CSGI - Italian Center for Colloid and Nanoscience

Università degli Studi di Firenze

CSGI stands for "*Consorzio per lo sviluppo dei Sistemi a Grande Interfase*"

The main goal of CSGI is the preparation and study of new supramolecular "smart" devices and colloidal systems, and the development of their different industrial applications such as pharmaceutics, bio-medical, inks and paintings, oil and energy, metallurgic, detergents, cosmetics, textile, paper, food industry, de-polluting procedures, conservation of cultural heritage, and so forth.

CSGI is also intended to provide Industrial companies with the basic scientific and technological knowledge to improve and optimize specific industrial processes.

CSGI promotes research projects and high level educational training in the field of Colloid and Surface Science in collaboration with other Universities, Research Centers and Industries, with the scientific contribution of the European Union, of the Italian Ministry of University and Scientific Research.

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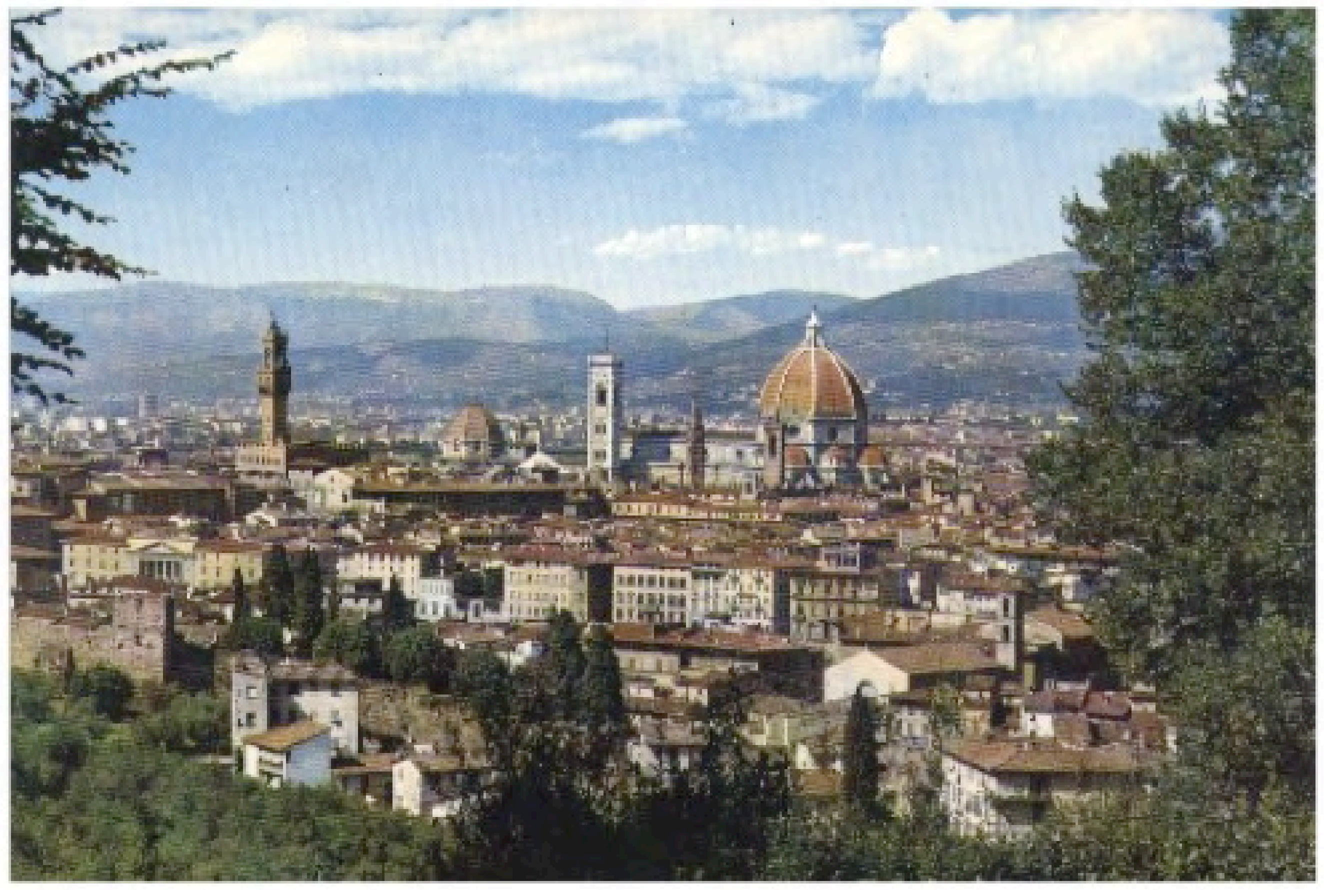

Tasks in the project

CSGI is mainly responsible for the development of materials and techniques based on nanotechnology for the presevation and conservation treatment of movable and immovable works of art (**WP_1**).

CSGI will be also involved in the physico-chemical characterization of nanomaterials (**WP_2**) and in the preliminary applicability assessment (**WP_3**).

The development of nanomaterials will start in the first phase of the project and will continue interactively with the other partners during the following phases, in order to maximize the materials performances and durability.

CSGI will also monitor RTD activities as technical coordinator of the project, and will be thus involved in the overall management including financial and IPR management, the general overview of the project progress, organization of work-activities, and definition of roles (**WP_9**).

CSGI will also participate in the upscale and commercialization of developed nanotechnology (**WP_4, 5**), and in its dissemination (**WP_8**)

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Instrumentation available for the Nanoforart project

Dynamic Quasi-Elastic Light scattering (QELS) - Brookhaven apparatus (BI200SM with BI9000AT correlator)

Nanoviewer SAXS spectrometer Rigaku based on Confocal Max-Flux™ Optics (Osmic) coupled with a 2-D CCD detector (Mercury70) mounted on a MicroMax 007 rotating anode generator

Simultaneous Small/Wide Angle X-Ray Scattering (SWAXS) - Hecus X-Ray System GmbH Kratky camera coupled with two 1-D Position-Sensitive Detectors (Methane/Ar) mounted on a ID3003 generator (Seifert)

Differential Scanning Calorimeter (DSC) - Perkin Elmer DSC-7

Differential Scanning Calorimeter (DSC) - TA-Instruments Q-1000

Differential Thermal Analysis coupled with ThermoGravimetry (DTA-TG) - TA Instruments Q-600

Viscometry-Rheometry - Paar Physica UDS-200

Fourier Transform InfraRed Spectroscopy (FTIR) - BioRad FTS-40

Fourier Transform Infrared Spectroscopy (FTIR) - Nicolet Nexus 870

Optical Microscope (OM) Reichert Zetopan

Surface Area BET Apparatus (SA-BET) - Coulter SA-3100

Static Tensiometry (ST) - KSV Sigma 70

Dynamic Surface Tensiometry (DST) - IT Concepts The Tracker

Scanning Electron Microscopy (SEM) Cambridge Stereoscan S360 coupled with Energy dispersive x-ray spectrometry (EDS) - Oxford Instruments INCA X-Sight

FEG electron microscope Gemini (Zeiss) coupled with Energy dispersive x-ray spectrometry (EDS) – Bruker Quantax 200

Contact Angle Goniometer - (Rame'-Hart, USA) Advanced Automated Goniometers Model 300

Spectrofluorimeter - Perkin Elmer, Italy; LS50B

UV/Vis/NIR Spectrophotometer - Perkin Elmer, Italy; Lambda 900

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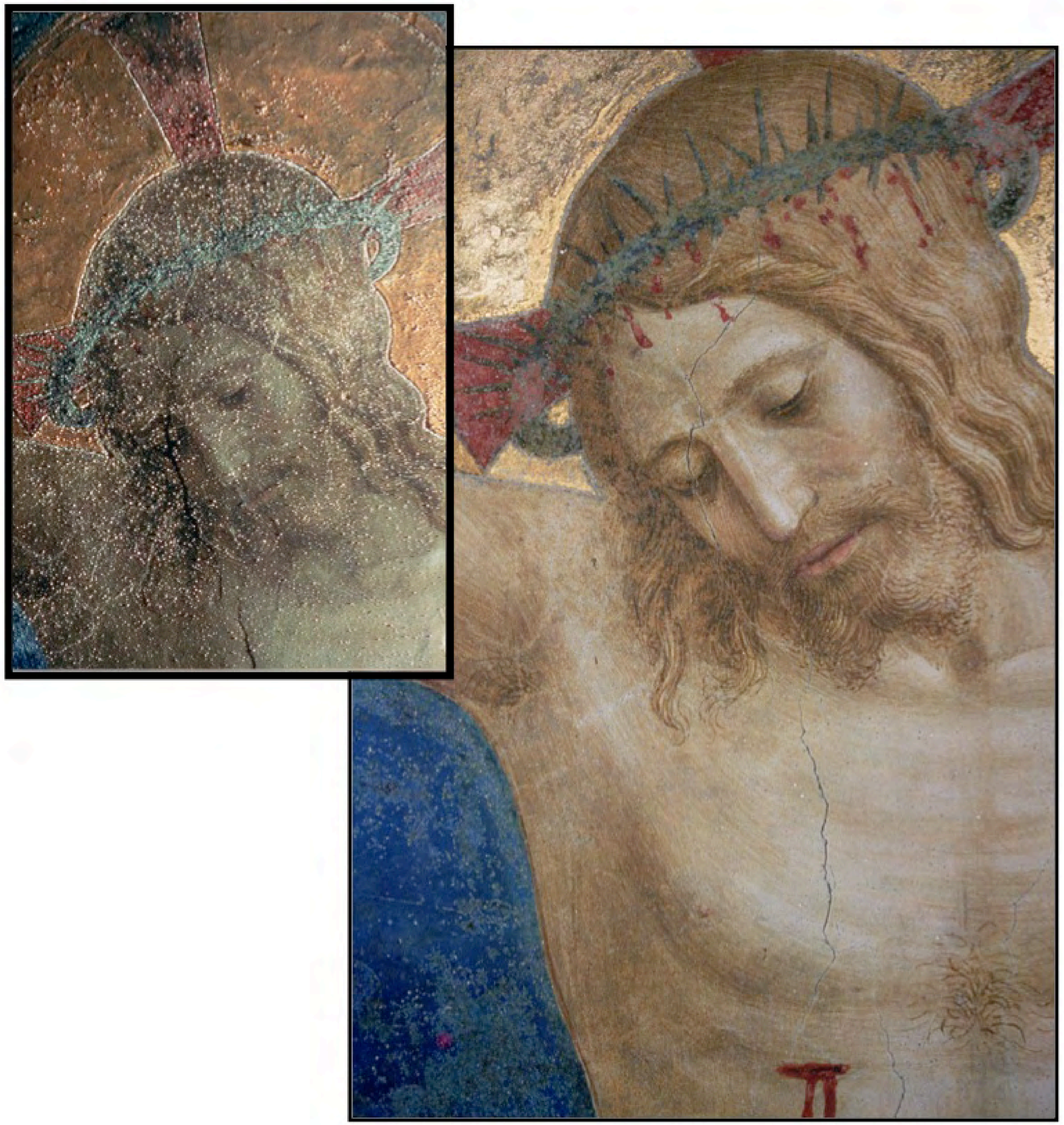

Wall paintings by Beato Angelico
-Italian Renaissance-

Consolidation and de-sulphatization by using Ferroni method

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CONSOLIDATION OF WALL PAINTINGS AND CARBONATIC STONES with NANOPARTICLES


nature scienceupdate
updated at midnight GMT today is wednesday, august 29
search nature science update go advanced search
news

Nanotechnology restores flaking frescos.

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

PHILIP BALL

A beautiful bottom - thanks to the restorative effects of nanotechnolgoy.

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.

Piero Baglioni and colleagues from the University of Florence have salvaged a lesser-known work: Gli Angeli Musicanti 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 skilfully, 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

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 bond[1].

