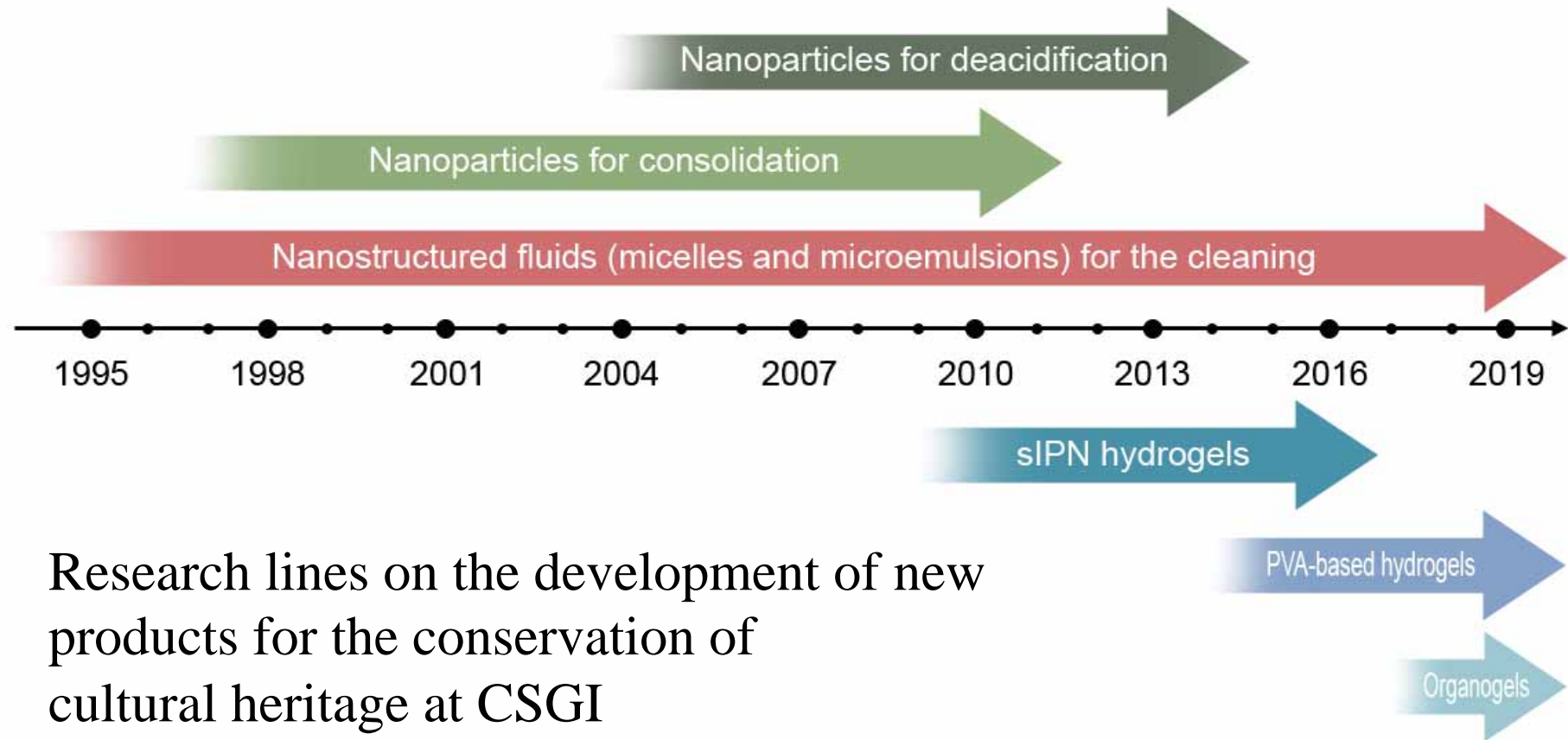


Prof. Enzo Ferroni



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Conservation Science at CSGI



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RESTORING THE CONSERVED

The use of micelles solutions and microemulsions

CLEANING

Restoring the conserved

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Sunday 31 August 2003

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Restoring the conserved

Micellar systems remove damaging polymer layers from works of art

21 August 2003

Jane Morris

Researchers in Italy¹ have developed micellar and microemulsion systems specifically for the removal of acrylic and vinyl polymers from works of art. These polymers have traditionally been used as a protective layer on paintings and frescoes — but they have their own drawbacks. Thermal and photochemical activity on the polymer surfaces cause depolymerization and crosslinking reactions, resulting not only in a yellowing effect, but also mechanical stress on the paint layers and the formation of microfractures. The authors tested several four- and five-component micellar or microemulsion systems for solubilizing the polymers away from the artwork. They found, for example, that a particular composition of the quaternary micellar system containing propylene carbonate (PC), 1-pentanol (PeOH), the surfactant sodium dodecyl sulphate (SDS) and water, completely removed the vinyl polymer layer covering the 16th-century fresco by Pozzoserrato in Conegliano, northern Italy. The authors suggest that the removal mechanism can be explained by the synergism between the highly active surface formed by the presence of SDS micelles, and their interface rich in PeOH and PC — a mixture forming a good solvent for aged vinyl polymers.

References

1. Carretti E., Dei L. & Baglioni P. *Langmuir* advance online publication 12 August 2003 | [article](#) |

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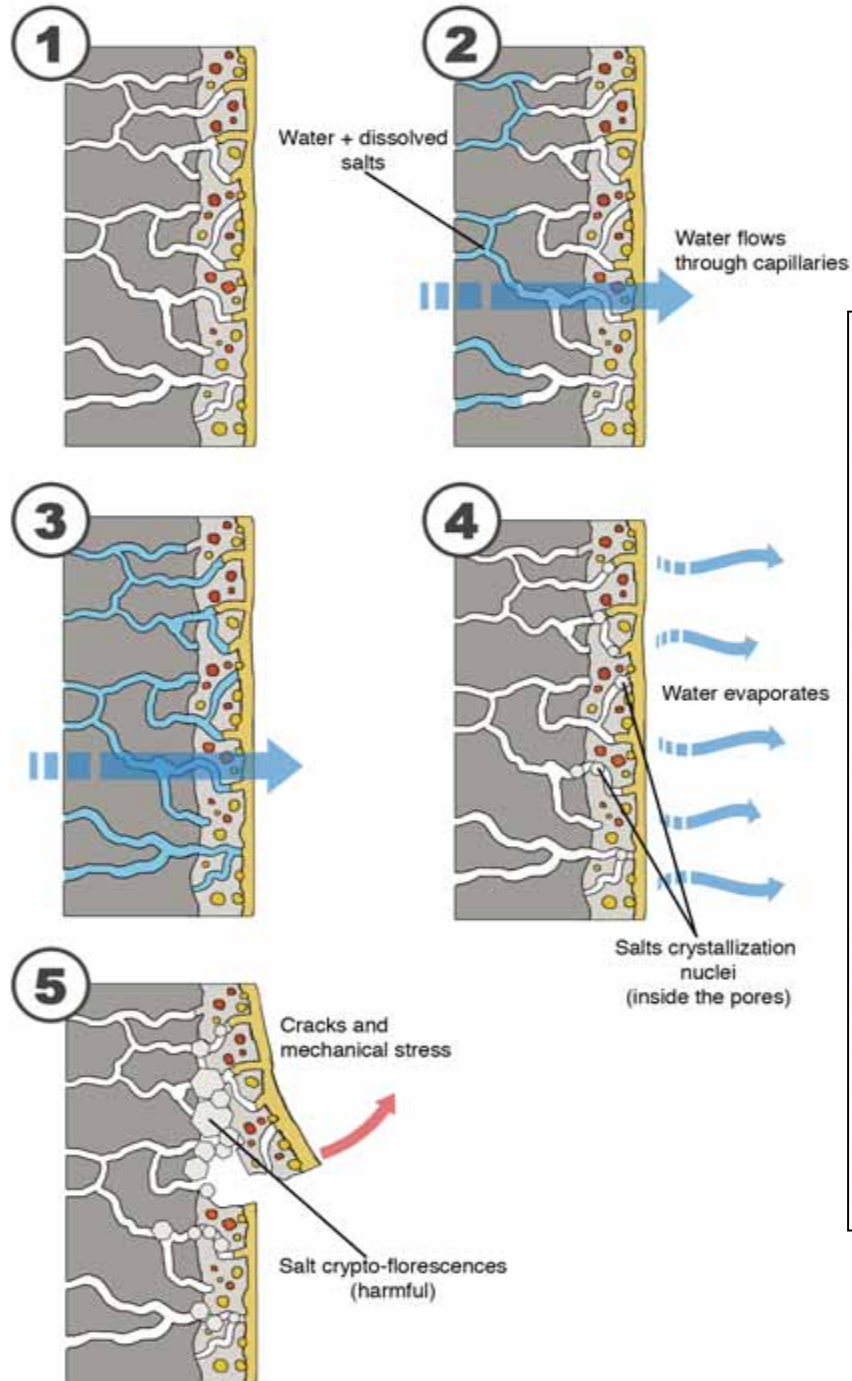


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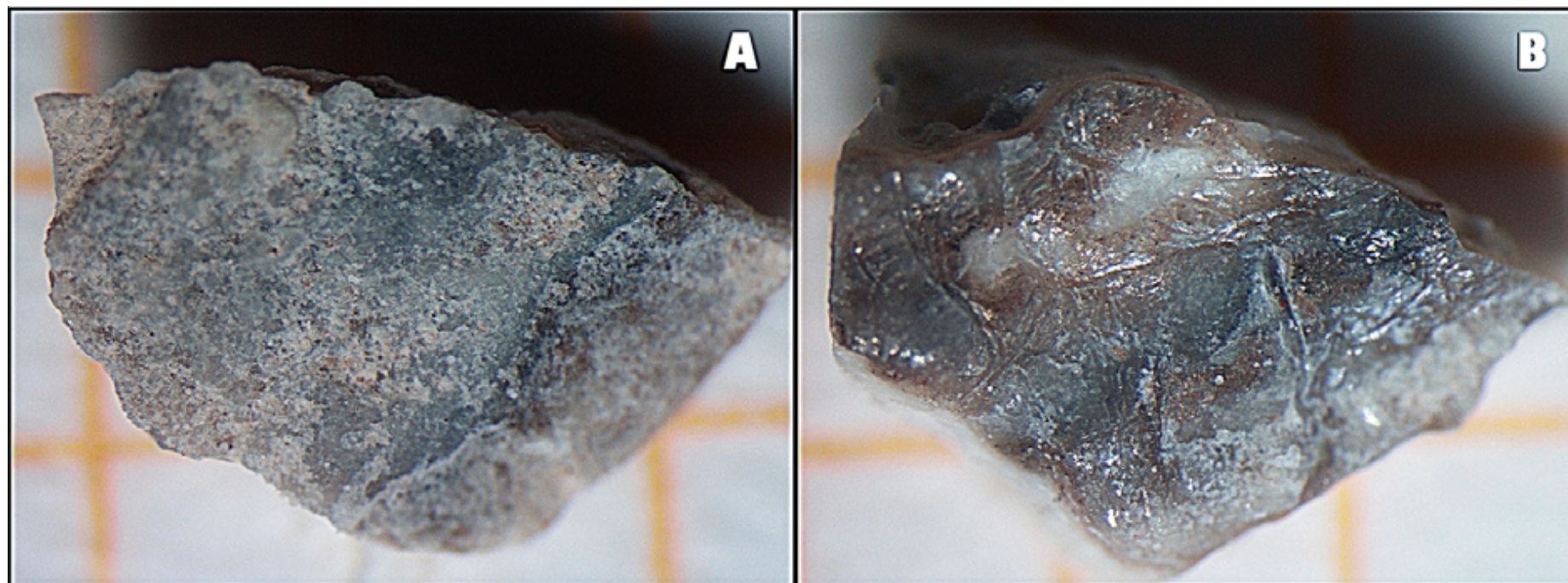
Mayapan, Messico



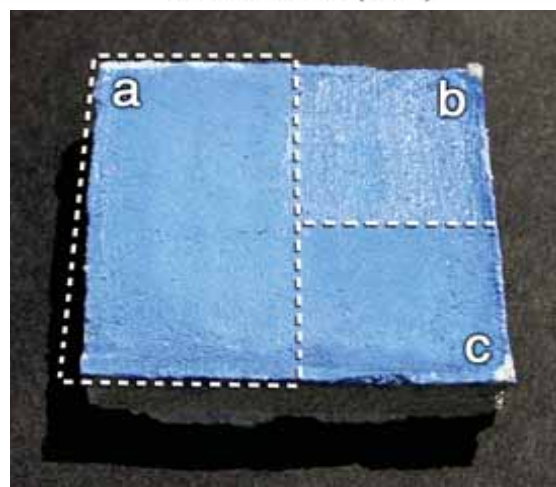
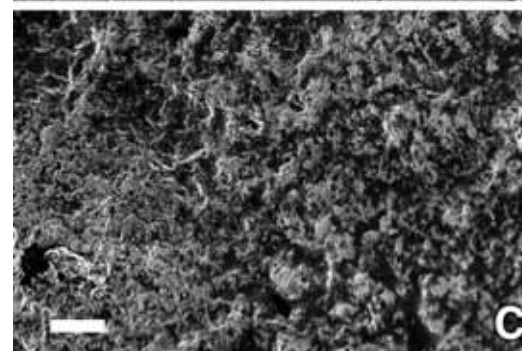
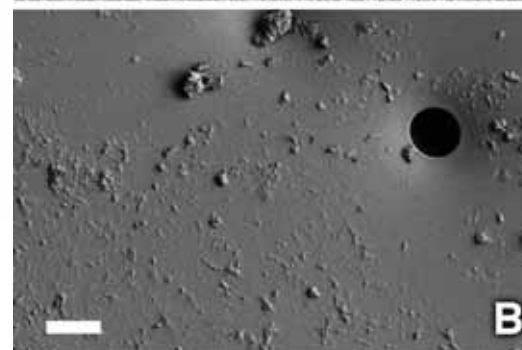
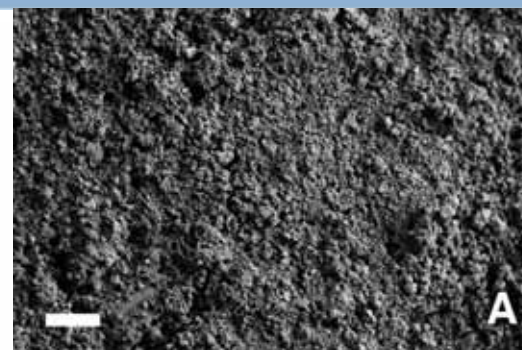
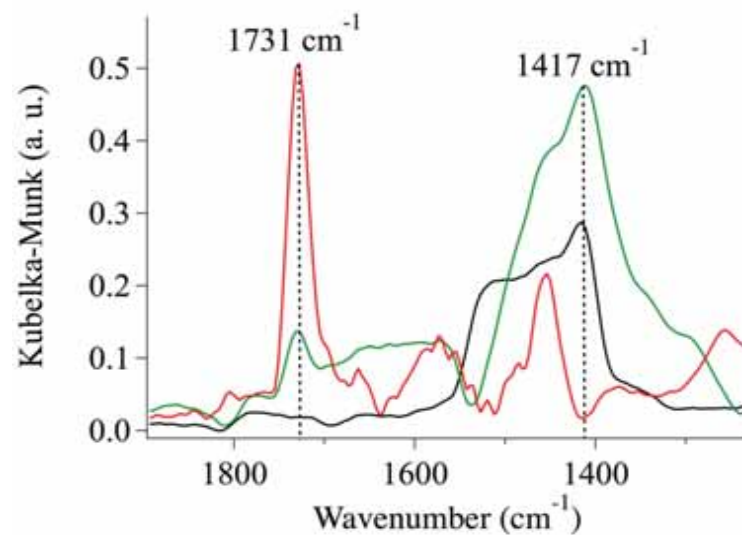
Oaxaca, Messico



