

Wednesday, December 11th

Session: SURFACE, INTERFACE & NANOCONFINEMENT 1

Morey – St Denis room

14h30 - 16h30

Keynote speaker: Anne-Sophie DUWEZ

Probing single molecules with AFM: Force, motion, dynamics, and function

Abstracts



Thematic Session: Surface and interface at the nanoscale

Keywords: QCM, SERS, molecular interaction, detection

Development of a QCM/SERS biosensor for the observation of molecular interactions

Médéric Lequeux^{1,2}, Nestor Quilis³, Jakub Dostalek³, Michèle Salmain⁴, Wolfgang Knoll³, Souhir Boujday², Marc Lamy de la Chapelle⁵

1. Laboratoire CSPBAT, CNRS UMR 7244, UFR-SMBH, Université Paris 13, 74 rue Marcel Cachin, 93017 Bobigny, France
2. Sorbonne Université, CNRS, UMR 7197, Laboratoire de Réactivité de Surface, F-75005 Paris, France
3. Biosensor Technologies, AIT-Austrian Institute of Technology GmbH, Muthgasse11, 1190 Vienna, Austria
4. Sorbonne Université, CNRS, Institut Parisien de Chimie Moléculaire (IPCM), F-75005 Paris, France
5. Institut des Molécules et Matériaux du Mans (IMMM - UMR CNRS 6283), Le Mans Université, Avenue Olivier Messiaen, 72085 Le Mans cedex 9, France

In order to observe the molecular interaction between a bioreceptor and its targeted analyte, we combine in a single transducer two detection methods: Quartz Microbalance (QCM) and Surface Enhanced Raman Scattering (SERS). QCM is based on the measurement of the change in resonance frequency of a quartz crystal resonator sandwiched between two gold electrodes whereas the SERS is based on the huge enhancement of the Raman signal of molecules deposited at the surface of metallic nanostructures.

To couple both techniques, we add gold nanocylinders on the gold electrode of a QCM chip. The nanocylinder grating were made by e-beam lithography with diameter of 250 nm and a periodicity of 400 nm. Gold nanostructures deposited on gold thin film exhibit specific optical properties with the observation of specific plasmon modes and it has already been demonstrated that the SERS signal is higher in this configuration compared to the one recorded for gold nanostructures on dielectric substrate.¹

Thanks to such transducer, we were able to perform simultaneously QCM and SERS measurements on the same molecules.

We provided the proof of concept of such coupling using Streptomycin, an antibiotic used to treat some bacterial infections. In this case, our bioreceptor is an aptamer which could recognize streptomycin with good affinity and selectivity.² We were then able to detect and quantify the streptomycine chemisorbed on the nanostructured chip using the QCM signal and to observe the streptomycine/aptamer interaction through the SERS spectrum.

The authors acknowledge the International ANR project Nanobiosensor (ANR-15-CE29-0026) for financial support.

¹ R. Gillibert, et al., Nanotechnology, 27/11, 115202, 2016

² Soheili, V., et al. Microchim Acta, 183, 1687, 2016

Thematic Session: Surface and interface at the nanoscale

Keywords: IR spectroscopy, AFM imaging, 3D tomography, cells, polymers

UA- AFM-IR platform for chemical and subsurface element density analysis

E. Lesniewska¹, E. Bourillot¹, S. Oliveri¹, N. Pocholle¹, O. Pietrement¹, M.J. Virolle², A. Deniset-Besseau³, A. Dazzi³

1. ICB UMR CNRS 6303, Université Bourgogne Franche-Comté, Dijon, France
2. I2BC UMR 9198, Université Paris-Sud, Orsay, France.
3. LCP UMR CNRS 8000, Université Paris-Sud, Orsay, France

