

52nd Meeting of the Italian Association of Crystallography



**52nd AIC Meeting
Bari**

8-11 September 2026

Edited by Benedetta Carrozzini
Institute of Crystallography - National Research Council (CNR), Bari, Italy

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Conference Schedule

Conference at glance

	8-Sept		9-Sept	10-Sept		11-Sept
		9:00-9:45	Plenary 1	Plenary 3	9:00-9:45	Plenary 5
		9:45-10:00	Sponsor talk	Sponsor talk		
		10:00-10:30	Coffee break	Coffee break	9:45-10:15	Coffee break
		10:30-12:20	MS1 MS2	MS5 MS6	10:15-12:05	MS9 MS10
		12:20-14:00	Lunch	Lunch	12:05-12:35	Nardelli Prize
14:00-15:15	Registration	14:00-14:45	Plenary 2	Plenary 4	12:35-13:15	Conclusions
15:15-15:45	Opening Ceremony	14:45-16:35	MS3 MS4	MS7 MS8		
15:45 – 17:15	Awards	16:35-17:05	Flash presentations	Flash presentations		
17:15-17:45	Coffee break	17:05-17:35	Coffee break	Coffee break		
17:45-18:15	Nardelli Prize	17:35-19:15	AIC Meeting	Poster session		
19:30-22:00	Welcome party	20:30-23:30	Social dinner			

Detailed program

Tuesday 8th September

14:00 - 15:15	Registration
15:15 - 15:45	Opening ceremony - Aula Magna
15:45 - 17:15	AIC Awards - Chair: Chiara Massera <i>MSc Thesis Award</i> Francesco Leone Margherita Vacchini <i>PhD Thesis Award</i> Lisa Baratelli Emanuela Santagata <i>Research Study Abroad Award</i> Giovanni Barra Emmanuele Parisi <i>Advanced Training Award</i> Mara Colombo Giorgio Cagossi Manuel Dedola
17:15 - 17:45	Coffee break
17:45 - 18:15	Nardelli Prize - Chair: Chiara Massera Paolo Swuec <i>Human Technopole, Milan, Italy</i> From Crystals to Cells: Building Structural Biology Across Scales
19:30 - 22:00	Welcome Party at Atrio Cherubini

Wednesday 9th September

9:00 - 9:45	Plenary Lecture 1 - Chair: Angela Altomare Alessandro Genoni <i>Polytechnic University of Milan, Italy</i> Quantum crystallography: from early days to modern tools for the investigation of the electronic structure of matter	
9:45 - 10:00	Sponsor talk Vernon Smith <i>Bruker AXS SE</i> PHOTON IV - Exploring New Possibilities in High Energy Crystallography	
10:00 - 10:30	Coffee break	
10:30 - 12:20	MS1 - Perspectives of crystallographic methods and techniques Chairs: Marco Milanesio, Enrico Mugnaioli	MS2 - Advances in crystal engineering Chairs: Valentina Colombo, Ferdinando Costantino
10:30 - 10:55	Laura Contini (Invited) <i>University of Bologna, Italy</i> Data Mining as a tool for understanding and predicting solid-state behaviour and properties of molecular materials	Len Barbour (Invited) <i>University of Lincoln, United Kingdom; Stellenbosch University, South Africa</i> Unravelling transient porosity: structural insights into flexible porous crystalline materials
10:55 - 11:20	Paolo Pio Mazzeo (Invited) <i>University of Parma, Italy</i> Multitechnique approach for the structural characterization of materials	Marijana Dakovic (Invited) <i>University of Zagreb, Croatia</i> Choreographed Flexibility in Molecular Crystals: Atomic-Level Insights into Stimuli-Responsive Coordination Polymers
11:20 - 11:35	Stefano Righi <i>University of Milan, Italy</i> Fast and Accurate Modelling of Harmonic and Anharmonic Motions in Molecular Crystals from Classical Molecular Dynamics	Luca Catalano <i>University of Modena and Reggio Emilia, Italy; University of Rochester, USA</i> Dynamics-structure-properties relation to design molecular crystals
11:35 - 11:50	Monica Dapiaggi <i>University of Milan, Italy</i> The structure of amorphous mold fluxes for steel continuous casting	Marco Moroni <i>University of Pavia, Italy</i> From metal substitution to halide alloying: structure-property relationships in chiral hybrid metal halides
11:50 - 12:05	Sara Monico <i>IGI, Milan - University of Milan, Italy</i> Integrated Multi-Scale Characterization of Chrysocolla: A Framework for Complex Mineral Materials Lacking Long-Range Order	Lorenzo Gatti <i>University of Bologna, Italy</i> Stimuli-Responsive Photophysical Behavior of Cu(I)-Based Coordination Polymers: A Structural Investigation
12:05 - 12:20	Roberto Borriello <i>University of Venice and University of Pisa, Italy</i> From the solar nebula to planetary interiors: two applications of 3DED	Martina Lippi <i>University of Florence, Italy</i> Guest-Responsive Structural Dynamics in 1D Bispidine-based Coordination Polymers
12:20 - 14:00	Lunch	

14:00 - 14:45	Plenary Lecture 2 - Chair: Emanuela Schingaro Fabrizio Nestola <i>University of Padua, Italy</i> News about natural diamond-forming fluids	
14:45 - 16:35	MS3 - High-throughput and dynamic studies on biosystems Chairs: Rocco Caliendo, Simona Fermani	MS4 - From ambient to non-ambient conditions: exploring mineral structures and transformations Chairs: Giovanni Orazio Lepore, Paolo Lotti
14:45 - 15:10	Daniele De Sanctis (Invited) <i>ESRF, France</i> Ambient crystallography at the fourth-generation synchrotron to resolve protein dynamics on biological timescales	Ella M. Schmidt (Invited) <i>University of Bremen, Germany</i> Disorder in Minerals and Mineral Analogues - A Design Principle for Functional Materials?
15:10 - 15:35	Michele Cianci (Invited) <i>Marche Polytechnic University, Italy</i> Cryo-EM structure of <i>Homarus americanus</i> α -crustacyanin reveals the astaxanthin molecular tuning in marine invertebrate colouration	Giorgia Confalonieri (Invited) <i>Sapienza University of Rome, Italy</i> Structure-Property Relationships in Zeolites: Crystallographic Insights from Ambient to Non-Ambient Conditions
15:35 - 15:50	Andrea Ilari <i>CNR-IBPM, Rome, Italy</i> Exploiting a Druggable E2 Conjugating Enzyme and an E3 Ligase for PROTAC-Based Therapy against Leishmaniasis	Marta Morana <i>University of Florence, Italy</i> Exploring structural distortions and ion-exchange properties in pyrochlores: the case of elsmoreite
15:50 - 16:05	Alessandra Del Giudice <i>Sapienza University of Rome, Italy</i> Coupling protein conformational cycles to time-programmable solution pH: an albumin unfolding–refolding case study	Lisa Baratelli <i>University of Hamburg, Germany</i> Thermal activation of charge carriers in amphiboles: influence of octahedral aluminium and A-site occupancy
16:05 - 16:20	Alessandro Grinzato <i>University of Padua, Italy</i> A novel extended conformation of mitochondrial complex I stabilized by ACAD9 interaction	Davide Delmonte <i>IMEM-CNR, Parma, Italy</i> Thermodynamic, kinetic, and density-driven pathways in the selective transformation of amorphous silica to α -quartz
16:20 - 16:35	Federica Mastropirro <i>University of Bari, Italy</i> Unraveling PCSK9 Structural Conformational Landscape and Evaluating Novel Small-Molecule Inhibitors: An Integrated In Silico, SAXS, and Cellular Approach	Abdelhamid Ouladmansour <i>University of Bari, Italy</i> CRM nanoscale analysis of Portovesme red muds, SW Sardinia
16:35 - 17:05	Flash presentations	
17:05 - 17:35	<i>Coffee break</i>	
17:35 - 19:15	AIC Meeting	
20:30 - 23:30	<i>Social dinner</i>	

Thursday 10th September

9:00 - 9:45	Plenary Lecture 3 - Chair: Cinzia Giannini Simona Galli <i>University of Insubria, Como, Italy</i> Functional coordination polymers: opportunities and challenges of powder diffraction	
9:45 - 10:00	Sponsor talk Alessandro Torboli <i>G.N.R. srl, Agrate Conturbia (NO), Italy</i> GNR Analytical Instruments Group - Worldwide Solutions for Advanced Materials Analysis	
10:00 - 10:30	Coffee break	
10:30 - 12:20	MS5 - New frontiers in X-ray sources for materials science Chairs: Cinzia Giannini, Vincent Olieric	MS6 - Crystal growth, from fundamentals to applications Chairs: Rosalba Fittipaldi, Linda Pastero
10:30 - 10:55	Nicola Casati (Invited) <i>Paul Scherrer Institute, Villigen, Switzerland</i> Evolving Diffraction for Materials Science at the Swiss Light Source	Gianfranco Ulian (Invited) <i>University of Bologna, Italy</i> Experimental and theoretical analysis of Biomaterial-Biomolecules surface interactions
10:55 - 11:20	Vincenzo Formoso (Invited) <i>University of Calabria, Rende, Italy</i> STAR — A Compact Inverse Compton Scattering X-ray Source for Multiscale Materials Characterisation: Design, Commissioning, and Perspectives	Liberato Manna (Invited) <i>Istituto Italiano di Tecnologia, Genoa, Italy</i> Halide Perovskite Nanocrystals Heterostructures
11:20 - 11:35	Cecilia Pozzi <i>University of Siena, Italy</i> Serial crystallography studies on the ferroxidase and biomineralization mechanisms of human H ferritin	Silvia Marino <i>University of Bologna, Italy</i> Manganese substitution in monetite: influence of the synthesis route on phase evolution and crystal structure
11:35 - 11:50	Teresa Sibillano <i>CNR-IC, Bari, Italy</i> From scattering to structural maps: advanced SAXS/WAXS characterization of fibrous materials	Onofrio Tau <i>University of Salento, Lecce, Italy</i> From Benzene to Triphenylene: Atomistic Mechanisms of Early Graphene Nucleation on Cu(111) During Low-Temperature CVD
11:50 - 12:05	Jeta Lica <i>University of Bari, Italy</i> Micro-CT study of biomass-induced porosity in clay bricks	Alessia Bacchi <i>University of Parma, Italy</i> Co-crystals formation from low melting eutectic mixtures
12:05 - 12:20	Fabiana Crucitti <i>University of Bologna, Italy</i> Structural and morphological characterization of bio-inspired superhydrophobic coatings for sustainable agricultural applications	Rabbia Tahir <i>University of Milano-Bicocca, Italy</i> Growth of strain-free InGaAs/InAlAs QDs via LDE as C-band single photon emitter
12:20 - 14:00	Lunch	

14:00 - 14:45	Plenary Lecture 4 - Chair: Anna Moliterni Fabia Gozzo <i>Excelsus Structural Solutions (Swiss) AG, Villigen, Switzerland</i> Synchrotron XRPD for Pharmaceutical Solids: From Trace Phase Detection to Quantitative Characterization	
14:45 - 16:35	MS7 - The role of applied mineralogy in bridging resources, materials, environment and health Chairs: Alessandro Gualtieri, Nicoletta Marinoni	MS8 - Protein structures for modeling, function and drug design Chairs: Filomena Sica, Paola Storici
14:45 - 15:10	Ángeles G. de la Torre (Invited) <i>University of Malaga, Spain</i> Unlocking the potential of 2:1 clays as supplementary cementitious materials	Enrico Di Cera (Invited) <i>Saint Louis University, USA</i> Cryo-EM of coagulation factor V
15:10 - 15:35	Alessandro Pacella (Invited) <i>Sapienza University of Rome, Italy</i> Understanding Carcinogenic Mineral Fibers through a Mineralogical-Crystallographic Lens	Laura Cendron (Invited) <i>University of Padua, Italy</i> Structure-guided inhibition of the bacterial SOS response as an anti-virulence strategy
15:35 - 15:50	Tommaso Magni <i>University of Modena and Reggio Emilia, Modena, Italy</i> Structural characterization of the aluminosilicate network in alkali-activated binders from thermally treated mineral wool with ssNMR	Matteo Ardini <i>University of L'Aquila, Italy</i> Ring-Interface Plasticity and Client Recognition in a Stress-Activated Peroxiredoxin Revealed by Cryo-EM
15:50 - 16:05	Carlotta Giacobbe <i>Thermo Fisher Scientific, Cheseaux-sur-Lausanne, Switzerland</i> Resolving symmetry in complex clinker systems: the crystallographer's role in cement plant decision-making	M. Giovanna E. Papadopoulos <i>CNR-IC, Bari, Italy</i> Comparative Molecular Dynamics Study of MPS I-Associated Mutations in α -L-Iduronidase
16:05 - 16:20	Riccardo Bono <i>University of Milan, Italy</i> Nano ferrites via sol-gel synthesis for Pb and Ni adsorption: analysis on ion-structure relationships	Ilaria Silvestri <i>CNR-IC, Caserta, Italy</i> Structural basis of selectivity in small molecules targeting carbohydrate binding site in galectins
16:20 - 16:35	Enrico Mugnaioli <i>University of Pisa, Italy</i> Using three-dimensional electron diffraction and TEM to address environmental issues	Flavia Squeglia <i>CNR-IBB, Naples, Italy</i> Structure-function insights into phage-derived depolymerases targeting multidrug-resistant <i>Klebsiella pneumoniae</i> : potential antibiotic adjuvants
16:35 - 17:05	Flash presentations	Flash presentations
17:05 - 17:35	<i>Coffee break</i>	<i>Coffee break</i>
17:35 - 19:15	Poster session	Poster session