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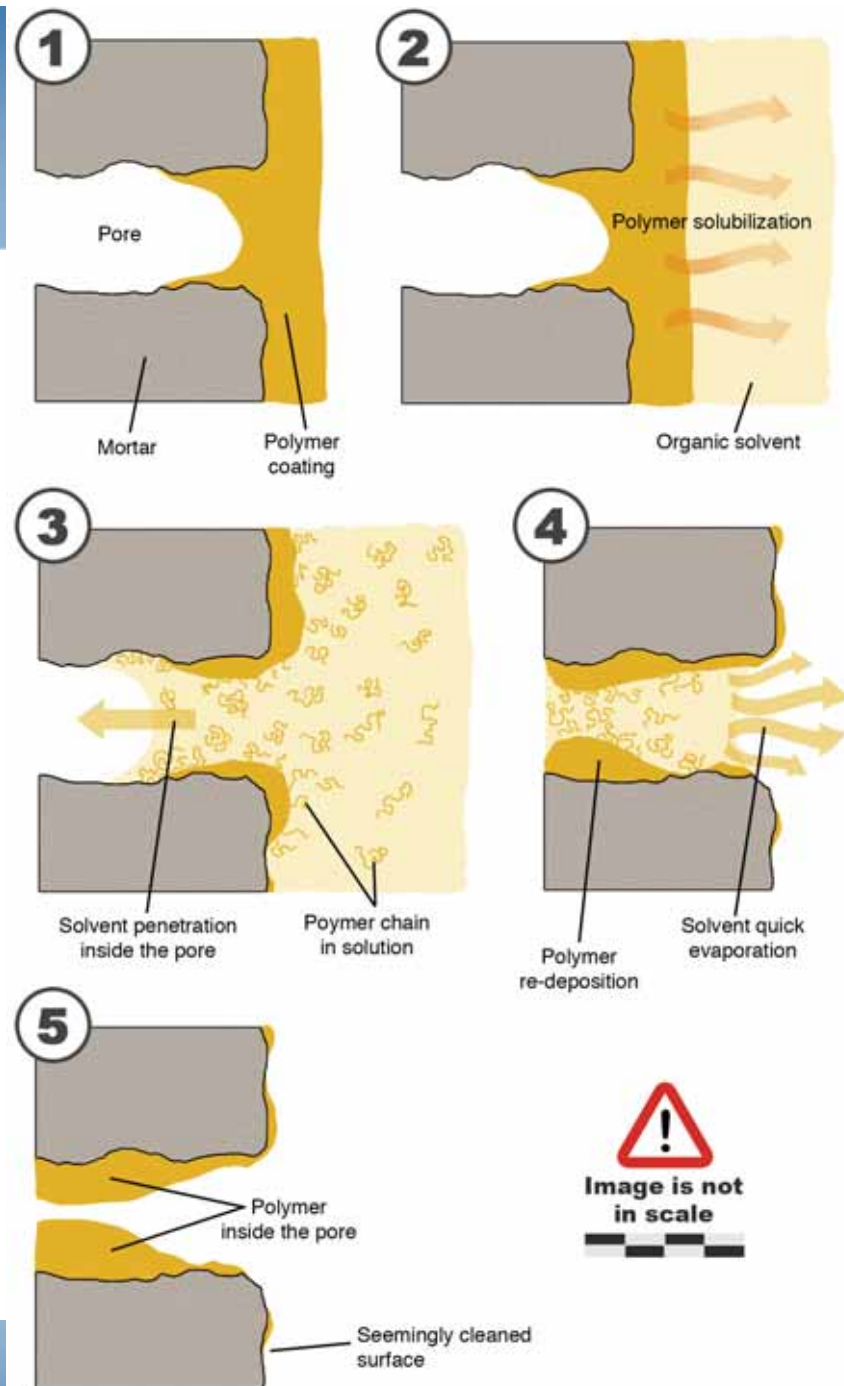
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The use of pure solvents can lead to swelling of binders and to the leaching of pigments

Microemulsion prevent polymer/
grime redeposition within pores

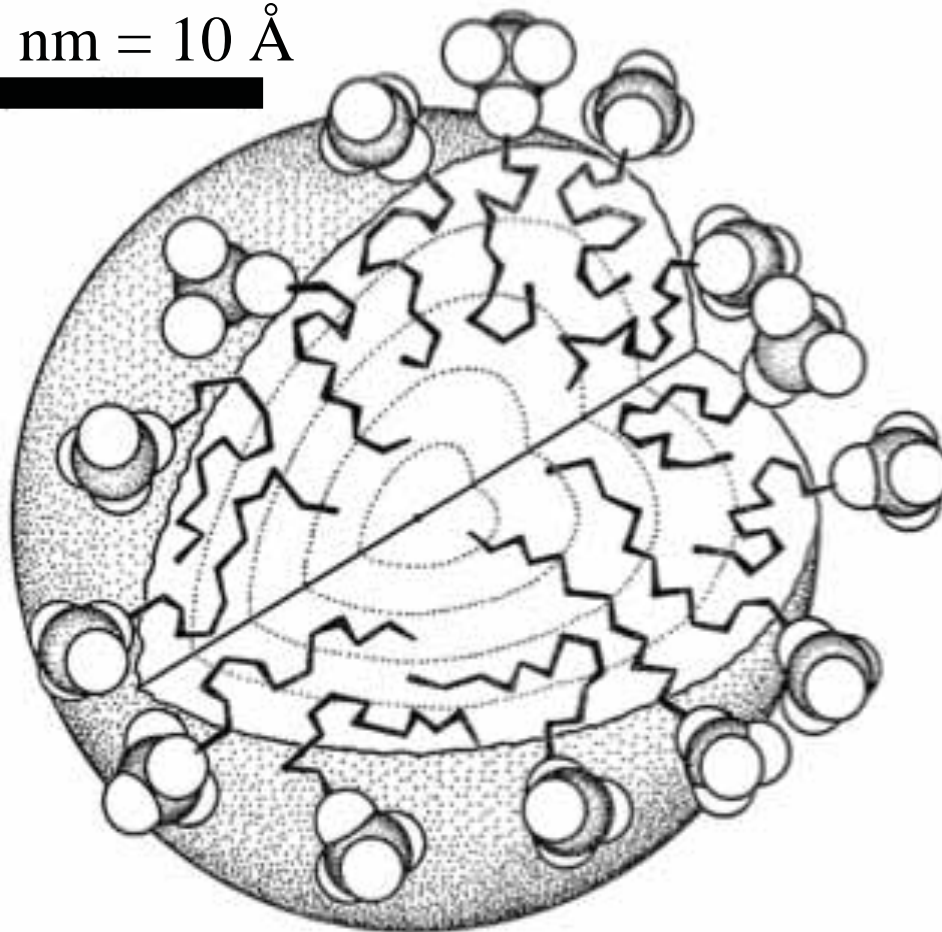
Piero Baglioni, Debora Berti, Massimo Bonini, Emiliano Carretti, Luigi Dei, Emiliano Fratini and Rodorico Giorgi, Micelle, microemulsions, and gels for the conservation of Cultural Heritage, *Advances in colloid and interface science*, 205, 2014, p. 361-371



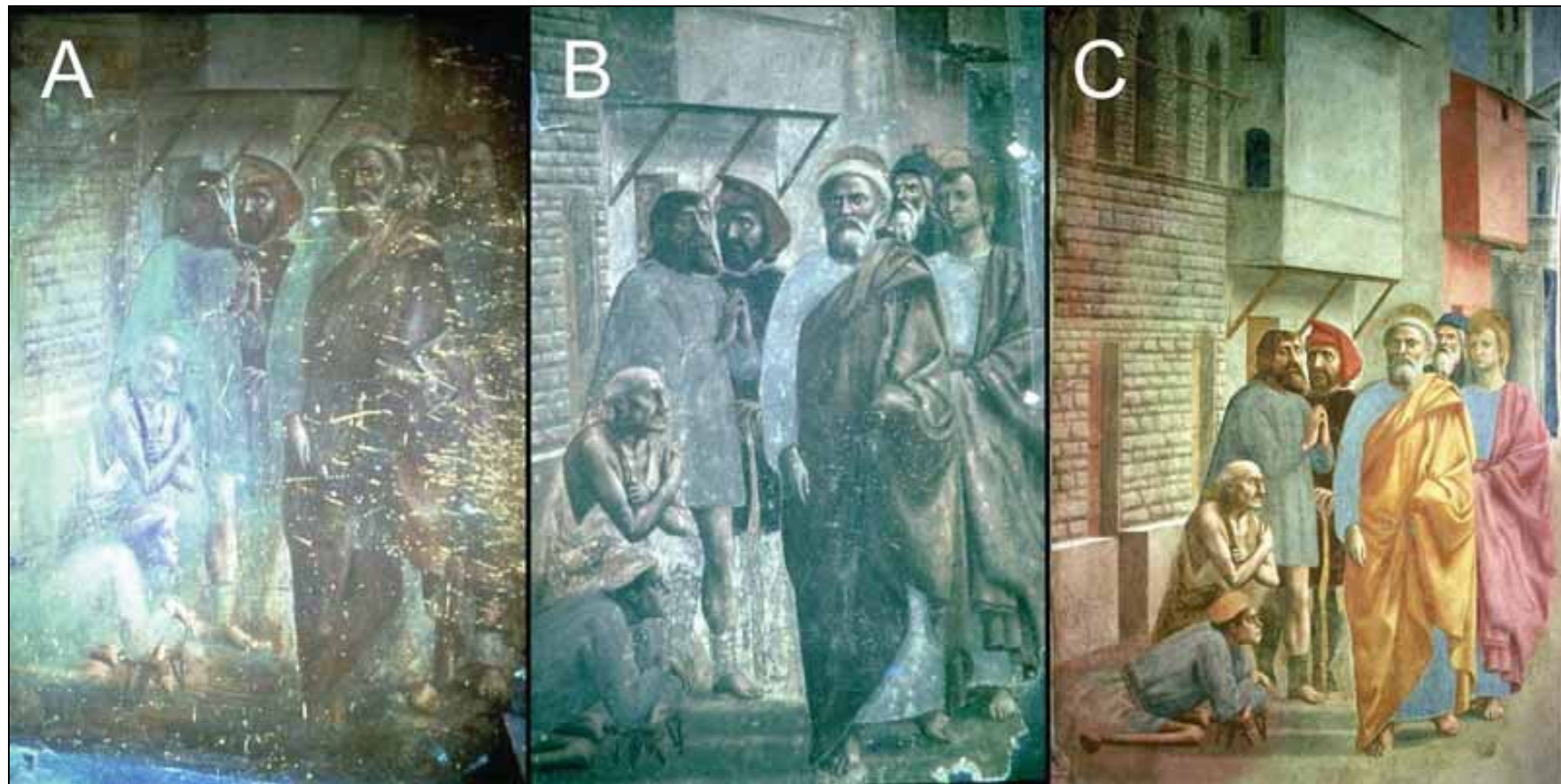
Surfactants and micelles

The term “surfactant” was coined in 1950 as a contraction of “surface active agent”, to indicate a class of organic chemical compounds that have the ability of locating themselves at interfaces, thereby altering significantly the physical and chemical properties of those interfaces

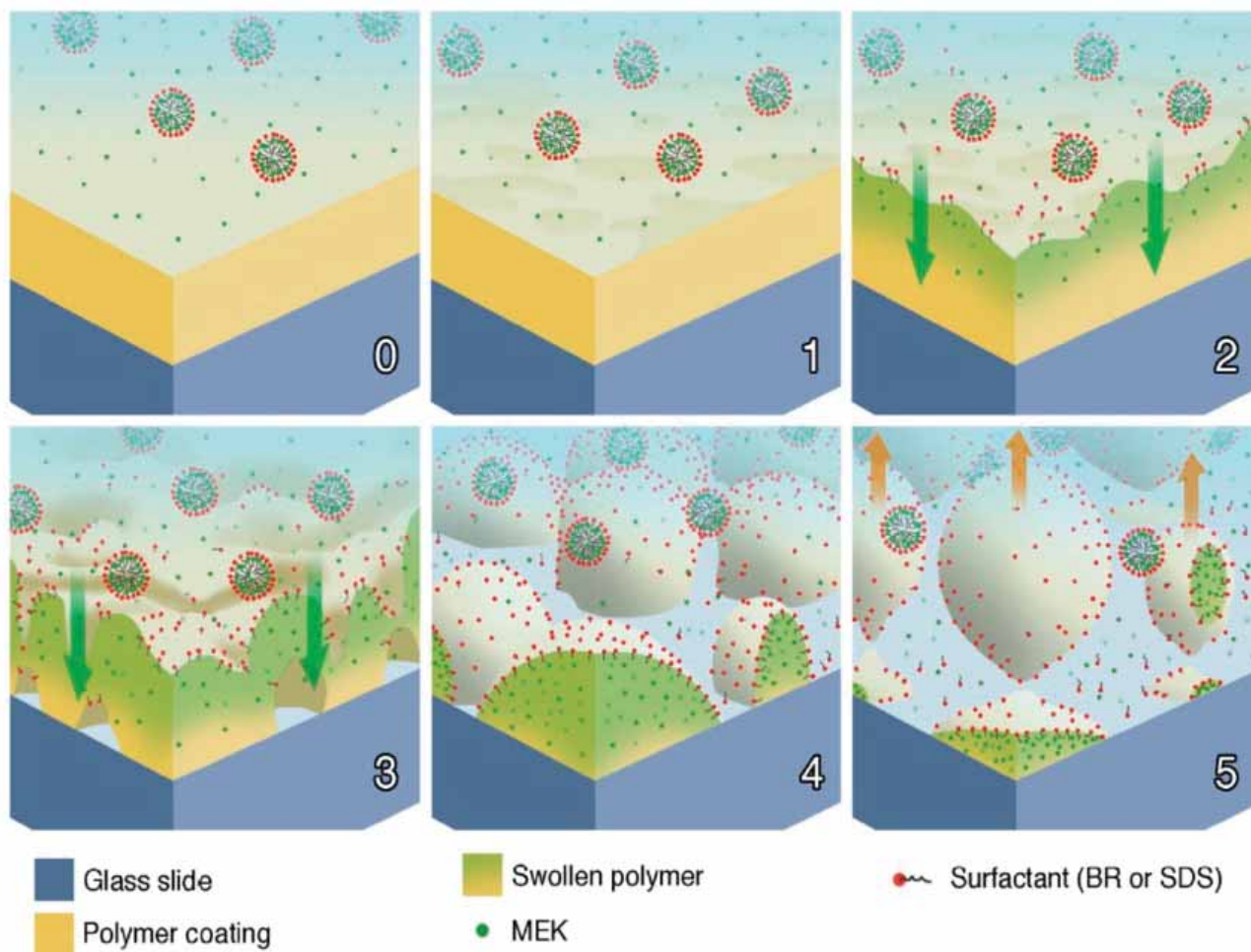
$$1 \text{ nm} = 10 \text{ \AA}$$



The Brancacci chapel in Florence



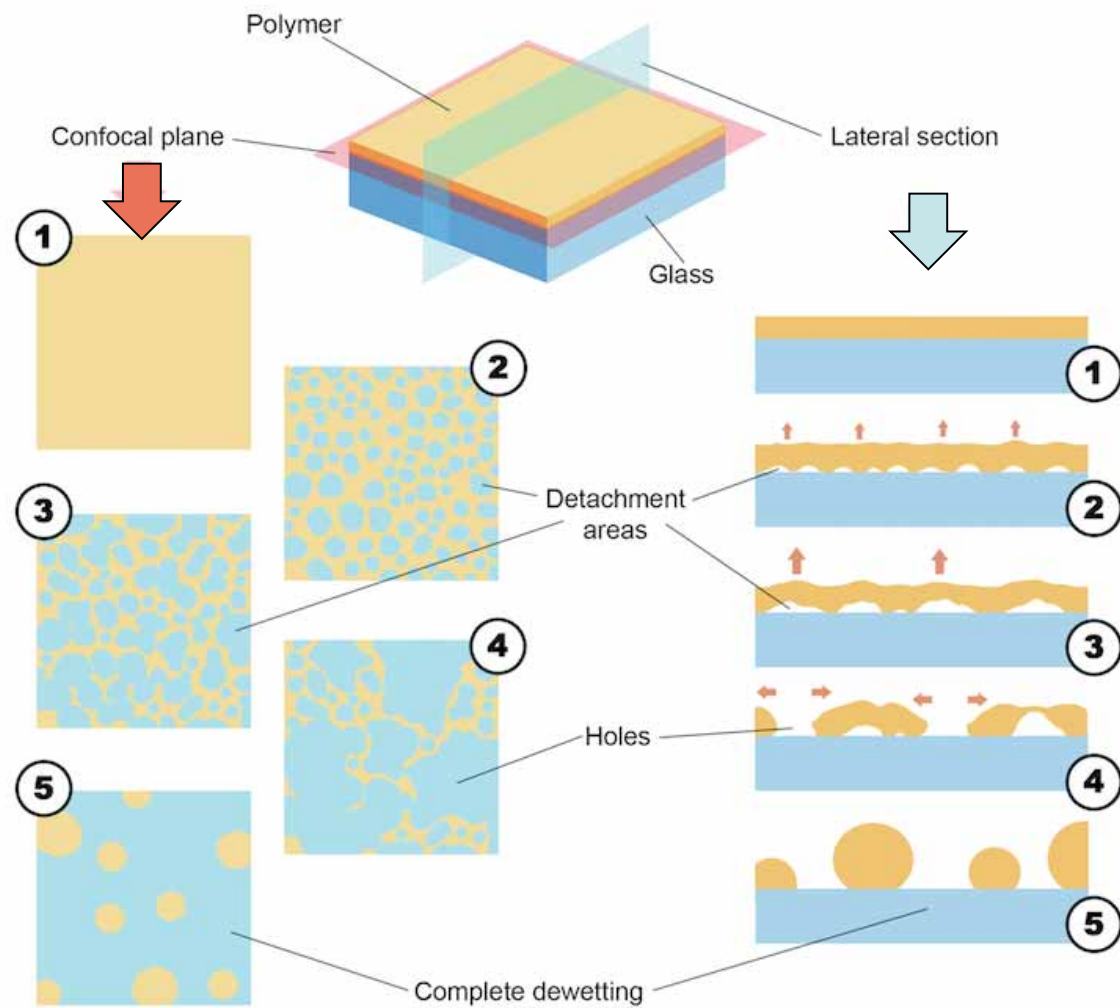
Polymer resin removal using nanofluids



Raudino, Selvolini, Montis, Baglioni, Bonini, Berti, and Baglioni,
ACS Appl. Mater. Interfaces 2015, 7, 6244–6253