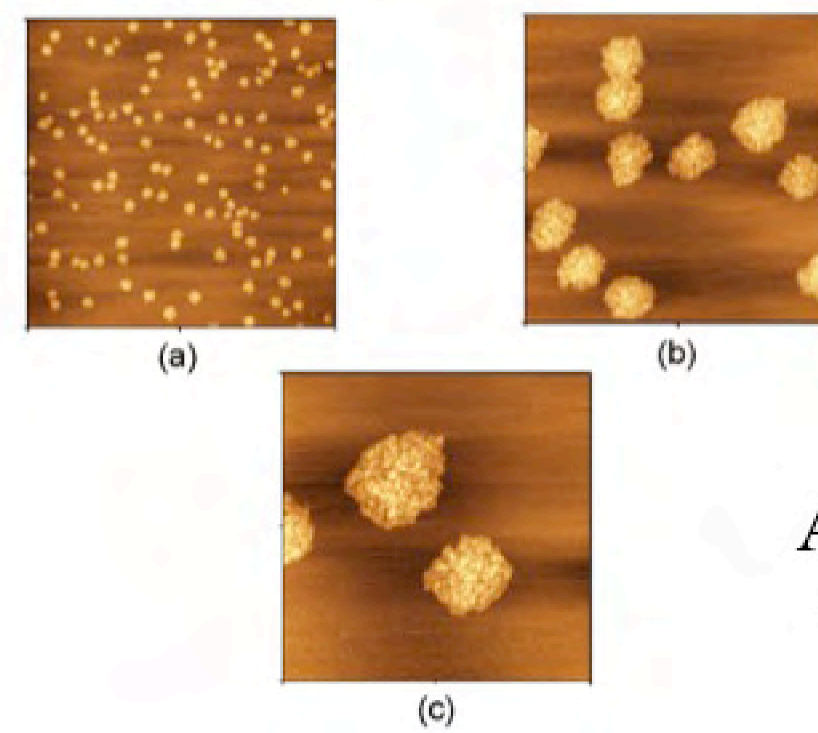
Ordinary ground-up calcium hydroxide doesn't work too well. More than a thousandth of a millimetre across, commercial powder particles are too big to penetrate deeply into all the cracks of the paint layer. Worse still, they tend to settle out from the solvent, producing an indelible white film on the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

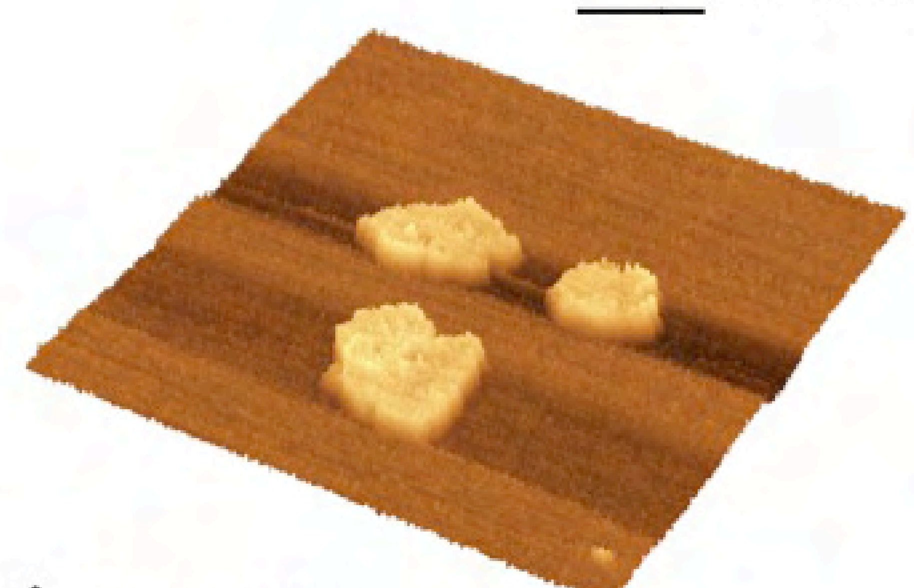
References

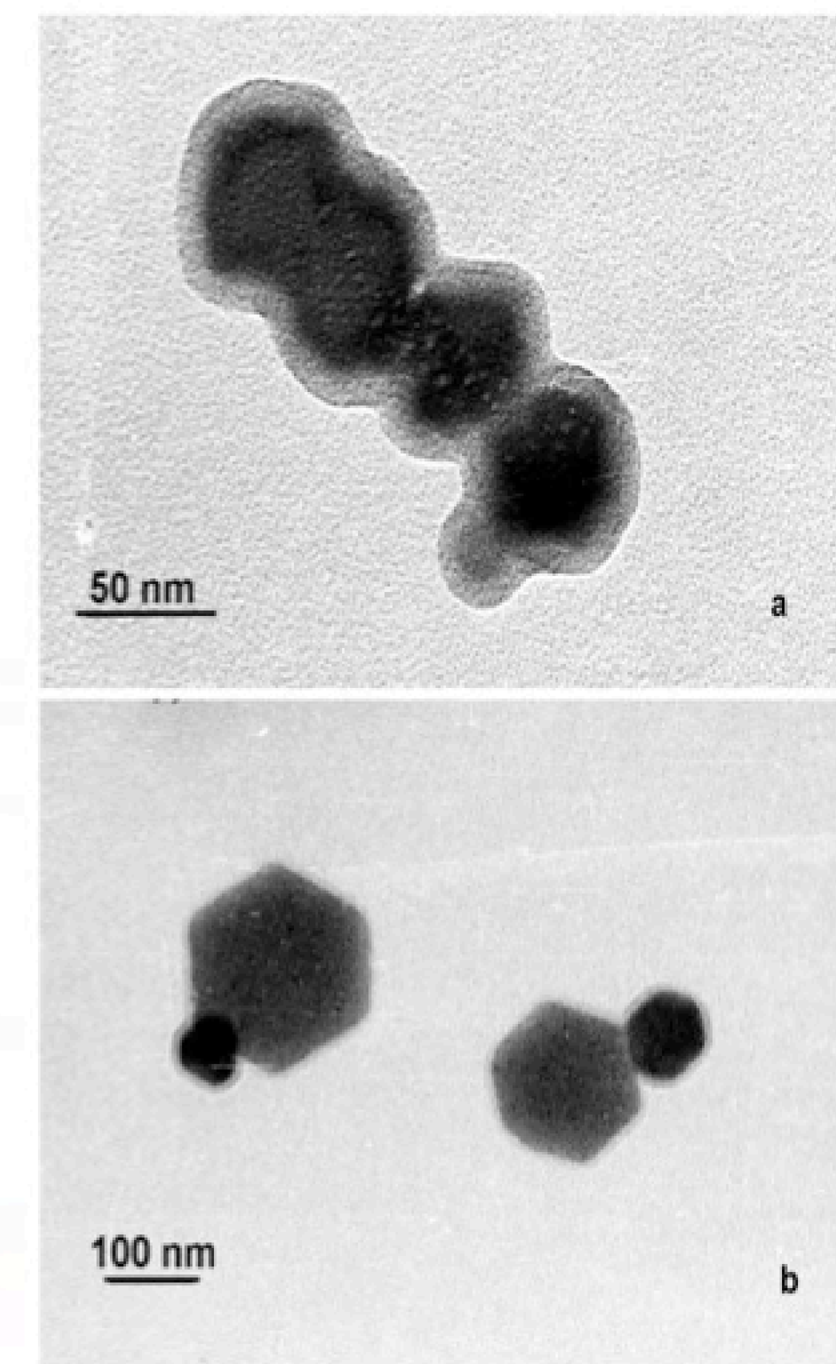
1. Ambrosi, M., Dei, L., Giorgi, R., Neto, C. & Baglioni, P.Colloidal particles of Ca(OH)2: properties and applications to restoration of frescoes. ***Langmuir***, **17**, 4251 - 4255, (2001).