Friday 11th September

9:00 - 9:45	Plenary Lecture 5 - Chair: Rita Berisio Andrea Musacchio <i>Max Planck Institute of Molecular Physiology, Dortmund, Germany</i> Cell division: a structural perspective	
9:45 - 10:15	Coffee break	
10:15 - 12:05	MS9 - Advanced materials for Sustainable Energy Chairs: Riccardo Fantini, Serena Chiara Tarantino	MS10 - Sharing and disseminating Crystallography Chairs: Annalisa Guerri, Marta Morana
10:15 - 10:40	Elisa Borfecchia (Invited) <i>University of Turin, Italy</i> Deciphering Selective Oxidation Chemistry in Copper-Based Porous Catalysts through X-ray Spectroscopy: From Zeolites to MOFs	Consiglia Tedesco (Invited) <i>University of Salerno, Italy</i> STEAM in action: from educational robotics to materials science
10:40 - 11:05	Daniele Callegari (Invited) <i>University of Pavia, Italy</i> Advanced X-ray characterization into Next-Generation Energy Materials: Tracking Structural Evolution in Lithium and Sodium-Ion Cathodes	Michele Zema (Invited) <i>University of Bari, Italy</i> Crystallography as a Vehicle for Stimulating Research and Education on a Global Scale
11:05 - 11:20	Maria Chiara di Gregorio <i>Sapienza University of Rome, Italy</i> Engineering metal-organic frameworks for highly nonlinear photonics	Carlotta Giacobbe <i>Thermo Fisher Scientific, Cheseaux-sur-Lausanne Switzerland</i> Crystallography behind the button: dissemination, automation and decision-making in industrial environments
11:20 - 11:35	Luciano Marchiò <i>University of Parma, Italy</i> Selective Complex Dissociation of REEs using Photoswitchable Ligands	Martina Lippi <i>University of Florence, Italy</i> Beyond the Invisible: Stories, Science, and Careers in Crystallography
11:35 - 11:50	Marzia Guerrini <i>University of Milan, Italy</i> Exploring Metal Exsolution in Titanate-Based Perovskites	Andrea Ienco <i>CNR-ICCOM, Florence, Italy</i> Words of Crystallography in the Italian Legal Language: A Laboratory for High School Students
11:50 - 12:05	Andrea Vitale <i>University of Bologna, Italy</i> Reordering Frustration in Enantiomer-Enriched Solid Solutions of Ionic Plastic Crystals for Proton-Conducting Materials Design	Alessia Bacchi <i>University of Parma, Italy</i> VISION3D: Augmented and Virtual Reality approach for engaging the public in structural science
12:05 - 12:35	Nardelli Prize 2026 Elena Simone <i>Polytechnic University of Turin, Italy</i> Experimental and multi-scale modelling techniques to engineer crystalline formulations for pharmaceutical and food applications	
12:35 - 13:15	Conclusions	

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AWARDS

Best MSc Thesis Award

Structural and functional characterization of novel inhibitors targeting β -lactamase NDM-1

Francesco Leone¹, Filippo Vascon¹, Giovanni Loprete¹, Rahma Ben Abderrazek², Emna Hamdi², Laura Cendron¹

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The worldwide increase in antimicrobial resistance (AMR) poses a major public health concern. In this regard, β -lactamases play a crucial role as they confer bacteria resistance to β -lactam antibiotics as penicillin, monobactam and carbapenems. Among them, New Delhi Metal- β -lactamase-1 (NDM-1), first identified in New Dehli, has also been reported in countries around the world, including United States, Japan, Australia and the United Kingdom. The NDM-1 gene was identified for the first time in 2009 in *Escherichia coli* and *Klebsiella pneumoniae* and has since spread among diverse bacterial species via horizontal gene transfer [1].

This thesis project aims to investigate nanobodies as potential inhibitors of NDM-1, with a particular focus on structural characterization and binding determinants. Nanobodies derived from a phage display library, were produced in *E. coli* and subsequently purified. Their binding to NDM-1 was assessed through analytic size-exclusion chromatography, which enabled observing the complex formation, by thermal fluorescence and by grating-coupled interferometry (GCI), which provided thermodynamic and kinetic parameters. The structure of NDM-1 and nanobodies were analyzed by X-ray crystallography.

[1] Walsh TR. New Delhi metallo- β -lactamase-1: detection and prevention. *CMAJ*. **2011**;183, 1240-1241.

Chasing the nucleus: experimental and computational investigation of molecular recognition mechanisms in benzoic acid

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Understanding the molecular-scale events preceding crystal nucleation remains one of the most elusive problems in physical chemistry. This thesis combines experimental and computational approaches to investigate benzoic acid (BZA), a notoriously monomorphic compound that crystallizes exclusively in the $P2_1/c$ space group as cyclic hydrogen-bonded dimers, despite crystal structure prediction studies indicating the existence of low-energy catemeric polymorphs with an estimated energy difference of only ~ 5 kJ/mol [1]. This was tested via standard *ex-situ* techniques (evaporation, sublimation) and, notably, *in-situ* crystallization in capillary geometry under unconventional harsh conditions – never previously applied to BZA. In all cases, single-crystal X-ray diffraction confirmed persistent crystallization in $P2_1/c$, reinforcing BZA's robust monomorphism.

To probe intermolecular recognition processes that might precede nucleation, classical Molecular Dynamics (MD) simulations were performed using the Milan Chemistry Molecular Simulation (MiCMoS) package developed in our group [2,3]. Since timescales limitations of unbiased MD prevent direct observation of nucleation, we adopted a “top-down” strategy, by simulating the dissolution of BZA nanoparticles (< 7 nm) embedded in a nanodroplet of their own liquid in high degree of undercooling ($\Delta T \approx 100$ K): the goal was to slow dissolution and enable detection of persistent, symmetry-retaining aggregates, assuming that solid-liquid and liquid-solid transitions share fundamental dynamical features.

After identifying stable, non-trivial conditions across varying morphology, temperature, and size, we observed that: (i) polar surface groups reduce stability, highlighted by a marked drop in rotational correlation; (ii) the experimentally most stable facet (002) is also the most stable computationally, validating the MD results; (iii) melting proceeds from the surface inward, with solvent progressively infiltrating the bulk.

The nanoparticle was then remodeled to match the experimental morphology, allowing us to assess facet stability and explicitly characterize surface carboxylic groups. A focused follow-up studied the dissolution of a ~ 5 nm (576-molecule) BZA nanoparticle in a ~ 14 nm liquid nanodroplet at 300 K. The nanoparticle invariably melted, shown by the global loss of rotational correlation and RDF features relative to the crystal. However, we developed a correlation-based crystallinity criterion that allowed us to detect some metastable supramolecular aggregates that persist for more than 30 ps that, despite being fluxional and partly liquid structures, share some packing features with crystalline BZA – geometrical analysis under approximate local equilibrium supported this resemblance. Subsequent findings [4] show that Bragg peaks could be recognizable in principle from partially crystalline structures down to ~ 3 nm, providing some suggestions on how an experiment should be designed to probe these elusive structures at their minimum length scale.

- [1] G. Macetti, L. Sironi, M. Vacchini, L. Lo Presti, *Cryst. Growth Des.*, **2025**, 25, 8382-8292,
- [2] A. Gavezzotti, L. Lo Presti, and S. Rizzato, *CrystEngComm*, **2022**, 24, 922–930,
- [3] A. Gavezzotti, L. Lo Presti, MiCMoS 2.4 Users' Manual
https://sites.unimi.it/xtal_chem_group/index.php/research/5-micmos
- [4] M. Vacchini, L. Sironi, G. Macetti and L. Lo Presti, *Cryst. Growth Des.*, **2025**, 25, 8969-8979,

Best PhD Thesis Award

The interplay between cation ordering and elasticity of omphacites to unravel PTt paths of metamorphic rocks

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Omphacite, a common clinopyroxene occurring in diverse geological environments, is one of the main rock-forming minerals of eclogites and is also found in blueschist facies and ultra-high-pressure rocks. Its widespread occurrence makes it a valuable candidate for elastic geothermobarometry, a method that uses the residual strain preserved in mineral inclusions to determine the pressure and temperature conditions of their entrapment [1]. Because Raman scattering is highly sensitive to structural deformation, Raman spectroscopy is particularly well suited for this methodology, as it provides detailed information on crystal-structural changes induced by pressure and temperature. However, Raman scattering is also influenced by chemical composition and the state of cation order in omphacite, that has so far prevented this mineral from being used for elastic geothermobarometry.

The aim of this study is to provide constraints to the history of crustal rocks subducted to high-pressure or ultra-high-pressure conditions by developing the necessary tools to exploit the chemical, structural and elastic properties of omphacite. *In situ* high-pressure Raman spectra were collected from both natural ordered and experimentally disordered omphacites. Two sets of Fe-poor crystals with different chemical compositions, previously studied by [2], were used as starting materials. By analysing omphacite crystals with different degrees of order, achieved via isothermal annealing experiments, we observed that increased cation disorder led to broader Raman peaks, while pressure variations shifted peak positions. Then, because Raman peak positions are also affected by chemical composition, an additional suite of Fe³⁺-rich omphacites was investigated to calibrate the compositional dependence of the Raman spectra.

The experimental results were complemented by *ab initio* calculations to model the elastic properties and atomic dynamics of omphacite. This integrated approach provides new insights into the closure temperature of cation ordering at the *M* polyhedra and the residual pressures preserved by omphacite inclusions in garnet, thereby improving our understanding of the pressure-temperature history and geological processes that led to the formation of their host rocks. By quantifying the effects of chemical composition and cation ordering on the Raman response and elastic behaviour of omphacite, this study establishes a procedure for its application in Raman elastic geobarometry, expanding the range of minerals available for reconstructing the pressure-temperature conditions of high- and ultra-high-pressure metamorphic rocks.

Acknowledgements

This work was financially supported by the Italian Ministry of University and Research and the University of Milan, project “Dipartimenti di Eccellenza 2023–2027”.

[1] R.J. Angel, M. Murri, B. Mihailova, M. Alvaro Z. *Kristallogr. Cryst. Mater.* **2019**, 234, 129–140.

[2] T. Boffa Ballaran, M.A. Carpenter, M.C. Domeneghetti, V. Tazzoli *Am. Miner.* **1998**, 83, 419–433.

New crystalline materials for telecommunications and advanced functional applications

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Crystal engineering is the scientific discipline that investigates how intermolecular interactions dictate crystal packing, enabling the rational design of solids with predetermined structures and functions. The design pathway, therefore, begins with the target application, extends to the selection of suitable synthons, and culminates in the choice of molecular functional groups [1]. Within this framework, this PhD project employs crystal engineering principles across two distinct research lines, both relying on the idea that controlling intermolecular interactions enables the design and understanding of advanced organic crystalline materials. The first research line focuses on the development of new ionic and neutral acentric organic crystals for nonlinear optical (NLO) applications, with targeting on terahertz (THz) telecommunications. Organic NLO crystals offer advantages over alternative sources (e.g. inorganic crystals, organic polymers) including higher nonlinear response and chemical versatility and outperform poled polymers in thermal and temporal stability [2]. The primary goal was the design and synthesis of compounds crystallising in polar, acentric space groups, an essential requirement for NLO applications. Several of these compounds crystallized in non-centrosymmetric space groups, as confirmed by single-crystal X-ray diffraction. Measurements of first hyperpolarizability (β) in solution via hyper-Rayleigh scattering (HRS) demonstrated values exceeding those of the benchmark organic NLO crystal DAST, while powder SHG experiments showed comparable efficiencies. The second research line, instead, investigates polymorphism, solid–solid phase transitions, and mechanical responsive materials [3]. Across these studies, we identified cases of exceptionally rich polymorphism in an imine crystallising in six distinct forms that collectively display an unusually broad range of phase-transition mechanisms [4]. In a mesogenic liquid crystal, a thermosalient SCSC transition was observed at remarkably low temperatures and combined structural and spectroscopic analyses revealed that the transformation is driven by a specific low-frequency phonon mode, providing insight into the microscopic origins of mechanically responsive behaviour [5]. Finally, an ionic push–pull compound crystallising in two distinct crystal forms represented a rare case of small organic molecule that exhibit a photosalient effect under UV, white, monochromatic visible and IR light. The mechanical response was found to be strictly polymorph dependent, demonstrating once more how subtle differences in crystal packing dictate the macroscopic outcome of photoinduced processes [6].

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Research Study Abroad Award

Structural and Functional Investigation of Novel Immunogenic Protein Candidates from *Klebsiella pneumoniae*

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Antimicrobial resistance (AMR) poses a serious threat to global human and animal public health. The excess and improper use of antibiotics is increasing the number of reported resistant microbial strains, compromising the conventional clinical treatments. To respond to AMR threat, an immediate action is required. A group of six scary nosocomial pathogens (*Enterococcus faecium*, *Staphylococcus aureus*, *Klebsiella pneumoniae*, *Acinetobacter baumannii*, *Pseudomonas aeruginosa* and *Enterobacter spp.*) is named with the acronym 'ESKAPE' because capable of 'escaping' the biocidal action of antibiotics classified as highly important for human medicine [1-3]. Of particular concern is the highly resistant Gram-negative (Gram-) *K. pneumoniae*, typically associated with infections in severely ill hospitalized patients, for which new strategies are strongly needed [2-3]. In this context, the identification of novel antigenic targets represents a promising strategy for the development of alternative therapeutic and preventive approaches against multidrug-resistant pathogens. In collaboration with Professor M.G. Pizza at Imperial College London, we investigated a pool of previously uncharacterized proteins of *K. pneumoniae*, identified through reverse vaccinology as promising vaccine antigens. To date, these proteins lack comprehensive structural and functional characterisation and their biological role in bacterial physiology and pathogenesis remains largely unknown. Despite this limited knowledge, preliminary analyses suggest that these proteins may possess significant immunogenic potential, making them attractive candidates for further investigation. Therefore, we aim to explore their structural features to identify their biological role and provide new insights into their possible exploitation for the development of innovative vaccine-based strategies against *K. pneumoniae* infections.

The AIC International Training Award has been crucial to my scientific training and to the research I am conducting.

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Multi-scale and multi-technique approach for rational design of crystalline products and crystallization process

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Crystallization is a key isolation and purification process in the pharmaceutical, chemical, and food industries. Crystal properties such as polymorphic form, particle size, morphology, and surface chemistry have a direct impact on critical product attributes, including solubility, dissolution rate, bioavailability, mechanical behaviour, and processability. Therefore, the ability to predict and control crystalline features is essential for the rational design of high-performance solid products. Crystal Structure Prediction (CSP)[2] has emerged as a powerful computational approach to explore the possible polymorphic landscape of molecular compounds. By relying on lattice-energy calculations, CSP can generate and rank putative crystal structures. However, conventional CSP workflows often predict a much larger number of structures than those that can be realistically obtained under experimental or industrial crystallization conditions. This overprediction limits the practical use of CSP for solid-form selection and product design. During the AIC-funded research stay at University College London, targeted crystallization experiments were integrated with molecular dynamics simulations to improve the reliability and transferability of CSP methodologies. The aim was to move beyond the identification of theoretically possible crystal structures and towards the prediction of polymorphs with a realistic probability of experimental occurrence. In particular, MD simulation were performed and incorporated into conventional CSP workflows to account for finite-temperature and finite-pressure effects, enabling the identification of crystal forms with higher nucleation propensity, including metastable but experimentally accessible polymorphs. The resulting MD-CSP framework will be further combined with Particle Informatics tools.[3] This integration will enable the rapid screening, clustering, and ranking of predicted crystal structures according to key bulk and surface properties, including thermodynamic stability, morphology, surface chemistry, roughness, mechanical resistance, and hydrophilic or hydrophobic character. Overall, this research contributes to the development of a computational toolbox for the rational design and selection of tailor-made molecular crystals for specific applications, such as improved dissolution performance, enhanced mechanical robustness, or controlled surface behaviour.

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Advanced Training Award

Stacking Fault and Morphology in the X-ray Total Scattering Patterns of CuInS₂ Quantum Dots

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For colloidal quantum dots (QDs) a few nanometers in size, particle shape, surface termination and planar defects act simultaneously on the same features of the X-ray scattering pattern, entangling structural and morphological information in a way that conventional peak-shape analysis cannot resolve. Atomistic modeling within the Debye Scattering Equation (DSE) framework [1] offers a way to treat shape and defectiveness jointly rather than as independent corrections, reading both directly from the same total scattering pattern.