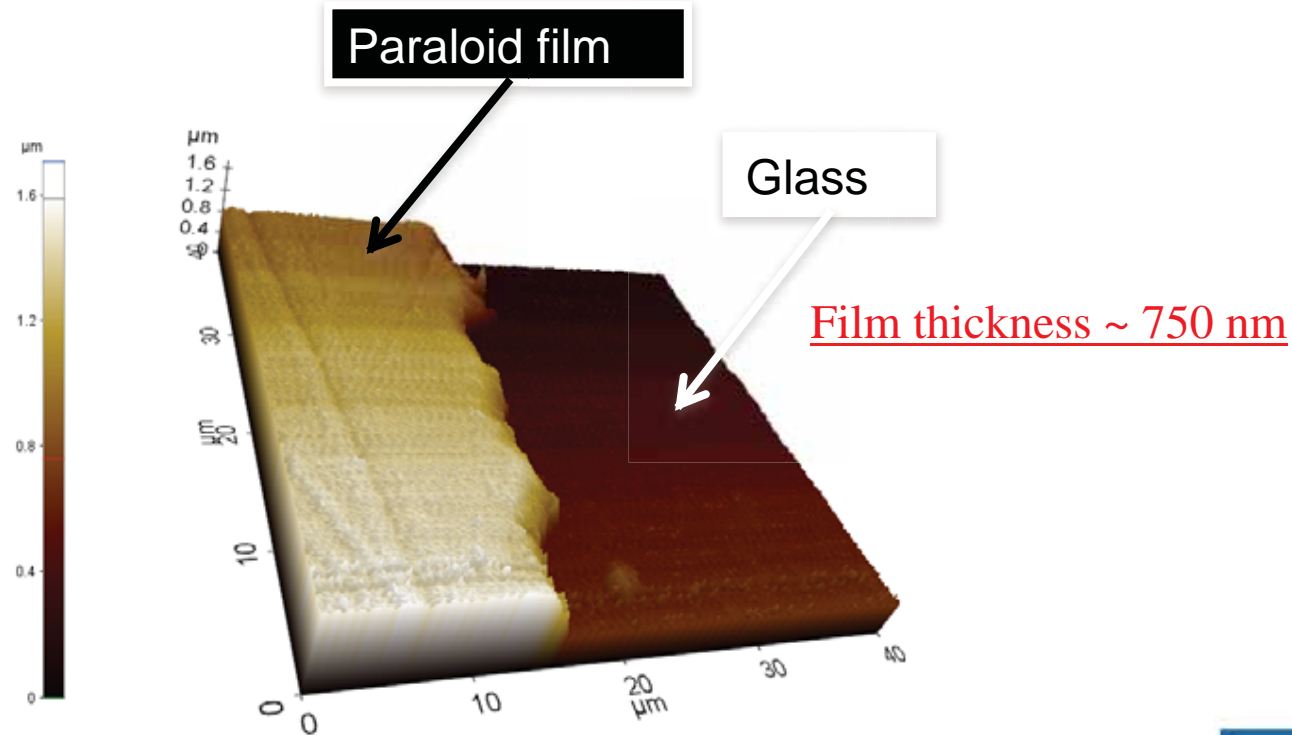


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Spatial control of cleaning

- Film of Paraloid B72 in Ethyl Acetate 10% w/w deposited by spin coating (1000 rpm, 120 sec) on glass
- Film Thickness measured by AFM



Polymer removal using nanofluids

Polymer film in red

Liquid phase in green

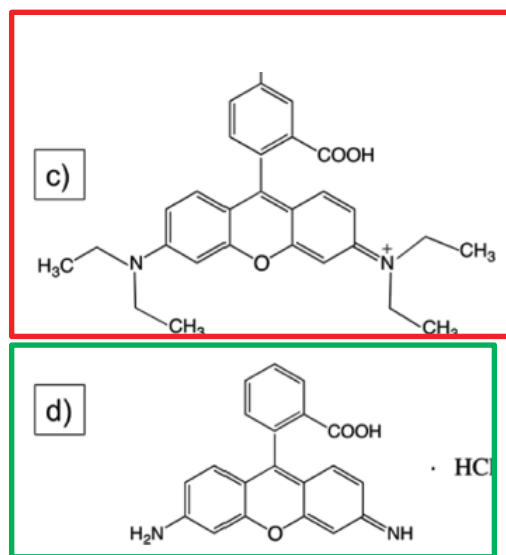


Figure 1. Structure of the used chemicals: (a) BR surfactant, (b) sodium dodecyl sulfate, (c) Rhodamine B isothiocyanate, and (d) Rhodamine 110 chloride.

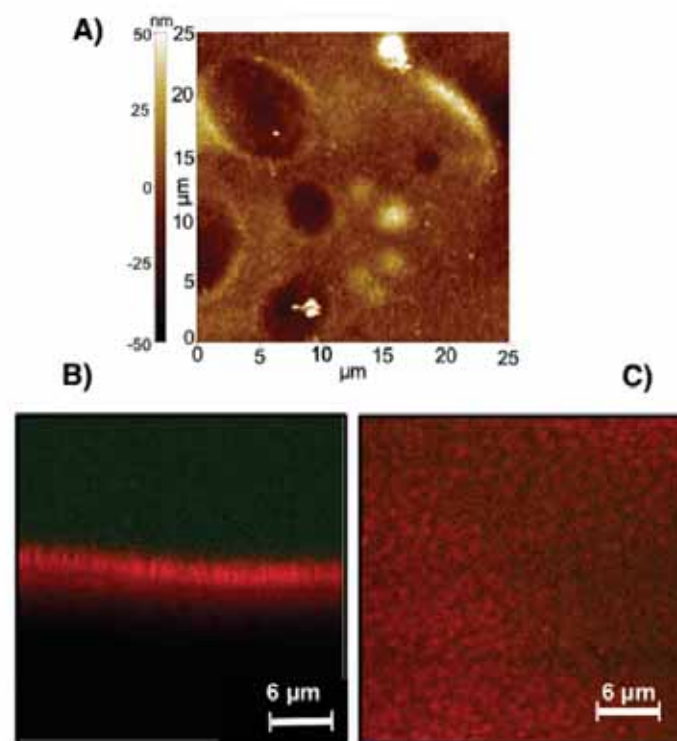


Figure 3. Paraloid B72 film on glass incubated with water for 3 h. (A) AFM image of a 750 nm thick film. CLSM vertical (B) and horizontal (C) sections of a 6 μm thick film. The polymeric film is stained with Rhodamine B isothiocyanate (red), while the liquid phase contains Rhodamine 110 chloride (green).

Polymer removal using nanofluids

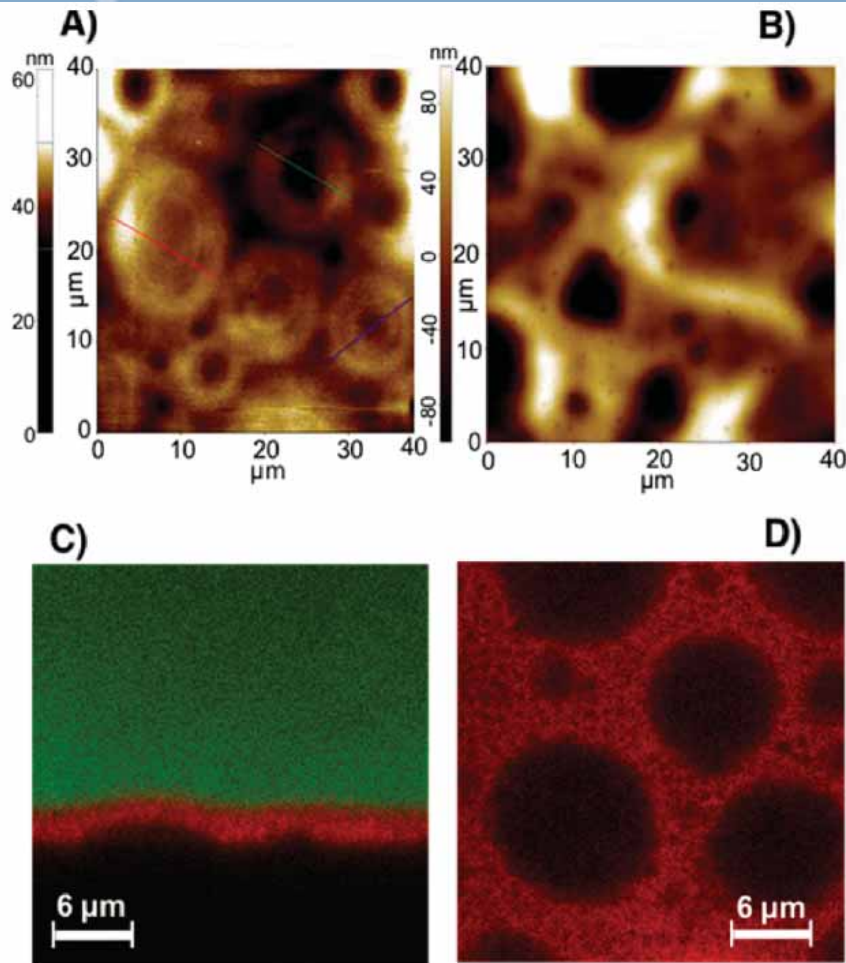


Figure 4. Paraloid B72 film deposited on glass incubated with a BR/ MEK 0% mixture for 3 h (A) and 8 h (B) investigated by AFM. CLSM vertical (C) and horizontal (D) sections of a 6 μm thick film incubated for 8 h. The polymeric film is stained with Rhodamine B isothiocyanate (red), while the liquid phase contains Rhodamine 110 chloride (green).

Surfactant and 0% MEK

The deformation of the polymer layer is clearly visible with CLSM experiment in the vertical section (Figure 4C).

The green fluorescent probe remains confined in the aqueous phase and the red tracer is not released by the film itself, indicating limited swelling.

A comparison of Figure 4C,D indicates that the film is lifted from the glass substrate in a few round areas (darker circles in Figure 4D).

This is a direct consequence of the surfactant presence, since no such effect is observed in neat water.

Polymer removal using nanofluids

Water/MEK blend

The vertical scan (Figure 6B) shows the presence of two simultaneous processes: swelling of external polymeric layers, indicated by a gradual release toward the bulk phase of the red tracer originally embedded in the film, and formation of yellow round areas at the glass polymer interfacial region.

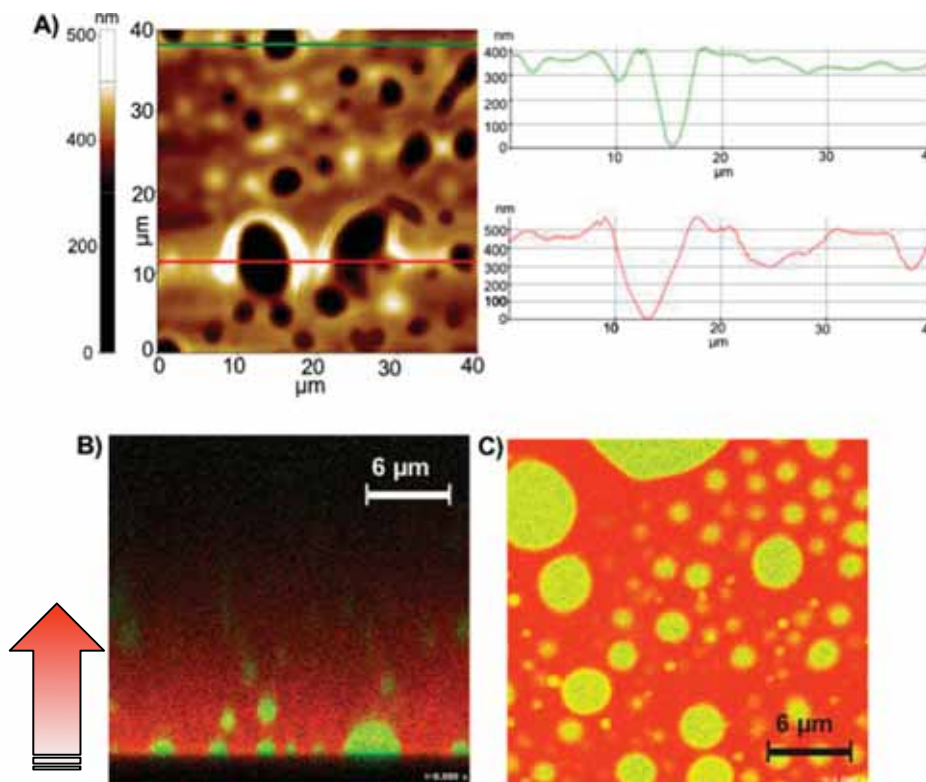
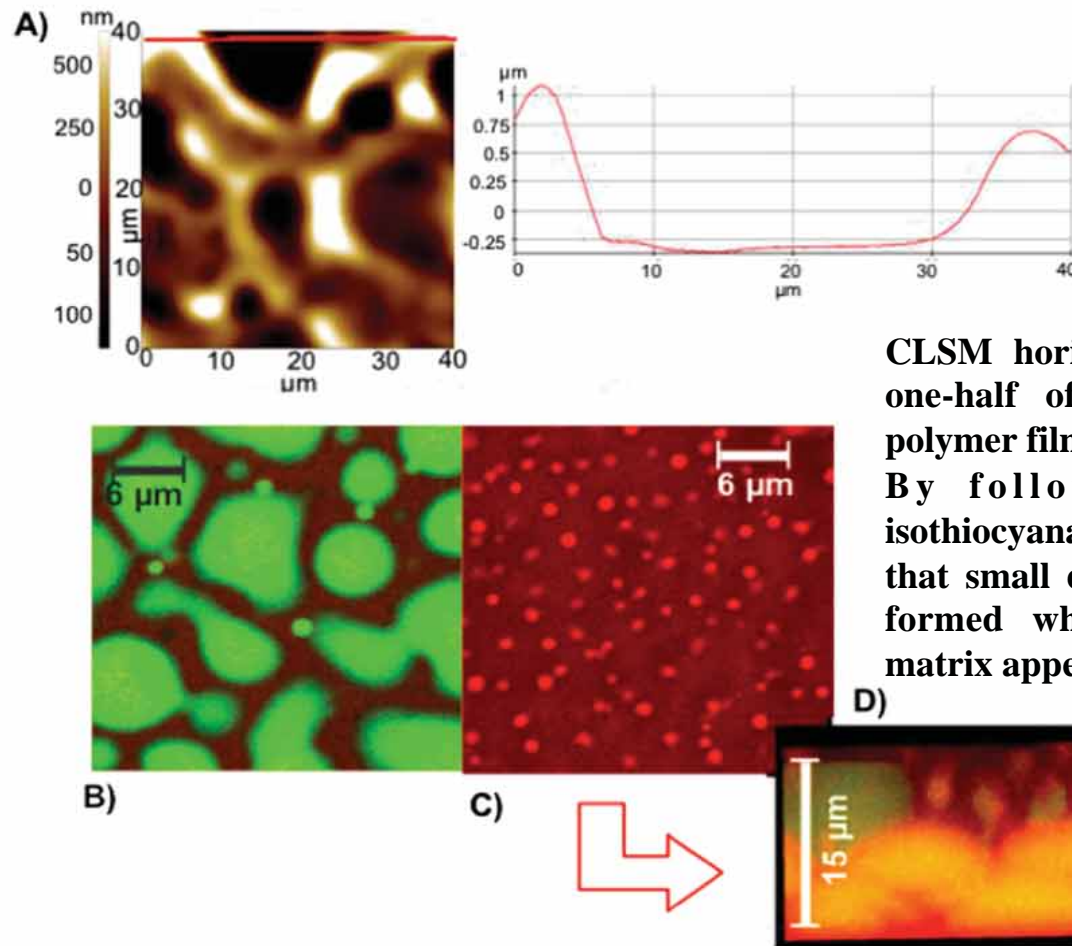


Figure 6. Paraloid B72 film deposited on glass incubated with a water/MEK mixture for 3 h: AFM image (A) of a 750 nm thick film. The height profile along two lines is also reported. CLSM vertical (B) and horizontal (C) sections of a 30 μm thick film incubated for 30 min. The polymeric film is stained with Rhodamine B isothiocyanate (red), while the liquid phase contains Rhodamine 110 chloride (green).