A new AFM-based platform combining IR spectroscopy¹ and ultrasound acoustic 3D tomography² was developed to detect specific bodies inside organic or inorganic elements. To illustrate the potential of combining these imaging and high-resolution spectroscopy techniques (Fig. 1), we will present the detection and size distribution (accuracy under 10 nm) of triglycerides vesicles in *Streptomyces* using high-resolution infrared microscopy and chemical identification. Acoustic mode UA-AFM³ has been used to detect change in density of lipidic vesicles present in the bacteria *Streptomyces*. Similar analysis will be carried out with oleaginous (*Yarrowia lipolytica*) and non-oleaginous yeasts (*Saccharomyces cerevisiae* yeasts) as well as with buccal cells⁴ in order to demonstrate the great potential of acoustic and microwave microscopy (Fig. 2) More classical samples have been studied such as co-polymers, pharmaceutical powders. We have achieved a comparative study of AFM-IR and acoustic analysis. Our results indicate that the coupling of these techniques constitutes a great advantage to fully characterize chemical, topographical and volumetric parameters of biological sample or inorganic sample at the surface and sub-surface with nanometric resolution. This work has been supported by the French National Research Agency: ANR-17-EURE-0002, ANR-15-IDEX-03 PIA2/iSite-BFC, and ANR-15-CE09-0002-02.

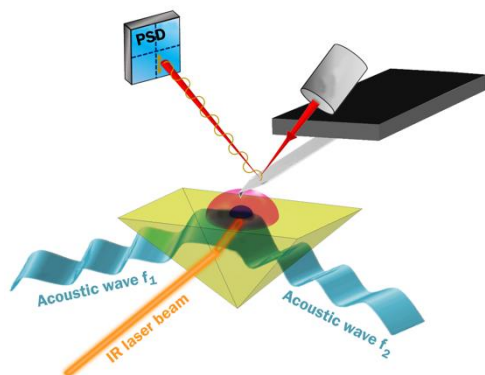


Fig. 1 - MS-AFM-IR platform

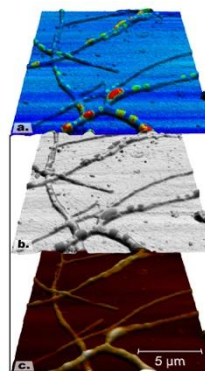


Fig. 2 – From top to bottom: AFM-IR at 1740 cm^{-1} UA-AFM at 640kHz

References:

1. A. Dazzi et al., *Applied spectroscopy* (2012) **66** (12), 1365.
2. P. Vitry et al., *Nano Research* (2015) **8**(7), 2199.
3. P. Vitry et al., *Nano Research* (2016) **9**(6), 1674.



Thematic Session: Surface and Interface at the Nanoscale

Keywords: cryogenic XPS, electric double layer, interface composition, low salinity effect

Probing the mineral / fluid interface using cryogenic XPS – a look into the electric double layer

Nicolas Bovet¹, Stanislav Jelavic², Susan Stipp¹

1. Technical University of Denmark (DTU), Denmark
2. University of Copenhagen, Denmark

One of the main drawback of X-ray photoelectron spectroscopy (XPS) is the requirement of ultrahigh vacuum which is often far from realistic conditions. The study of the interface between surfaces and liquids is thus challenging, especially when analyzing the interaction between rocks and fluids where the composition of the interface is crucial for a better understanding of natural environments.

We have developed a cryogenic XPS technique, initiated by Shchukarev *et al.*, to provide information of the mineral – fluid interface. Our samples are vitrified and thus, the ion concentration in the near surface fluid, i.e. the portion of the solution that includes the electric double layer, is preserved. The analysis depth of XPS (10 nm) means that it can probe the ion concentrations in the fluid that was at equilibrium with the mineral surface at the instant it solidified.

Using cryogenic XPS, we have studied the surface composition of natural clays (kaolinite, illite and chlorite) during contact with solutions of various salinities, containing Ca^{2+} , Mg^{2+} and Na^+ as well as small organic molecules. The results demonstrate with direct spectroscopic evidence that the relative ion concentration near the surface is different than in the bulk, consistent with electric double layer theory. Of particular interest is that at the lowest bulk liquid concentration, the ratio negative charges / cations is decreasing, meaning the surface is losing its negative charges. The ability of cryogenic XPS to probe the electric double layer at the mineral – fluid interface can provides insight into processes governing natural systems.