In a first synthesis series of CuInS₂ QDs, for which no independent morphological prior was available from imaging, the wide-angle total scattering pattern displayed a clear stacking-fault fingerprint. Stacking fault, when present, leaves a signature on the total scattering pattern that is qualitatively distinct from a morphological one: individual hkl families shift and broaden according to their specific fault probability, rather than uniformly across the pattern. Because this fingerprint can otherwise be absorbed into shape-induced anisotropic broadening, we isolate it using shape-agnostic spherical models, following the approach established for stacking-faulted zincblende CdSe nanocrystals [2-3], while shape anisotropy was explored separately through elongated prismatic models.

A second synthesis route yielded, by contrast, essentially fault-free particles: applying the same diagnostic to these size-selected CuInS₂ QDs revealed no measurable stacking-fault fingerprint in the experimental patterns. With defectiveness ruled out, the residual features of the pattern are attributable to shape itself.

We therefore combine synchrotron (ESRF, ID22) wide- and small-angle total scattering data with DSE simulations and fitting to isolate the individual fingerprint left by four candidate morphologies: sphere, [100]- and [111]-elongated prism, tetrahedron, and triangular bipyramid, on the diffraction pattern. At wide angle, shape acts as a second-order but systematic modifier: each morphology redistributes intensity among reflections in an hkl-selective manner, sharpening some peaks while broadening others, with deviations from a spherical reference reaching 10–20% in agreement factor despite identical particle mass. At small angle, the same shapes act at leading order instead: faceted morphologies lacking parallel faces, the tetrahedron being the limiting case, damp the form-factor oscillations that a spherical model would otherwise show, generating an apparent degeneracy with size polydispersity that scattering data alone cannot resolve.

Taken together, these results provide a quantitative basis for separating shape- from defect-driven features in the total scattering pattern of faceted, defect-bearing quantum dots, a distinction that is a necessary prerequisite for extracting meaningful compositional and structural parameters, such as vacancy content or interface character, from real experimental data.

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Mechanochemistry redefines pillared MOFs: from twisting polymorphism to high entropy mixing

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Mechanochemistry is emerging as far more than a green alternative to solution synthesis [1,2]. By inducing reactivity in the solid state, mechanical activation can modify reaction pathways and influence selectivity, stabilizing unexpected phases and enabling levels of structural and compositional complexity that are often difficult to obtain in solution. In the field of metal–organic frameworks (MOFs), ball milling has proven capable of altering framework assembly, accessing new structural motifs, and facilitating the incorporation of multiple metals within a single material. Coordination polymers and MOFs are particularly sensitive to these effects [3], as subtle variations can drastically alter symmetry and topology.

A clear example is PUM198, a Zn-based mixed-ligand pillared MOF previously obtained solvothermally [4]. Under ball milling, the material forms in just 5 minutes a new polymorph, PUM198-m. Although composition and connectivity are preserved, the two phases differ in symmetry, interlayer arrangement, pore geometry, and topology, representing the first case of interlayer-twisting polymorphism in a pillared MOF. As PUM198-m is obtained only as a flexible, guest-filled microcrystalline powder, its structure was solved through a combined 3DED, plane-wave DFT, and Rietveld refinement workflow.

Mechanochemistry also enabled increased compositional complexity in PUM420(Zn), a similar double-interpenetrated pillared MOF reproduced in high yield by ball milling. Beyond Co- and Cu-analogues, a high-entropy [5] pentametallic variant containing equimolar Zn²⁺, Cu²⁺, Ni²⁺, Co²⁺, and Mn²⁺ was obtained with homogeneous metal distribution (~20% each). Preliminary proton conductivity measurements show a positive trend for the high-entropy analogue, highlighting the functional impact of entropy-driven multimetal mixing. Overall, mechanochemistry emerges as a powerful tool to redefine MOFs granting access to more complex frameworks while reducing environmental impacts.

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Metal Snowdrift: Ultraporous Base Metal Monoliths with Fully Addressable Reactivity

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Metallic foams have long been constrained by a 'density floor' - fabrication limits like surface tension and sintering - blocking them from achieving the ultralow densities of silica aerogels [1,2,3]. Here, we report a template-free, bottom-up synthesis of self-supporting iron foams with a record-low density of $\sim 1 \text{ mg cm}^{-3}$ and porosity of 98.32%. Formed via vapor-phase decomposition, these materials evolve through flow-driven fractal aggregation into a hierarchical network spanning from 10 nm grains to macroscopic branches. We demonstrate this framework as a versatile reactive scaffold, yielding conductive Cu and Au foams and $\text{TiO}_2\text{@Fe}$ composites, and polymer hybrids via galvanic replacement, co-decomposition, or infiltration. This approach establishes a scalable platform for multifunctional metallic aerogels that merge nanoparticle reactivity with macroscopic structural integrity.

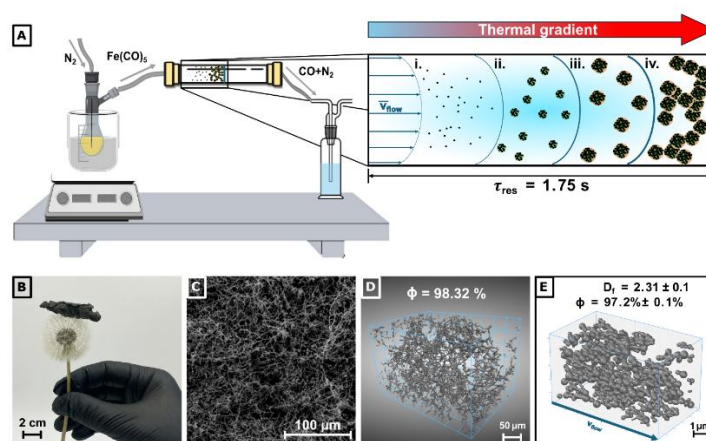


Figure 1: Synthesis and structure of ultralight Fe foams. (A) Vapor-phase decomposition of Fe(CO)_5 under N_2 flow. (B) Macro image of $\sim 1 \text{ mg/cm}^3$ foam on a dandelion. (C) SEM of the percolating nanoparticle network. (D) 3D nano-CT showing hierarchical porosity ($\phi=98.32\%$). (E) Brownian simulations of flow-driven fractal aggregation.

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Nardelli Prize

From Crystals to Cells: Building Structural Biology Across Scales

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Structural biology has undergone a profound transformation, expanding from the atomic description of purified macromolecules to the visualization of molecular machines in increasingly native environments. In this talk, I will present an overview of my contribution to this evolution, spanning integrative structural biology, single-particle cryo-electron microscopy, cryo-electron tomography, and the establishment of cryo-EM as an accessible and competitive approach in Italy. Through selected examples, I will discuss how cryo-EM and complementary structural methods have enabled mechanistic insight into diverse biological systems: the Fanconi Anemia core complex and the molecular basis of DNA interstrand crosslink repair [1]; patient-derived cardiac amyloid fibrils in AL amyloidosis [2]; anti-CRISPR inhibition of Cas9 [3]; ion permeation and gating in HCN4 pacemaker channels [4]; and, more recently, SOLIST, a cryo-lift-out strategy that enables nanoscale “biopsies” of native tissues for cryo-electron tomography [5]. Together, these studies illustrate a trajectory from isolated macromolecular assemblies to complex cellular and tissue contexts.

A central theme of the lecture will be the translation of cryo-EM expertise into infrastructure, training, and scientific opportunity. I will describe the creation of the first cryo-EM laboratory in Italy at the University of Milan, followed by the design and development of the Human Technopole Cryo-EM Core Facility and, subsequently, the National Facility for Structural Biology. These efforts have aimed not only to provide access to state-of-the-art instrumentation, but also to import best practices in sample preparation, data acquisition, processing, user training, and facility operation.

By combining methodological innovation, collaborative science, and national infrastructure building, this work reflects how we can reshape structural biology community and open new routes to understanding biological function across scales.

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Experimental and multi-scale modelling techniques to engineer crystalline formulations for pharmaceutical and food applications

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Multiphase systems (e.g., emulsions, foams, creams) are a convenient and effective encapsulation and delivery strategy, particularly for oral and topical formulations. Currently, synthetic excipients, surfactants and specialty polymers are used to create formulations with enhanced properties. However, these compounds are derived from non-renewable resources through some of the most greenhouse gas-intensive manufacturing processes. For this reason it is necessary to replace the common synthetic stabilizers used for these products with natural, biocompatible and biodegradable materials. These include natural micro and nano-particles (Pickering stabilizers) such as proteins, polysaccharides and various crystalline materials including cellulose, chitin, fat crystals and polyphenol crystals. Pickering systems are particularly promising since particles adsorb at interfaces more strongly than surfactants, providing significantly more stable formulations. The stability of Pickering systems is strongly affected by particle size and shape, but surface wettability is the most important property of Pickering particles. For faceted, anisotropic crystals surface wettability is not easy to determine. In fact, crystals present multiple crystallographic facets, whose surface properties (e.g., polarity, wettability) depend on the type and directionality of the intermolecular interactions that characterize each facet.

The purpose of the presented work is to understand how crystal properties (size, shape and polymorphism) of Pickering particles affect their surface properties, hence their orientation and adsorption behavior at interfaces. In order to achieve this goal and enable the design of optimized multiphase formulations, we applied a combination of modelling techniques, ranging from the molecular (synthons analysis from crystallographic data, molecular dynamics), meso (population balance equations, interfacial particle stabilization) and continuum (diffusion, dissolution) scales, and experimental activities, including particle characterization (facet-specific and bulk measurements such as nano-IR and electron diffraction) and powder performance determination (kinetic parameters estimation, stability studies) measurements to better understand Pickering stabilization and enable optimal multiphase formulation design. Different model systems including mixtures of edible triacylglycerides and flavonoids of various nature (e.g., quercetin, curcumin, xanthone) were used and will be illustrated.

PLENARY LECTURES

Quantum crystallography: from early days to modern tools for the investigation of the electronic structure of matter

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Quantum crystallography [1] is often viewed as a brand-new frontier of science and particularly of crystallography, but it is far from a temporary trend. The discipline finds its historical roots in the early twentieth century, at the concurrent dawn of both quantum physics and modern structural science. Indeed, early pioneers in those fields already envisioned a profound dual relationship [2, 3]: exploiting X-ray diffraction data to map the electronic structure of matter, while simultaneously leveraging emerging quantum theory to interpret crystallographic experiments.

Although this intrinsic connection never ceased to exist, it long remained tacit, largely because crystallography was, and often still is, perceived merely as a structural science. On the one hand, the crystallographic community itself frequently overlooks how deeply embedded quantum mechanics is when a crystal structure is solved or when optical or electronic properties of crystals are evaluated. Furthermore, decades of rigorous work to determine electron densities from accurate X-ray diffraction data [4] were rarely recognized for what they truly were, namely fundamental investigations into quantum electronic structures. On the other hand, the broader quantum chemistry and condensed-matter communities frequently ignored how crystals provide the ultimate, perfectly ordered laboratories for observing electronic behavior and validating theoretical models in the solid state.

Today, driven by the modern convergence of high-resolution diffraction, intense radiation sources, and high-performance computing, quantum crystallography is reviving the underlying symbiosis between quantum mechanics and crystallography [1]. This plenary lecture will highlight this aspect and will show how modern quantum crystallographic methods go far beyond standard structure solution. In fact, while techniques such as the now widely popular Hirshfeld atom refinement [5] (HAR) have dramatically improved structural models and atomic positional parameters, an even broader challenge is the direct characterization of electronic behavior. We will explore how advanced methods like the X-ray restrained wave function (XRW) approach [6, 7] provide an unprecedented window into electron densities and electronic structures, revealing experimentally supported descriptors of chemical bonding, charge transfer, spin distribution, and intermolecular interactions, which are also critical for engineering next-generation functional materials. Finally, future outlooks for quantum crystallographic methods will be discussed, particularly their integration with emerging experimental and theoretical techniques, such as X-ray free-electron laser (XFEL), artificial intelligence, and quantum computing.

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News about natural diamond-forming fluids

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Natural gem-quality diamonds do not usually contain fluid inclusions and therefore the nature of the fluids or melts from which diamonds form has remained poorly understood. In contrast, “fluid-rich” diamonds (more generally referred to as “fibrous diamonds”) contain millions of submicrometric fluid inclusions (e.g., “high-density fluids” - HDFs). Although the fluid inclusions in fibrous diamonds represent the deepest natural fluids that can be directly investigated, they remain understudied compared to normal gem-quality diamonds. It is still unclear whether the fluids observed in fibrous diamonds are universally the same as those from which gem-quality diamonds form. Previous work identified, by Raman spectroscopy, a possible fluid in gem-quality diamonds occurring as thin films (~1–5 µm) surrounding a wide variety of mineral inclusions [1]. This fluid film is currently the only form of fluid recognized in gem-quality diamonds and although its composition remains largely unknown, it appears to be a Si–O–H-bearing fluid. This year, researchers [2] attempted to measure the composition of this fluid film using slow LA-ICP-MS depth profiling and they were able to distinguish the respective contributions of the diamond, fluid film, and mineral inclusions, and detected elements such as Ba, Sr, Ce, Nb, La, Nd, and Th within the fluid film. These elements are typically found in HDF inclusions in fibrous diamonds, suggesting a possible genetic link between the two types of diamond-forming fluids. In order to provide new insights into the composition and possible sources of both gem-quality and fibrous diamond-forming fluids, we investigated a unique diamond from the Democratic Republic of Congo (DRC) which consists of a gem-quality core overgrown by a fibrous rim (i.e., a “coated” diamond) (Figure 1). Within the gem core we observed clusters of isolated circular droplets of a fluid with similar composition to that found in films around mineral inclusions, but in this case not associated with solid inclusions. Such fluids have been described previously as isolated inclusions along twinning planes in only one gem-quality diamond. In addition, we observed, for the first time, the same fluid in the fibrous region of the studied diamond close to the boundary between the gem core and fibrous rim. Several coesite inclusions, surrounded by a fluid film, were also observed at the core-rim boundary. Nitrogen-aggregation (FTIR) thermochronometry suggests the gem core resided within the mantle at ~1100 °C. Elastic geobarometry applied to coesite inclusions show the fibrous rim formed at a pressure of ~5.5 GPa.

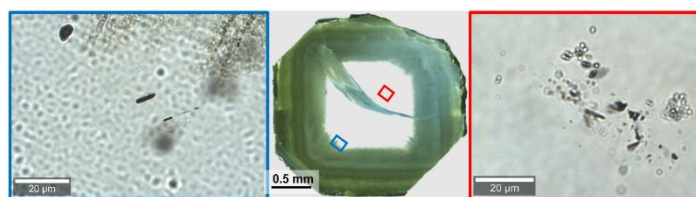


Figure 1. The coated diamond here investigated (central). On the left, three coesite inclusions (one on the top left and two elongated in the middle); on the right, pseudo-spheres of high-density fluids.