Polymer removal using nanofluids



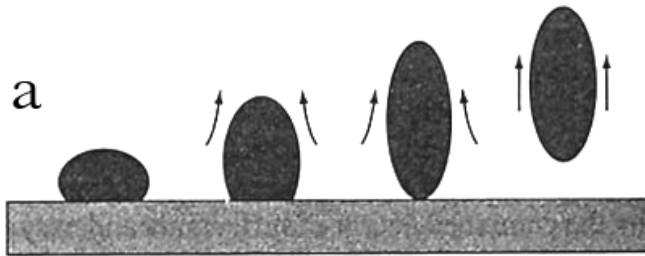
Surfactant
& 20% MEK

CLSM horizontal scan acquired at about one-half of the initial thickness of the polymer film (Figure 7C).

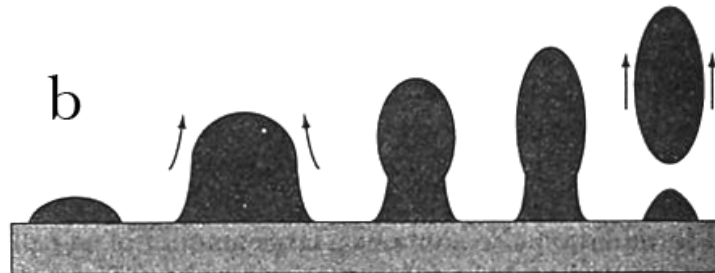
By following the Rhodamine B isothiocyanate emission (red), it appears that small droplets of swollen polymer are formed while the surrounding polymer matrix appears disrupted.

Figure 7. Paraloid B72 film deposited on glass incubated with ternary system BR/MEK 20% for 8 h (A) investigated by AFM. Horizontal CLSM scan at the polymer/glass interface (B), horizontal CLSM scan at about one-half of the initial thickness (C) where only the fluorescence of the probe encapsulated into the polymer is recorded, and CLSM 3D section (D) of a 30 μm thick film incubated for 30 min. The polymeric film is stained with Rhodamine B isothiocyanate (red), while the liquid phase contains Rhodamine 110 chloride (green).

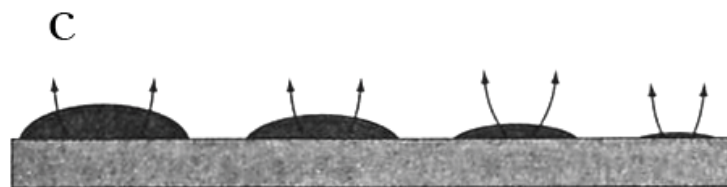
Polymer film de-wetting



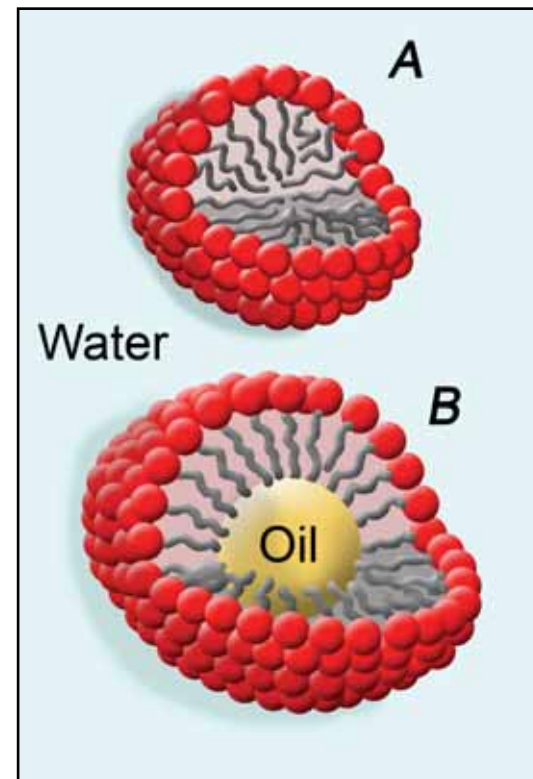
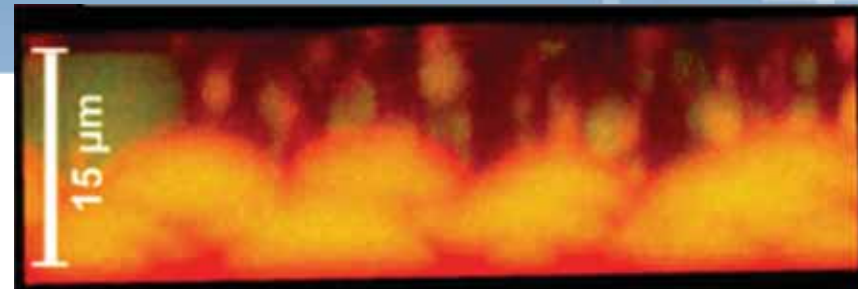
Rolling-up



Emulsification



Solubilization



Confocal
Laser
Scanning
Microscopy



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Nazareth, Israel

5th century murals



Nazareth, Israel

5th century murals

Removal of alkyl, aryl-silane



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Nazareth, Israel

5th century murals



Water sensitive artifacts

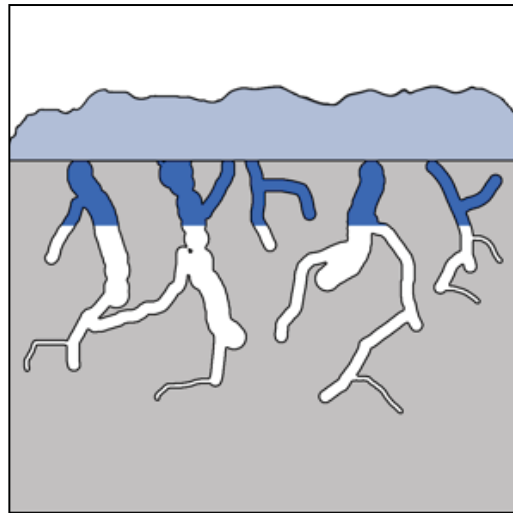
Thang-Ka

(Tibetan votive artifact based on *tempera magra* painting on canvas).

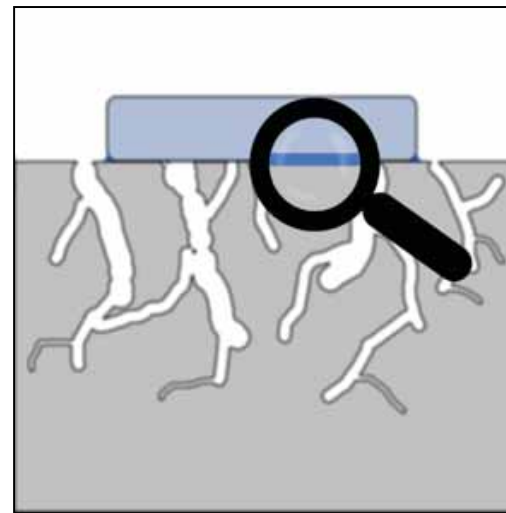


Water sensitive artifacts

Water-sensitive artifacts interact strongly with water, favoring mechanical stresses between the hydrophilic substrate and paint layers, which can lead to detachment or leaching of paint.



*Present methods to confine
water-based systems*



Highly retentive hydrogels



Gel technology

Ideal properties of gels for cleaning

- Gel cohesion: no gel residues
- Good adhesion to most of surfaces
- High retention of cleaning systems
- Stability and mechanical strength
- Transparency
- Release feature suitable for water-sensitive materials



GELS FOR CLEANING

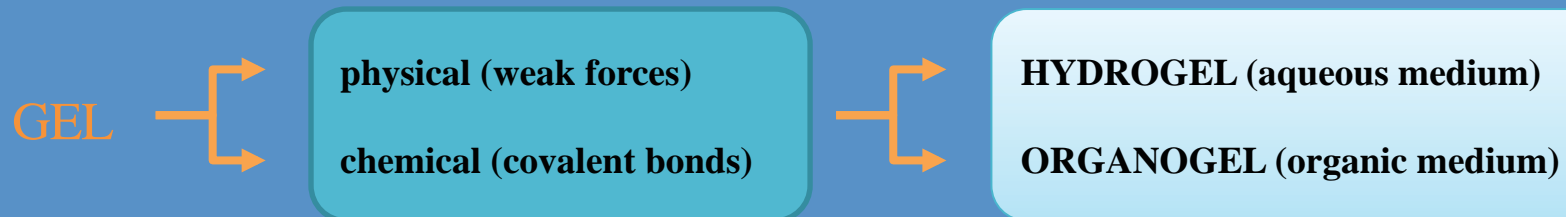
Definition

a totally agreed definition probably does not exist!

Gel status : “more easy to recognize than define”

D.J.Lloyd, The problem of gel structure, Colloid Chemistry 1926

- **Kind of intermolecular interactions**
- **medium: aqueous or nonaqueous (organic).**

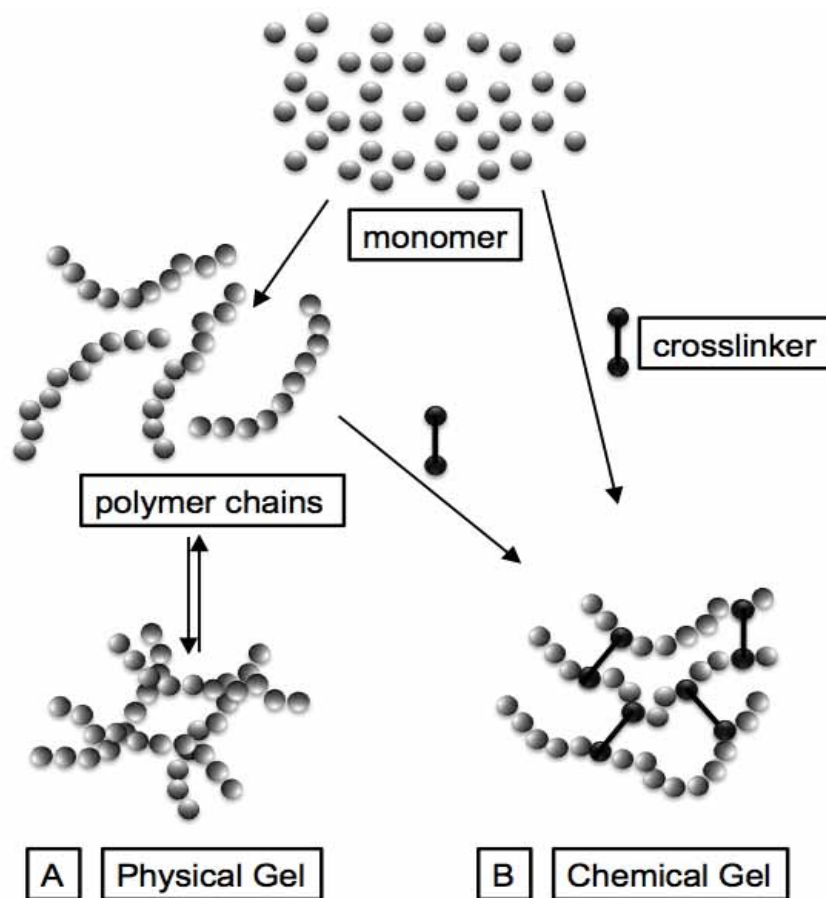


POLYMER GEL



energies of interactions
in physical gels

$1-120 \text{ kJ mol}^{-1}$



energies involved in
covalent bonds

$200-650 \text{ kJ mol}^{-1}$



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POLYMER GEL



energies of interactions
in physical gels

$1-120 \text{ kJ mol}^{-1}$



energies involved in
covalent bonds

$200-650 \text{ kJ mol}^{-1}$



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Semi-IPN p(HEMA)/PVP Hydrogels

High control of cleaning action is achieved by reaching a good equilibrium between release/retention features of the hydrogel

Semi-IPN

Semi-interpenetrating polymer networks



“Polymer blend” permits to benefit of both mechanical strength (p(HEMA)) and hydrophilicity (PVP)

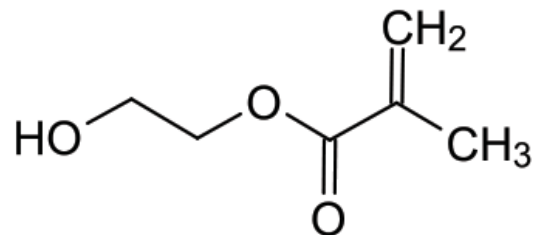
Joana Andreia Lameiras Domingues, Nicole Bonelli, Rodorico Giorgi, Emiliano Fratini, Florence Gorel, Piero Baglioni, Innovative Hydrogels Based on Semi-Interpenetrating p(HEMA)/PVP Networks for the Cleaning of Water-Sensitive Cultural Heritage Artifacts, *Langmuir*, 2013, 29 (8), p. 2746–2755



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Semi-IPN p(HEMA)/PVP Hydrogels

HEMA (monomer)



2-hydroxyethyl methacrylate

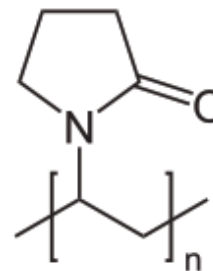
- mechanical strength
- not sufficient hydrophilicity (max. equilibrium water content ca. 40% w/w)



Polymerization



PVP (Polymer $M_w \approx 1300\text{kDa}$)



polyvinylpyrrolidone

- scarce mechanical features
- highly hydrophilic



Embedding in the forming p-HEMA network



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Semi-IPN p(HEMA)/PVP Hydrogels

Obtained hydrogels are transparent or translucent and easy to manipulate. They have enough mechanical strength to be synthesized as film-shaped (ca. 2mm thick).

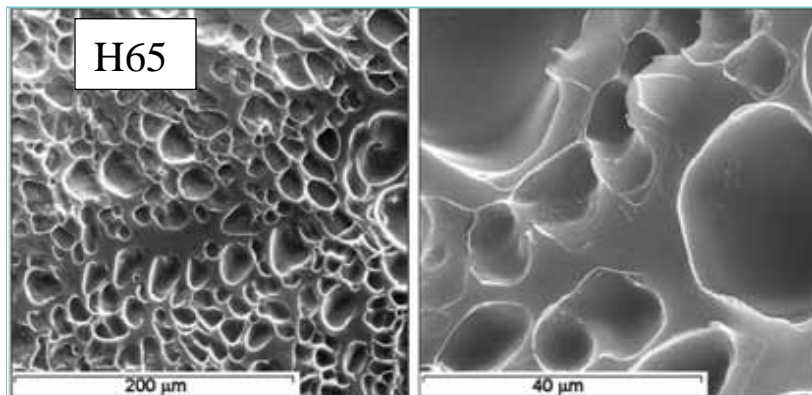
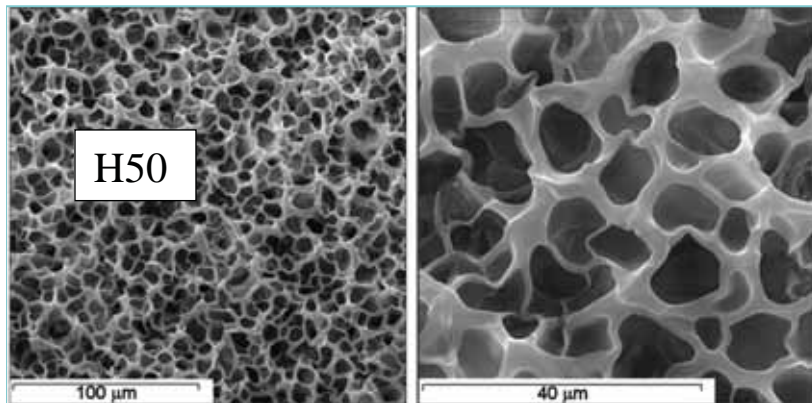


Semi-IPN p(HEMA)/PVP Hydrogels

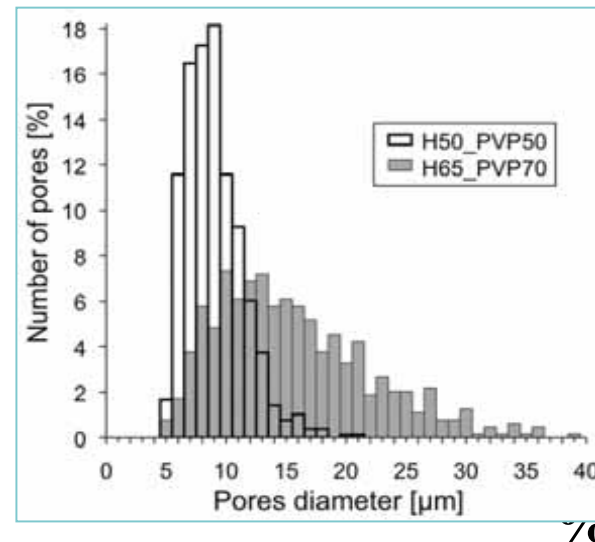


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Semi-IPN p(HEMA)/PVP Hydrogels