Thematic Session: (Nanoconfined liquids & gases)

Keywords: Monte-Carlo simulation, gaz hydrates, mixture selectivity, ideal adsorption solution theory

Guest Trapping and Selectivity within mixed Clathrate Hydrates: a Grand Canonical Monte Carlo Study coupled with thermodynamic modelling

A.Patt^{1,2}, J._M Simon¹, J.M. Salazar¹, S. Picaud²

1. Laboratoire Interdisciplinaire Carnot de Bourgogne UMR 6303, 9 Av. Alain Savary, F-21078 Dijon, France
2. Institut UTINAM UMR 6213, 41 bis Av. de l'Observatoire, 25010 Besançon, France

Abstract

Naturally occurring clathrate hydrates are at the heart of important environmental concerns and are also subjects and/or means of study for astrophysicists (O. Mousis et al.; Faraday Discuss. 147 (2010) 509). Among the clathrate hydrates of interest for extraterrestrial environments are the N₂, CO, and mixed N₂-CO hydrates (O. Mousis et al.; Astrophys. J. 691 (2009) 1780). A better understanding of the trapping capabilities of those hydrates can help providing constraints on the chemical abundances of astrophysical environments of planetesimals and planetary atmospheres.

To that end, Grand Canonical Monte Carlo (GCMC) simulations were used. In the case of the mixed hydrate, we report a significant selectivity towards CO, in agreement with experimental work (C. Pétuya, Ph.D. thesis, 2017), especially at low temperatures. A two-site adsorption behavior is evidenced for structure II hydrates considered in this work: the small cages being more likely filled than the large ones. Additionally, we show that the Ideal Adsorbed Solution Theory (IAST) gives results which are in excellent agreement with those of our binary GCMC simulations (A. Patt et al.; J. Phys. Chem. C 122 (2018) 18432). The influence of the gas phase composition on the molecular selectivity is highlighted from both GCMC and IAST calculations, for which we obtained qualitative agreements with experiments.



Thematic Session: Confined liquids and gases

Keywords: confinement, binary liquids, nanostructure, porous organosilicates

TOLUENE/BUTANOL BINARY SOLVENTS CONFINED IN PERIODIC ORGANOSILICATES

Aïcha JANI¹, Benedikt MIETNER², Viviana CRISTIGLIO⁴, Jean Mark ZANOTTI⁵, Patrick HUBER³, Michael FRÖBA², Denis MORINEAU¹

1. Institute of Physics of Rennes (IPR), CNRS-University of Rennes 1, France
2. Department of Chemistry, University of Hamburg (UHH), Germany
3. Institute of Materials Physics and Technology, Hamburg University of Technology (TUHH), Germany
4. Institut Max von Laue - Paul Langevin (ILL), Grenoble, France
5. Laboratoire Léon Brillouin (LLB), Saclay, France

Abstract:

The confinement of liquid mixtures in porous channels provides new insight into fluid ordering at the nanoscale. In this presentation, we address a phenomenon of microphase separation, which appears as a novel fascinating confinement effect for fully miscible binary liquids. This phenomenon was firstly investigated for tert-butanol-toluene mixtures confined in the straight and mono-dispersed cylindrical nanochannels of MCM-41 and SBA-15 mesoporous silicates ($D = 3.6$ nm and 8.3 nm) [1,2].

The present study aims at extending this approach to different fluid-wall interactions. It shows that novel types of nanostructures can be generated using functionalized porous organosilicates with periodic alternating surface chemistry along the pore channel.