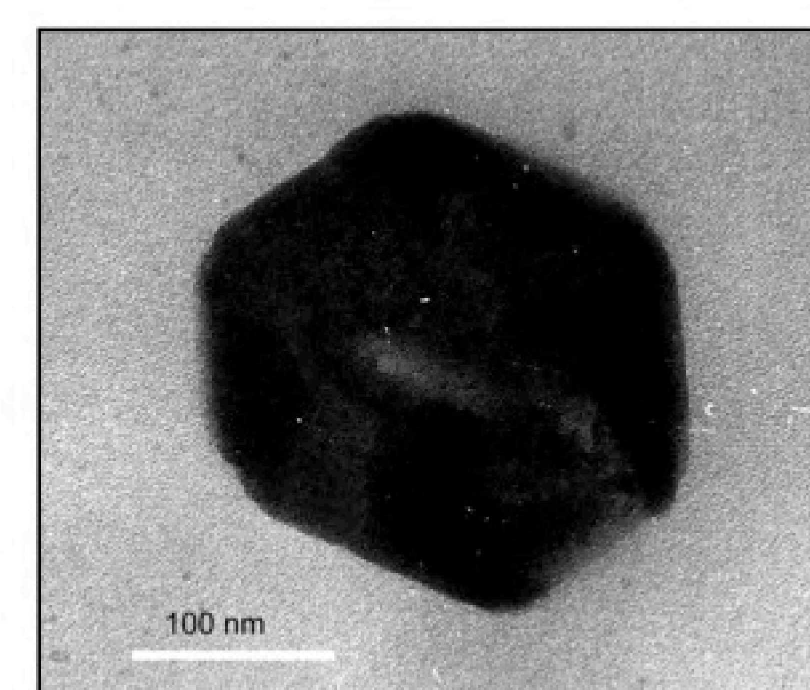
Atomic force microscopy AFM

___50 nm

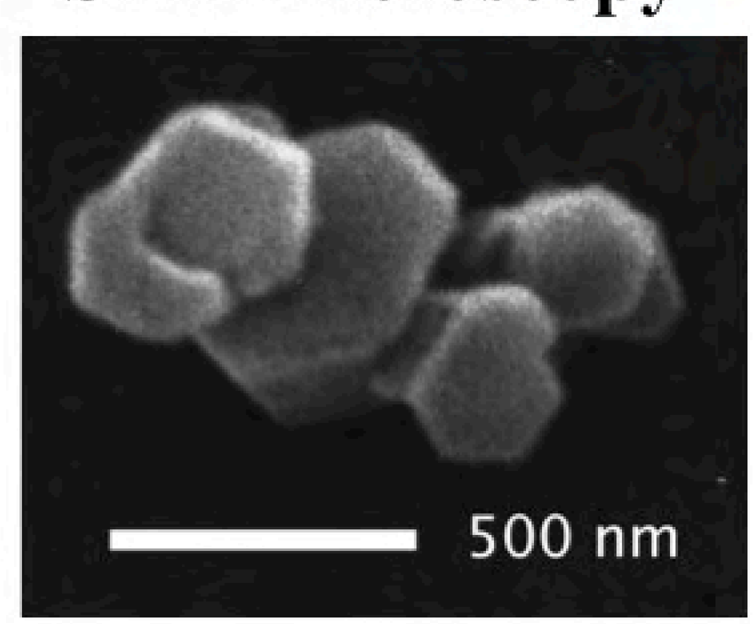




TEM microscopy



SEM microscopy



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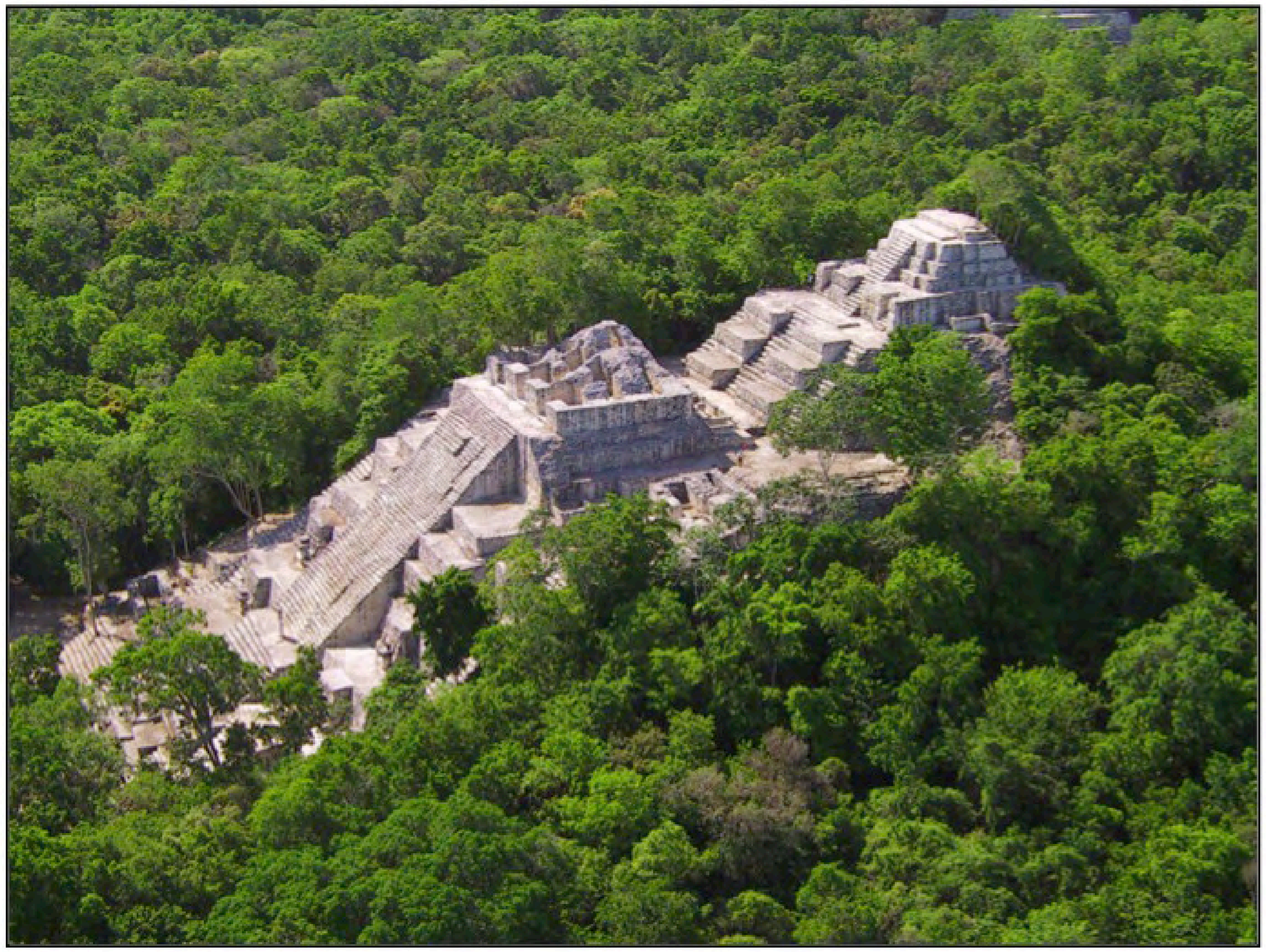
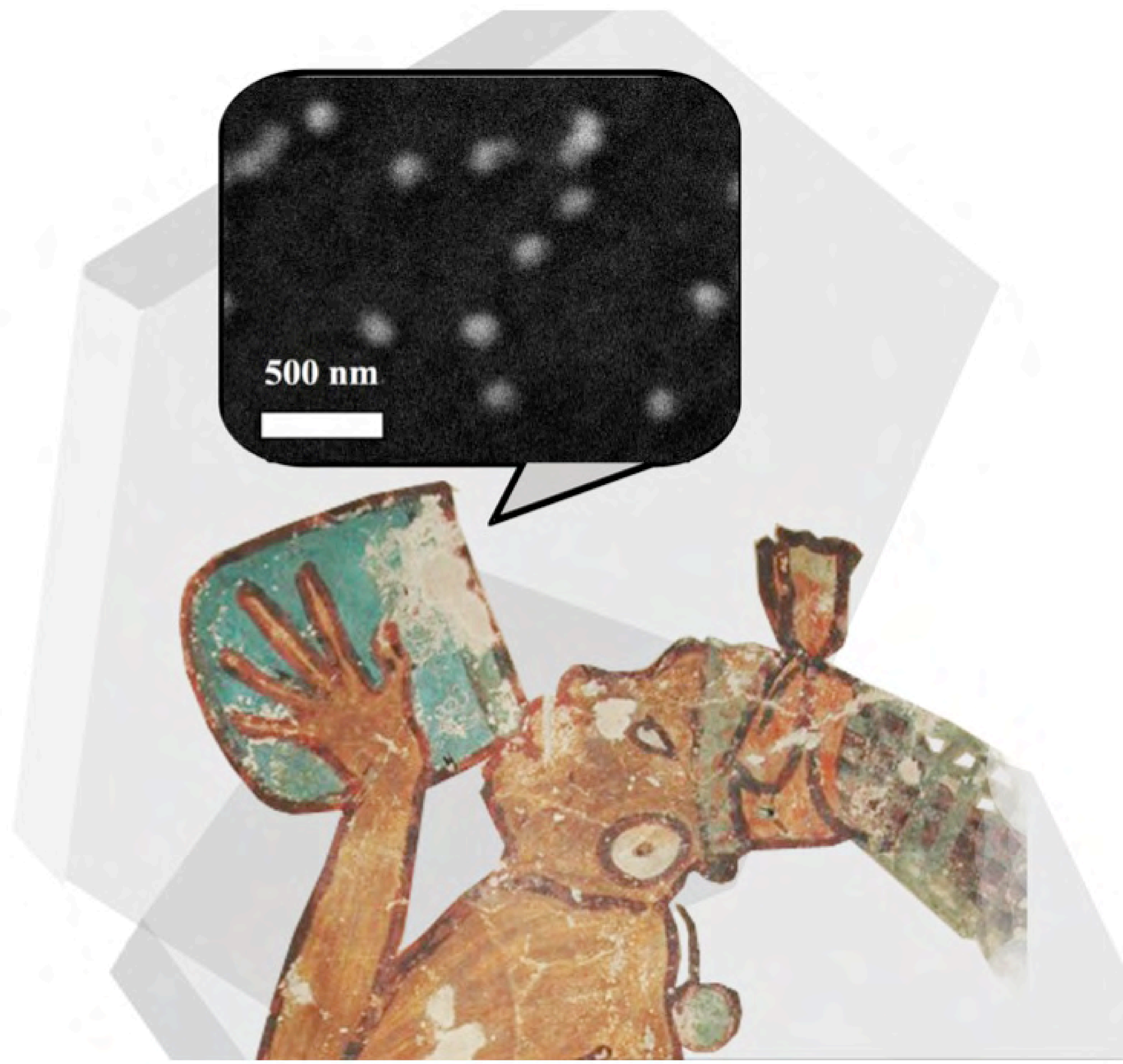
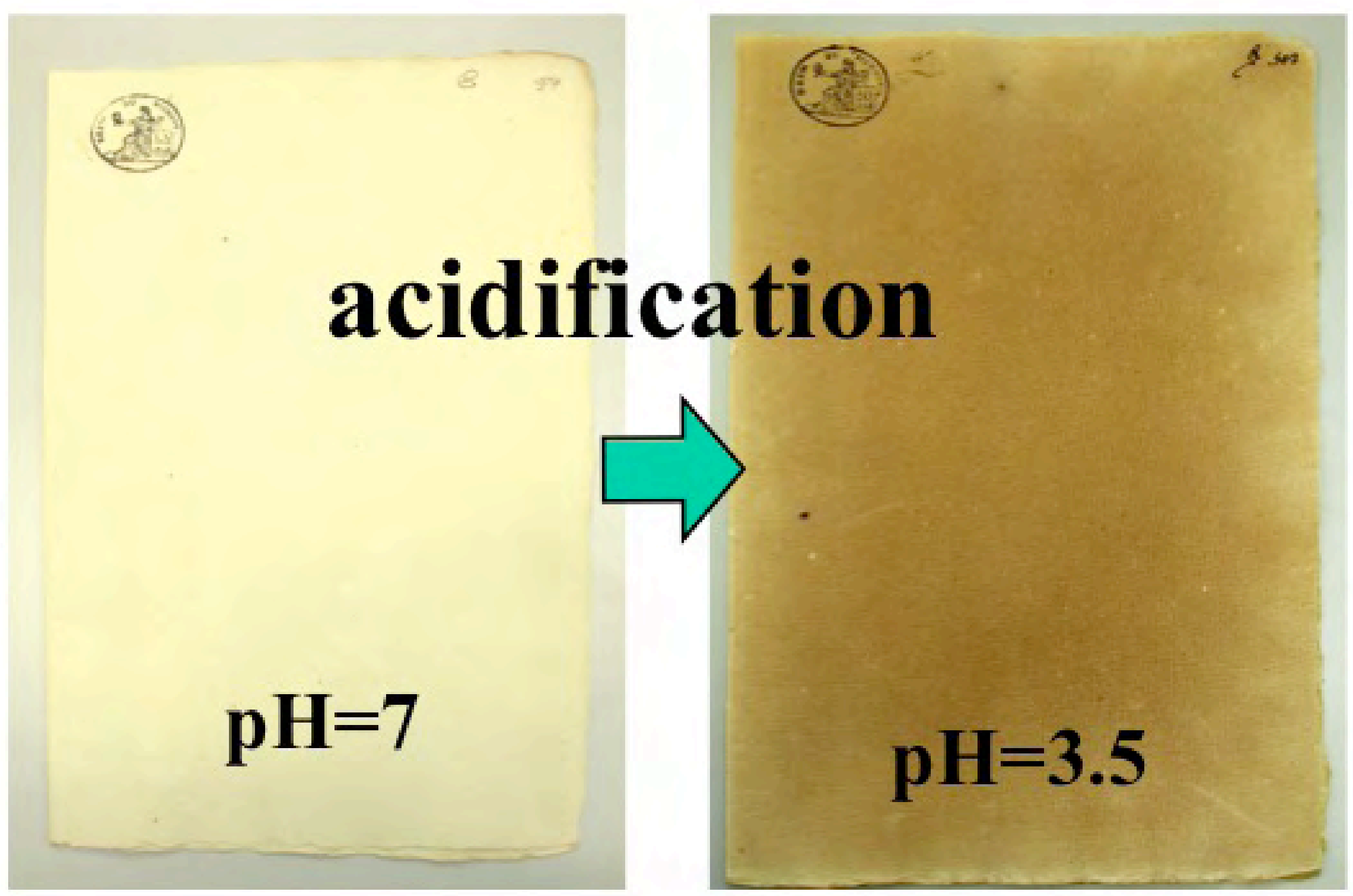


Unesco Heritage List site

The Maya site of Calakmul

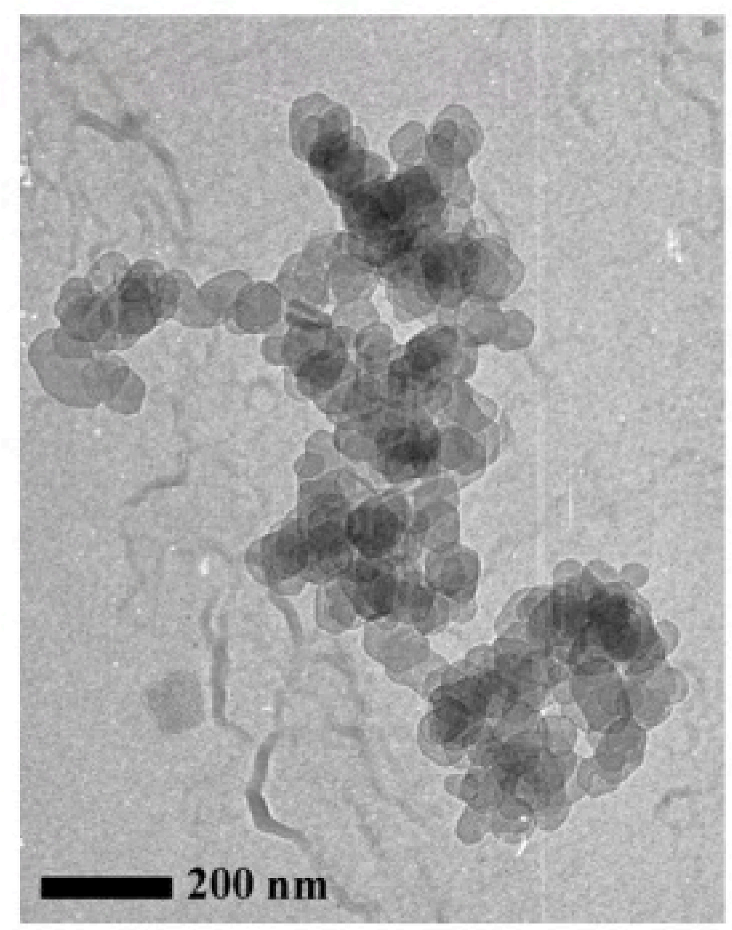


Alkaline nanoparticles for PAPER DEACIDIFICATION

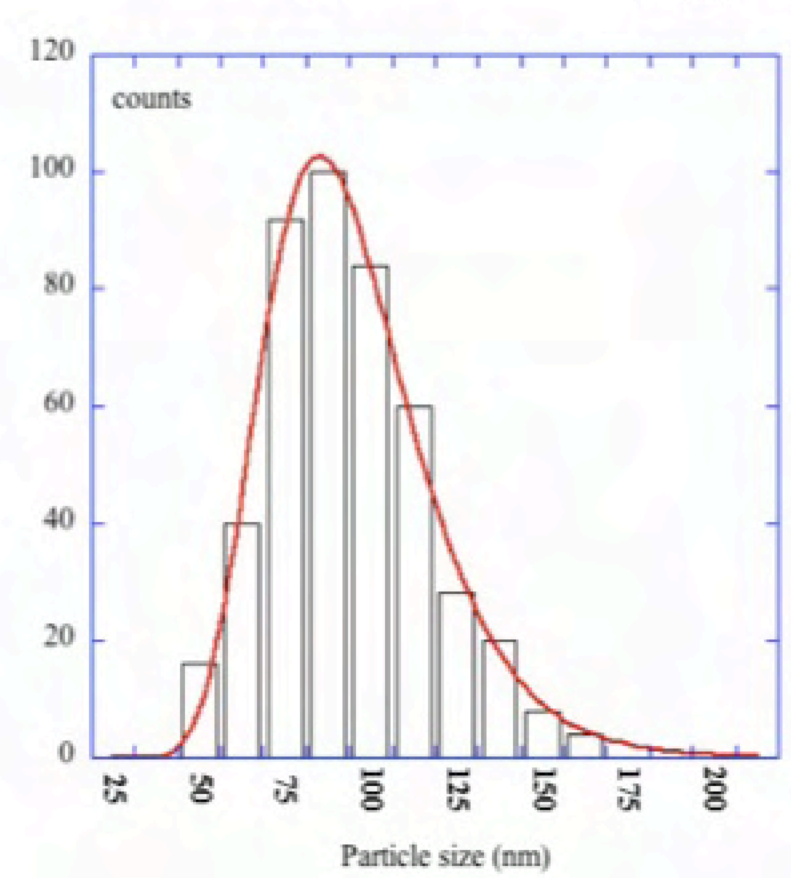


Acid-catalyzed processes

Hydrolysis of glycosidic bonds (cellulose depolymerization)



$Mg(OH)_2$



nature scienceupdate news

Nanoparticles save paper

A sprinkling of slaked lime conserves old documents.