Structure and porosity of hydrogel network:



More compact network for :

- < **PVP content**
- < **water %**
- > **cross-linker**

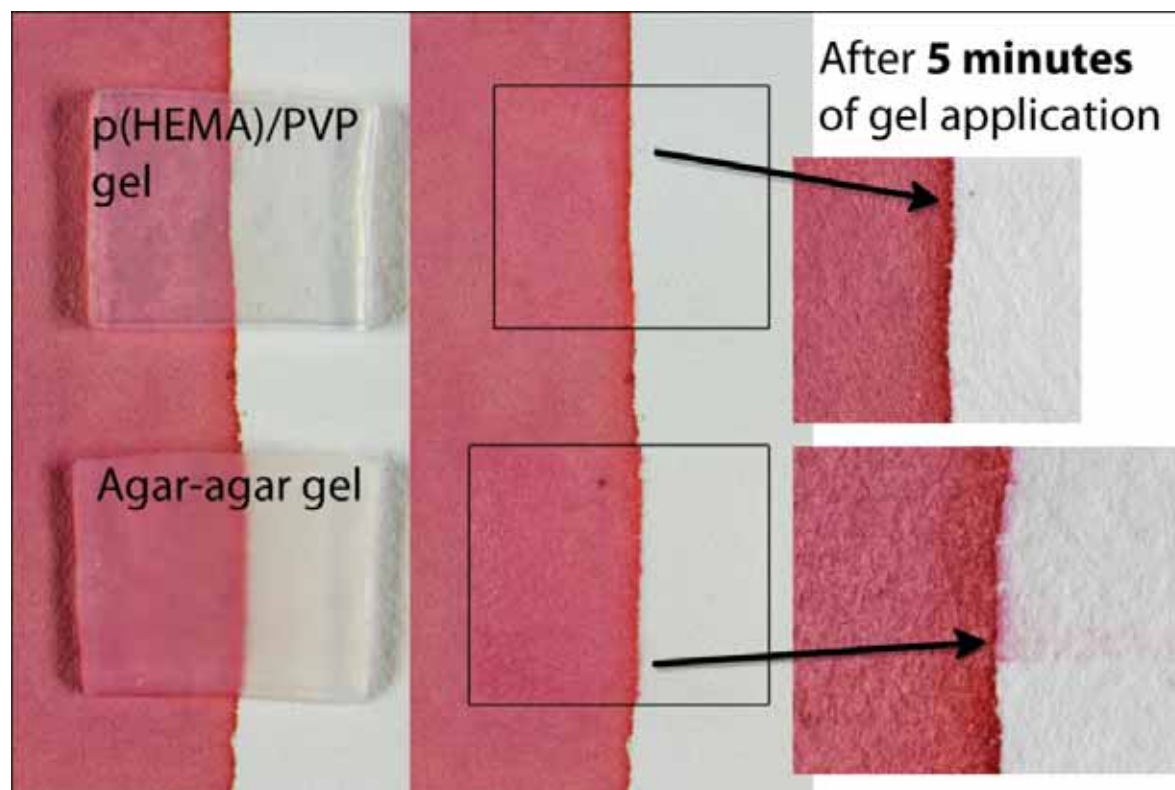
Pore size distribution for *H50* and *H65* xerogels (*ImageJ*® software analysis).

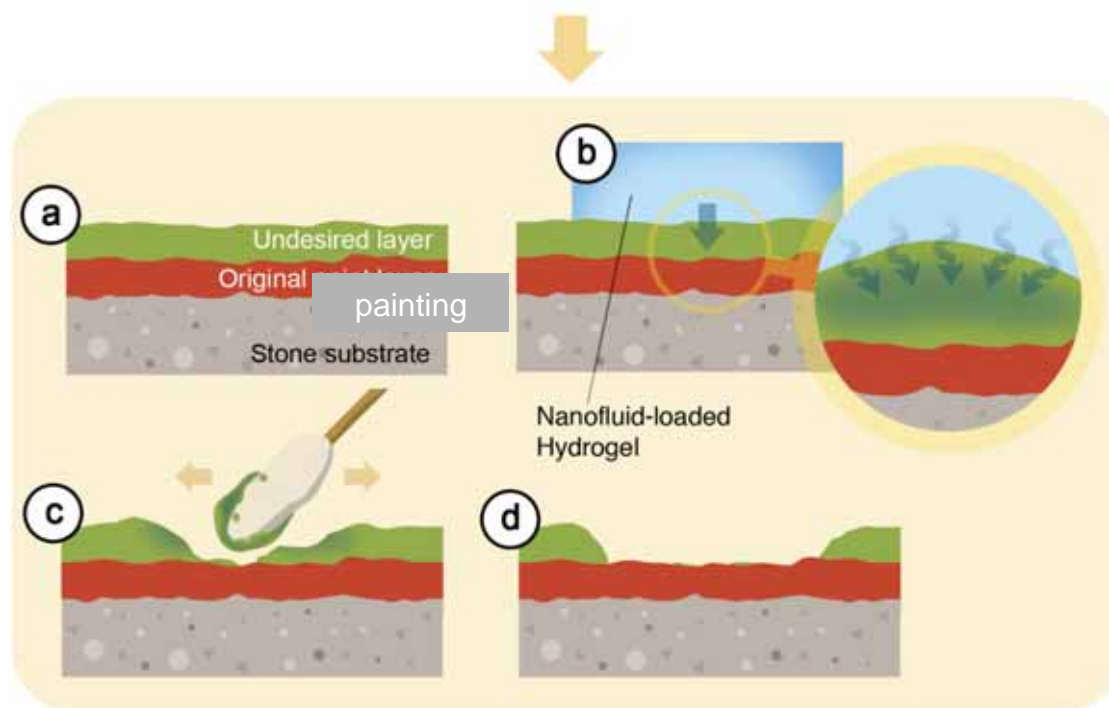
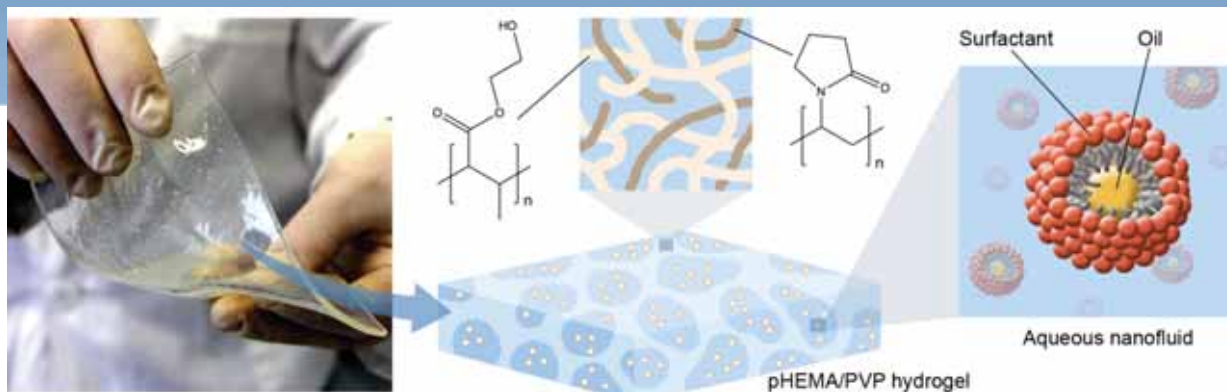
Retention and release properties

Water release test on
paper (mg/cm²)
(30 min on Whatman® filter
paper)

Gel H50	16
Gel H65	30
Agar gel	166

Comparison with
Agar-agar gel

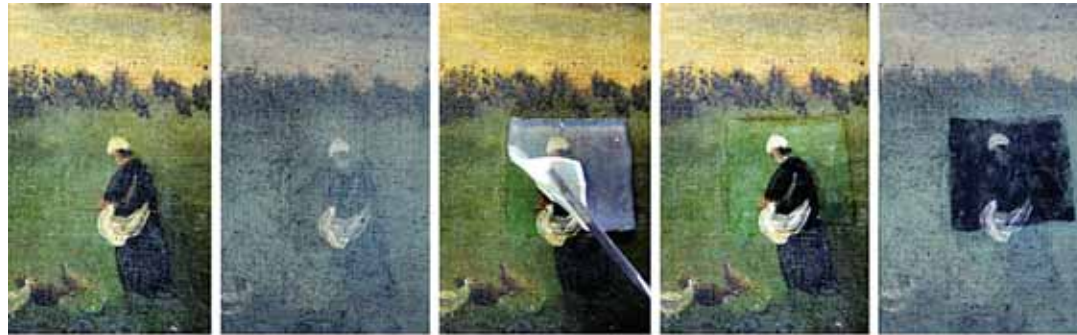






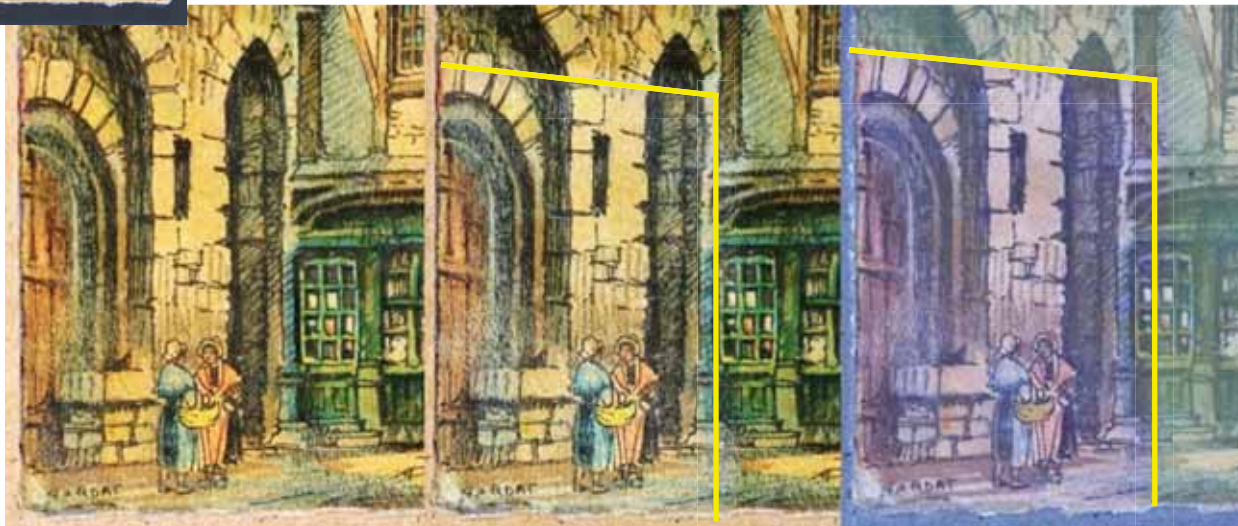
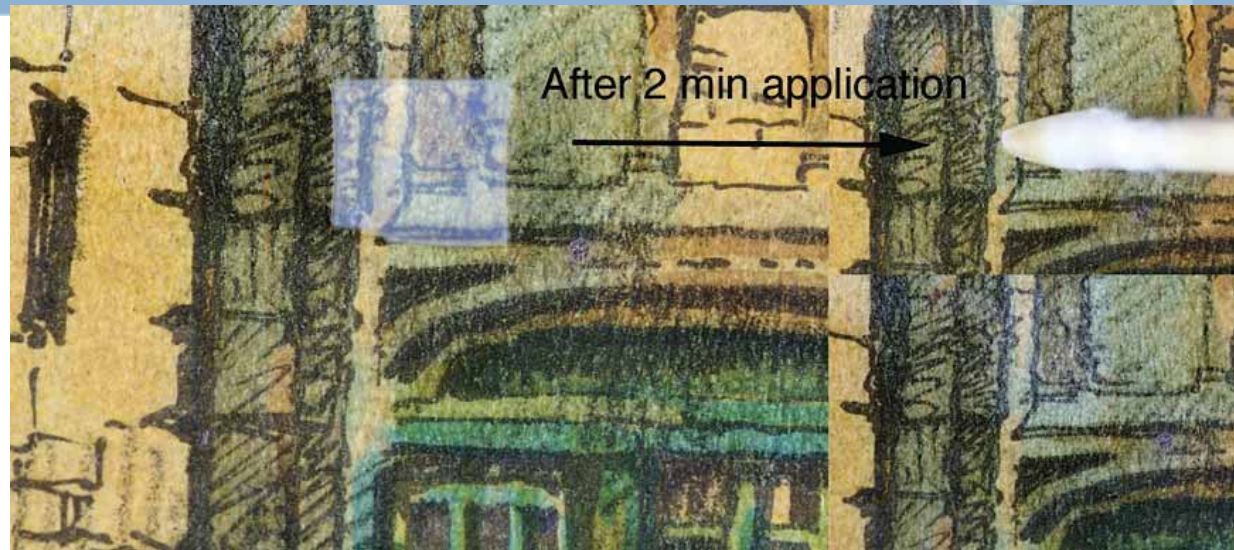
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Aged dammar varnish: canvas painting



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Varnishes removal from watercolors on paper



Gel loaded with nanostructured o/w microemulsions for the removal of hydrophobic polymeric materials

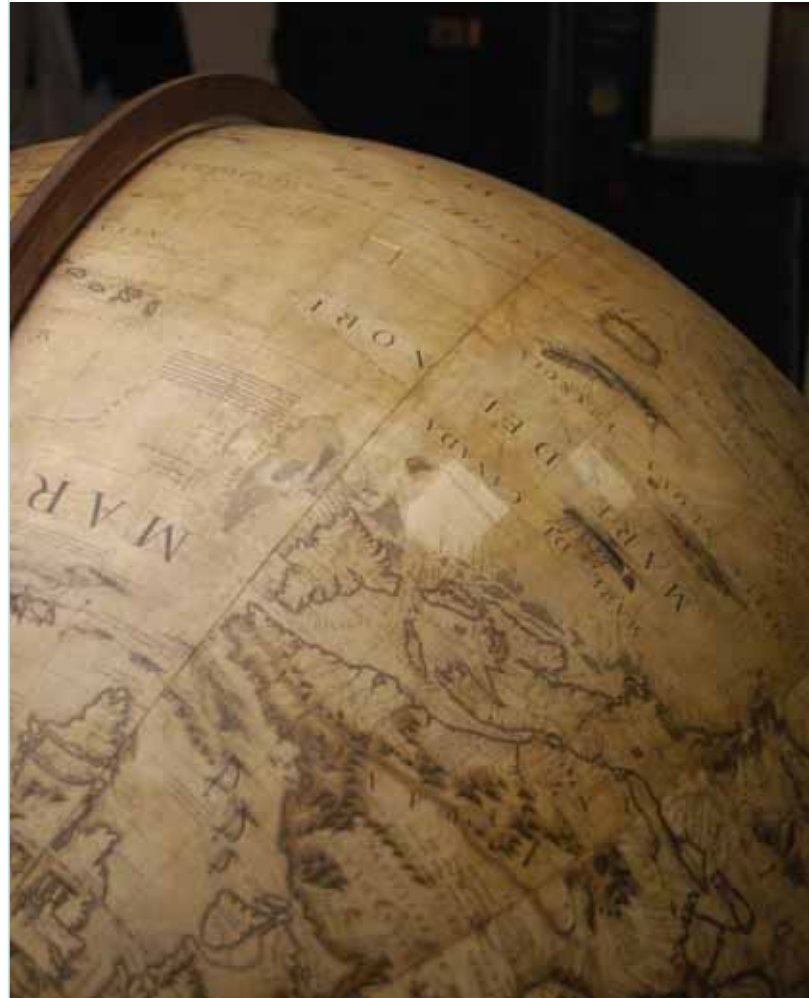


Water sensitive artifacts:
Terrestrial globe (Coronelli, 17th century)



Removal of aged
Vinavyl (polyvinylacetate)

Cleaning agent: EAPC μ -emulsion



Good handling & transparency for treating “FLAT” surfaces ...