[1] 'More room for microphase separation: An extended study on binary liquids confined in SBA-15 cylindrical pores', R. Mhanna, A.R. Abdel Hamid, S. Dutta, R. Lefort, L. Noirez, B. Frick, D. Morineau, *Journal of Chemical Physics*, 146, 024501 (2017) <https://doi.org/10.1063/1.4972126>

[2] 'Microphase Separation of Binary Liquids Confined in Cylindrical Pores', A.R. Abdel Hamid, R. Mhanna, R. Lefort, A. Ghoufi, C. Alba-Simionesco, B. Frick, D. Morineau, *Journal of Physical Chemistry C*, 120, 9245-9252 (2016) <https://doi.org/10.1021/acs.jpcc.6b01446>

Thematic Session: Nanoconfined liquids and gases

Keywords: Brownian ratchet, Nanopores, Translocation, DNA, Nuclear Pore Complex

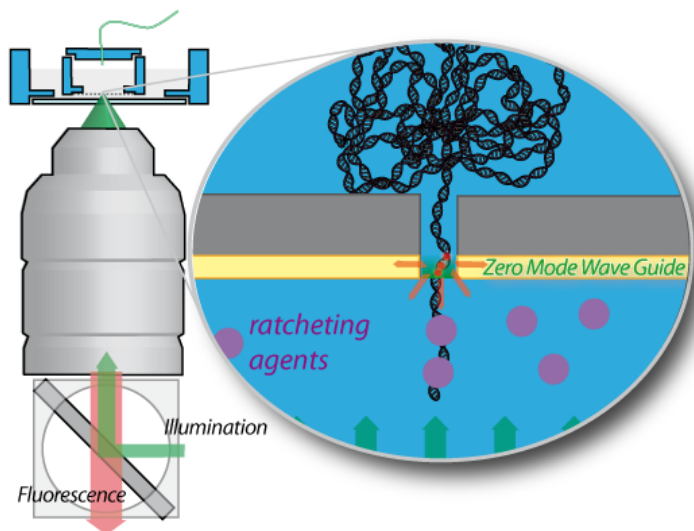
Directional transport of biomolecules through nanopores: an experimental approach of a nanoscale Brownian ratchet

Bastien Molcrette¹, Fabien Montel¹

1. UMR 5672 – Laboratoire de Physique, ENS de Lyon

Abstract : Some natural systems known as Brownian ratchets use thermal fluctuations to extract mechanical power. In these systems, diffusion is biased in order to achieve unidirectional biological processes. Good examples are the molecular motors as kinesins or myosins that walk on the cytoskeleton and use ATP to bias their movement. Another case is the transport of mRNA between the nucleus and the cytoplasm through Nuclear Pore Complexes (NPC). The NPC is a large molecular complex which selects molecules that enter or exit the nucleus. It has been shown to be highly selective and directional.

To study the directional transport of biomolecules through the NPC, a simplified mimetic device based on nanoporous membranes was designed. Our approach used near-field microscopy technique, Zero Mode Waveguide for Nanopores. With this method single DNA molecules translocation through nanopores was observed in real time. Ratchet agents which are polycations that bind strongly to the DNA and cannot diffuse through the pore were added on the exit side of the membrane. We quantified the effect of the ratchet agent on the translocation frequency of DNA molecules. An experimental measurement of the free energy gain associated with the Brownian ratchet was extracted and compared with coarse grained modeling.



Thursday, December 12th

Session: SURFACE, INTERFACE & NANOCONFINEMENT 2

Morey-St Denis room

14h00 – 16h30

Keynote speaker: Denis MORINEAU

Fluids in nanoporous confinement: how are they different?

Abstracts



Thematic Session: Nanoconfined liquids & gases

Keywords: mechanical energy storage, high pressure intrusion, zeolites, mesoporous silica, ZIFs

High-pressure intrusion of electrolyte aqueous solutions in hydrophobic micro- and mesoporous solids for mechanical energy absorption and storage

Andrey Ryzhikov^{1,2}, Bénédicte Lebeau^{1,2}, Claire Marichal^{1,2}, Gérald Chaplais^{1,2}, Habiba Nouali^{1,2}, T. Jean Daou^{1,2}

1. Université de Haute-Alsace (UHA), CNRS, Institut de Science des Matériaux de Mulhouse (IS2M), Axe Matériaux à Porosité Contrôlée (MPC), UMR 7361, Mulhouse, France
2. Université de Strasbourg, 67000 Strasbourg, France