22 October 2002

PHILIP BALL

Acids slowly break down the cellulose fibres in paper.
© GettyImages

Tiny particles of a strong alkali are helping preserve historical documents.

Piero Baglioni of the University of Florence and his coworkers have treated manuscripts dating back to the fourteenth century with a sprinkling of calcium hydroxide grains just 200 millionths of a millimetre across.

The nanoparticles of what is commonly called slaked lime penetrate between paper's fibres. They combat the ravages of acids introduced when paper is made, without altering documents' apperance[1]. The technique is cheap and green and could also be used on canvas.

The researchers subjected one nineteenth-century sample to accelerated ageing by keeping it in a hot, moist environment for three weeks. Untreated, the paper turned a deep brown because of acid damage. A treated sample showed very little discolouration.

Neutral territory

Before the nineteenth century, paper was made primarily from shredded rags; thereafter wood pulp has been used. Acids slowly break down the cellulose fibres in paper, making it yellow, brittle and weak.

Currently museum conservators and librarians use chemical treatments to prevent or retard this degradation. A common method, the Book-keeper process, involves scattering grains of magnesium oxide over the paper. These stick to the fibres and react with water to form an alkali that neutralizes acids.

Baglioni's team penetrate paper more thoroughly by spraying it with nanoparticles dispersed in propanol, a volatile alcohol. Paper could even be brushed with or immersed in this liquid, they suggest.

Water would simply dissolve the calcium hydroxide to form a strong alkali solution that might itself harm the document. Water could also dissolve and smudge the inks and paints used in some old manuscripts.

Once stuck to the cellulose fibres, the nanoparticles react slowly with carbon dioxide in the air to form calcium carbonate - in effect, a very fine powdered chalk. This creates a stable and durable reservoir of alkali to neutralize acids.

Paper samples from the fourteenth to the twentieth centuries, some of which were initially mildly acid, became near-neutral or mildly alkaline after nanoparticle treatment. They will probably stay this way, the researchers suspect.

References

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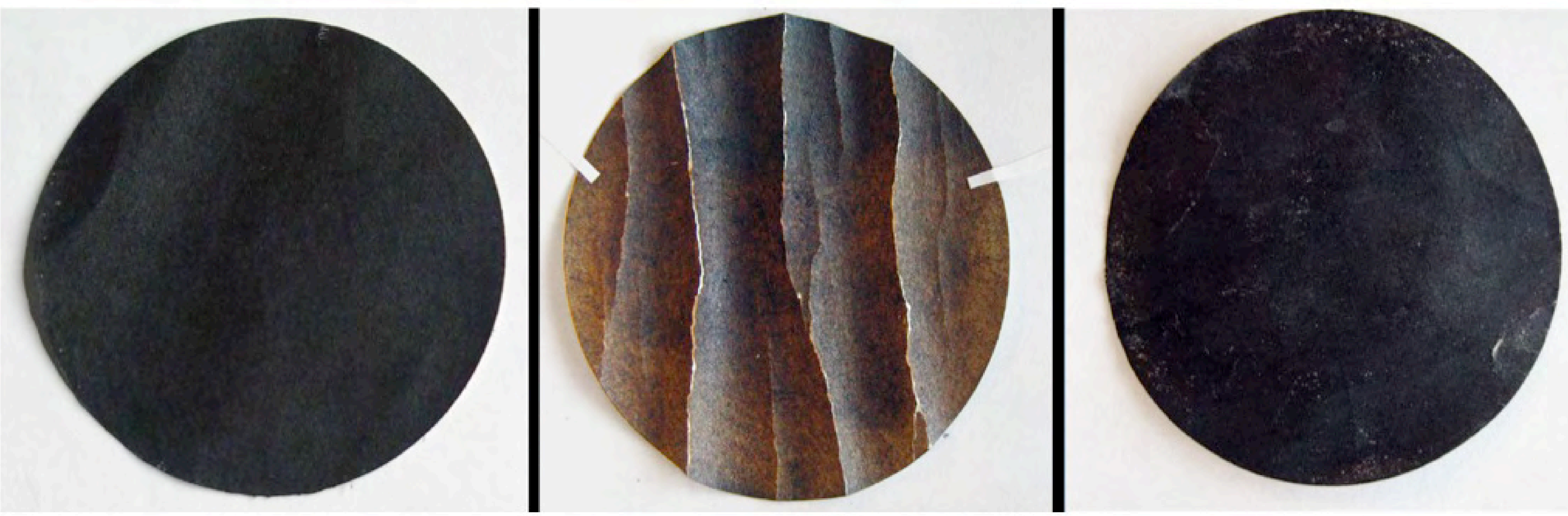
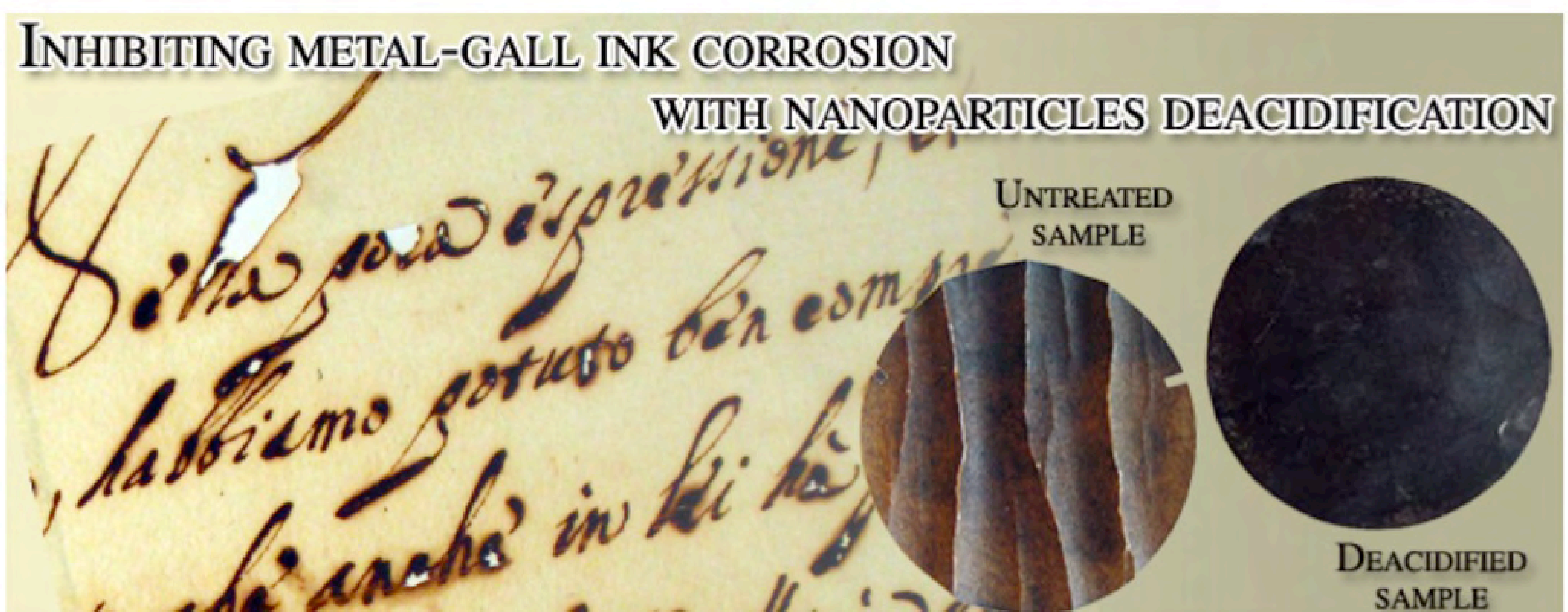
Paper protection from oxidation (Fenton reaction) using $Mg(OH)_2$ nanoparticles



BEFORE AGING AGED AGED NANOPARTICLE



$Mg(OH)_2$ size from 30-50 nm

1) formation of peroxides in <u>acidic medium (Fenton reaction)</u>:

$Fe^{2+} + O_2 + H^+ \rightarrow Fe^{3+} + OOH\bullet$

$Fe^{2+} + OOH\bullet + H^+ \rightarrow Fe^{3+} + H_2O_2$

2) homolytic decomposition of peroxides by transition metal ions:

$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH- \quad OH\bullet$
(Fenton reaction)

$Cu^{+} + H_2O_2 \rightarrow Cu^{2+} + OH- \quad OH\bullet$
(Fenton-like reaction)

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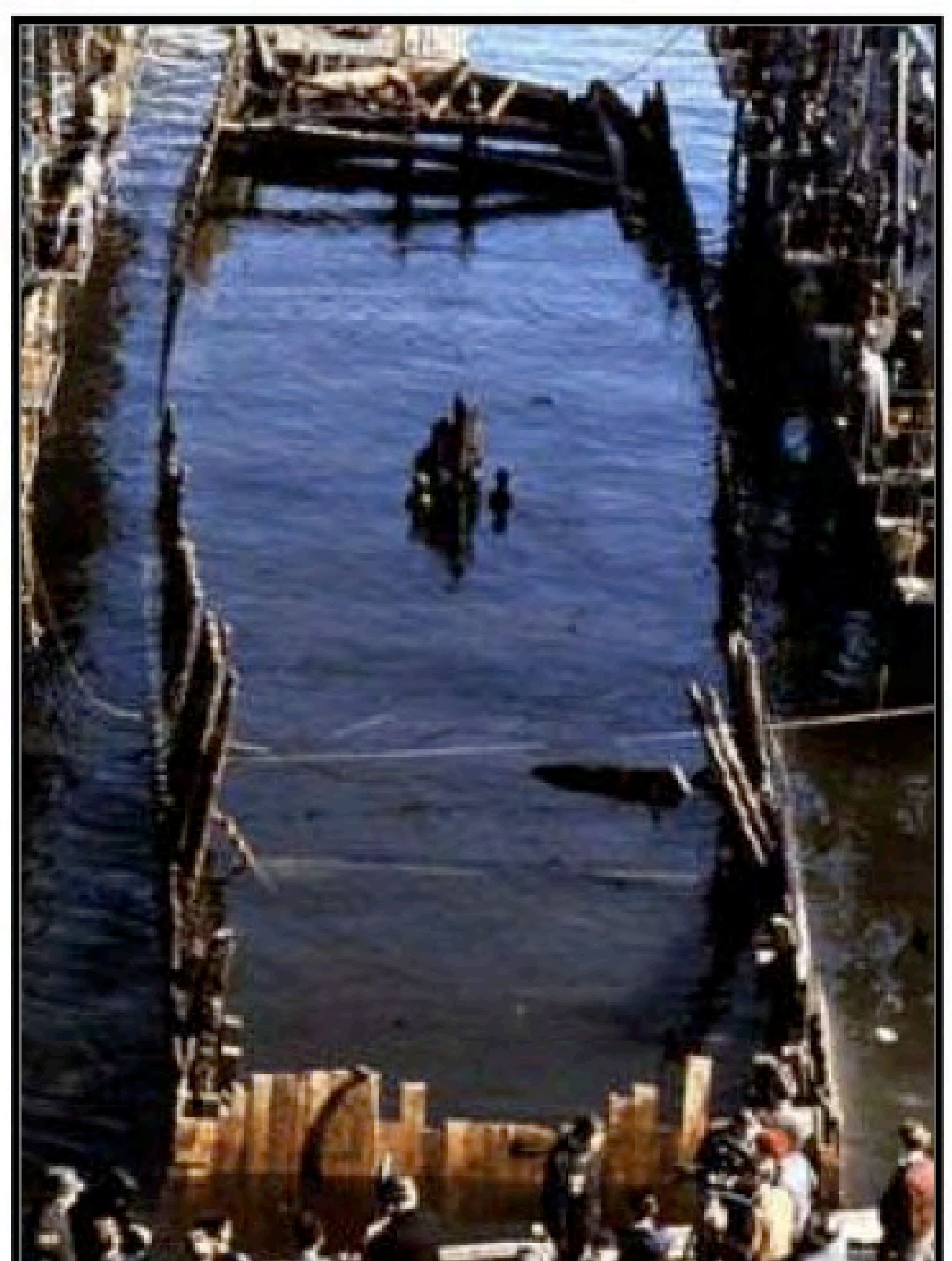
The Vasa warship history

10 Agosto 1628



1956: Anders Franzen discovered the shipwreck

1961 (April 24th) : after 333 anni in the seabed, the Vasa wreck was recovered

1962-1979: for 17 years the shipwreck was treated with PEG to prevent, after drying, the ship from shrinking