CLEANING

A very hard work
with conventional
technologies

*Jackson
Pollock*

Dripping technique



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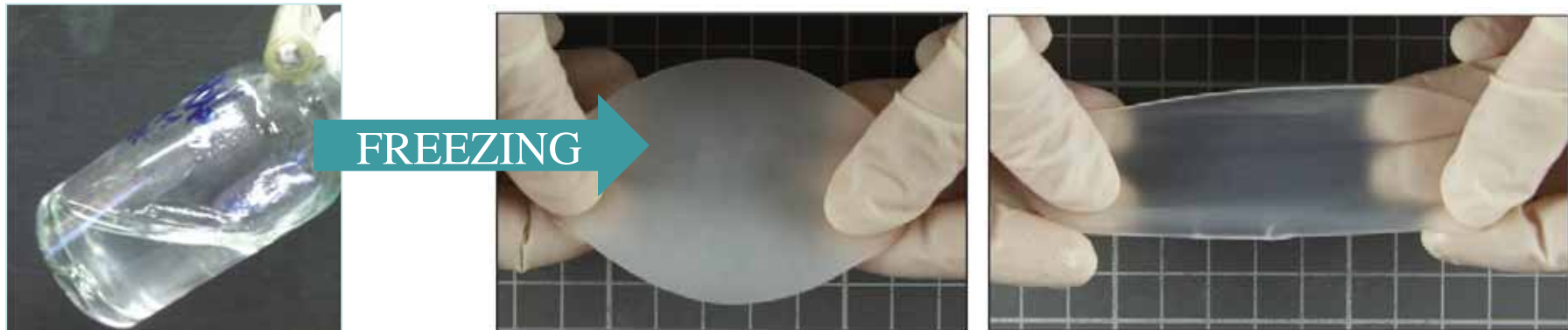
PEGGY GUGGENHEIM
COLLECTION



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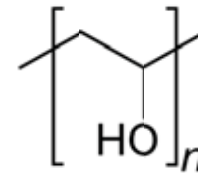
Highly retentive PVA-based hydrogels

‘Physical’ (pseudo-chemical) gels obtained from a PVA solution (freezing-thawing)

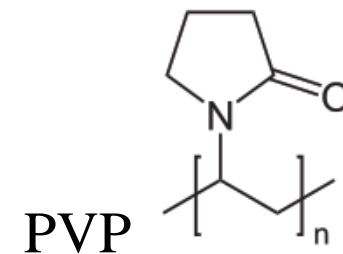


Originally designed for human tissue replacement:

- High water content ✓
- Excellent mechanical properties ✓
- No release of toxic substances (e.g. cross-linkers) ✓



PVA

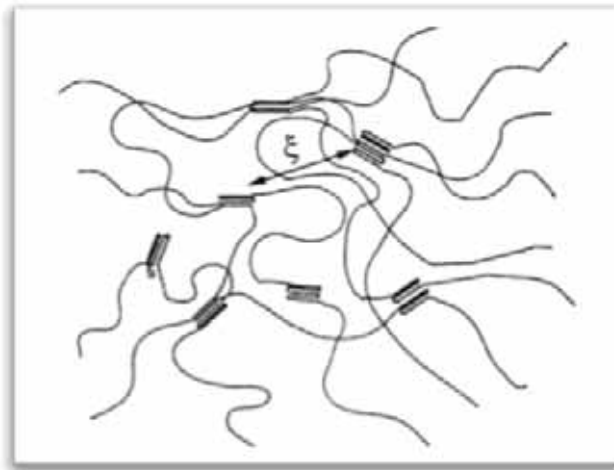


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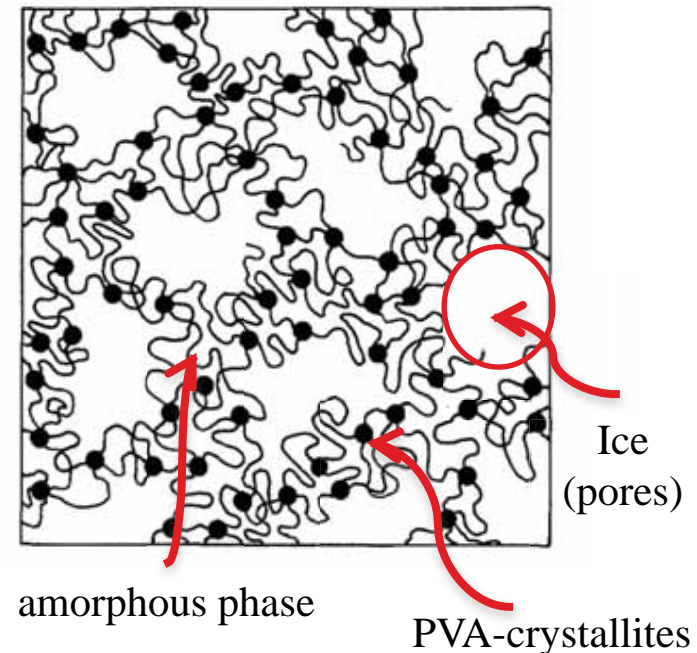
Highly retentive PVA-based hydrogels

‘Physical’ (pseudo-chemical) gels obtained from a PVA solution

Crystallite: highly ordered region, PVA chains are aligned and strongly bound by a very high number of h-bonds

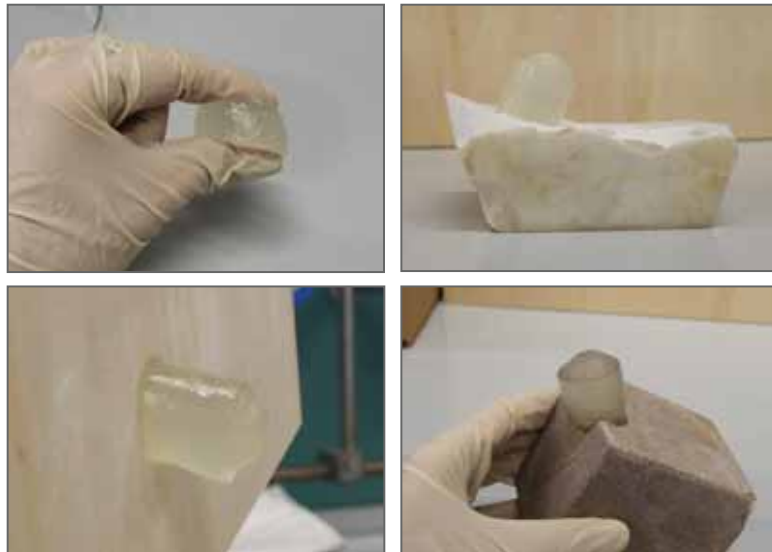


A single polymer chain may participate to more than one crystallite, passing through the amorphous region



PEGGY GELS

Highly retentive
PVA-based hydrogels

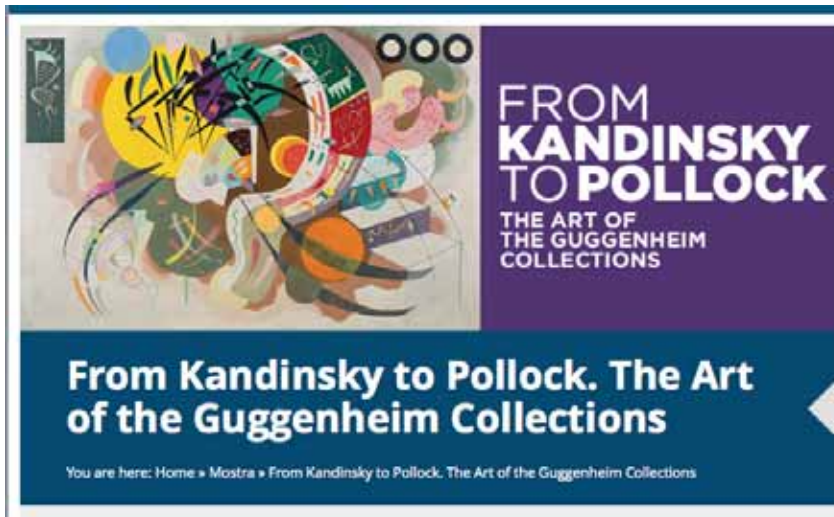


PEGGY GELS

Two
(1943-1945)
Jackson Pollock
193 x 110 cm
Oil on canvas



The new method developed in the framework of the EU-funded project NANORESTART has successfully used for the cleaning of Pollock's painting "Two"; recently exhibited at Palazzo Strozzi, in Florence:
"From Kandinsky to Pollock. The Art of the Guggenheim Collections"





The Studio

(1928)

Pablo Picasso

161 x 129,9 cm

Oil and black crayon on canvas

Lined in 1969 with wax-resin adhesive and
varnished with a PVA-based varnish

Margaret Watherson's Conservation Report, 1969

PEGGY GUGGENHEIM
COLLECTION

CONSERVATION REPORT

PICASSO = "The Studio", oil on canvas, 61-7/8" x 49-3/8",
dated 1938.

Condition before Treatment

Surface of painting had become extremely dirty with grime and discolored varnish making whites appear brownish-yellow. In areas of heavy impasto, scattered groups of pressure cracks had developed with lifting of the paint layer along the edges of these cracks. The vertical rectangular panel, bordered in yellow, was executed in relatively thin paint with the texture of the canvas apparent in places through the paint layer; the rest of the surface was heavily coated and the pressure cracks mentioned above were most apparent here.

Conservation Treatment

Surface of painting was faced. Since lining would accentuate the difference in level between the thinly painted and heavily painted parts of the surface, the thinly painted vertical rectangle was given additional thickness on the reverse of the canvas - this part of the painting was first coated thinly with white Magna Medium color and then brush coated with gesso which was sanded down so that the texture of the canvas began to be visible.

Painting was then treated with water and chemicals on the vacuum table to minimize pressure cracks. After this, painting was lined, again on the vacuum table, with wax-resin adhesive and fiberglass and then was mounted on a honeycomb panel with further wax-resin adhesive. Facing paper was removed and the surface cleaned to remove grime and discolored varnish. Special attention had to be given to discolored varnish caught in the brushwork and impasto. Surface was sprayed with synthetic resin varnish.

Photographic Record

- 8 x 10 black and white photograph, before restoration, with areas of test cleaning marked in red.
- 8 x 10 black and white photograph, before restoration, taken in raking light to show impasto and numerous pressure cracks in paint layer.

(continued)

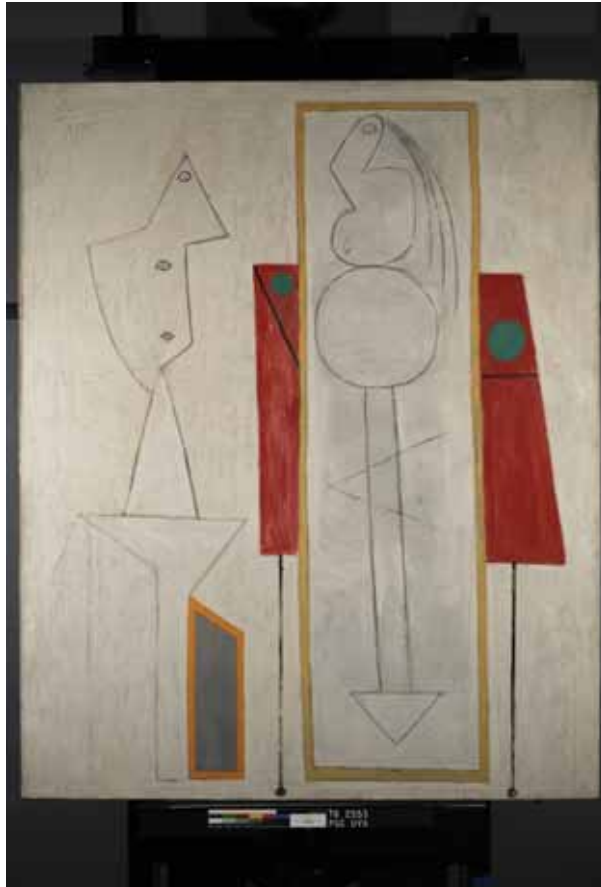
Wax+ colophony for
relining

And re-varnishing
with PVAc





CSGI



Luciano Pensabene Buemi is gratefully acknowledged



Removal of soot from wall paintings in Skandia (Sweden)



Cooperation with Hanna Eriksson, conservator (Sweden)



CSGI

Removal of soot from wall paintings in Skandia (Sweden)



CSGI

Removal of soot from wall paintings in Skandia (Sweden)



CONSOLIDATION



Nanotechnology restores flaking frescos.

An off-the-wall application of tiny particles re-unites paint and plaster.
11 July 2001

PHILIP BALL



Had Leonardo da Vinci known about nanotechnology, his Last Supper might not be in its present sorry state. Italian chemists have shown that particles of slaked lime - a staple of the Renaissance palette - just a few millionths of a millimetre across can rescue old frescoes from decay.

Leonardo's painting is one of the worst affected by the ravages of time. The damage was largely the result of ill-informed experimentation with materials - Leonardo was no chemist. Similar fresco deterioration is a common problem for conservators.

A beautiful bottom -

thanks to the restorative effects of nanotechnology.

Muscenti painted in the sixteenth century by Santi di Tito in the Santa Maria del Fiore Cathedral in Florence. This image is disfigured where flakes of paint-impregnated plaster, having lifted off the wall below, are threatening to fall off, damaging the painting irreparably.

Before Italian painters began to use canvas in the fifteenth century, many made frescoes. They applied pigment directly to damp plaster on a wall, so that it bound fast as the plaster dried. Giotto and Michelangelo were masters of this technique.

Done skillfully, the results were robust. Unfortunately half a millennium later, flaking of the top layer has become a common problem, especially in damp areas.

Plaster was typically made from sand and lime (calcium

oxide), which becomes slaked lime (calcium hydroxide) when wet. As it dries, slaked lime reacts with the carbon dioxide in air to make chalky calcium carbonate.

Baglioni and his colleagues use humble slaked lime as a kind of glue to re-adhere flaking paint. They apply it as a suspension of tiny calcium hydroxide crystals in alcohol. As the alcohol evaporates, the crystals absorb water and carbon dioxide, and merge with the calcium carbonate in the paint layer and the underlying plaster, welding them together with an almost invisible bond¹.

Ordinary ground-up calcium hydroxide doesn't work too well. More than a thousandth of a millimetre across, commercial powder particles are too big to penetrate deeply into all the cracks of the paint layer. Worse still, they tend to settle out from the solvent, producing an indecipherable white film on the paint surface.

The Italian chemists' particles are smaller: they are hexagonal plates about 100-250 nanometres (millionths of a millimetre) across. These penetrate a fresco more thoroughly, and, being light, do not settle out. The particles' flat shape makes them very water-absorbent, aiding their transformation to calcium carbonate as the alcohol evaporates.

Creating such small crystals is one of the objectives of nanotechnology, the manipulation of matter at the nanometre level. Nanotechnology, usually portrayed as the futuristic pursuit of molecular-scale machines, has something to offer the past too, it seems.

References

1. Androsi, M., Dei, L., Giorgi, R., Netti, C. & Baglioni, P. Colloidal particles of Ca(OH)₂: properties and applications to restoration of frescoes. *Langmuir*, **17**, 4211-4225, (2001).

© Nature News Service / Macmillan Magazines Ltd 2001



Wall paintings degradation



©Desprat, INAH 2010



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Templo Mayor
(Mexico City)



Stone degradation



**Angera stone
(dolostone)**

Milan, Italy



The use of physico-chemocal compatible materials

Lime is the original binder of the most of mural paintings and ornamental stones



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Why nano-particles

The advantage in using these particles are:

- 1) high reactivity**
- 2) Nano particles penetrate easily inside the artifact**
- 3) particles have enhanced physico-chemical properties**
- 4) Reactivity and properties can be controlled and modulated controlling the particle size**



Several methods to produce nanoparticles

- **Heterogeneous phase synthesis - slaking of lime**
- **Homogeneous phase synthesis in water solution**
- **Heterogeneous phase synthesis - hydrolisis of alkoxides in alcohol (solvo-thermal method)**
- **Homogeneous phase synthesis in diol solution**
- **Synthesis in a 'confined' environment, i.e. microemulsions**
- **Particle size/shape modifications by using co-solute/co-ions (Hofmeister-like behavior)**

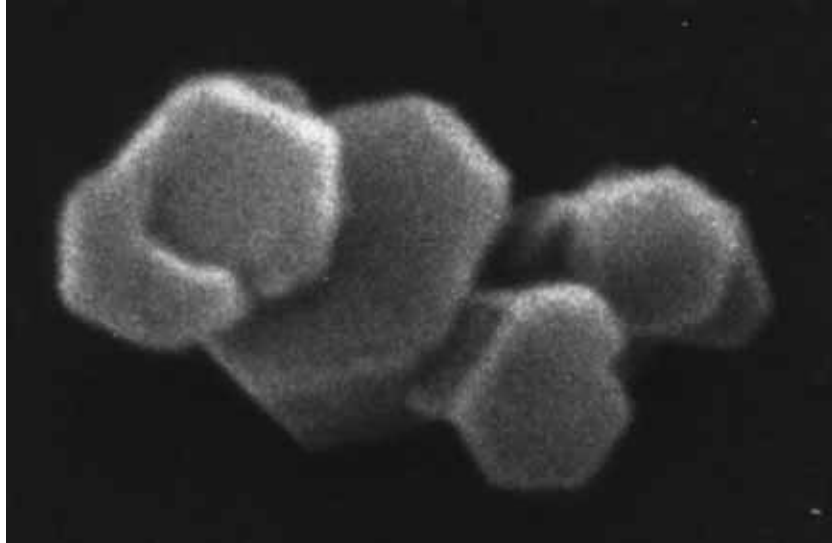


Baglioni, P.; Giorgi, R. - *Soft Matter* 2006, 2, 293-303.