Heterogeneous lyophobic systems, which consist of a lyophobic porous matrix and a nonwetting liquid, are one of the promising technologies for absorption and storage of mechanical energy. Mainly, they are based on high-pressure intrusion of water in hydrophobic porous solids such as pure silica zeolites (zeosils), fonctionnalized mesoporous silicas and Zeolitic Imidazolate Frameworks (ZIFs). Depending on the nature of porous material, the systems, when the pressure is released (extrusion), are able to restore, dissipate or absorb the supplied mechanical energy during the compression step (intrusion) and therefore to display a spring, shock-absorber or bumper behavior. Recently, it has been found that the use of aqueous salt solutions is a promising way to improve the energetic performance of such systems by an increase of the intrusion pressure. This increase is particularly pronounced for highly concentrated solutions and depends on the salt nature and its concentration as well as the type and the structure of porous material and its pore size. In the case of hydrophobic mesoporous silicas, the increase is relatively weak and seems to be determined by a higher surface tension and a lower wettability of the solutions according to Laplace-Washburn equation. In the case of zeosils and ZIFs, the pressure rises much stronger and this rise is mainly related to the desolvation of solvated ions under forced penetration into the micropores. It has been also observed that the use of highly concentrated salt solutions can also change the behavior of the systems.



Thematic Session: (Nanoconfined liquids & gases, Surface & interface at the nanoscale)

Keywords: nanothermodynamics properties, molecular simulations, water at the nanoscale, Kirkwood-buff integrals

Nanothermodynamics from theory to applications.

J._M Simon¹

1. Laboratoire Interdisciplinaire Carnot de Bourgogne UMR 6303, 9 Av. Alain Savary, F-21078 Dijon, France

Abstract

Small systems (nano size) cannot be described by classical thermodynamics. One reason is that the surface area becomes important, in contrast to the case for systems in the thermodynamic limit. The increased experimental interest in nanosized systems, has created however a demand on the theoretical side.

The thermodynamics of small systems (also called nanothermodynamics) that was first developed by T. L. Hill in the sixties gives a general background to systematically express thermodynamic properties in terms of system size [Schnell et al, Mol. Phys. 110, 1069 (2012)]. It was tested using molecular simulations (Monte-Carlo and Molecular dynamics) on different system types: Lennard-Jones mixtures, ternary molecular systems, reactive mixtures ($2H=H_2$), CO adsorbed on graphite, ...

The results validate the use of the nanothermodynamics for molecular systems and clearly show that they are consistent both with the Kirkwood-Buff approach and Gibbs' thermodynamics for surfaces.

The new approach will be illustrated through different examples and in particular on nanoscale volume of water [Strom et al, PCCP 19, 9016 (2017)]. Beyond the application to molecular simulated data, this new approach opens new ways to study systems where the individual particle sizes are large compared to the system size.

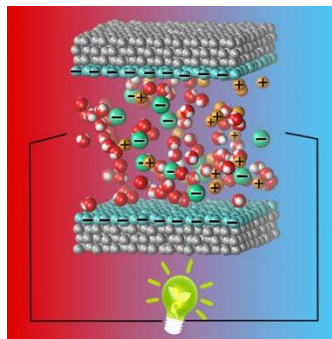
Thematic Session: Nanoconfined liquids & gases

Keywords: nanofluidics, thermoelectricity, modeling, molecular dynamics

Giant thermoelectric response of nanofluidic systems driven by water excess enthalpy

Li Fu¹, Laurent Joly¹, Samy Merabia¹

1. Univ Lyon, Univ Claude Bernard Lyon 1, CNRS, Institut Lumi`ere Mati`ere, F-69622 Villeurbanne, France



Nanofluidic systems could in principle be used to produce electricity from waste heat, but current theoretical descriptions predict a rather poor performance as compared to thermoelectric solid materials. I will present a recent work where we investigated the thermoelectric response of NaCl and NaI solutions confined between charged walls, using molecular dynamics simulations. We computed a giant thermoelectric response, two orders of magnitude larger than the predictions of standard models. We showed that water excess enthalpy – neglected in the standard picture – plays a dominant role in combination with the electroosmotic mobility of the liquid-solid interface. Accordingly, the thermoelectric response can be boosted using surfaces with large hydrodynamic slip. Overall, the heat harvesting performance of the model systems considered is comparable to that of the best thermoelectric materials, and the fundamental insight provided by molecular dynamics suggests guidelines to further optimize the performance, opening the way to recycle waste heat using nanofluidic devices.