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Functional coordination polymers: opportunities and challenges of powder diffraction

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Owing to their modular architecture, remarkable crystallochemical versatility, and (ultra)high porosity, metal–organic frameworks (MOFs) have steadily evolved from a primarily academic research topic into a mature and widely recognized class of materials, demonstrating outstanding performance across a broad spectrum of cutting-edge applications [1]. Integration of advanced solid-state characterization techniques is particularly powerful for unveiling the atomic-scale features underneath bulk functional properties. In the case of MOFs, a rational combination of solid-state complementary techniques enables the successful investigation of primary adsorption sites, adsorbent–adsorbate interactions, short- and long-range structural dynamics, thermodynamic properties [2–4]. Within this context, this contribution presents a selection of MOF case studies [5–9] by the research group in Structural Chemistry of Advanced Materials at the Università dell'Insubria. The selected examples will illustrate how multi-technique strategies based on (*in situ*) powder X-ray diffraction are pivotal to elucidating the atomic-scale details (Figure 1) responsible for the observed functional performance. These findings are not only significant for the material under study but also provide valuable guidance for the rational design of next-generation MOFs with optimized crystallochemical—and, consequently, functional—properties.

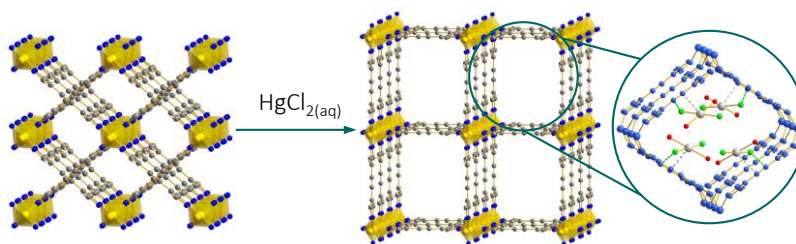


Figure 1. Interpenetration to non-interpenetration phase transition triggered by HgCl_2 capture and MOF- HgCl_2 interactions insurgence.

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Synchrotron XRPD for Pharmaceutical Solids: From Trace Phase Detection to Quantitative Characterization

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The solid-state form of an active pharmaceutical ingredient (API) plays a critical role in determining the properties, stability, manufacturability, and performance of pharmaceutical products [1,2]. Consequently, the detection, identification, and quantification of crystalline and amorphous forms are essential throughout the pharmaceutical product lifecycle, from development and manufacturing to intellectual property protection [3].

The exceptional angular resolution and high signal-to-noise ratio offered by synchrotron X-ray powder diffraction (S-XRPD) have enabled analytical capabilities that extend well beyond those of conventional laboratory instrumentation. At Excelsus Structural Solutions, these advantages have been leveraged to develop methodologies for the detection and quantification of pharmaceutical crystalline forms at trace levels, reaching limits as low as 0.01 wt% in favorable cases, as well as for the characterization of complex mixtures containing both crystalline and amorphous components [3].

This presentation will illustrate how high-resolution synchrotron XRPD can be used to address a range of industrially relevant challenges in pharmaceutical development. Selected case studies will demonstrate the characterization of polymorphic mixtures, the assessment of phase purity in drug substances and drug products, the monitoring of solid-state transformations during stability studies, and the quantification of trace crystalline impurities. Particular emphasis will be placed on the factors governing detection limits, including counting statistics, angular resolution, and diffraction-angle calibration, and on how these parameters can be exploited to distinguish and quantify closely related pharmaceutical solid forms.

Through these examples, the presentation will highlight the growing role of synchrotron XRPD as a powerful tool for the quantitative characterization of pharmaceutical solids and for supporting critical scientific, regulatory, and intellectual property decisions.

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Cell division: a structural perspective

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Kinetochore are multisubunit complexes assembled on specialized chromosome regions named centromeres [1,2]. Kinetochore provide chromosomes with points of attachment to spindle microtubules during cell division, and are therefore essential for genome inheritance and the propagation of life [3]. In addition to binding microtubules, kinetochores control mitotic surveillance mechanisms that promote chromosome bi-orientation (the error correction mechanism) and prevent premature mitotic exit in presence of incomplete or incorrect microtubule attachments (spindle assembly checkpoint, SAC). Elimination of the NDC80 complex, the main microtubule receptor of kinetochores, causes a SAC deficiency, identifying this complex as a crucial regulatory focus for checkpoint function. In recent years, there has been considerable progress in understanding how the SAC effector, known as the mitotic checkpoint complex (MCC), assembles from its individual components to inhibit its target, the anaphase promoting complex/cyclosome (APC/C). Conversely, how microtubule attachment to kinetochores regulates the SAC remains incompletely understood. From a molecular perspective, answering this question implies investigating the mechanisms that promote targeting of the SAC proteins to unattached kinetochores, and suppress it upon microtubule binding and biorientation. In our recent work, we have combined biochemical reconstitutions, structural biology/modelling, and cell biology to gain insights into this fundamental biological question.

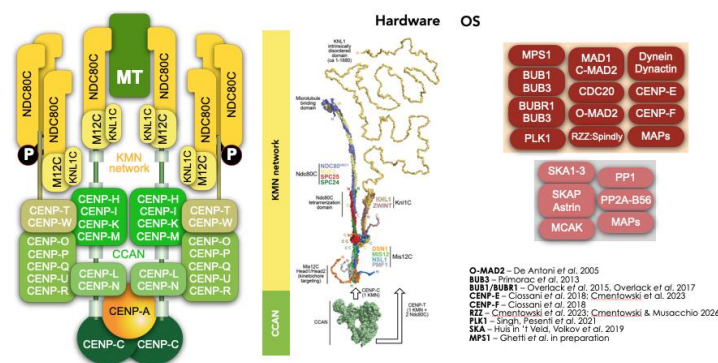


Figure 1. This should include a brief caption to the B/W figure.

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ORAL PRESENTATIONS

MS1

Perspectives of crystallographic methods and techniques

Data Mining as a tool for understanding and predicting solid-state behaviour and properties of molecular materials

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Structural analysis of crystal structures deposited in crystallographic databases offers a powerful route to rationalise the behaviour of molecular materials, accelerating the understanding of the structural reasons behind experimentally observed properties and informing the development of predictive tools [1,2]. In this context, the continuous growth of crystallographic databases provides an increasingly rich dataset for data mining and knowledge-based approaches to molecular materials design, complementing traditional experimental methods [1,3]. This contribution presents two case studies that illustrate this approach in complementary contexts.

The first case concerns the challenge of predicting the behaviour of molecules that, although possessing functional groups that make them ideal candidates for co-crystallization with a large number of apparently suitable coformers, resist co-crystals formation and largely prefer self-aggregation [4,5]. Allantoin represents an ideal model system for this class of molecules. An extensive experimental co-crystal screening, combined with structural analysis, revealed that its limited propensity for co-crystal formation is driven by strong, recurring self-association motifs that impose structural and energetic constraints on potential coformers. These insights guided the development of a novel knowledge-based descriptor for virtual co-crystal screening, demonstrating how low-yielding experimental campaigns can generate the structural knowledge needed to build improved predictive strategies.

The second case illustrates a combined approach of data mining with structural analysis to investigate the mechanochromic behaviour of Cu(I)-based halide coordination polymers (HCPs). Mechanochromism is a property of significant interest for applications in sensing and damage detection [6] but, despite its practical relevance, the physical and structural origins of this phenomenon remain poorly understood [7,8]. Through crystallographic data mining and structural analysis of a series of CuI-based HCPs, this work aims to establish a framework for rationalising mechanochromic propensity in terms of structural features, contributing to the broader goal of property-driven crystal engineering.

Together, these two case studies demonstrate how integrating crystallographic methods, structural databases, and data-driven strategies can yield tools for predicting materials properties.

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Multitechnique approach for the structural characterization of materials

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Metal–organic frameworks are versatile crystalline materials whose structural flexibility and host–guest responsiveness can be exploited for molecular recognition, adsorption, and controlled release [1–3]. Within this context, the PUM family of Zn(II)-based pillared-layer MOFs has provided a valuable platform to investigate how supramolecular interactions inside the pores govern framework adaptability and guest organization [1–3]. Mechanochemistry offers a complementary route to these materials, enabling rapid, low-solvent synthesis while accessing crystalline phases not available under solvothermal conditions [4].

I will here present PUM198-m, a new mechanochemically accessed polymorph of the Zn(II)-based pillared-layer MOF PUM198. Although PUM198-m has the same composition and connectivity as the parent solvothermal phase, it displays a distinct framework arrangement arising from interlayer rotation and twisting of the bis-amidic pillars. This reorganization changes the symmetry, pore architecture, and topology of the framework, revealing a mechanochemistry-induced polymorphic form encoded within the same chemical system [5]. Because PUM198-m is obtained only as a microcrystalline, guest-containing powder, conventional single-crystal X-ray diffraction and direct structure solution from powder diffraction could not be applied. Its structure was therefore determined through an integrated workflow combining three-dimensional electron diffraction, plane-wave DFT optimization, and DFT-assisted Rietveld refinement. 3DED provided the initial framework model, DFT enabled construction of a chemically meaningful DMF-loaded structure, and Rietveld refinement against PXRD data validated the as-synthesized phase. This work highlights how advanced crystallographic methods can overcome the limitations of individual techniques and reveal latent structural degrees of freedom in flexible MOFs [5].

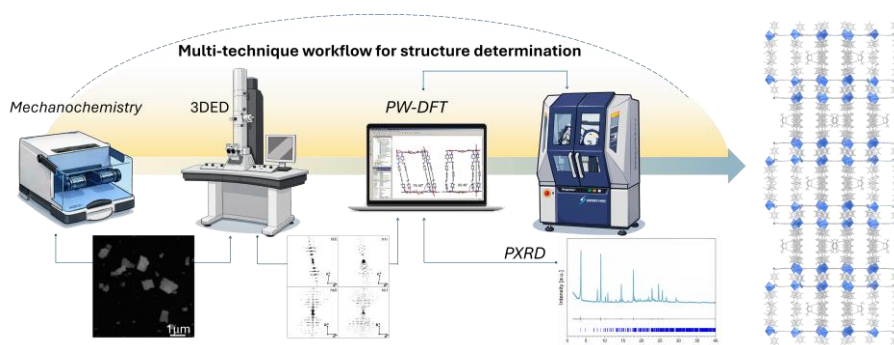


Figure 1. Schematic workflow followed for the structural elucidation of PUM198-m

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Fast and Accurate Modelling of Harmonic and Anharmonic Motions in Molecular Crystals from Classical Molecular Dynamics

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Harmonic atomic displacement parameters (ADPs) provide useful information about the magnitudes and the directions of average atomic vibrations. However, anharmonic effects may be far from negligible, especially above cryogenic temperatures. Even estimating the relative weight of anharmonic and harmonic displacements is experimentally challenging, as it requires diffraction data at very high resolution, which are seldom available at room temperature, precisely where anharmonic effects are expected to be least negligible.

Here, we leverage classical Molecular Dynamics (MD) methods available MiCMoSv2.4 [1] to sample atomic probability density functions (pdfs) in form I of crystalline urea [2] as a function of temperature and pressure with the LJC Force Field [3]. We provide approximate yet quantitatively consistent estimates of the harmonic Debye-Waller parameters, comparable with those derived from X-ray and neutron diffraction (Figure 1), and periodic DFT simulations. The anharmonic effects, quantified by the Grüneisen parameter and by explicit temperature-dependent evaluation of Gram-Charlier tensors, are in excellent agreement with available experimental data. Our results confirm that anharmonic effects may be non-negligible even at cryogenic temperatures in molecular materials. Overall, we provide a cheap semi-quantitative method to estimate the relative weight of harmonic and anharmonic displacement amplitudes as a function of temperature, as well as their mutual interplay in describing the crystal dynamics in the real space. Implications on accuracy of X-ray datasets are also discussed.

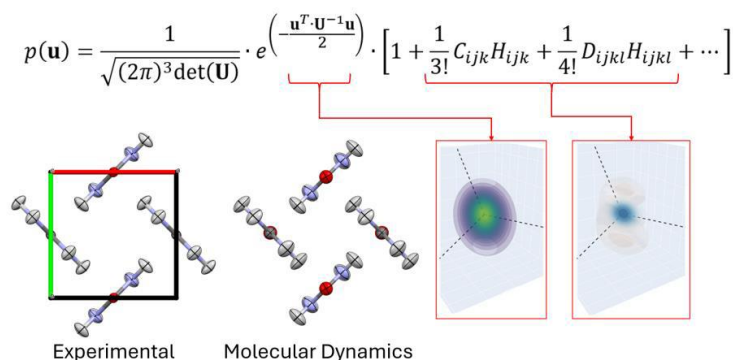


Figure 1. MD simulations reproduce reasonably well the ADPs of phase I (*P*-421*m*) of urea at *T* = 123 K and allows to model anharmonic (Gram-Charlier) contributions to real-space atomic probability distribution functions (pdfs)

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The structure of amorphous mold fluxes for steel continuous casting

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Steel production is made through continuous casting, with the need to reduce the frictional force between molten steel (which is solidifying on the outer shell) and the copper mould. Fluxes are especially made for this purpose: they are synthetic oxy-fluoride glass-ceramic systems used also to control the amount of heat transfer rate [1, 2]. Mold fluxes form various layers, in contact with molten steel, the most interesting one being the interface between the amorphous and the liquid one. In this work, the structure of the amorphous layer is studied by means of total scattering, with spallation neutrons and high energy X-rays.

The performance of these materials is ideal when the ratio CaO/SiO_2 is larger than one, but the relationship between this empirical value and actual structure of the amorphous layer is yet to be fully understood. The samples were prepared with a CaO/SiO_2 varying from 0.65 to 1.3, and with different amount of fluorine (which also has important effects on viscosity), in order to check how the structural features of the materials vary with the chemical composition. The data collection was performed at ISIS (beamline NIMROD) and at ESRF (beamline ID11), both ideally suited for amorphous materials. Studying the structure of amorphous materials is not easy and requires specific knowledge and some good statistical indicators for the quality of the fit between the experimental data and the model for the structure. With no periodicity and with short-length order, amorphous materials are challenging. The structural model obtained for an amorphous material can therefore be one of the possible models, and understanding its reliability can be particularly intriguing and difficult. The structural information were obtained using the EPSR (Empirical Potential Structure Refinement) approach [3], using a large cell of 50000 atoms, for better statistics.