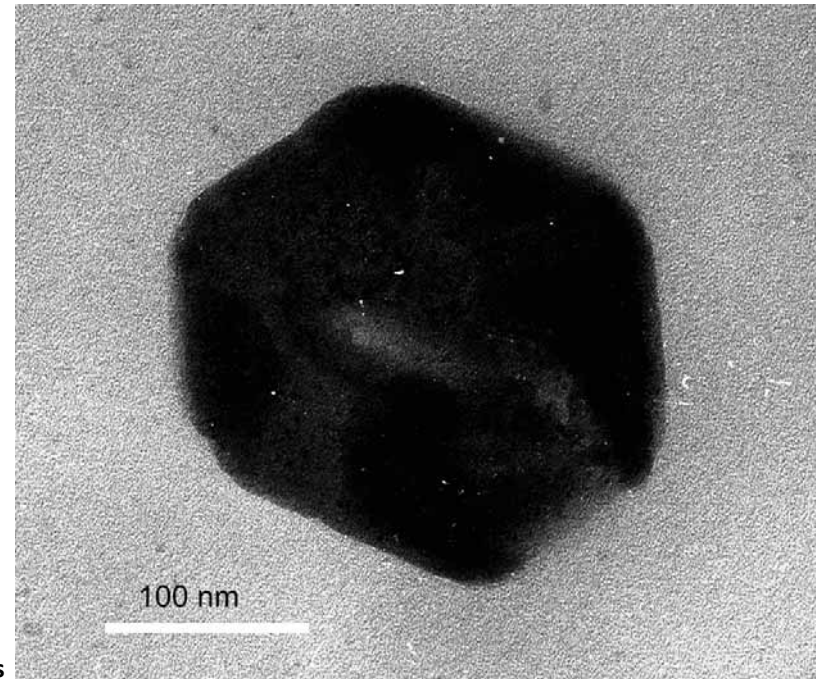
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Characterization of nanoparticles



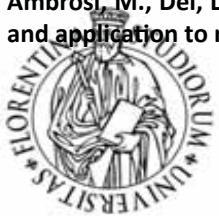
_____ 100 nm

SEM micrograph of Ca(OH)_2 particles obtained from homogeneous phase reaction at 90 °C;



TEM micrograph of a single Ca(OH)_2 particle prepared from homogeneous phase at 90 °C showing the hexagonal habitus.

Ambrosi, M., Dei, L., Giorgi, R., Neto, C., Baglioni, P. Colloidal particles of Ca(OH)_2 : properties and application to restoration of frescoes, *Langmuir* 17, 2001, 4251-4255.



Chemical composition

Mineralogical composition

AVERAGE SIZE

SIZE DISTRIBUTION

POLYDISPERSITY

CRYSTALLINITY

CRYSTAL HABITUS

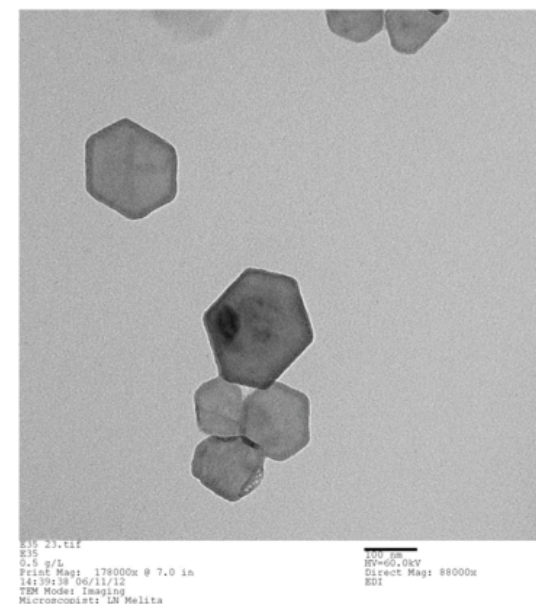
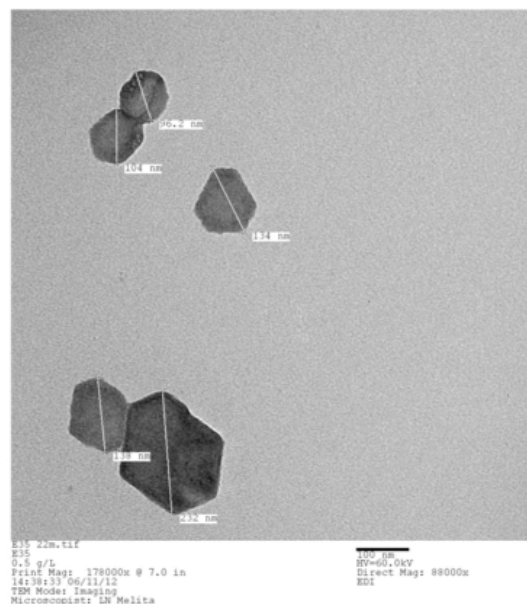
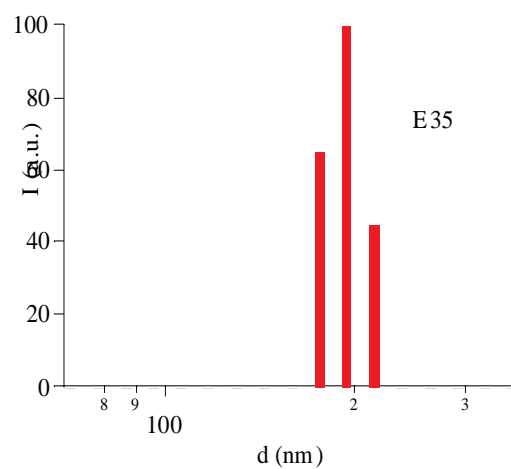
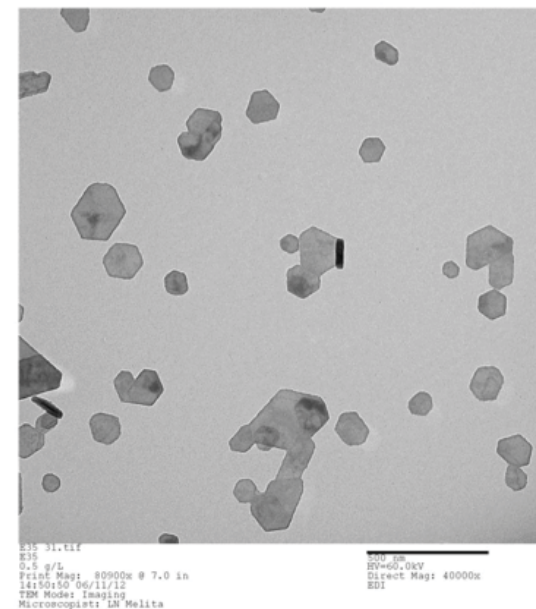
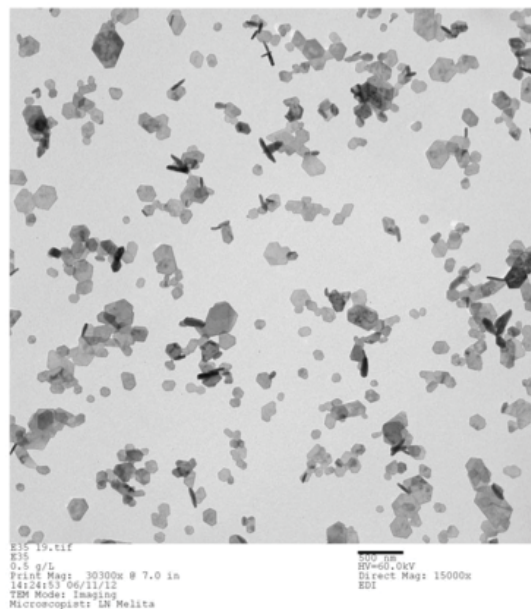
SPECIFIC SURFACE AREA

Which is the
relationship
between these
characteristics
and the chemical
reactivity ?



Calcium hydroxide
nanoparticles in ethanol
(CSGI sample E35)

Solvothermal process



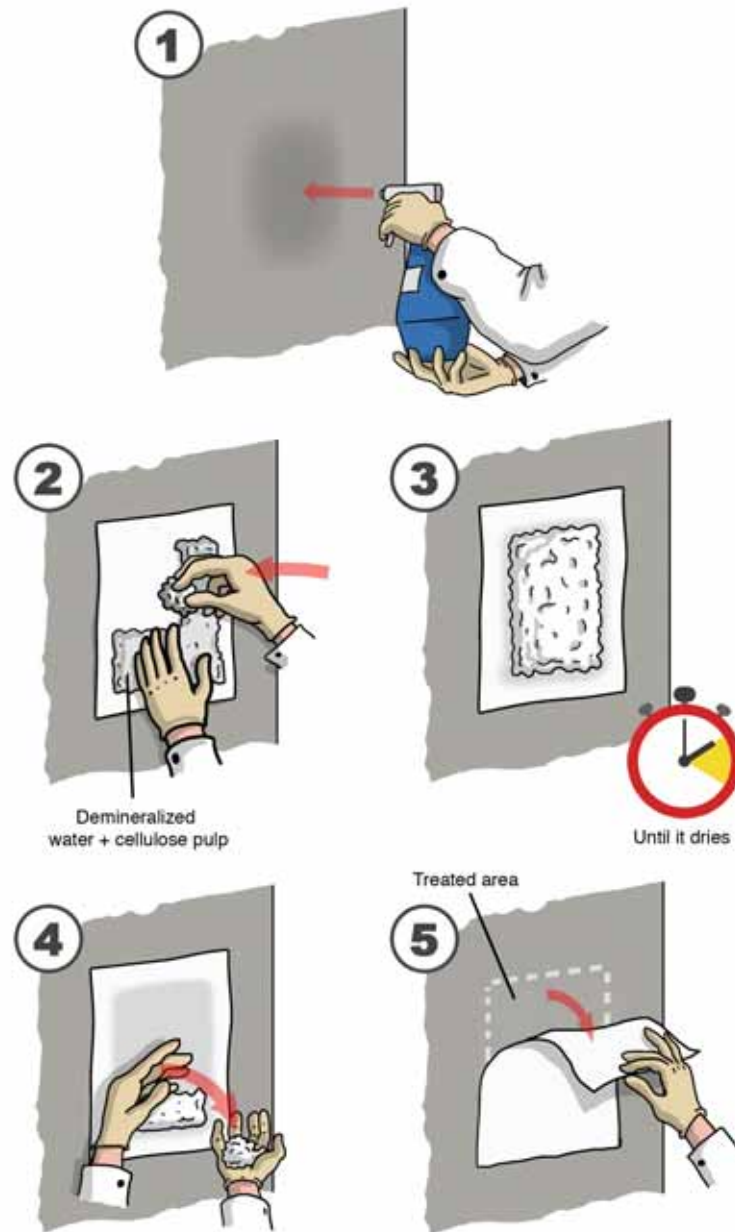
Application of Nanoparticles by brushing

Images source: Baglioni, Piero, Chelazzi, David, Giorgi, Rodorico, Nanotechnologies in the Conservation of Cultural Heritage - A compendium of materials and techniques, Springer, 2014

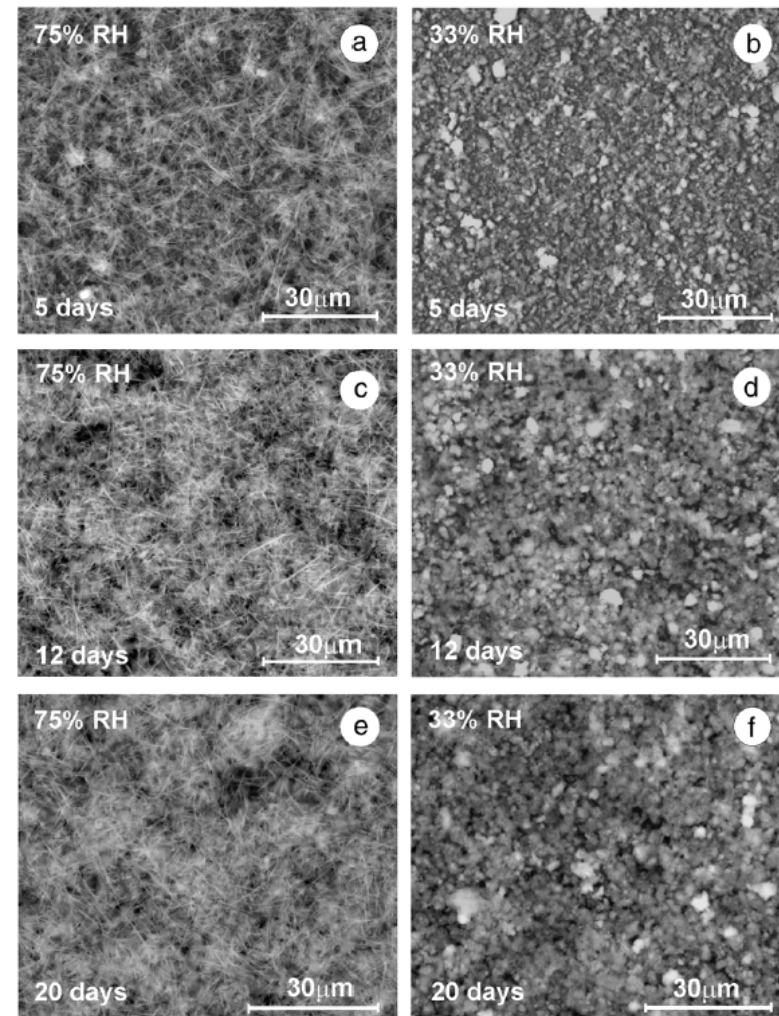
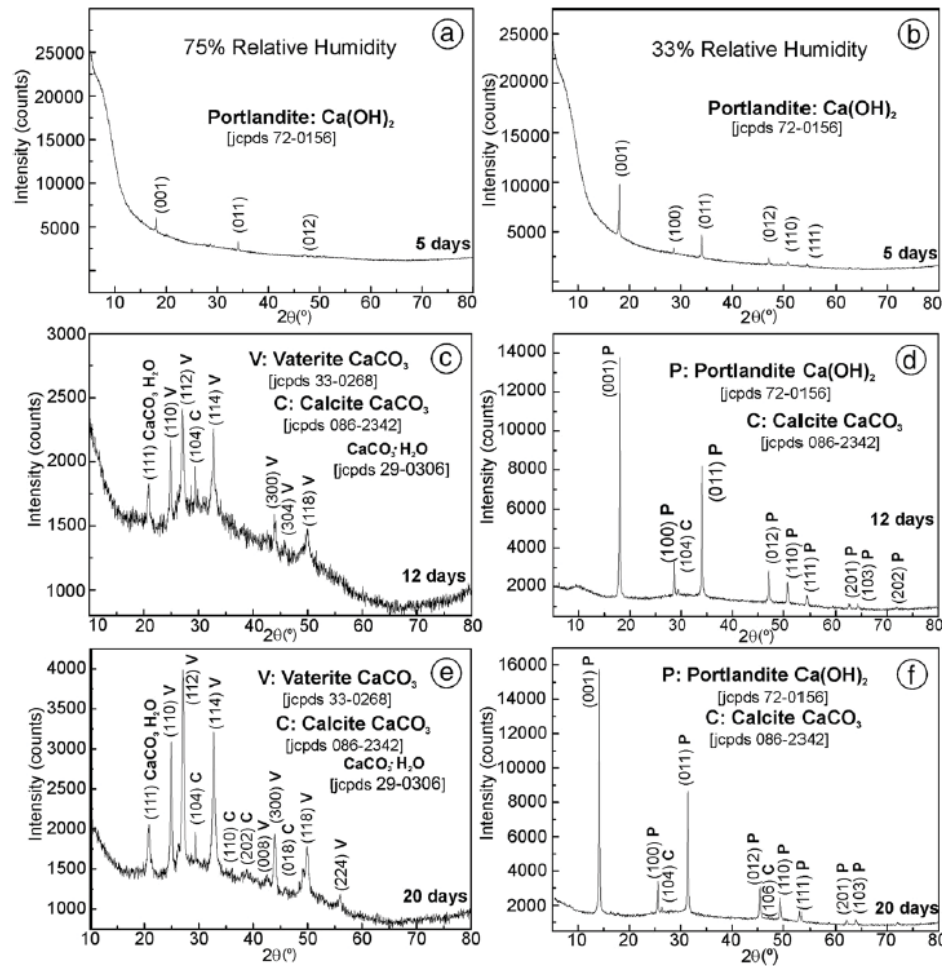


Application of nanoparticles with the spraying technique

Images source: Baglioni, Piero, Chelazzi, David, Giorgi, Rodorico, Nanotechnologies in the Conservation of Cultural Heritage - A compendium of materials and techniques, Springer, 2014



Carbonation process



Carbonation process

Dependence on the environmental conditions

XRD quantification of portlandite and calcium carbonate polymorphs depending on RH (relative humidity) and exposure time (days).

