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Automated crystal harvesting, freezing and X-ray diffraction

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This system is an evolution of the G-Rob systems. G-Rob system was developed on protein crystallography beamline FIP-BM30A at the ESRF. G-Rob, A 6-axis robotic arm based system, is a fully integrated device for crystallography beamlines and laboratories. G-Rob is an "all in one" system. Thanks to its tool changer, it goes automatically from one application to another.

A new tool for the robot and an adapted environment has been developed to allow G-Rob *in situ* crystal manipulation from crystallization plates. The harvesting step allows mounting the crystal on the robot's fishing tool with X-ray transparent terminal organs. Once the crystal has been fished, an automated cycle prepares the crystal for diffraction trough cryoprotection and flash cooling steps. Thus the sample is ready for X-ray diffraction without dismounting and human manipulation. At this point, the classical G-Rob's goniometer function [1] is used for data collection.

A visualization bench with an inverted 90° angled microscope and an image processing programs has been developed to offer the ability to evaluate crystals positions in Greiner CrystalQuick™ X plates. The video acquired from the microscope is processed to find three corners of the square well containing the crystallization drop. Accordingly, a click on the centre of an interested crystal will save the coordinates of the crystal in the well frame. The same coordinates are used in situ X-ray analysis of the crystal, in its crystallization drop using G-Rob [2], prior to harvesting.

With this new function, G-Rob can go from *in situ* analysis to data collection on frozen crystal with no need of manual manipulation. All the process can then be operated remotely.

[1] Jacquamet *et al.*, *Structure,* **2004** *12*, 1219. [2] Jacquamet *et al.*, *Acta Cryst.,* **2004** *D60*, 888.

Keywords: crystal_fishing, robot goniometer, x-ray screening automation

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Biomimetic carbonate-apatite nanoparticles functionalized with doxorubicin for applications in nanomedicine

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Inorganic nanosized drug carriers are one of the most promising fields in nanomedicine applied to cancer therapies. These materials must be non-toxic, highly biocompatible and biodegradable. Hydroxyapatite (HA), which is the major inorganic component in hard tissues of vertebrates, may be an attractive biomaterial as nanocarrier for drugs, proteins and genes delivery [1]. HA presents the following advantages: favourable biodegradability, biocompatibility and pH-dependent dissolution; soluble and less toxic than silica, quantum dots, carbon nanotubes, or magnetic particles; low production costs and excellent storage abilities (not easily subjected to microbial degradation); in particular they are dissolved at low pH, e.g. in lysosomes after the cellular intake or in the environment of solid tumours, thereby releasing incorporated drugs or biomolecules. In addition, most of the synthetic micro- and nanocrystalline HA find important biomedical applications such as osteologic implant coatings, grafts and scaffolds for bone cavity fillings [1]. For these reasons, nanocrystalline apatites have been the object of extensive research in several interdisciplinary areas with objectives ranging from better understanding of the formation mechanisms in natural mineralization processes to its applicability as a biomedical or industrial material [2].

In this work we present two different methods for the synthesis of carbonate-HA (cHA) nanocarriers to be functionalized with a chemotherapeutic drug. Firstly, we have employed batch precipitation based on thermal decomplexing of Ca/citrate/phosphate solutions [3]. Secondly, the HA has been synthesized by dropping a solution of H_3PO_4 into a $Ca(CH_3COO)_2$ suspension, keeping the pH at a constant value of 10 by addition of $(NH_4)OH$ solution. The nanoparticles have been characterized by HREM, XRD, FTIR and Raman and subsequently functionalized with Doxorubicin hydrochloride (Doxo), a drug commonly used in cancer chemotherapy.

The adsorption isotherms of this drug onto the different cHA nanocarriers plot the adsorbed amount, Γ_{Doxo} (mg/mg_{HA}), calculated from the difference between the concentrations of the Doxo solutions before and after adsorption on cHA, against the drug concentration remaining after adsorption, C_{Doxo} (mg/mL). An initial slope related with the drug affinity for the cHA surface characterizes all the isotherms. The amount of drug adsorbed on cHA increases with the concentration of Doxo in the solution until it reaches the saturation concentration. Both the affinity and the saturation concentration are much higher for Doxo adsorbed on cHA synthesized by the thermal decomplexing batch method. A model describing the interaction between Doxo molecules and cHA surface is proposed from Raman and FTIR data.

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[1] M. Iafisco, B. Palazzo, M. Marchetti, et al.. *J. Mater. Chem.* **2009**, *19*, 8385-8392. [2] M. Iafisco, J. Gómez-Morales, M. A. Hernández-Hernández et al. *Adv. Eng. Mater.* **2010**,*12*, B218-B223. [3] A. López-Macipe, J. Gómez-Morales, R. Rodríguez-Clemente. *Adv. Mater.* **1998**, *10*, 49.

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Evolution of microstructure and crystallographic orientation during shell growth

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We have studied the evolution of the microstructure and crystallographic orientation of mollusc shells of different species, specifically, shells of *Nautilus belauensis* (Cephalopoda) and *Psilunio littoralis* (Bivalvia) using Scanning Electron Microscopy and X-Ray synchrotron diffraction. These mollusk shells are composites of aragonite ($CaCO_3$) crystals which are disposed in superimposed layers with different three-dimensional arrangements or microstructure types