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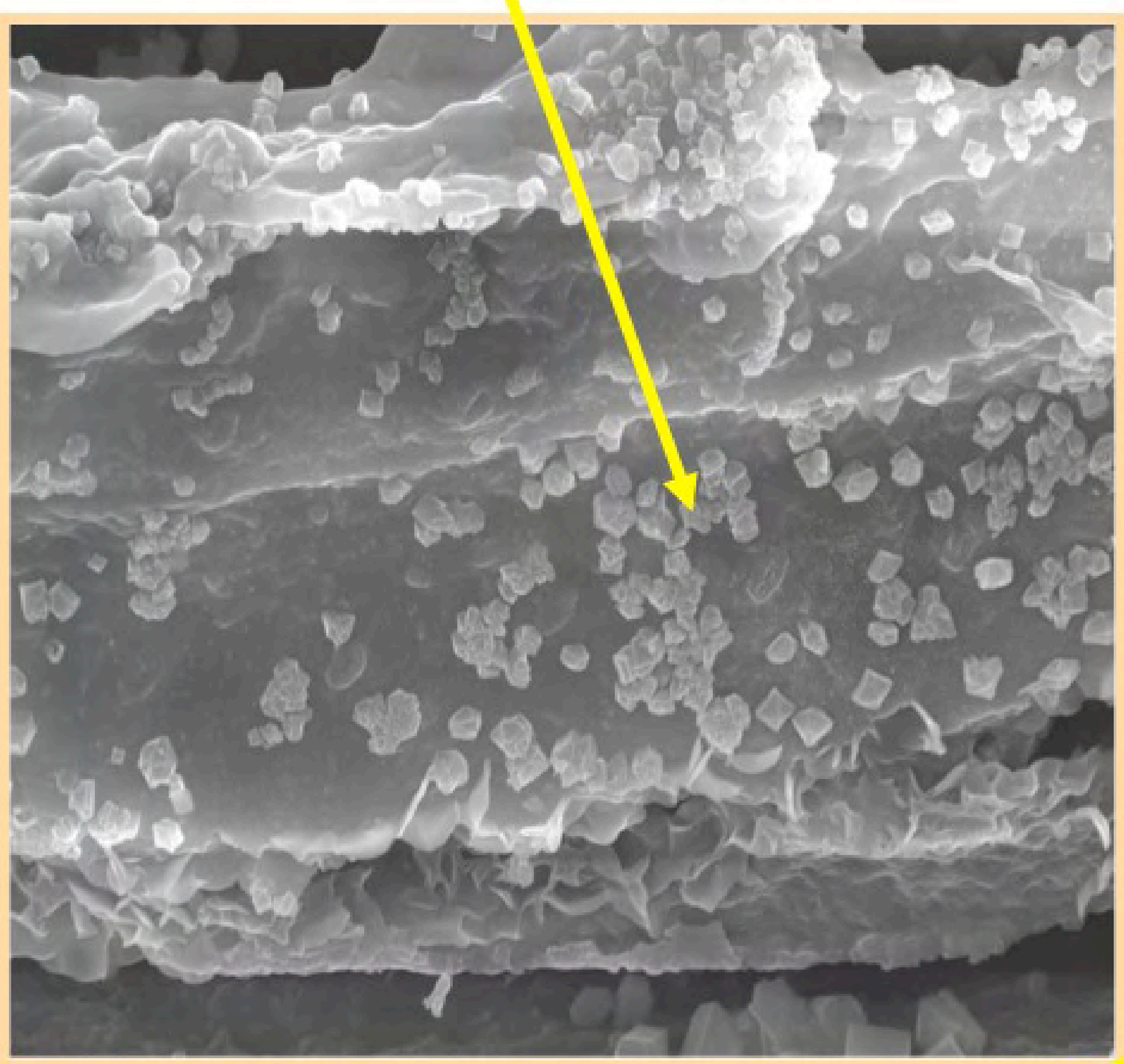
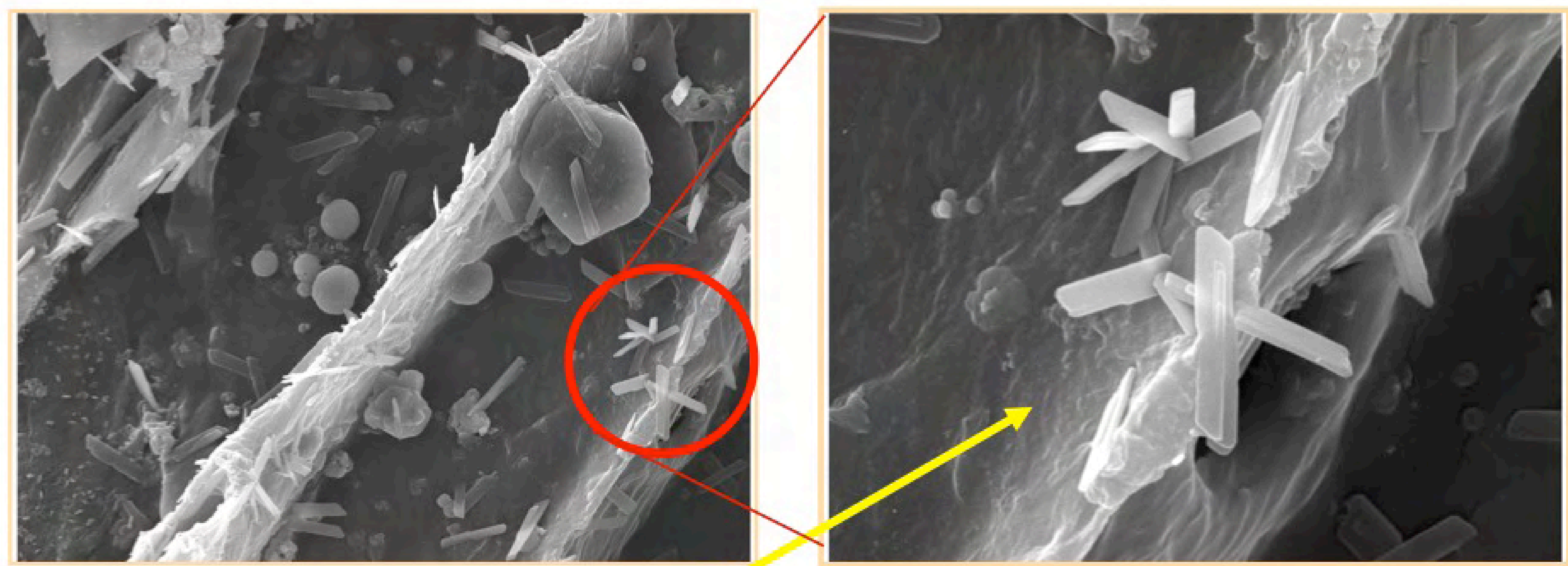
Wood deacidification treatment with alkaline nanoparticles

$Ca(OH)_2$

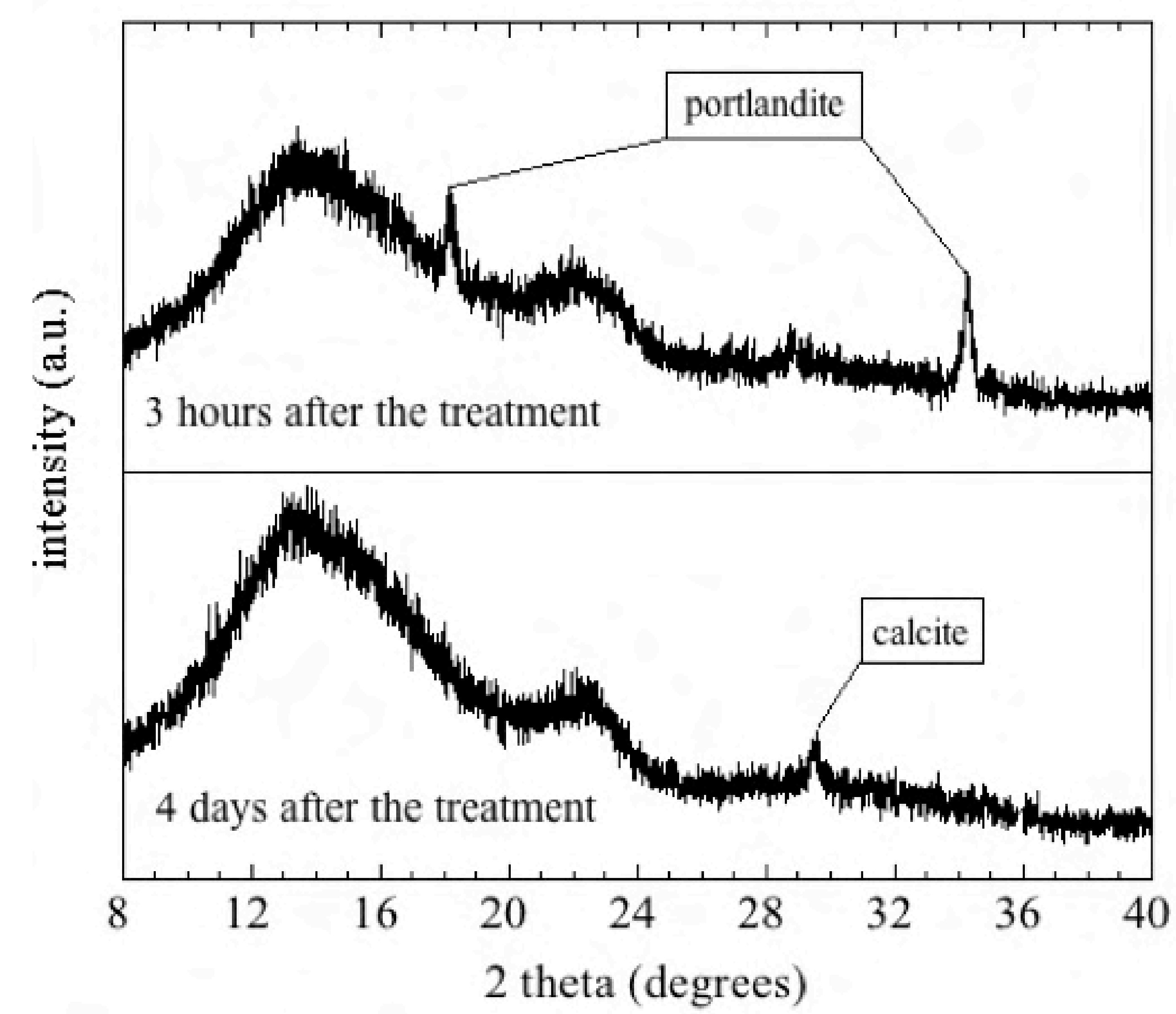




20μm

$CaSO_4 \cdot 2H_2O$

CARBONATE pH BUFFER formation by the excess of $Ca(OH)_2$



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RESTORING THE CONSERVED

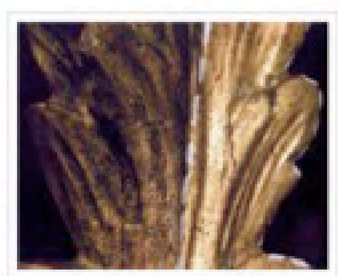
Research Highlights

Nature Nanotechnology
Published online: 30 October 2009 | doi:10.1038/nnano.2009.350
Subject Category: Nanomaterials

Nanocontainers: Clean and spotless art

Ai Lin Chun

Oil-in-water microemulsions embedded in a polymer network can remove aged protective coatings on works of art.



© 2009 Wiley

Stable nanodroplets form in microemulsions when two immiscible phases such as oil and water are mixed together in the presence of a surfactant. Many different types of microemulsions exist and they have been used in various cleaning applications. Researchers at the University of Florence now report that nanodroplets in oil-in-water emulsions when embedded in a polymer network can become effective cleaning agents for different pieces of art, including paintings and gilded surfaces.

To preserve the structure and performance of the microemulsion system, Piero Baglioni and colleagues[1] embedded a previously studied *p*-xylene-in-water microemulsion into a hydrophobic hydroxyethylcellulose polymer network with the aim of using the mesh size and viscosity of the network to control the diffusion of the nanodroplets; cleaning agents that are more viscous will penetrate less into the porous matrices of paintings. Small angle X-ray scattering and differential scanning calorimetry experiments confirmed that the structure of the embedded nanodroplets, which was essential for the cleaning process, was retained. The aged protective coating on a fifteenth-century painting and an eighteenth-century gilded frame was successfully removed using the material.

Such water-based cleaning agents offer a simple and less invasive way of cleaning works of art because these systems are optically transparent and cleaning can be visually monitored.