The structure of the samples are very similar to each other in terms of coordination numbers, bond lengths and bond angles of the network formers (Si and Al), both with a coordination number with O between 3 and 4. There is a good correlation between the 3-fold network formers and the 1-fold coordination number of fluorine with Si and Al, respectively, showing that there are Si-O (or Al-O) tetrahedra with 3 oxygens and one fluorine. Ca-Ca clusters play a very important role, *in operando*, for these materials, as they can be thought as the onset of the nuclei formation of the main crystalline phase (*i.e.* cuspidine). Various size distributions can be obtained, as a function of the cutoff distance: once the proper one is chosen, the most suitable distribution to describe observations was Gamma distribution with a peculiar shape (optimal $\alpha \approx 1.04$) and scale parameter [4], as it better captures long-tail behaviour, with respect to a Gaussian one. From the Gamma distribution parameters, it was possible to infer the probability density which, for atomic concentration higher than clustering critical atomic concentration, showed a flex point for CaO/SiO_2 across 1.0 which largely overlaps the trend of DSC crystallization index curve. The relationship between this structural feature and the performance of the mold flux seems then to be clear, but it needs to be confirmed by the results obtained from the same samples in the melted state.

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Integrated Multi-Scale Characterization of Chrysocolla: A Framework for Complex Mineral Materials Lacking Long-Range Order

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Chrysocolla is a secondary copper-bearing material whose mineralogical nature remains debated, making it a topic of significant scientific interest. Besides its mineralogical relevance, chrysocolla has long been exploited as a copper ore and gemstone, because of its intense blue-green colour. It is generally described as a hydrated copper silicate, with the formula $(\text{Cu}_{2-x}\text{Al}_x(\text{H}_{2-x}\text{Si}_2\text{O}_5)(\text{OH})_4 \cdot n\text{H}_2\text{O}, x < 1)$ [1]. Although accepted as a mineral species by the International Mineralogical Association (IMA) in 1969, its true nature is still controversial, particularly regarding whether it represents a distinct mineral phase or a colloidal gel containing Cu-bearing phases. These uncertainties arise from its low structural order, compositional heterogeneity, and susceptibility to alteration when subjected to some analytical protocols [2].

This study presents an integrated multi-analytical and multi-scale investigation of chrysocolla from compositional, structural, and mineralogical perspectives. Samples from two mining localities—Capo Calamita, Capoliveri (Italy), and Cornwall (Lebanon County), Pennsylvania (USA)—were analysed using an *ad-hoc* analytical set-up designed to preserve the material stability while highlighting its distinctive features.

Chemical characterization was performed through EMPA-WDS analyses under optimized low-beam-damage conditions, coupled with multivariate statistical analysis. High-resolution Pair Distribution Function (PDF) analysis, based on synchrotron X-ray data, provided insights into the short-range structural organization, overcoming the limitations of conventional X-ray diffraction techniques, while transmission electron microscopy (TEM) enabled nanoscale investigation of structural, textural, and chemical features. Thermodynamic modelling of the Cu–SiO₂–SCOH system further constrained chrysocolla stability and formation conditions.

The adopted workflow enabled progressive characterization from the microscale to the sub-nanoscale, providing complementary structural, compositional, and textural information inaccessible through conventional approaches alone. The results reveal previously unknown features of chrysocolla and highlight characteristics related to the distinct formation environments of the investigated samples [3]. Overall, this study contributes to the mineralogical characterization and classification of chrysocolla, and represents a significant case study in the application of advanced analytical protocols to complex mineralogical materials.

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From the solar nebula to planetary interiors: two applications of 3DED

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Minerals are the building blocks of rocky bodies in the Solar System. By definition, they are normally crystalline [1], meaning that, ideally, their atomic structure is ordered and periodic. In fact, real crystals often show short-range disorder and reduction in crystallinity, but these local features are usually averaged in a sufficiently long range. However, natural processes, like rapid cooling, often produce heterogeneous aggregates of sub-micrometric crystals where even such small imperfections may become significant. These features prevent the use of conventional crystallographic techniques like single-crystal X-ray diffraction. In contrast, transmission electron microscope (TEM) techniques, like selected area electron diffraction, are capable to analyse such challenging samples, but they only produce 2D sections of the reciprocal lattice. Three-dimensional electron diffraction (3DED) [2] combines the high spatial resolution of TEM with a 3D reconstruction of the reciprocal lattice, enabling deep micro-structural investigations of minerals formed in extreme environments. Here we present two applications of 3DED that cover different topics in planetary sciences.

1. Ca-Al-rich inclusions (CAIs) are thought to be the oldest solids formed in the solar nebula, recording the early evolution of the Solar System [3]. 3DED was performed to study mineralogical transformations in altered CAIs from the metasomatized type 3 Ornans-type carbonaceous chondrite (CO3) Frontier Mountain 90006. Particular attention was paid to those involving 1) hibonite and hercynite, 2) clinopyroxenes with variable Al-Ti-Mg contents and 3) anorthite and nepheline. The combination of TEM imaging, nanoscale energy-dispersive X-ray spectroscopy and 3DED guarantees a robust petrographic investigation, allowing to resolve cryptocrystalline and polyphasic matrices which contain information about both primary crystallization and secondary metamorphic reactions at non-equilibrium conditions.

2. Tranquillityite, currently defined as $\text{Fe}^{2+}_8\text{Zr}_7\text{Ti}_3\text{Si}_3\text{O}_{24}$, is a rare silicate first discovered in Apollo 11 samples [4] and subsequently found in lunar and Martian meteorites [5,6] and as an accessory phase in terrestrial mafic rocks [7]. To date, a description of its crystal structure is still missing. By combining 3DED with electron backscatter diffraction data, we managed to solve and refine, for the first time, the structure of tranquillityite from the Martian shergottite Northwest Africa 856.

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MS2

Advances in crystal engineering

Unravelling transient porosity: structural insights into flexible porous crystalline materials

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The past three decades have seen remarkable progress in the design and characterisation of new classes of porous materials. Yet, deciphering the complex interplay between molecular-level structure and physico-chemical properties – particularly in systems that undergo structural transformations upon guest uptake or release – remains a significant challenge. This is especially true for flexible porous materials with 0D pores, which exhibit transient porosity: the ability to transport guest molecules despite lacking permanent channels. These behaviours, often indicated by stepped sorption isotherms, call for innovative analytical approaches. This presentation will describe laboratory-based *in situ* single-crystal and powder diffraction techniques developed in our group to probe guest-induced structural changes. It will also highlight a relatively straightforward adsorption calorimetric method for determining the enthalpy changes associated with individual sorption and desorption events during gate-opening and -closing transitions.[1] Drawing on two decades of work, the presentation will demonstrate how these methods have yielded deep insights into the behaviour of soft porous crystals and clarified key mechanisms underlying transient porosity.[2-10]

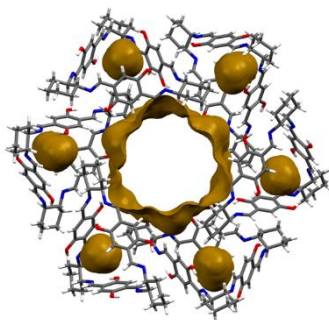


Figure 1. Perspective view of a porous molecular system containing open channels and 0D cavities.

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Choreographed Flexibility in Molecular Crystals: Atomic-Level Insights into Stimuli-Responsive Coordination Polymers

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Long regarded as rigid, highly ordered, and largely immobile, molecular crystals are now recognised as capable, in selected cases, of undergoing pronounced mechanical deformation while preserving their long-range order and structural integrity. This unusual combination of crystallinity and adaptability offers exciting opportunities for the design of responsive materials that convert external stimuli into controlled structural or functional changes [1]. Understanding such behaviour requires detailed insight into how molecular arrangement, supramolecular interactions, and crystal packing collectively govern a crystal's response to external stress.

One-dimensional coordination polymers (CPs) represent particularly powerful model systems for investigating the structural origins of mechano-flexibility. Owing to their modular architectures, directional coordination bonds, and tunable supramolecular interactions, these materials can exhibit a wide range of adaptive responses, from elastic bending to more complex reversible motions. Recent experimental studies have revealed close relationships between crystal structure, interaction energetics, and mechanical performance [2–4], while the identification of flexibility-enabling structural motifs has enabled more targeted approaches to improving mechanical behaviour [5].

In this contribution, we focus on the atomic-level mechanisms that govern mechanoresponsive behaviour in selected 1D CPs [6]. Synchrotron μ -SCXRD, supported by complementary spectroscopic, mechanical, and computational approaches, is used to correlate mechanically and thermally induced structural changes with macroscopic deformation. The results reveal distinct structural accommodation pathways in 1D CPs and show that their deformation mechanisms differ markedly from those previously reported for other classes of reconfigurable molecular crystals.

Acknowledgements

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Dynamics-structure-properties relation to design molecular crystals

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Functional properties of molecular crystals are often governed not only by static structure but also by collective lattice dynamics, including low-frequency vibrations, cooperative molecular motions, and soft phonon modes. Establishing clear dynamics-structure-properties relationships is therefore essential to move beyond empirical crystal engineering toward predictive material design. Recent advances in vibrational spectroscopy, in situ structural characterization, and multiscale molecular design enable direct access to these dynamic degrees of freedom and their rational modulation.

By integrating molecular and supramolecular engineering with external stimuli, such as temperature, pressure, and electric or electromagnetic fields, polymorphic energy landscapes can be reshaped on demand, reversible phase transitions can be programmed, and adaptive functionalities such as flexibility, self-healing, and tunable thermal and optoelectronic properties can be achieved [1-3]. Representative examples illustrate how lattice dynamics can be elevated from a descriptive parameter to an active design variable, enabling the development of dynamic functional molecular crystals.

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From metal substitution to halide alloying: structure–property relationships in chiral hybrid metal halides

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In the recent years, chiral hybrid organic–inorganic metal halides (HOIMHs) have attracted increasing attention for applications in optoelectronics, spintronics, photodetection, energy harvesting and beyond, owing to their ability to absorb and emit polarized light with tunable responses across the electromagnetic spectrum [1,2]. In these materials, the incorporation of chiral organic cations promotes crystallization in non-centrosymmetric structures, enabling intriguing nonlinear optical properties in addition to the features mentioned above. Despite the remarkable growth of the research field and the synthesis of numerous novel compounds, several fundamental questions remain unanswered. On one hand, most reported chiral HOIMHs are based on lead, raising concerns due to its well-known toxicity. On the other hand, a comprehensive understanding of how the chemical composition influences crystal structure, particularly the octahedral distortions responsible for non-centrosymmetry and leading to nonlinear optical behavior, is still lacking [3].

To address these challenges, we are developing new families of chiral HOIMHs through the systematic tuning of both the metal center and the halide component. Starting from $(R/S\text{-}3\text{AQ})_2\text{PbBr}_4\cdot 2\text{Br}$ ($R/S\text{-}3\text{AQ} = R/S\text{-}3\text{-aminoquinuclidine}$), we synthesized the corresponding Sn^{II} and Ge^{II} analogues, obtaining isorecticular Ruddlesden–Popper phases in which the degree of octahedral distortion increases with decreasing metal atomic weight. Circular dichroism measurements revealed comparable chiral dissymmetry factors across the series, indicating that this parameter is primarily governed by the materials dimensionality rather than by the nature of the metal center, in agreement with previous studies from our laboratory. In parallel, we are investigating iodide substitution in $(R/S\text{-}3\text{AQ})_2\text{PbBr}_4\cdot 2\text{Br}$ to induce a red shift of the band gap aiming to extend the optical response into the visible region. Besides the expected absorption shift, increasing iodide content triggers a structural transition toward a zig-zag two-dimensional perovskite phase, accompanied by enhanced photoluminescence emission. Through these studies, we aim to establish new structure–property relationships governing the chiro-optical response, ultimately providing design principles for next-generation chiral functional materials.

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Stimuli-Responsive Photophysical Behavior of Cu(I)-Based Coordination Polymers: A Structural Investigation

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We present a comprehensive study on the structural characterization and stimuli-responsive photophysical behavior of Cu(I)-X hybrid coordination polymers (HCPs), with a specific focus on their mechanochromic properties. These materials, composed of coordination clusters, exhibit significant and reversible changes in their emission spectra when exposed to external stimuli, including mechanical and thermal inputs.

Our investigation primarily focused on the mechanochromic response induced by mechanical stress. The synthesized materials displayed notable shifts in emission characteristics correlated with the intensity and duration of the applied mechanical stimulus. Spectroscopic analyses highlighted how these variations are associated with structural changes induced by the external perturbation.

To further understand the impact of mechanical stress, structural analysis revealed that, despite the introduction of lattice distortions, the crystalline integrity of the material was preserved. Additionally, complementary studies were conducted to investigate the thermal response, exploring the relationship between structural dynamics and photophysical behavior. This work provides valuable insights into the dynamic luminescence of Cu(I)-based HCPs, emphasizing their potential applications in responsive optical devices and adaptive materials. The results contribute to advancing the understanding of structure-property relationships in coordination polymer systems.

Guest-Responsive Structural Dynamics in 1D Bispidine-based Coordination Polymers

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Made of metal salts and organic linkers, Coordination Polymers (CPs) have emerged as highly versatile materials for applications ranging from electronics and magnetism to medicine and volatile organic compound (VOC) adsorption. Despite the absence of permanent porosity in many conventional 1D CPs, these systems can exhibit remarkable dynamic behavior, accommodating or exchanging guest molecules through substantial structural reorganizations.[1] In this work, we report a new family of 1D CPs based on differently functionalized bispidine ligands combined with metal salts such as CuCl₂, MnCl₂, and HgCl₂. Depending on both the nature of the building blocks and the incorporated solvent molecules, the resulting compounds adopt distinct structural topologies and display markedly different dynamic responses toward solid-vapor adsorption and guest-exchange processes. (Figure 1) Significant structural rearrangements, together with notable resilience in accommodating or replacing solvent molecules, were observed upon exposure to VOC-saturated atmospheres. The adsorption and exchange processes are directly associated with pronounced phase transformations, investigated by single-crystal and powder X-ray diffraction (SCXRD and PXRD), thermal analysis (DSC and TGA-FTIR), and hot-stage microscopy. The studied systems exhibit a variety of transformation pathways, including single-crystal-to-single-crystal, single-crystal-to-microcrystalline powder, amorphous-to-microcrystalline powder, and microcrystalline-to-microcrystalline transitions. The kinetics of these processes span a broad timescale, ranging from hours to several weeks. In selected cases, the materials also demonstrated selective adsorption behavior toward specific VOCs. Overall, these findings highlight the exceptional structural flexibility and tunability of bispidine-based 1D CPs, offering new insights into their dynamic solid-vapor responsiveness and underlining their potential as promising materials for selective VOC adsorption and separation technologies.