RH (%)	Time (days)	Portlandite (%)	Calcite (%)	Vaterite (%)	Aragonite (%)	MHC (%)
33	7	96	4	N/d	N/d	N/d
	28	84	8	N/d	N/d	8
54	7	92	N/d	8	N/d	N/d
	28	36	N/d	52	7	5
75	7	N/d	3	20	41	36
	28	N/d	11	39	23	27
90	7	N/d	32	37	N/d	31
	28	N/d	37	25	24	16

N/d (No detected); MHC (monohydrocalcite).



Maya paintings discovered in 2005 in CALAKMUL

The city was inhabited for more than twelve centuries starting around 400 b.C. (Pre Classic period) and been slowly abandoned until the year 900 A.D. (Post Classic period), reaching its maximum development between 600-800 A.D. (Late Classic period).



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Maya paintings discovered in 2005 in CALAKMUL



Acropolis Chik Naab - Building 1



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Maya paintings discovered in 2005 in CALAKMUL



May 2005



September 2005



CSGI

Maya paintings discovered in 2005 in CALAKMUL



Arqueologo Ramon Carrasco Vargas

Calakmul – cooperation with Arq. Ramon Carrasco Vargas

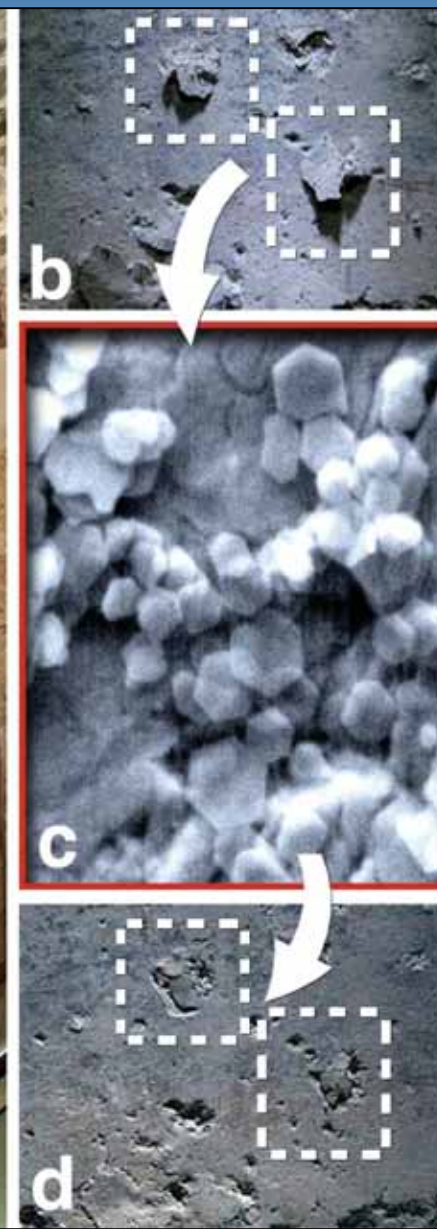
Pre-



Post-



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Earthen Masonry

- Over half world population lives in unbaked earth houses
- Earthen architectural heritage includes archaeological sites and modern buildings.

Adobe: handmade, mixing earth and water. Mixture adjusted with stabilizers (straw, dry grass) or artificial products, often including lime in small percentages

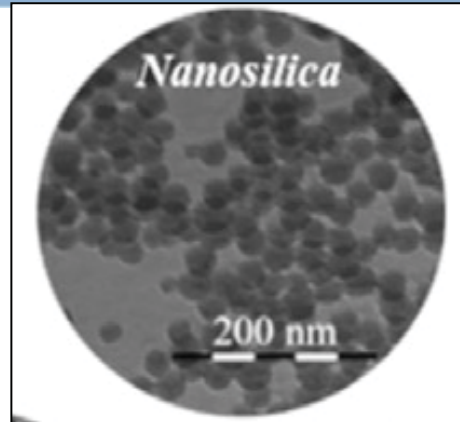
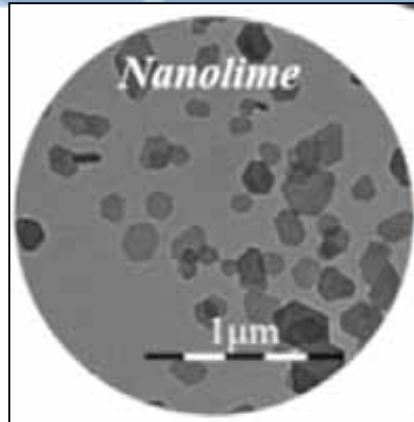


The largest adobe city on earth – Chan, Peru; Smithsonian Magazine, 2009



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NEW CONSOLIDANTS

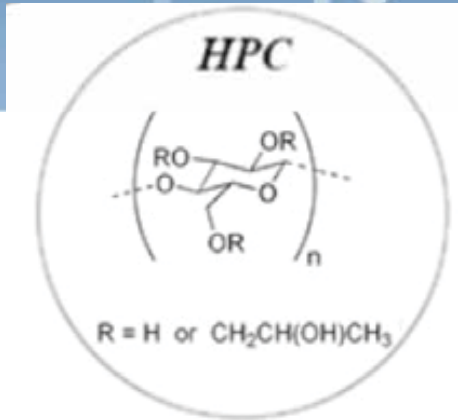


IN SITU formation of calcium silicate hydrate
(cement chemistry)

Organic-inorganic nano-composites
in (4:1) ethanol:water blend

Ethanol: optimal volatility, surface tension, and boiling point, for the application of nanoparticles to mortars and stone.

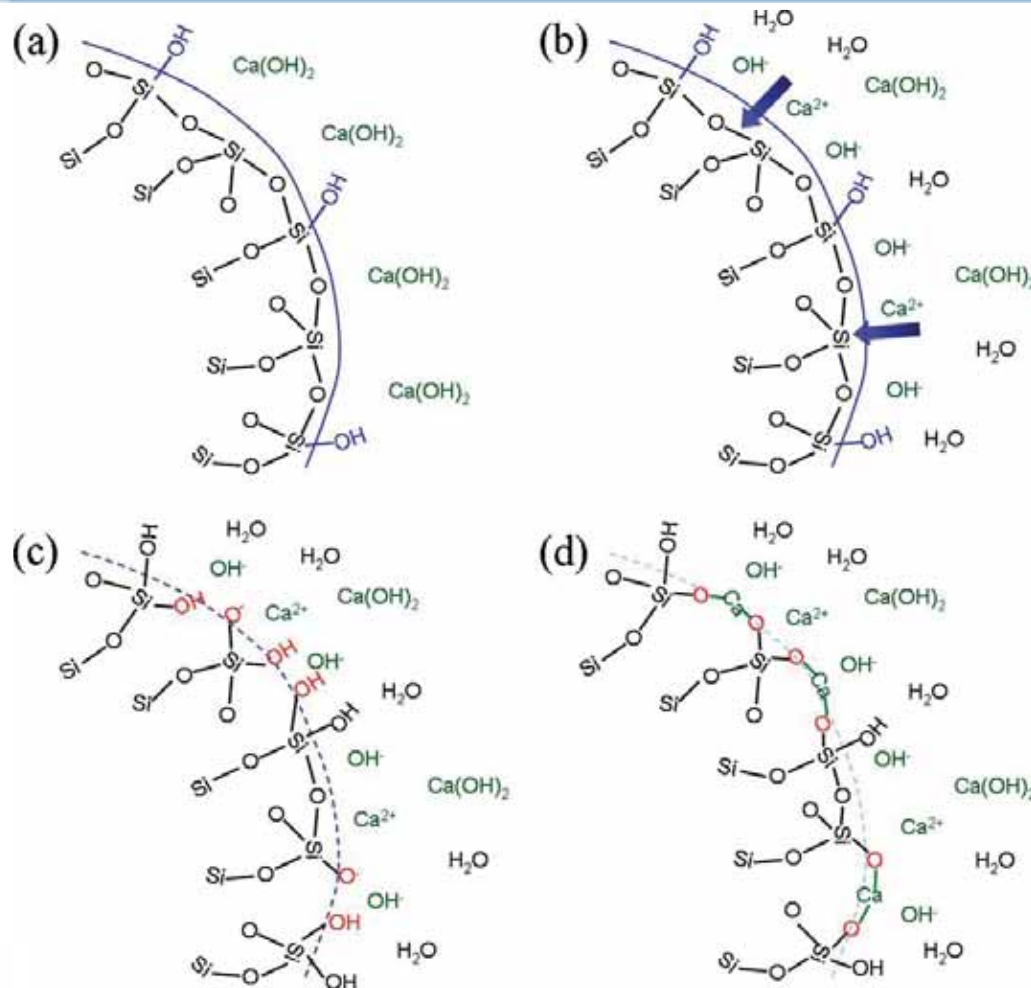
The amount of water was reduced, maintaining it suitable for the setting of CSH.



- viscosity-modifier
- provide flexural strength, reduce hygrometric shrinkage during drying
- regulator of water release during hydration reaction, increasing hydration efficiency, and promoting formation of CSH



SiO₂ particles and Ca(OH)₂ in water ...



Reaction affected by phase,
particle size, Ca(OH)₂ content
and L/P ratio in the paste

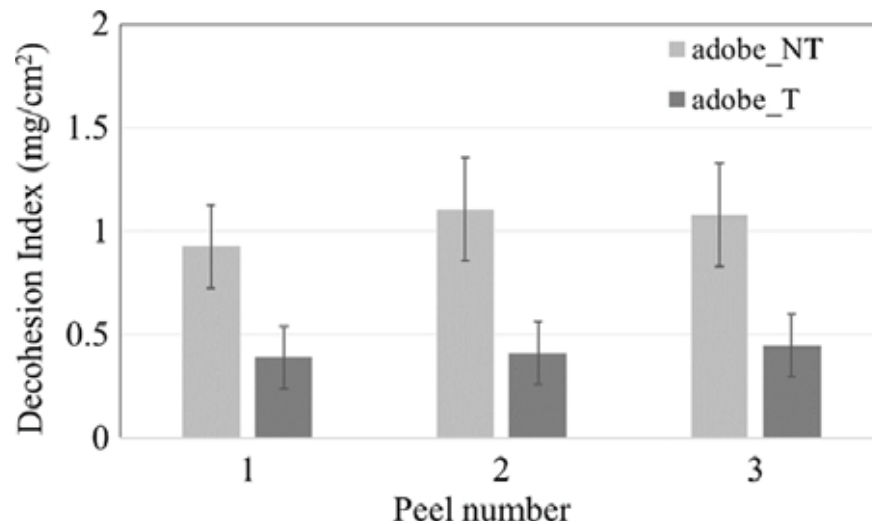
Basic unit
of CSH gel

Q. Lin, Z. Xu, X. Lan, Y. Ni, C. Lu. J. Biomed. Mater.
Res. B Appl. Biomater. 2011, 99(2):239-46



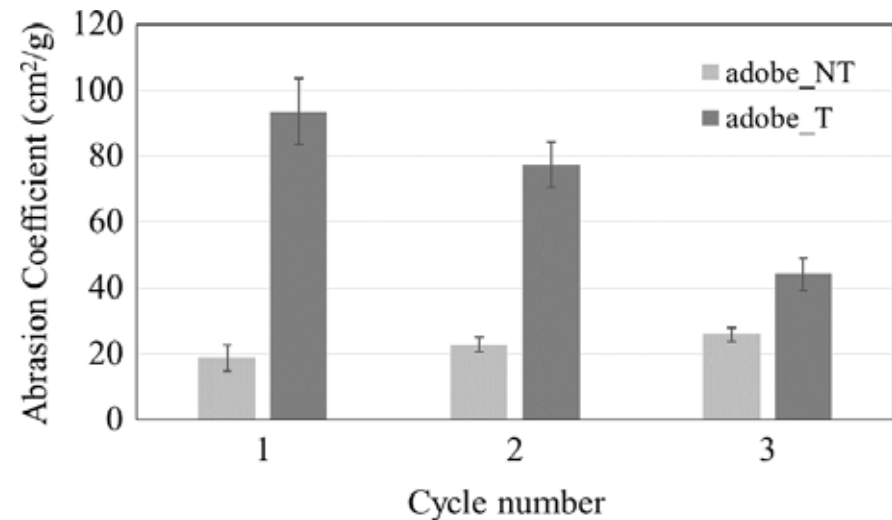
Adobe treatment

INCREASE OF COHESION



De-cohesion index (obtained with the scotch tape test) of adobe samples, untreated (NT), and treated with SiO₂_HPC_lime (T)

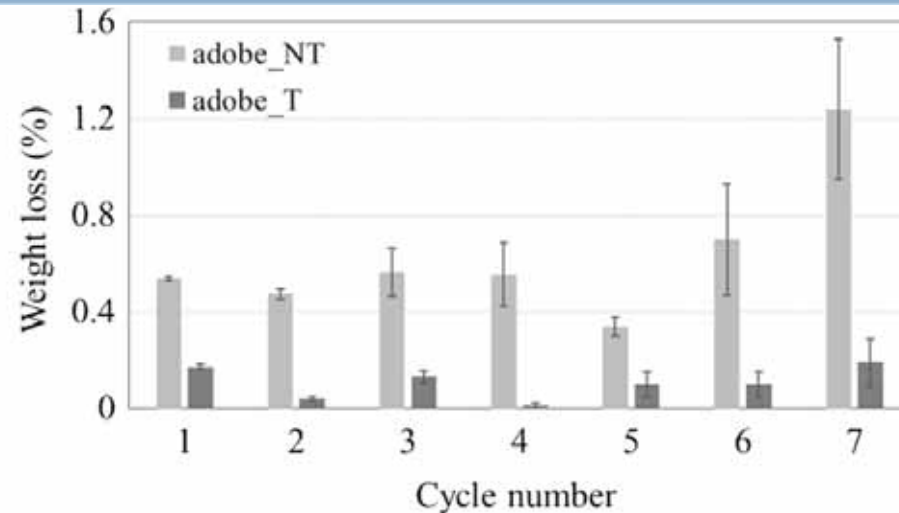
IMPROVED RESISTANCE TO ABRASION



Abrasion coefficient of adobe samples, untreated (NT), and treated with SiO₂_HPC_lime (T)

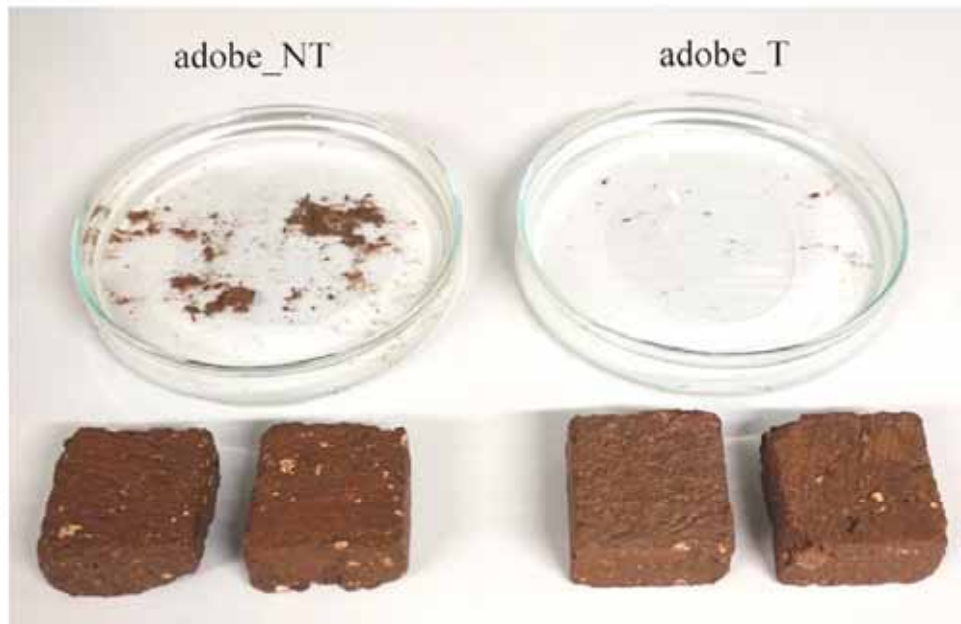


Adobe treatment



WEIGHT LOSS DURING WET/DRY CYCLES

Weight loss of adobe samples, untreated (NT), and treated with SiO₂_HPC_lime (T), during wet/dry cycles



The adobe samples (untreated and treated) after the seventh wet/dry cycle.



*Thanks for your attention
and kind invitation*

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