Reference

top

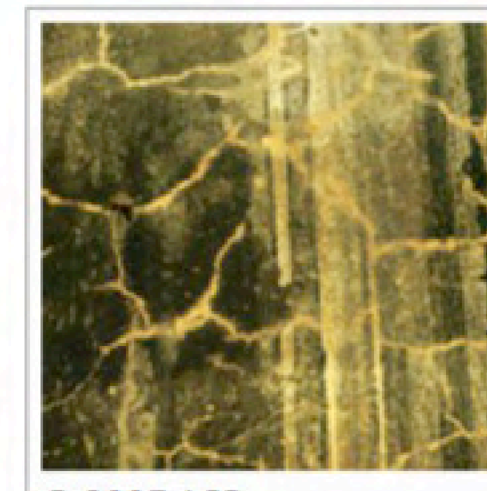
1. Carretti, E., Fratini, E., Berti, D., Dei, L. & Baglioni, P. Nanoscience for art conservation: Oil-in-water microemulsions embedded in a polymeric network for the cleaning of works of art. *Angew. Chem. Int. Ed.* doi:10.1002/anie.200904244 (2009). | Article

Nature Nanotechnology
Published online: 11 May 2007 | doi:10.1038/nnano.2007.173
Subject Category: Nanomaterials

Art restoration: Keeping it clean

Ros Portman

Unwanted materials can be cleaned from the surfaces of works of art without disturbing the original painting by using oil-in-water microemulsions



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The appearance of works of art can be disrupted by unwanted substances being deposited on their surfaces. Restoring them to their original state requires a delicate cleaning process that removes the coatings without disturbing the underlying materials. Organic solvents are efficient removal agents, but are often very invasive. However, when these solvents form nanodroplets in an oil-in-water microemulsion they have proved to be effective without any detrimental side effects.

Piero Baglioni and colleagues[1] at the University of Florence, Italy have used this approach to clean a specific type of wall fresco that has very fragile and detachable paint. A pure organic solvent would penetrate the porous structure of the painting and disrupt the original paint layers. But by using a microemulsion, a much lower volume of oil can efficiently solubilize the coatings because the nanodroplets have such a high surface area, making this method a lot safer. Furthermore, they have also been effective in removing a particularly insoluble coating found on many paintings, containing salts as well as polymers. Both components can be removed in a single step — the polymers are dissolved by the oil nanodroplets and the salts are dissolved by the water.

The technique has been successfully tested on two works of art, proving its potential as a low-impact cleaning tool in cultural heritage conservation.

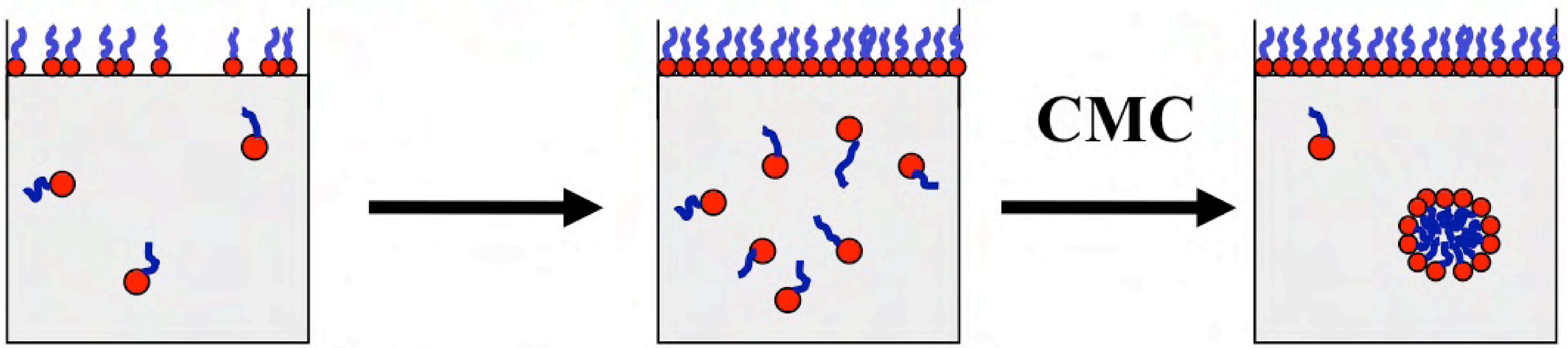
REFERENCES

top

1. Caretti, E., Giorgi, R., Berti, D. & Baglioni, P. Oil-in-water nanocontainers as low environmental impact cleaning tools for works of art: two case studies. *Langmuir* doi: 10.1021/la700487s (2007). | Article |

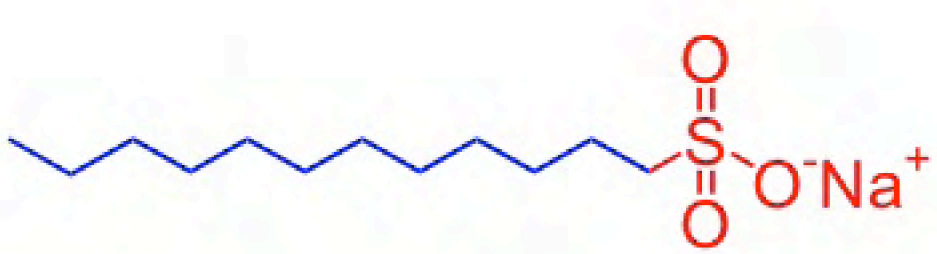
New strategies for cleaning artworks

Self-assembly properties of surfactants



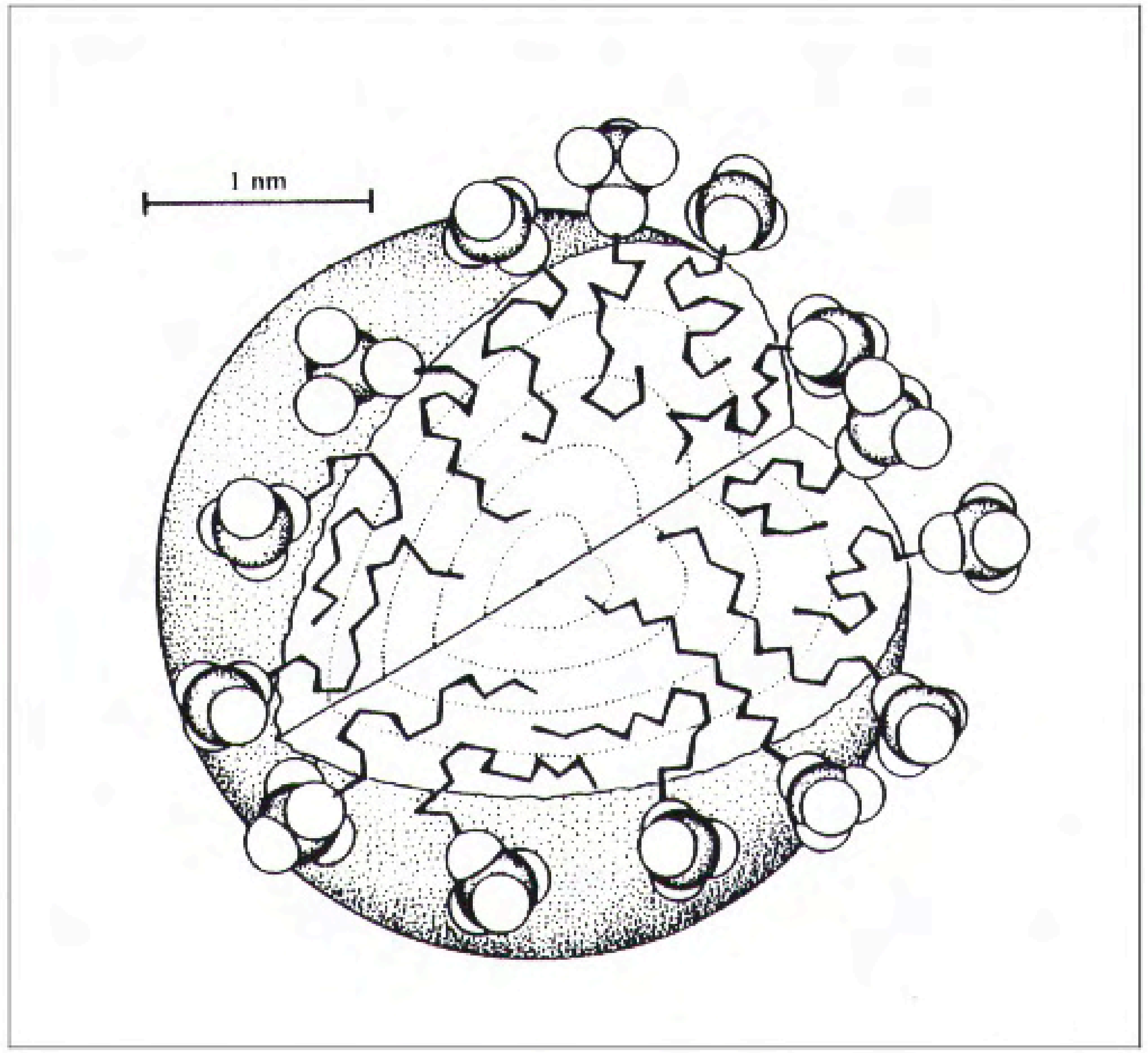
Normal micelles

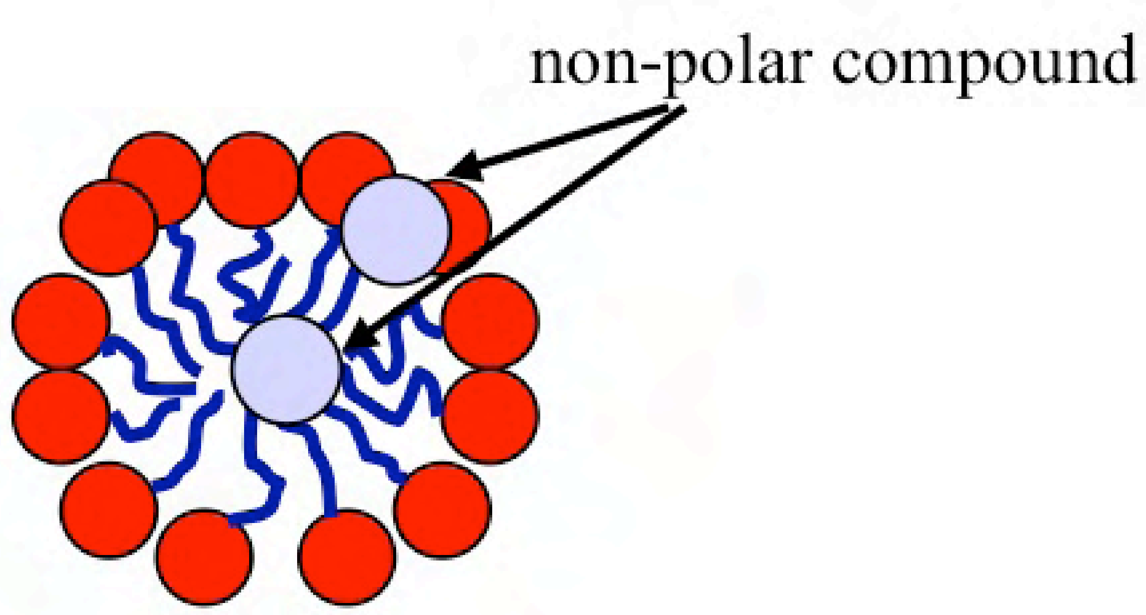
Amphiphilic surfactants contain a non-polar portion and a polar portion



Sodium dodecylsulfate (SDS)

Polyoxyethylene(4) lauryl ether (Brij 30)





Solubilization sites

Spontaneous transfer of a compound insoluble in the bulk solvent into solution due to incorporation into the surfactant micelles

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materials update

Restoring the conserved

Micellar systems remove damaging polymer layers from works of art

21 August 2003

Jane Morris

Researchers in Italy[1] 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 frescoe 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.