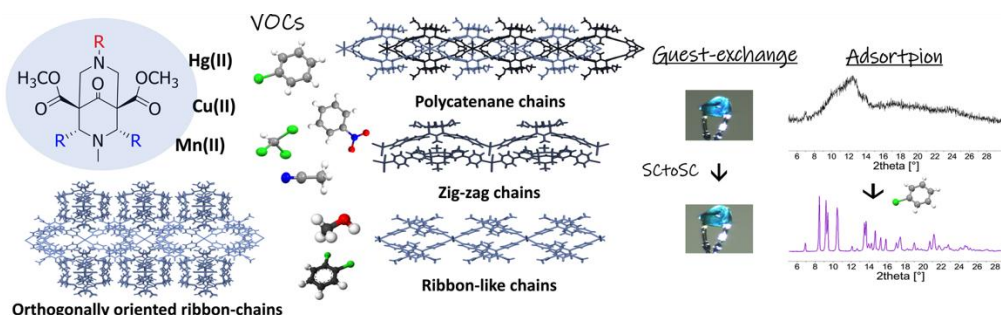


Figure 1. Different topological chains constituting the 1D bispidine CPs based on Mn(II), Cu(II), Hg(II) and the example of their ability to guest exchange and adsorption via solid-state transformations.

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MS3

High-throughput and dynamic studies on biosystems

Ambient crystallography at the fourth generation synchrotron to resolve protein dynamics on biological timescales.

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Room-temperature (RT) data collection in macromolecular crystallography (MX) is experiencing a resurgence due to its ability to provide structural information under near-physiological conditions. In contrast to cryogenic data collection, RT methods offer the advantage of revealing functionally relevant conformational states, dynamics, and transient structural changes in a time-resolved manner. This capability is particularly valuable for structure-based drug design, as it enables the identification of hidden or rare binding states. Moreover, RT collection eliminates the potential artifacts introduced by cryoprotectants and cryocooling, ensuring more reliable and accurate data. The European Synchrotron Radiation Facility's Macromolecular Crystallography, Imaging, and Scattering group operates various beamlines with complementary capabilities to study protein structures in ambient conditions. These capabilities include in situ data collection on ID30A1 and ID23-2, where diffraction data can be directly collected from crystallization plates. ID30A1 also offers automated sample harvesting, which enables the automatic collection of large crystals from plates and their preservation in a humidity-controlled environment. Additionally, ID29 employs time-resolved serial crystallography, using microsecond-long pulses to expose microcrystals to extremely high flux density, revealing dynamics at this time scale [1]. This presentation will provide a broad overview of the methods available at these beamlines and delve deeper into the time-resolved capabilities of ID29, highlighting some scientific results.

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Cryo-EM structure of *Homarus americanus* α -crustacyanin reveals the astaxanthin molecular tuning in marine invertebrate colouration

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Carotenoproteins are essential in generating vibrant colors through complex interactions between carotenoids and proteins. One key example is the *Homarus americanus* (American lobster), whose distinctive blue coloration arises from the interaction of the carotenoid pigment astaxanthin (AXT) with the α -crustacyanin (α -CR) protein complex. α -crustacyanins are constituted of multiple copies of the heterodimer β -crustacyanin in which the chromophore, namely the carotenoid astaxanthin (3,3'-dihydroxy- β,β -carotene-4,4'-dione), is associated stoichiometrically 1:1 with the protein subunits (Zagalsky, 1985).

Astaxanthin is a carotenoid that exhibits a characteristic red-orange color, absorbing at 472 nm in its free form. However, when bound to the crustacyanins, it undergoes a bathochromic shift, shifting its absorption maximum to dark-blue ($\lambda_{\text{max}} = 632$ nm) upon interaction with α -crustacyanin, resulting in the blue hue of the lobster carapace. We obtained a high-resolution cryo-EM structure of α -crustacyanin from *H. americanus* complemented by small angle X-ray scattering and X-ray diffraction, revealing groundbreaking key features behind the molecular mechanisms responsible for the significant bathochromic shift to 631 nm. The cryo-EM structure of *H. americanus* α -crustacyanin to a resolution of 2.75 Å reveals, besides the expected crustacyanin subunits H1 and H2, the presence of a third protein belonging to the heptatricopeptide repeat protein (HPR) family, which interconnects a variable number of β -crustacyanin heterodimers and interacts asymmetrically with the two astaxanthin molecules, leading to the observed bathochromic shift to $\lambda_{\text{max}} = 631$ nm [1]. The study provides not only a successful understanding of the lobster blue coloration but also an exhaustive advance of how protein-carotenoid interaction influences the spectral properties of astaxanthin laying the foundation for the rational design of astaxanthin-based scaffolding compounds.

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Exploiting a Druggable E2 Conjugating Enzyme and an E3 Ligase for PROTAC-Based Therapy against Leishmaniasis

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Leishmaniasis is a neglected tropical disease endemic in low- and middle-income countries, affecting up to one million people annually according to the World Health Organization. Visceral leishmaniasis, caused by *Leishmania infantum*, is the most severe form of the disease and can be fatal if left untreated. Current therapies are limited by toxicity, variable efficacy, lengthy treatment regimens, and the emergence of drug resistance [1].

The parasite survives the oxidative burst generated by host macrophages through a unique trypanothione-based redox system, whose central enzyme, trypanothione reductase (TR), maintains trypanothione in its reduced state. Owing to its essential role in parasite survival, absence in humans, and favorable druggability profile, TR has long been considered an attractive therapeutic target. However, despite decades of research, no TR inhibitors have advanced to clinical development, mainly because of insufficient efficacy and safety concerns [2].

Targeted protein degradation has recently emerged as an alternative therapeutic strategy capable of eliminating, rather than inhibiting, disease-relevant proteins. PROteolysis TARgeting Chimeras (PROTACs) are bifunctional molecules that induce ubiquitination and subsequent proteasomal degradation of a target protein by recruiting components of the ubiquitin–proteasome system (UPS) [3]. As first step, we developed TR-targeting PROTACs designed to recruit a putative *Leishmania* E3 ligase, but their activity was modest and the recruited E3 remains poorly characterized. To overcome these limitations, we propose an innovative E2-recruiting PROTAC strategy that bypasses the requirement for a defined E3 ligase. As a first step, we structurally and functionally characterized a *Leishmania* major ubiquitin-conjugating enzyme (E2). Crystallographic fragment screening identified potential ligandable sites on the enzyme, while DeepFrag-guided fragment expansion and AutoDock Vina docking were employed to generate and prioritize E2-binding chemotypes. Together, these studies establish the molecular framework for the rational development of E2-recruiting PROTACs targeting TR and open new opportunities for exploiting the ubiquitin–proteasome system in antileishmanial drug discovery.

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Coupling protein conformational cycles to time-programmable solution pH: an albumin unfolding–refolding case study

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The intimate connection between the structure and the function of biological macromolecules makes the temporal control of their conformational state a powerful handle to regulate specific functionalities, with prospective applications ranging from protein-based actuators to programmable catalysis and drug release. A general strategy to achieve such control is to couple the conformation of a macromolecule to the gradual, programmed shift of a solution parameter – most conveniently pH – over time. Two complementary chemical approaches can drive a smooth pH change in a single sample without further operator intervention. The first relies on the slow hydrolysis of glucono- δ -lactone, which produces a continuous and tunable acidification ($\text{pH}_{\text{high}} \rightarrow \text{pH}_{\text{low}}$); the second exploits “activated” carboxylic acids, such as nitroacetic acid (NAA), whose conjugate base undergoes base-catalysed decarboxylation and acts as a chemical fuel to generate dissipative $\text{pH}_{\text{high}} - \text{pH}_{\text{low}} - \text{pH}_{\text{high}}$ sequences, whose amplitude and recovery time can be tuned by varying the absolute and relative concentrations of acid and base [1,2]. Coupled to a conformation-sensitive read-out, these methods turn time into a structural reaction coordinate.

We previously established this paradigm on the acid-induced unfolding of Human Serum Albumin (HSA), a heart-shaped, three-domain carrier protein that reversibly switches among native and partially unfolded acid forms below pH 4.5.

By combining the glucono- δ -lactone acidification with simultaneous small-angle X-ray scattering (SAXS) and intrinsic tryptophan fluorescence, the multistep unfolding pathway was resolved as a function of pH within a single sample, isolating the scattering signature of the intermediate F form and assigning it to a drifting apart of the three domains [3]. The same continuous-acidification approach further revealed that the binding of fatty acids stabilises the native conformation toward lower pH, accounting for the long-standing conformational heterogeneity observed near pH 4 [4].

Building on these foundations, we show how NAA enables a complete, time-programmable unfolding–refolding cycle of HSA monitored by fluorescence and synchrotron SAXS. Starting from the native protein, the fast acidification triggers unfolding to a partially denatured state, followed by a time-tunable slow pH rise that drives full refolding. The conformational states accessed during the jump recapitulate those obtained by static acid addition at the same pH values, confirming the reversibility of the structural transition, while the time-resolved scan of a single sample removes the variability of multiple preparations and improves the identification of the structural components. The permanence in the partially unfolded state can be programmed from below one minute to about twenty minutes, after which the native structure is fully recovered without further intervention. Being albumin a versatile macromolecular carrier, this strategy could be exploited for time-programmable ligand sequestration or release protocols.

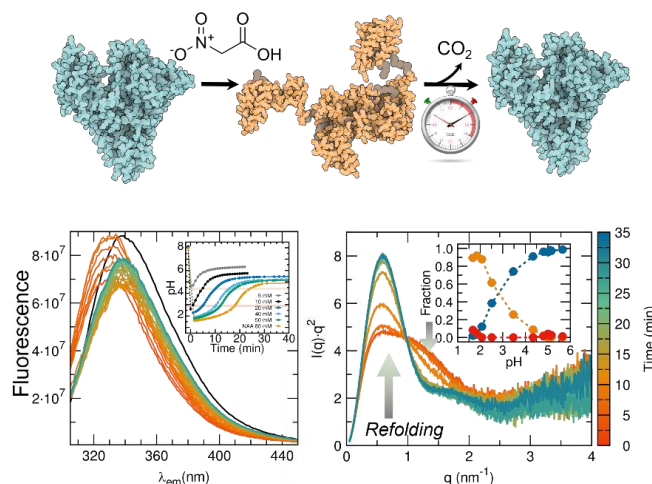


Figure 1. Pictorial view of an unfolding–refolding cycle of albumin triggered by NAA decarboxylation; intrinsic fluorescence and SAXS data (Kratky plot) report the time-dependent refolding to the native state.

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A novel extended conformation of mitochondrial complex I stabilized by ACAD9 interaction

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Complex I (CI) is the largest enzyme of the mitochondrial respiratory chain, coupling NADH oxidation to proton pumping across the inner mitochondrial membrane. The acyl-CoA dehydrogenase family member 9 (ACAD9) is a dual-function mitochondrial protein involved in both long-chain fatty acid β -oxidation (FAO) and CI assembly[1,2]. Although ACAD9 is essential for mitochondrial function and is implicated in CI deficiency, its structural and functional interplay with assembled CI remains poorly understood.

Here, we combined cryo-EM and solution biophysics to characterize the ACAD9–CI interaction. Cryo-EM analysis shows that bovine CI alone adopts the previously reported deactive conformation [3], whereas the structure obtained from the CI–ACAD9 co-eluted SEC fraction revealed a novel extended state at 2.3 Å resolution. Although the ACAD9–CI interaction occurs on a timescale not directly visible on the cryo-EM grid, we captured the structural consequences of that interaction within the same dataset: both ACAD9 and CI exhibit marked conformational changes. Fully assembled CI adopts a novel extended state—its membrane arm elongates and inter-subunit separations increase, with these changes propagating into the catalytic core while canonical subunits and cofactors remain clearly present in the maps.

Complementary, SEC-SAXS, SEC-MALS, and microscale thermophoresis indicate a low-micromolar interaction consistent with a 1:1 stoichiometry between one ACAD9 dimer and one CI. Crosslinking mass spectrometry maps ACAD9 contacts to matrix-exposed subunits in the membrane arm, defining a specific interaction interface, while EPR measurements show no detectable alteration of the redox-cofactor signals, indicating that cofactors remain functionally intact. Together, these data identify ACAD9 as a structural modulator of Complex I, with a regulatory role that extends beyond assembly.

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Unraveling PCSK9 Structural Conformational Landscape and Evaluating Novel Small-Molecule Inhibitors: An Integrated *In Silico*, SAXS, and Cellular Approach

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Cardiovascular diseases represent the leading cause of mortality worldwide, with hypercholesterolemia being a major associated risk factor [1]. Elevated low-density lipoprotein cholesterol (LDL-C) promotes atherosclerotic plaque formation and increases cardiovascular risk [2]. While statins remain the first-line treatment, a subset of patients exhibits intolerance or an inadequate response, highlighting the need for alternative pharmacological options [2,3]. In this context, Proprotein Convertase Subtilisin/Kexin Type 9 (PCSK9) plays a central role in cholesterol homeostasis by promoting LDL receptor (LDLr) degradation [2–4]. To date, between 200 and 300 genetic variants, including both gain- and loss-of-function mutations, are known to modulate this protein's activity, directly impacting cholesterol regulation. In this study, we combined structural biology techniques with functional cellular assays to characterize PCSK9 variants and explore novel regulatory strategies. Human WT PCSK9 and three disease-associated variants, two gain of function: E32K and R496W and one loss of function: R237W, were overexpressed in cellular systems (HEK293) and purified. Small-angle X-ray scattering (SAXS) analyses were performed to investigate their solution structure, overall conformational organization, and flexibility under near-physiological conditions. Comparative SAXS analysis revealed mutation-dependent conformational changes that may significantly affect domain organization and functional interactions. In parallel, we pursued the identification of novel small-molecule inhibitors. Starting from an *in silico* molecular docking analysis, a series of structurally related compounds were evaluated. The candidate molecules were then screened using human HepG2 cells constitutively expressing the protein. Cytotoxicity and cell viability were assessed using the Alamar Blue assay, demonstrating a favorable safety profile with no significant toxic effects at the tested concentrations and time points. To evaluate the inhibitory potential of the compounds, cellular expression levels and intracellular maturation of PCSK9 were further analyzed and validated via Western blot. Taken together, the SAXS-based characterization of PCSK9 variants establishes a detailed structural framework, while the identification and functional screening of novel small molecules open promising avenues for the development of alternative therapeutic strategies against hypercholesterolemia.

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MS4

**From ambient to non-ambient conditions:
exploring mineral structures and transformations**

Disorder in Minerals and Mineral Analogues: A Design Principle for Functional Materials?

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We often think of crystals as perfectly ordered structures composed of a single repeating building block: the unit cell. In reality, nature rarely forms perfectly ordered structures, and compositional and configurational degrees of freedom are inherent to many mineral classes. The atomic structure of a material ultimately determines its physical properties, including thermal expansion, mechanical stiffness, ionic conductivity, and heat transport.

On the microscopic scale, disorder introduces variability in local building blocks, and this flexibility in atomic arrangements can profoundly alter macroscopic physical properties [1]. In this contribution, I explore how structural disorder in minerals and their synthetic analogues can be understood as a design principle for functional materials. Specific examples are drawn from hollandites [2], whose tunnel structures can host disorder relevant to ionic transport, aluminophosphate frameworks such as $\text{AlPO}_4\text{-5}$ [3] and cubic zirconia [4], a classic example of stabilized disorder with implications for ionic conductivity and thermal barrier applications.