Thematic Session: (Surface & Interface at the nanoscale)

Keywords: (hybrid interface, epitaxial molecular layer, dynamic ordering, interface thermodynamics)

Dynamic and heterogeneous ordering of perylene layer on Ag(110)

Laurent Guillemot¹, Nataliya Kalashnyk¹, Kirill Bobrov¹

1. ISMO (Institut des Sciences Moléculaires d'Orsay), CNRS/Université Paris-Saclay, Bât. 520, 91405 Orsay

We present a room temperature STM study of dynamics of individual flat lying perylene molecules shaping diffusive monolayers on Ag(110) driven by substrate site recognition. Thermal diffusion, balancing the intermolecular interaction and the site recognition, give rise to remarkable properties of the molecular layer. The balance precludes molecular network formation and favours unidirectional string-like molecule motion. The site recognition induces dynamic heterogeneity and separates the diffusing molecules spatially into solid-like and liquid-like metabasins exchanging via the string-like motion. Analysis of the spatial frequencies indicates that the force field of the naturally ordered Ag(110) substrate, modified by the intermolecular interaction, arranges both metabasins into a single long-range ordered thermodynamically stable $(\sqrt{3}\sqrt{3})$ network. The monolayer persists the permanent substrate restructuring driven by silver adatom diffusion and is able to consistently spread over mono- and bi-atomic surface steps. Then, this dynamic heterogeneity bestows to the monolayer dynamics, characteristic to a liquid, and long-range order, characteristic to a crystalline solid. We anticipate that this duality, unattainable under conventional nanofabrication, opens perspectives to fabricate epitaxial self-assembled nanostructures of arbitrary lateral shape.

Thematic Session: Surface and interface at the nanoscale

Keywords: STM, molecule, IETS, Exchange coupling, Spin polarized transport.

Atomic-scale spin sensing with a single-molecule at the apex of a scanning tunneling microscope

B. Verlhac¹, N. Bachellier¹, L. Garnier¹, M. Ormaza¹, P. Abufager², R. Robles³, M.-L. Bocquet⁴, M. Ternes^{5,6}, N. Lorente^{3,7}, L. Limot¹.

1. Université de Strasbourg, CNRS, IPCMS, UMR 7504, F-67000 Strasbourg, France
2. Instituto de Fisica de Rosario, CONICET and Universidad Nacional de Rosario, Av. Pellegrini 250 (2000) Rosario, Argentina
3. Centro de Fisica de Materiales CFM/MPC (CSIC-UPV/EHU), 20018 Donostia-San Sebastian, Spain.
4. PASTEUR, Département de Chimie, Ecole Normale Supérieure, PSL Research University, Sorbonne Universités, UPMC Univ. Paris 06, CNRS, 75005 Paris, France
5. Institute of Physics II B, RWTH Aachen University, 52074 Aachen, Germany
6. Peter Grunberg Institut (PGI-3), Forschungszentrum Jülich, 52425 Jülich, Germany
7. Donostia International Physics Center (DIPC), 20018 Donostia-San Sebastian, Spain

The field of surface magnetism has undergone major developments in recent years thanks to scanning tunneling microscopy (STM). As a new path to complete these developments, the study presented here, focuses on how to give quantum-based magnetic properties to the STM tip, in order to give it a sensibility to both exchange coupling with the surface and spin-polarized transport. This is done by attaching to the STM tip apex a molecule, the nickelocene [NiCp₂], which preserves its spin 1 character and its uniaxial magnetic anisotropy that is detected through inelastic spin excitation in the molecular conductance. The functionalized tip is then used to probe the magnetism of an on-surface object such as an adatom adsorbed on Cu(100) and a ferromagnetic surface. Due to the nature of the spin excitations of the molecule, we are able to determine the strength of the exchange coupling between the on-surface object and the tip. It has been done by varying with a distance with a precision (<10pm) such as atomic and subsurface resolutions are reached. This coupling allows also to discriminate the different contributions related to the spin populations in the transport through the tunneling junction.