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

Removal of degraded polymers by using nanocontainer

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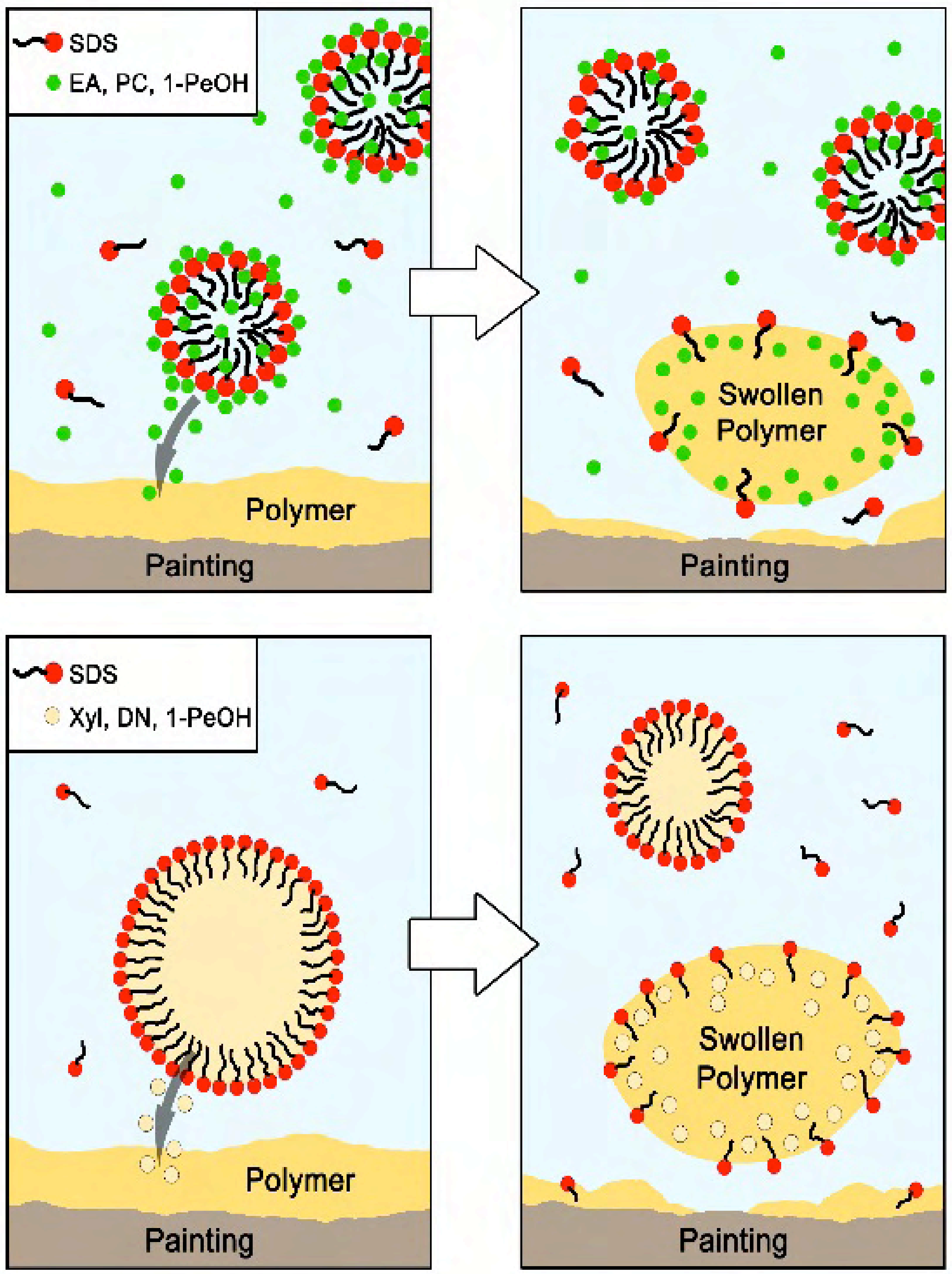
Micelles



Microemulsion

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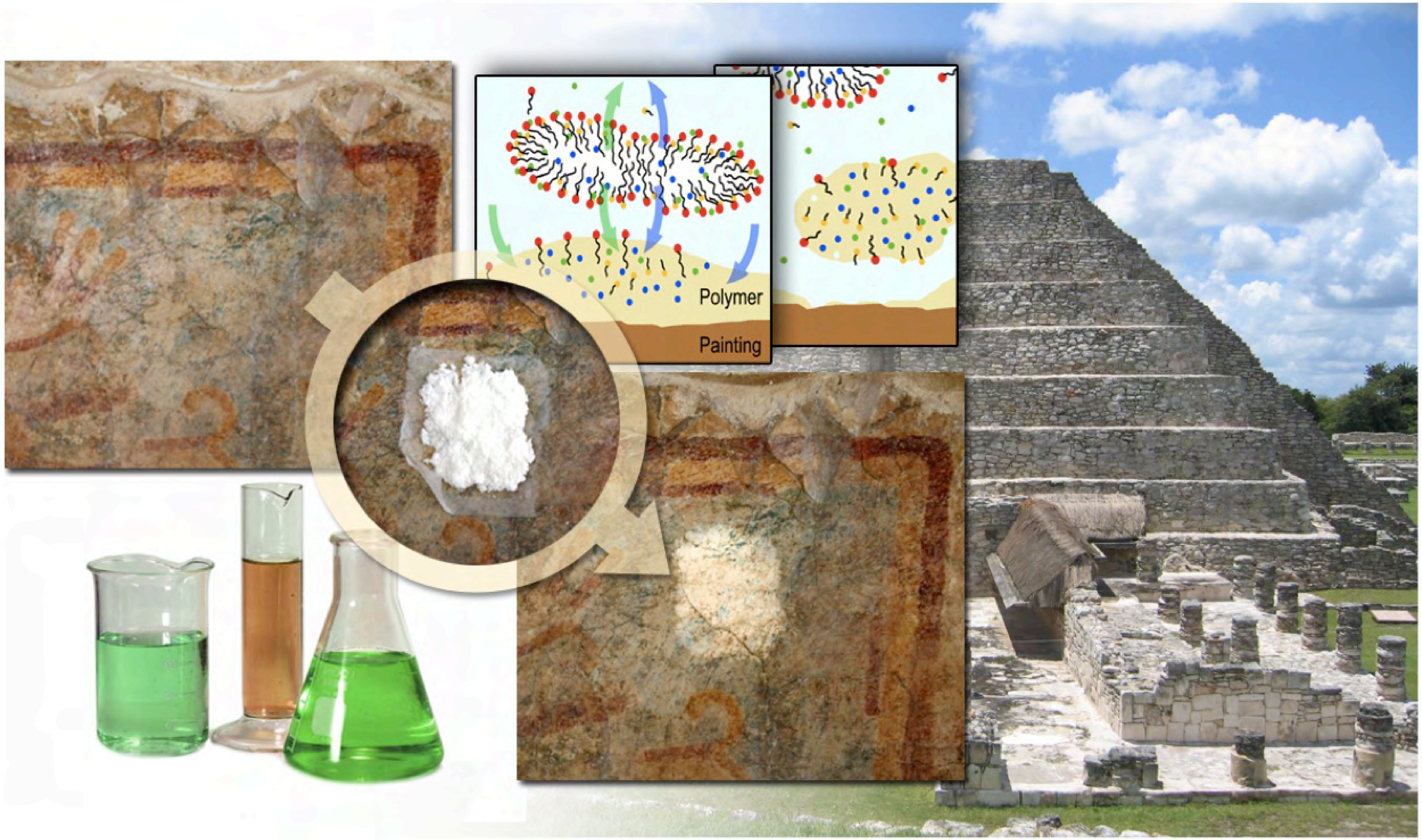

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CLEANING WITH GELS

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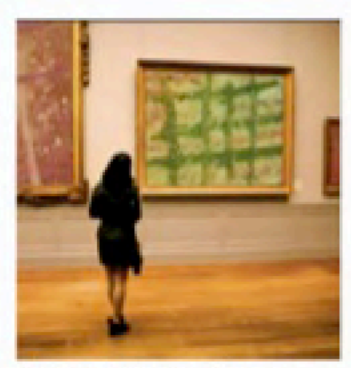
Published online: 3 September 2007; | doi:10.1038/news070903-1

Magnets harnessed to clean artwork

Sponges filled with iron nanoparticles make lifting dirt easy.

Daniel Cressey

When a priceless work of art becomes dirty, running it through the dishwasher obviously isn't an option. But even the most sensitive of modern cleaning techniques come with problems — which one researcher says he can solve with a gel sponge and a magnet.



Clean me: getting dirt off artwork can be a sticky task.

Punchstock

Many current conservation techniques use specially designed gels that can be placed on isolated dirty spots of a painting. In this way, specific cleaners can be applied to a small area of an artwork without the cleansing gel running or removing the paint along with the dirt.

But getting the sticky gel off at the end can be tricky. Residue of gel and cleaner can remain both on and under the surface being cleaned, marring the art. And using any kind of abrasive removal technique on a delicate painting comes with its own problems.

Piero Baglioni, a chemist at the University of Florence, Italy, says he has found a way around these problems — by creating a gel that can be removed with a magnet.

"It will replace the old method because it's easier," Baglioni says.

Baglioni and his colleagues detail their production of a gel sponge containing magnetic nanoparticles in the latest issue of the journal *Langmuir*[1].

Sponge bath

Baglioni's gel consists mainly of a polymer (polyethylene glycol and acrylamide) impregnated with iron nanoparticles. This gel is firm enough to be cut with scissors into 'sponges' of specific shapes required for cleaning. Such sponges can be loaded with a wide array of cleaning materials, as required for oil paintings or marble sculptures for example, and applied to the parts of the artwork that need cleaning.

> **"It will replace the old method because it's easier"**
>
> Piero Baglioni

Once applied, the cleanser leaches onto the top surface of the artwork, where it should dissolve the dirt to be removed. As the upper surface of the gel dries during the cleaning process, an osmotic pressure gradient is set up within the gel that then pulls the cleaning solution back into the sponge and away from the surface of the painting or sculpture. This trick works thanks to the fact that the gel is generally more inclined to soak up water-based substances than the surfaces being treated.

Simply placing a magnet above the piece of gel then removes it from the painting's surface without damaging the art.

"To the best of our knowledge, the ... responsive chemical gel represents one of the most advanced, versatile systems for cleaning works of art, avoiding any side effects," the researchers write in their paper.

Aviva Burnstock, head of the Department of Conservation & Technology at London's Courtauld Institute of Art, notes the importance of being able to remove the sponge as well as the cleaning material that the sponge was impregnated with.

What's interesting about Baglioni's system, she says, is how his sponge can be used to treat such small areas; other gels are not necessarily so good at limiting how much of the painting is exposed to the cleaner. "It's not a bad idea," she says. "It could be useful for very delicate surfaces."

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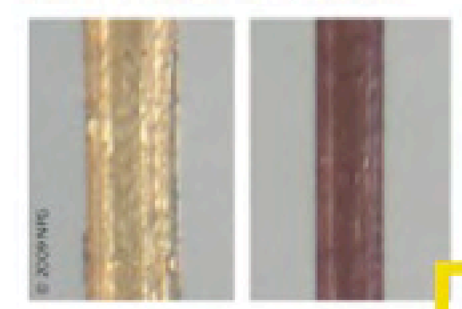
References

1. Bonini, M. *et al. Langmuir* **23**, 8681-8685 (2007).

research highlights

The pressure mounts

Nature doi:10.1038/nature07970 (2009)



Mechanical stimuli can have beneficial effects on biological materials, for example in muscle contraction, and bone and tissue growth. Reproducing this behaviour in synthetic polymers is problematic because mechanical stresses normally cause random covalent bond cleavage, destroying the material. Nancy Sottos and co-workers prevent breakdown in glassy and elastomeric polymers by incorporating selected polymer strands of a spiropyran into the bulk solid material. On application of a mechanical load, the spiropyran directs the resulting force towards a relatively small number of bonds, triggering an electrocyclic ring-opening reaction via the C–O bond. By attaching poly(methyl acrylate) or poly(methyl methacrylate) to specific points on the spiropyran, it can be used to create crosslinked bulk polymers that show mechanical transduction. The reversible covalent bond activation in the spiropyran is accompanied by a colour change, making mechanically induced changes in the bulk polymers easy to see. This should lead to materials that are less prone to damage caused by strain. Future developments based on this approach could create polymers that regenerate in response to a mechanical stress.

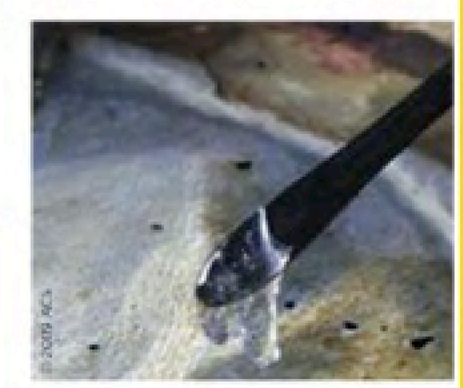
Sensitive palladium

Nano Lett. doi:10.121/nl9008474 (2009)

Fast and selective hydrogen sensors are crucial to ensure the safety and optimize the performance of electrochemical devices, such as fuel cells, powered by hydrogen. Because of their rapid response, palladium nanowires are prime candidates, but their sensing characteristics require a high operating temperature, and they have problems with mechanical failure and interference from other molecules such as water and ammonia. Reginald Penner and colleagues have now prepared nanocrystalline palladium nanowires that show unprecedented sensitivity to hydrogen, and are capable of resisting fracture on repeated exposure to hydrogen at any concentration. The nanowires were prepared by lithographical electrodeposition from a plating solution containing ethylenediaminetetraacetic acid, which results in a mean grain diameter size of up to 15 nm. The sensors showed a rapid increase in resistance in the presence of hydrogen (a maximum value was reached within two seconds at 10% concentration). The response scaled with hydrogen concentration spanning five orders of magnitude down to 2 ppm, with excellent reproducibility and baseline stability at room temperature.