I introduce how local order and disorder can be probed in diffraction experiments through diffuse scattering, and how complementary analysis approaches can be employed to link local atomic arrangements to functional material properties.

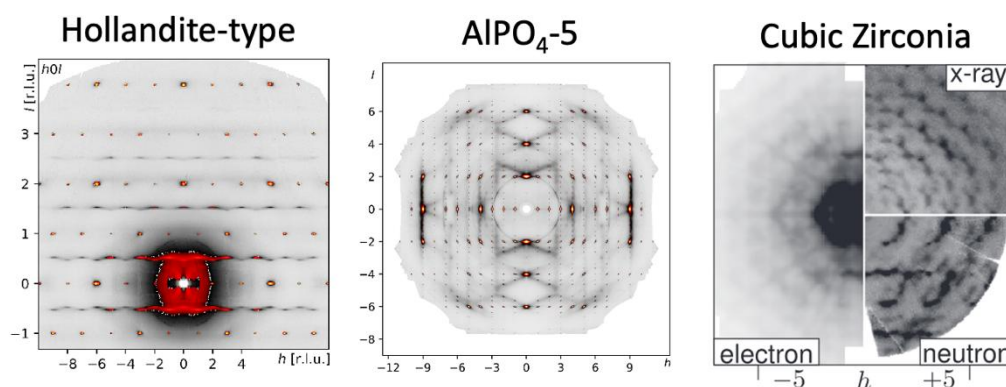


Figure 1. Examples of diffuse scattering in minerals and mineral analogues. (left) $h0l$ -layer in Mannardite, a hollandite-type mineral; (middle) $h0l$ -layer in the zeotype $\text{AlPO}_4\text{-5}$; (right) hhl -layer in Y-stabilized cubic zirconia.

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Structure-Property Relationships in Zeolites: Crystallographic Insights from Ambient to Non-Ambient Conditions

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Zeolites are natural and synthetic microporous aluminosilicates of major mineralogical and technological importance, owing to their remarkable chemical and structural diversity and to their widespread applications in adsorption, catalysis, ion exchange, molecular separation, and environmental technologies.

Their structures consist of ordered systems of cages, channels, and cavities [1] that host extra-framework cations, water molecules, and a wide variety of guest species. This open-framework architecture makes zeolites highly responsive to chemical and physical stimuli, including cation exchange, hydration–dehydration processes, molecular adsorption, gas pressure, temperature variations, and hydrostatic compression [2].

Even subtle variations in these parameters may induce framework deformation, symmetry changes, cation redistribution, disorder, or phase transitions. Such structural responses are fundamental for understanding and tuning zeolite properties and can be exploited in technological applications including gas capture and separation, molecular confinement, energy storage, and environmental remediation [3].

In this context, zeolites represent ideal model systems for investigating the relationships among crystal chemistry, structural flexibility, and functionality in porous materials. Crystallography plays a central role in this field, as it enables these transformations to be investigated at the atomic scale. *In situ* experiments allow the structural evolution of zeolites to be monitored under controlled pressure, temperature, gas atmosphere, or fluid composition, whereas *ex situ* studies provide complementary information on recovered phases, metastable states, and host–guest configurations. Representative examples include CO₂-induced framework flexibility and hydration-driven metastability in RHO zeolites, pressure-induced ion intrusion in pure-silica chabazite, reversible Ce exchange in zeolite L, and the encapsulation of UV filters within LTL zeolite channels.

The integration of *in situ* and *ex situ* approaches is therefore essential for understanding how external stimuli are reflected into structural modifications. This knowledge provides the basis for the rational design and optimization of new zeolite-based materials with targeted structural and functional properties.

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Exploring structural distortions and ion-exchange properties in pyrochlores: the case of elsmoreite

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Pyrochlores are natural and synthetic transition metal oxides, with general formula $A_{2-m}B_2X_{6-w}Y_{1-n}$, where m , w and n represent structural vacancies [1]. They have an ideal cubic structure ($Fd\bar{3}m$), characterized by the presence of a framework of BX_6 octahedra that form tunnels along $[110]$. Such tunnels accommodate the interstitial A and Y sites that often exhibit disorder and are associated with a broad distribution of electron density. A wide variety of cations and anions of different sizes and charges can be found in these sites, which may also participate in ion-exchange processes. This aspect makes pyrochlores interesting materials for environmental applications, particularly for trapping large heavy-metal cations.

In this context, we explored the structure and ion-exchange properties of a synthetic analogue of the W-dominant pyrochlore, elsmoreite [2]. Powder samples of $Na_{0.88}(W_{1.82}Mg_{0.18})O_6(OH)_{0.16}$ were synthesized [3] and characterized by Particle Induced X-ray emission (PIXE), X-ray diffraction (XRD) and X-ray absorption spectroscopy (XAS) before and after imbibition with solutions containing the targeted metal cation, Tl^+ , Pb^{2+} , Zn^{2+} at different concentrations.

From the XRD data, it is clear that synthetic elsmoreite deviates from the ideal cubic structure, even before treatment with heavy metal solutions. Analysis of the electron densities in the tunnel sites suggests that a significant disorder is present within the tunnels, both before and after imbibition, and that Tl^+ , Pb^{2+} and Zn^{2+} can, in different quantities, enter the structure. However, monovalent Tl^+ shows a different behaviour with respect to the divalent cations. In fact, a larger amount of Tl^+ is hosted in the tunnels, making elsmoreite a selective sink for monovalent cations, an interesting and promising feature for prospective environmental applications. Additionally, XAS data show that Pb^{2+} and Zn^{2+} tend to replace the cations in the A sites, whereas Tl^+ is disordered among the Y sites, showing a markedly asymmetrical environment. Our results highlight the importance of a detailed structural characterization, comprising both average and local structure, to fully understand structure-property relationships in minerals and their synthetic analogues as well as in potential functional materials.

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Thermal activation of charge carriers in amphiboles: influence of octahedral aluminium and A-site occupancy

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Characterising mineral properties under non-ambient conditions is critical for understanding large-scale geological processes. Hydrous minerals, such as amphiboles (general formula $AB_2C_5T_8O_{22}W_2$), are abundant in the mid-crust and subduction zones and they are key contributors to volatile cycling and anomalous high-conductivity layers (HCLs) through their role in charge transport, influencing fundamental processes such as water cycling, seismicity, and arc magmatism. Recent research demonstrates that heating iron-bearing amphiboles triggers a stepwise redox reaction: ${}^C\text{Fe}^{2+} + {}^W\text{OH}^- \leftrightarrow {}^C\text{Fe}^{3+} + e^- + {}^W\text{O}^{2-} + \text{H}^+ \rightarrow (\text{with } \frac{1}{4} \text{O}_2^{\text{ext}}) \rightarrow {}^C\text{Fe}^{3+} + {}^W\text{O}^{2-} + (\frac{1}{2} \text{H}_2\text{O})_{\text{released}}$. This process generates two types of charge carriers: small electronic polarons (conduction electrons coupled with longitudinal optical phonons) and delocalized H^+ ions. Direct evidence of these carriers can be provided by Raman spectroscopy through the detection of resonance Raman scattering (RRS) and the reversible delocalization of H^+ cations [1]. The activation temperature as well as the concentration and mobility of charge carriers are highly sensitive to crystal chemistry. In Fe-rich species like riebeckite, polaron activation occurs at relatively low temperature (~ 500 K), matching the pressure and temperature conditions of HCLs [2]. In contrast, in Fe-poor species like actinolite, even minor iron content triggers polaron formation, but at significantly higher temperatures (~ 1150 K) and with poorly aligned dipoles [3].

Our recent studies focused on Fe^{2+} -bearing magnesian amphiboles of similar ${}^C\text{Fe}/({}^C\text{Fe}+{}^M\text{g})$ ratio, namely, glaucophane and a specimen on the richterite-ferri-winchite join. Glaucophane was used to analyse the influence of the octahedrally coordinated Al (${}^C\text{Al}$), whereas richterite-ferri-winchite was used to examine the effect of partially filled A site. In glaucophane, a three-stage oxidation process occurs: an initial Fe^{2+} oxidation (~ 600 K), a further oxidation, mainly at the $M(1)$ sites, highlighted by RRS (~ 900 K), and final delocalization of H^+ (above ~ 1000 K). While ${}^C\text{Al}$ does not inhibit the delocalization of electrons and H^+ , electrical conductivity measurements reveal that it suppresses the mobility of FeO_6 -related polarons, resulting in very low electrical conductivity. In richterite-ferri-winchite, polaron activation, revealed by RRS, occur at approximately the same temperature as in glaucophane (~ 950 K), whereas H^+ delocalization begins at lower temperature (from ~ 650 K). Electrical conductivity measurements show that a partially filled A site does not inhibit polaron mobility, as evidenced high electrical conductivity value along the octahedral stripes ($0.01 \text{ S}\cdot\text{m}^{-1}$ at 1050 K) and its strong anisotropy.

These findings provide essential atomistic data to refine geophysical models of the subducting lithosphere, demonstrating that the activation of mobile charge carriers is a universal characteristic of Fe-bearing amphiboles.

Acknowledgements

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Thermodynamic, kinetic, and density-driven pathways in the selective transformation of amorphous silica to α -quartz

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Silica is one of the most abundant and technologically important materials, owing to its remarkable physicochemical properties and the wide range of polymorphs it can adopt. Among these, α -quartz is the thermodynamically stable phase under ambient conditions and is extensively employed in applications spanning electronics, optics, piezoelectric devices, and advanced ceramics. Although α -quartz can, in principle, be obtained by calcination of amorphous silica, its direct formation in the absence of mineralizers, such as alkaline salts, remains a major challenge. Under conventional thermal treatments, metastable polymorphs, particularly cristobalite, preferentially nucleate and grow, even within temperature ranges where α -quartz is thermodynamically favored, highlighting the dominant role of kinetic factors in the crystallization process.

In this work, we develop an optimized mineralizer-free calcination strategy to selectively produce polycrystalline α -quartz from amorphous silica pellets. The effects of key processing parameters, including target temperature, dwell time, and cooling rate, are systematically investigated to identify the conditions that maximize quartz formation while suppressing competing polymorphs. Quantitative phase analysis by X-ray powder diffraction, complemented by microstructural characterization using scanning electron microscopy, reveals that the initial density of the amorphous precursors plays a crucial role in governing both the thermodynamics and kinetics of the phase transformation. Based on these observations, we propose a phenomenological model in which the density and morphology of the starting silica dictate the crystallization pathway by influencing nucleation and growth mechanisms. Finally, complementary high-pressure/high-temperature experiments provide further insight into the interplay between thermodynamic stability and kinetic constraints, emphasizing their combined influence on the selective formation of α -quartz and offering new perspectives for the mineralizer-free synthesis of quartz-based materials.

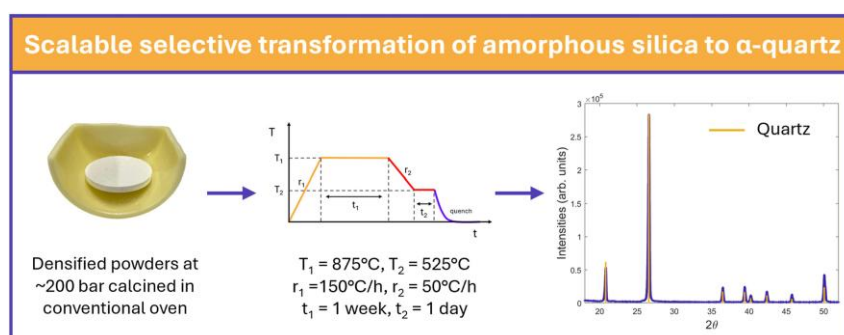


Figure 1. Optimized process for obtaining pure α -quartz from calcination process at ambient pressure without the employment of mineralizers

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CRM nanoscale analysis of Portovesme red muds, SW Sardinia

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The Porto Vesme disposal area in southern Sardinia, Italy, hosts approximately 30 million tonnes of bauxite residue generated during alumina production through the Bayer process. Recent mineralogical and geochemical studies have highlighted the potential of these residues as secondary resources for rare earth elements (REEs) and other critical or valuable elements, including Ga, Sc, and V [1,2]. The mineral assemblage of the investigated residues is mainly composed of hematite, goethite, ilmenite, gibbsite, boehmite, bayerite, anatase, rutile, quartz, sodalite, cancrinite, andradite, calcite, dolomite, and halite. Accessory REE-bearing phases, such as lanthanide-rich phosphates and LREE-bearing ferrotitanates, have also been occasionally detected. From a geochemical perspective, the residues are dominated by Fe₂O₃, Al₂O₃, SiO₂, Na₂O, TiO₂, and CaO, while significant concentrations of Zr, Cr, Ce, V, Sr, Ni, Zn, Ga, and Y are also present. Compared with the original bauxite material, the residues show an enrichment in REEs, with LREEs prevailing over HREEs + Y and Sc. Among the LREEs, Ce is the most abundant element, and chondrite-normalized REE patterns display marked positive Ce anomalies. Nevertheless, the identification of the mineral hosts of REEs and other valuable elements in bauxite residues remains challenging and requires nanoscale investigation. Preliminary TEM observations reveal the frequent occurrence of lamellar Fe–Ti oxide particles containing Ca, Cr, and LREEs. These particles show lattice fringe spacings of approximately 2.70 Å, which may be compatible with a ferrotitanate-type phase. However, determining their exact composition by EDS is complicated by strong spectral overlaps among LREE, Ti, and Cr X-ray lines in the 4.5–6 keV energy range, particularly in Ti-rich domains [3]. Despite these limitations, detailed EDS analyses made it possible to distinguish the La L α peak at 4.65 keV, although it partly overlaps with the Ti K α line at 4.52 keV. Cerium was mainly recognized through the Ce L β emission at 5.26 keV, since the Ce L α line at 4.84 keV is commonly masked by the Ti K β line at 4.93 keV. Chromium, in contrast, was more straightforward to identify through its Cr K α emission at 5.41 keV.

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MS5

New frontiers in X-ray sources for materials science

Evolving Diffraction for Materials Science at the Swiss Light Source

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The synchrotron machine at the Swiss Light Source has undergone a major upgrade to a diffraction limited storage ring, named SLS 2.0[1]. At the same time several beamlines have upgraded or are upgrading the source points and end-stations, to improve capabilities and fully exploit the new characteristics of the synchrotron source.

The Materials Science beamline had already significantly evolved from the initial design and the first upgrade of the source and optics in 2011[2]. The machine upgrade has given new opportunities to further improve the beamline capabilities, stability and operability, and the new emerging beamline, named ADvanced Diffraction for Materials Science (ADDAMS) offers to users the benefit of improved brilliance, flux, workable energy range and more easily tuneable wavelength.