Thematic Session: Surface and interface at the nanoscale

Keywords: Thin films, Growth, sputtering, Electrical resistivity

Structural and electrical properties of nanostructured W-Ag thin films

Housseem BOUKHALFA¹, Valérie POTIN¹, Nicolas MARTIN²

¹ Laboratoire Interdisciplinaire Carnot de Bourgogne (ICB), UMR 6303 CNRS Université Bourgogne Franche-Comté, 9 Av. A. Savary, BP 47 870, F-21078 Dijon Cedex, France

² Institut FEMTO-ST, UMR 6174 CNRS Univ. Bourgogne Franche-Comté, 15B, Avenue des Montboucons, 25030 Besançon Cedex, France

Using GLAD co-sputtering technique, W-Ag thin films were deposited by using two targets: Tungsten W and Silver Ag where each one was tilted from the center of the substrate by 80°. Different samples were prepared at the same pressure, with the same thickness and the same tungsten target current (140mA) but different silver target currents (from 20 to 80mA). Several characterizations were carried out to study the structure of these films; their morphology, crystallography, elemental composition and their electrical resistivity. The obtained thin films present a columnar growth where the form, angle of tilt and the distance between columns depend on the experimental parameters. Elemental composition is influenced by the evolution of the Ag target current. Moreover, silver is deposited on tungsten-based columns as grains. Finally, electrical resistivity measurements show an important influence of the Ag target current evolution and the temperature variation.



Thematic Session: Surface & interface at the nanoscale.

Keywords: gold nanoparticles, proteins adsorption, correlation spectroscopy, hydrodynamic radius and binding affinity.

Protein corona study by scattering correlation spectroscopy:

A comparative study between spherical and urchin-shaped gold nanoparticles

Hanane Moustouai,¹ Justine Saber,¹ Ines Djeddi,¹ Qiqian Liu,¹ Dania Movia,² Adriele Prina-Mello,² Marc Lamy de la Chapelle,³ Jolanda Spadavecchia,¹ and Nadia Djaker^{*1}

1. Université Paris 13, Sorbonne Paris Cité, UFR SMBH, Laboratoire CSPBAT, CNRS (UMR 7244), 74 rue Marcel Cachin, F-93017 Bobigny, France.
2. Department of Clinical Medicine, School of Medicine, Trinity College Dublin, Dublin 2, Ireland.
3. Institut des Molécules et Matériaux du Mans (IMMM - UMR CNRS 6283), Université du Mans, Avenue Olivier Messiaen, 72085 Le Mans, France.

*Corresponding author. Tel.: +33 148388526 ; E-mail address: nadia.djaker@univ-paris13.fr

The study of the protein interactions with gold nanoparticles (GNP) is a key step prior any biomedical application. These interactions depend on many GNP parameters such as size, surface charge, chemistry, as well as shape. In this work, we propose to use a sensitive technique named scattering correlation spectroscopy or SCS to study protein interactions with GNP. SCS allowed the investigation of the GNP hydrodynamic radius with a very high sensitivity before and after interaction with proteins. No labeling is needed. As proof-of-concept, two of the most used morphologies of GNP-based nanovectors have been used within this work: spherical-shaped GNP (GNS) and branched-shaped GNP (GNU). The measurement of several parameters such as the number of proteins binding to one GNP, the binding affinity and the cooperativeness of binding for three different plasma proteins on the GNP surface was carried out. While GNS showed an increase in the hydrodynamic radius, indicating that each kind of proteins binds on the GNS in a specific orientation, GNU showed different orientations of proteins due to their multi-oriented surfaces (tips) with a higher surface to volume area. Quantitative data based on Hill model were extracted to obtain the affinity of the proteins to both GNS and GNU surfaces. The data variations can be understood in terms of the electrostatic properties of the proteins, which interact differently with the negatively charged GNP surfaces.



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4. S. Ployon et al. *Archives of Oral Biology* (2016) 61, 149