Hydrogels clean up

Langmuir doi:10.1021/la804306w (2009)



Hydrogels — polymer networks with a substantial proportion of incorporated water — are known largely for their use in the biomaterials field. Emiliano Carretti and colleagues have instead found an intriguing use for them in art conservation, made possible by the use of a co-solvent to control their rheological and solubilizing properties. The researchers use poly(vinyl alcohol) crosslinked with borate, and hydrate the network with water mixed with an organic liquid such as 1-propanol; propanol was chosen as it is a common solvent used when cleaning paintings to solubilize unwanted polymeric coatings. With only small concentrations of polymer, the addition of propanol enabled strong, elastic gels to be made, with the advantages of easy control over the extent of the region treated and straightforward removal from the surface after treatment. Carretti and colleagues have made use of the gel's advantages to clean oxidized varnish from the surface of the wood panel 'Santo Stefano', painted by Ludovico Cardi ditto il Cigoli in the late sixteenth to early seventeenth century.

No need for chromium

Phys. Rev. B **79**, 165208 (2009)

Room-temperature ferromagnetism has been observed in numerous semiconductors and oxides, raising hopes for applications in spintronics. But the trouble always seems to be the same: to be useful for spintronics, the magnetization should be 'carrier-mediated' (that is, arising from the interaction of electrons and holes with localized magnetic moments) and not arising from magnetic impurities. Among the ferromagnetic oxides, chromium-doped In_2O_3 does indeed seem to show carrier-mediated ferromagnetism. Gavin Lawes and colleagues investigated the material in depth and compared it with the case of undoped In_2O_3. Surprisingly, they observed room-temperature ferromagnetism in both cases. In addition, they made use of a low-temperature phenomenon known as Andreev reflection to measure the electron polarization, and found that in both cases electrons are 50% polarized. The results show on the one hand that chromium is not necessary for ferromagnetism. On the other hand, even if the polarization measurements were only possible at low temperature (1.3 K), they provide another confirmation that ferromagnetism in In_2O_3 is carrier-mediated.

Metallic glasses in good shape

Scripta Mater. **60**, 431–434 (2009)

Metallic glasses have some remarkable properties such as a higher strength than steel, making them interesting for aeronautical applications, medical devices and sports equipment. But at the same time they can be brittle, and this has hampered their commercial development. Simon Pauly and colleagues have now developed a bulk metallic glass composite that leads to improved compressive deformability while maintaining the high yield strength of the metallic glass. Their composite is based on Cu–Zr alloys. For some composition ranges, these alloys have shape-memory effects, where the crystals show a reversible transformation from austenite to martensite phases. To support the glass formation in this system, the researchers added small fractions of aluminium, which resulted in metallic glass composites that also contain varying amounts of crystalline phases. With increasing crystalline fraction, the compressive deformability of the composites improves, a result of what is believed to be the absorption of compressive strain through the martensitic phase transformation within the crystalline phase. These improved properties may therefore lead to new applications for metallic glasses.

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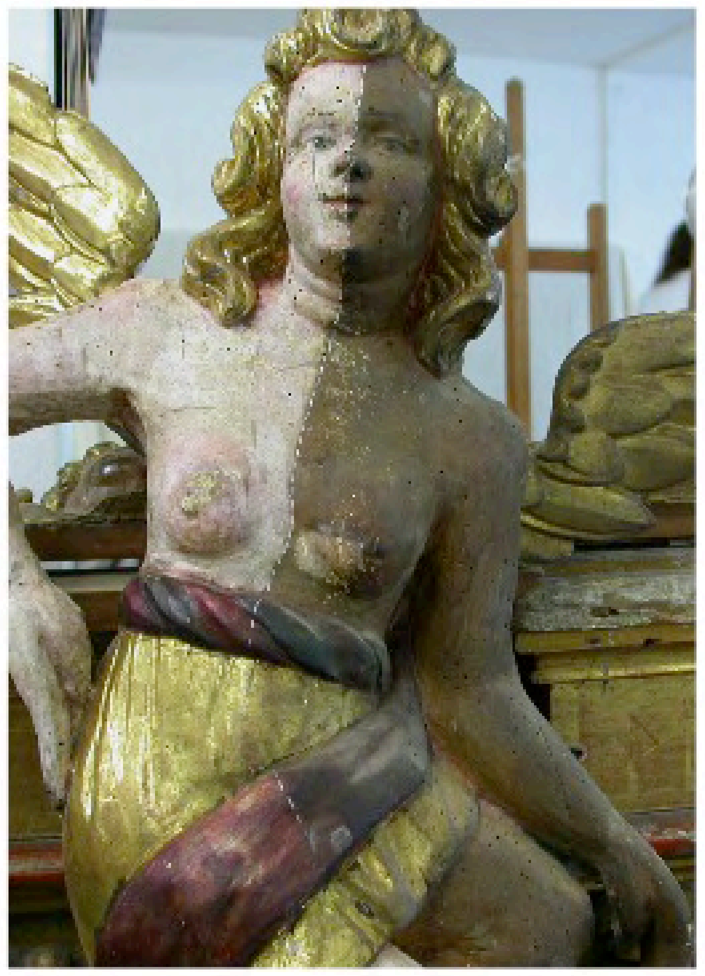
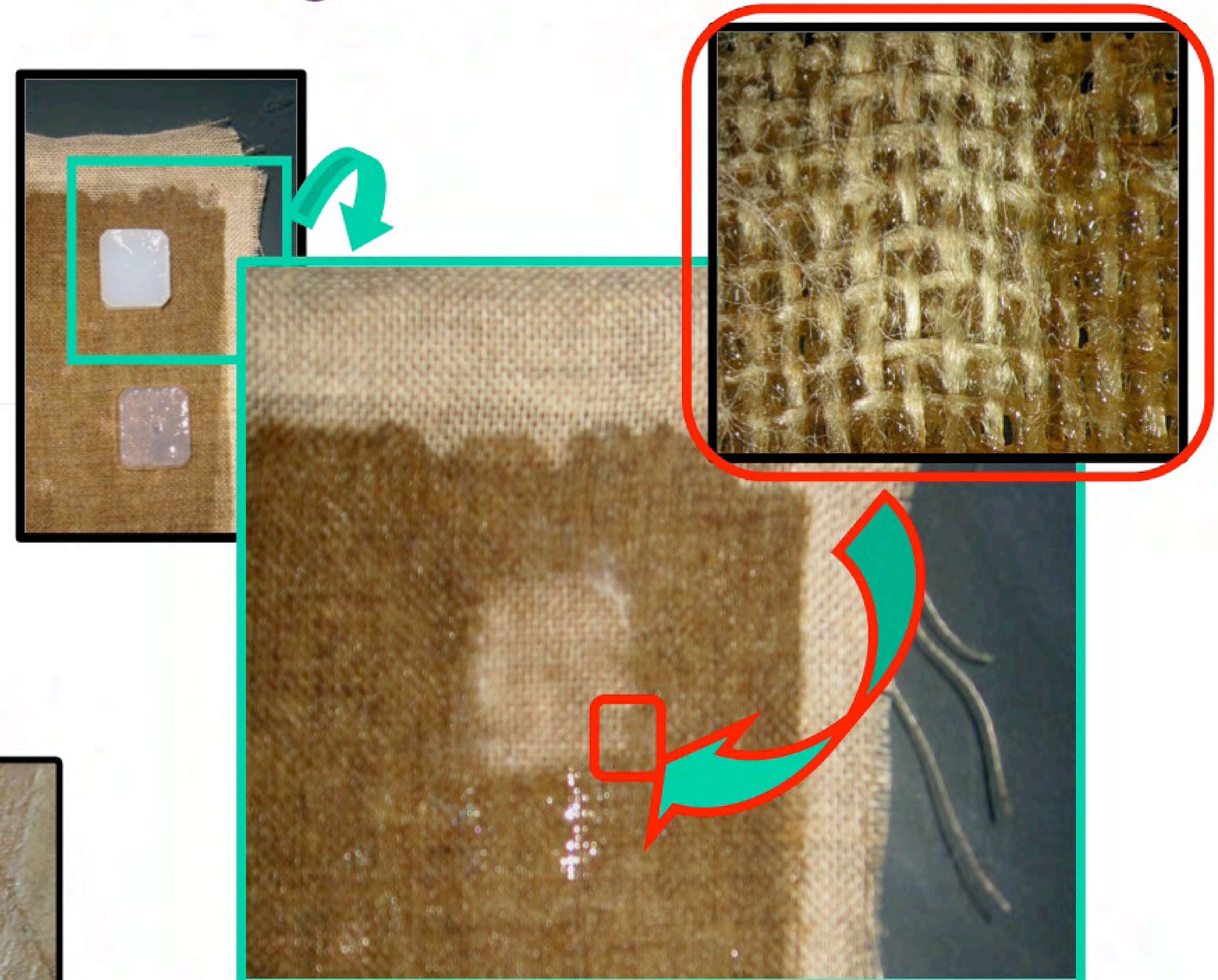


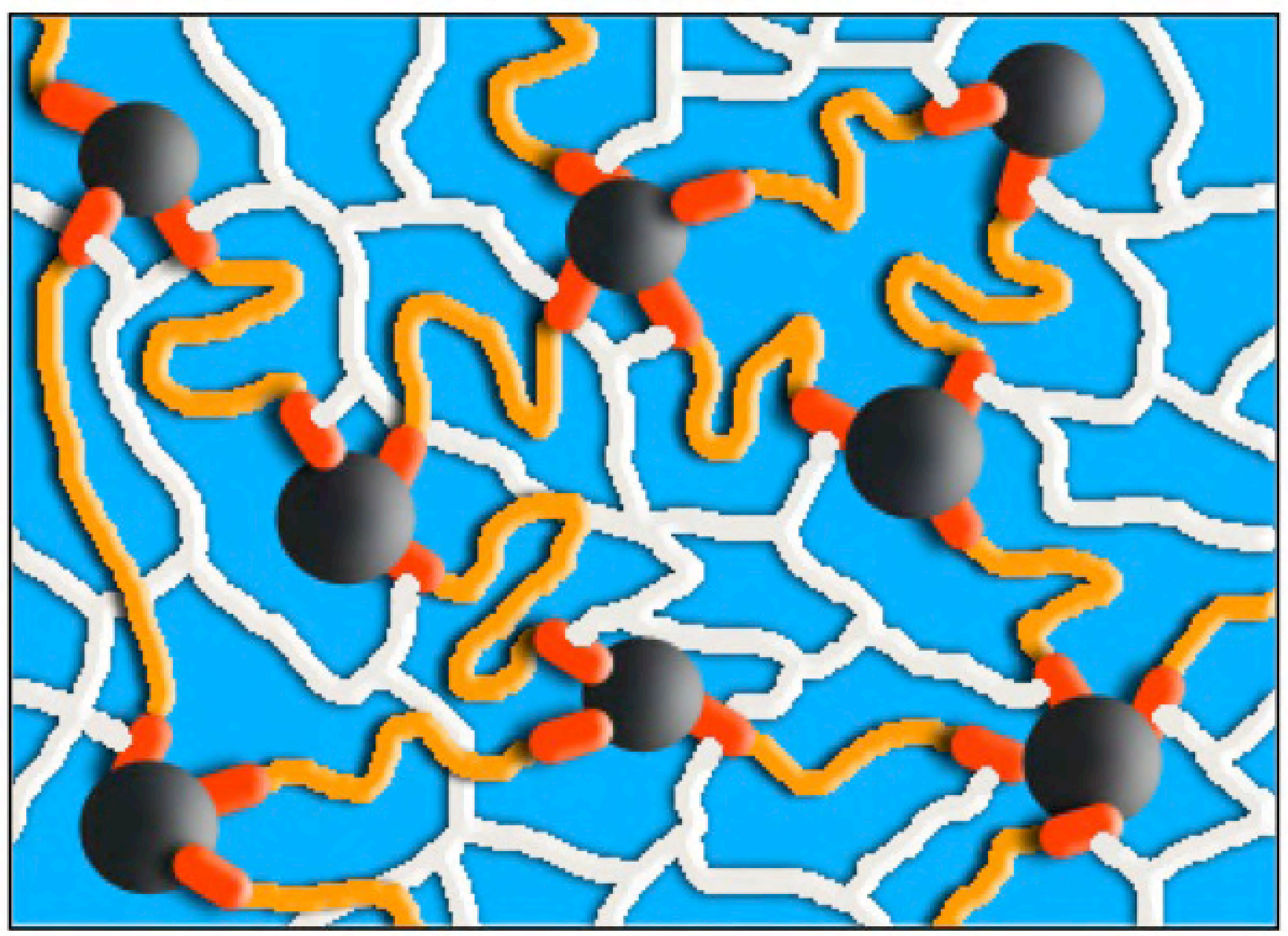
Innovative PVA/
Borax gels with
confined solvents

Cleaning tests: Mowilith DM5



Responsive chemical gels

We can include inside the polymer network some compounds that make the gel responsive to external stimulations



Magnetic nanoparticles

Crosslinker between nanoparticles and gel

Polymer gel

The magnetic nanoparticles are bound to the gel by special molecules with two functional part. In this way we obtain a magnetic chemical gel that can be completly removed by the action of a permanent magnet.

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New Nanomagenetic gels for polymer or organics removal