Three diffractometers are operated at the beamline, with a large variety of possible geometries and techniques ranging from single-crystal, surface, grazing-incidence, powder, total scattering, small angle and X-ray reflectivity capabilities. Local devices and sample conditions are available, but speciality equipment for specific experiments can be discussed, designed and realized together with users for special needs. Comparative data between the Materials Science beamline at SLS and the ADDAMS beamline at SLS 2.0 will be shown, along with first experiences with users.

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STAR — A Compact Inverse Compton Scattering X-ray Source for Multiscale Materials Characterisation: Design, Commissioning, and Perspectives

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The development of compact, high-brilliance X-ray sources represents one of the most significant advances in photon science of the past decade, opening the path toward *university-scale* facilities that deliver synchrotron-grade beam quality at a fraction of the cost and footprint of large-scale storage rings. STAR (Southern Europe Thomson Backscattering Source for Applied Research) is a national research infrastructure hosted at the University of Calabria (Rende, Italy) that embodies this paradigm: a compact hard X-ray source based on inverse Compton scattering (ICS), purpose-built for non-destructive, multiscale characterisation of materials.

In the ICS mechanism, a high-brightness relativistic electron beam collides head-on with an intense laser pulse; electrons up-scatter a laser photons to X-ray energies through an inverse Compton scattering process. At STAR, an S-band photoinjector followed by one S band and two C-band travelling-wave accelerating structures brings 5-ps electron bunches to energies up to 165 MeV, where it interacts with a high-power Yb:YAG laser delivering 500 mJ pulses of $\approx 5\text{--}9$ ps duration at a 100 Hz repetition rate. The resulting X-ray beam is quasi-monochromatic, continuously tuneable over the 20–350 keV range, highly collimated, and capable of fluxes up to 10^{11} photons/s within a 1%-10% tunable relative bandwidth. These characteristics position STAR alongside MuCLS (Munich) and ThomX (Paris) as one of only three ICS-based compact user facilities operational or nearing completion in Europe, and as the first such facility in southern Europe.

STAR reached a pivotal milestone on 20 October 2025, when the electron beam was successfully accelerated to 61 MeV and transported to the interaction chamber — the so-called *first spark* marking the transition from construction to experimental commissioning. The facility was officially inaugurated on 28 October 2025 and is currently progressing through the phased commissioning of the accelerator complex and the laser system toward first X-ray emission.

Two dedicated experimental end-stations have been designed to serve the materials science community. The **μ Tomo beamline** is optimised for high-energy X-ray microtomography, enabling three-dimensional reconstruction of large and optically dense samples — including metallic alloys, geological specimens, concrete cores, and composite structures — with micrometric spatial resolution across volumes inaccessible to conventional laboratory sources. The **SoftX beamline** operates at lower photon energies and is tailored for X-ray microscopy and phase-contrast imaging of polymeric, biological, and nanostructured materials, with applications ranging from biomedicine to cultural heritage non-destructive imaging. Both beamlines exploit the intrinsic monochromaticity and low divergence of the ICS beam to achieve contrast and resolution levels complementary to those available at large-scale synchrotron facilities.

The experimental stations are supported by six multidisciplinary service laboratories covering the complete workflow from sample preparation to data analysis: surface science and advanced microscopy (LSAM), sample preparation (LPM), materials characterisation (LCM), biological sample preparation (LPCB), rapid prototyping (LPF), and numerical modelling and simulation (LMSV). This integrated platform enables a seamless research cycle particularly suited to the needs of the materials science and engineering communities.

From a broader perspective, compact ICS sources such as STAR represent a qualitatively new class of X-ray facilities that bridge the gap between laboratory tools and large-scale synchrotron radiation. Their energy tuneability, monochromaticity, and relatively modest infrastructure requirements make them ideally suited for regional research ecosystems, industrial partnerships, and systematic screening campaigns that require extended and flexible beamtime allocations — use cases for which oversubscribed synchrotron beamlines are structurally ill-suited.

This keynote lecture will present the design rationale and the current commissioning status of the STAR source, the performance benchmarks of the accelerator and laser subsystems achieved during early operation, the optical design and readiness of the two beamlines, and the scientific programme envisaged for the first user calls. Particular emphasis will be placed on the potential of compact ICS sources as a new frontier in X-ray generation for multiscale materials characterisation.

Serial crystallography studies on the ferroxidase and biomineralization mechanisms of human H ferritin

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Ferritins are ubiquitous multimeric proteins that form nanocages able to store thousands of iron atoms as oxo-ferric biominerals. In mammals, these 24-mer shells are typically heteropolymers of heavy (H, 183 amino acids) and light (L, 175 amino acids) subunits. H chains catalyze ferroxidase activity by oxidizing Fe(II) to Fe(III) with dioxygen at the ferroxidase site, whereas L chains promote iron biomineralization through a nucleation site on the inner cage surface. A third type, mitochondrial ferritin (MTF), has also been identified in humans and other mammals. Although the catalytic center is structurally highly conserved, the ferroxidase mechanism remains only partly understood. To study this process, we soaked/flash-cooled ferritin crystals under both aerobic and anaerobic conditions upon Fe(II) addition, in combination with multi-wavelength anomalous diffraction for unambiguous iron identification [1-5]. This approach enabled us to observe, for the first time, iron-binding sites in X-ray crystal structures of human H ferritin (HuHF) and human MTF (hMTF), as well as their time-dependent population [2,5]. Transient accessory metal sites near the ferroxidase site were also detected and shown to be essential for reaction turnover [2,5]. Using the same strategy, we characterized the formation of a trinuclear iron-cluster at the nucleation site of human L ferritin (HuLF) and followed its growth into octanuclear clusters on the inner cage surface [6,7]. These species are key intermediates in biomineralization at the protein–mineral interface.

Recently, we introduced a synchrotron serial crystallography (SSX) approach to deeply investigate the catalytic mechanism of HuHF. While earlier structural snapshots had come from crystals measured at cryogenic temperature, the method inevitably limited the reaction landscape that could be observed. Using SSX, we captured the sequential binding of both iron ions at the ferroxidase site and observed dioxygen binding as the second substrate in the redox process. Drop-on-chip experiments captured the earlier stages of the reaction (in the millisecond/second time scale), while longer substrate-exposure times (minutes) revealed later intermediates.

HuHF-Fe(II) complexes were also studied under strict anaerobic conditions to define the pre-reactive state of the active site and validating the sequential substrate binding. Anomalous SSX combined data collected close to the Fe K-edge to generate strong anomalous signals that unequivocally confirmed the observed iron-binding sites. This combination of room-temperature serial crystallography experiments revealed a broader and more dynamic ensemble of intermediates, typically inaccessible to conventional cryogenic approaches.

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From scattering to structural maps: advanced SAXS/WAXS characterization of fibrous materials

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Recent advances in high-brilliance X-ray sources, from state-of-the-art laboratory instruments to next-generation synchrotron facilities, are enabling new possibilities for the structural characterization of complex materials across multiple length scales. Fibrous materials are particularly challenging systems due to their hierarchical organization and intrinsic anisotropy, requiring dedicated scattering and imaging approaches to correlate structure with function.

This contribution highlights the complementary use of Wide-Angle X-ray Scattering (WAXS) and Small-Angle X-ray Scattering (SAXS) for investigating fibrous materials from molecular to mesoscale levels. These techniques provide information on molecular packing, crystallinity, nanoscale organization, and supramolecular architectures. Furthermore, scanning SAXS/WAXS microscopy approaches, enable spatially resolved characterization of structural variations in complex fibrous tissues and biomaterials.

Examples from natural and bioinspired systems, including type I collagen [1,2], *Bombyx mori* silk fibroin [3,4], cellulose-based materials [5], and peptide-derived architectures [6–8] will be discussed to show how SAXS/WAXS analyses can reveal key structural parameters such as molecular orientation, domain size, and characteristic periodicities. These approaches provide a direct link between hierarchical organization and functional properties, supporting the design of biomaterials and technologically advanced fibrous systems.

The continuous development of high-brilliance sources and innovative X-ray methodologies is also contributing to more sustainable material strategies by enabling the valorisation, modification, and recycling of natural polymers and bio-derived resources. Such capabilities are paving the way toward environmentally responsible materials with tailored properties for biomedical, technological, and industrial applications.

Acknowledgements

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Micro-CT study of biomass-induced porosity in clay bricks

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Functionalizing new materials requires a comprehensive characterization of both the amount and the spatial organization of porosity. Traditional techniques, such as Mercury Intrusion Pycnometer (MIP) and N₂ gas pycnometer, provide information on the total porosity and pore size distribution, but are limited by their destructive nature and by the lack of direct three-dimensional information. In contrast, imaging techniques such as micro X-ray computed tomography (μ -CT) enable non-destructive 3D characterization of pore networks, including size, shape, distribution, connectivity, and tortuosity [1,2]. This is particularly relevant for materials where porosity is generated by sacrificial additives, and pore architecture strongly controls functional performance.

In this study, we investigated the porosity of clay bricks produced by adding a sewage sludge-derived biomass (“cake”) obtained via hydrothermal treatment (220 °C, 30 min) at the Cassano d’Adda (Milan, Italy) demonstration plant. Samples were prepared by replacing 10, 30 and 50 wt% of clay with cake and fired at 900 °C in an industrial kiln. The clay used in this study, hereafter labelled as VC, was quarried at Cascina Groppello (Valenza, Italy) and characterised by XRF, TG-DTA and XRPD. It contains mainly quartz, albite, microcline and muscovite, as non-clay minerals, accompanied by kaolinite, smectite, and illite.

Increasing cake content significantly reduces bulk density due to pore formation induced by thermal decomposition of organic matter. Conversely, real density increases, suggesting densification of the solid phase, possibly associated with the formation of calcium iron phosphate phases enhancing sintering. μ -CT confirmed the increase in porosity and, crucially, provided insights into pore architecture, revealing spatial distribution, homogeneity and connectivity of the pore network. The ability to distinguish between isolated and interconnected porosity is key to understanding transport properties and functional performance.

These results highlight the value of combining bulk techniques with μ -CT to establish robust links between processing, 3D microstructure and properties, supporting the optimization of sustainable sludge-based porous ceramics.

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Structural and morphological characterization of bio-inspired superhydrophobic coatings for sustainable agricultural applications

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Physiological alterations in agricultural crops represent an extremely negative consequence of the rise in temperature and humidity levels associated with climate change. These environmental conditions create a favorable environment for the proliferation of various pathogens, such as fungi, responsible for diseases like powdery and downy mildew, currently affecting crops such as grapevine and tomatoes.

In this regard, SafeWax coating has been identified as a promising and sustainable alternative to traditional synthetic or heavy-metal fungicides. It was developed to satisfy the requirements established by the European Green Deal concerning different crop protection strategies, whose aim is a 50% reduction of the use of harmful pesticides by 2030. A key advantage of this technology lies in its composition, being made of fatty acids (FA) derived from fruit and vegetables waste, such as stearic or palmitic acid, promoting circularity of resources and allowing to obtain a bio-based and bio-degradable formulation [1]. The aim of SafeWax is to modify the leaf surface exploiting fast FA crystallization, mimicking the natural cuticles present in some plants like lotus leaf or cauliflower, which are responsible for superhydrophobic and self-cleaning properties, as well as protection from dehydration and UV radiation.

SafeWax coating replicates a similar passive protective function by a solvent-based deposition method using a spraying technique. The selected solvents already used in agriculture are diethyl ether, ethanol and ethyl acetate.

Surface's roughness and morphology, observed by Scanning Electron Microscopy (SEM), prevent pathogens from adhering to the leaf and facilitate water droplets to roll off minimizing the level of moisture and carrying microbes away. Complementary X-ray scattering techniques, including synchrotron-based—Grazing Incidence Wide-Angle X-ray Scattering (GIWAXS), have revealed a hierarchical crystalline structure of the coating with marked texturing and have allowed the determination of the composition of the crystalline phase.

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MS6

Crystal growth, from fundamentals to applications

Experimental and theoretical analysis of Biomineral-Biomolecules surface interactions

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Mineral surfaces represent active interfaces controlling the exchange of matter and energy between the geosphere, hydrosphere and biosphere. Understanding the molecular mechanisms governing mineral–environment interactions is therefore essential not only for mineralogy and environmental sciences, but also for biotechnology, biomaterials research and studies on the origin of life [1].

This contribution presents a multidisciplinary investigation of the interactions occurring at the interface between layered silicate minerals and aqueous or biological molecular species, focusing on clinocllore, a chlorite-group phyllosilicate characterized by the alternation of chemically distinct brucite-like and talc-like layers. By combining atomic force microscopy, scanning probe techniques and first-principles Density Functional Theory simulations, the physical and chemical properties of mineral surfaces have been characterized from the nanometric down to the atomic scale, providing quantitative information on surface morphology, electrostatic potential, adsorption sites and intermolecular interactions. The results demonstrate that clinocllore surfaces are not passive substrates, but highly selective interfaces capable of driving the adsorption and organization of a wide range of molecular systems. Amino acids and short peptides preferentially interact with specific surface domains, forming ordered assemblies stabilized by hydrogen bonding and electrostatic interactions [2]. Nucleotides, RNA and DNA exhibit selective adsorption and self-organization behaviours, revealing the capability of mineral surfaces to act as molecular templates [3]. More recently, atomistic simulations have clarified the interaction mechanisms between phospholipids and chlorite surfaces, highlighting the potential role of layered silicates in the stabilization of membrane-like structures [4]. Complementary studies on water adsorption further elucidate the fundamental role played by hydration processes in modulating surface reactivity and molecular recognition [5, 6].

These investigations provide a unified picture of mineral–biomolecule interactions across different levels of molecular complexity, showing how the structural and electrostatic heterogeneity of clay mineral surfaces can influence adsorption, self-assembly and molecular organization. Such findings contribute to a deeper understanding of environmental interface processes and open new perspectives for applications ranging from nanobiotechnology and biomaterials engineering to prebiotic chemistry and the study of the emergence of complex molecular systems on the early Earth.

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Halide Perovskite Nanocrystals Heterostructures

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Halide perovskite semiconductors can merge the highly efficient operational principles of conventional inorganic semiconductors with the low-temperature solution processability of emerging organic and hybrid materials, offering a promising route towards cheaply generating electricity as well as light. Following a surge of interest in this class of materials, research on colloidal halide perovskite nanocrystals (NCs) has gathered momentum in the last decade. This talk will highlight several findings of our group on their synthesis, for example our recent studies on the influence of various factors on the growth of perovskite NCs, which can lead to the formation of NCs with peculiar shapes (for example hollow structures) and to NC heterostructures (for example CsPbBr₃/PbS, CsPbBr₃/Pb₄S₃B₂ and Bi/Bi₁₃S₁₈Br₂ heterostructures) by promoting/suppressing the heterogenous nucleation of selected materials. Detailed structural and optical investigations of these heterostructures will be covered, and their potential in a set of photocatalytic reactions will be discussed. I will also present how we can computationally predict the feasibility of such heterostructures, based on interfacial matching, using software tools developed in collaboration with other groups.

Manganese substitution in monetite: influence of the synthesis route on phase evolution and crystal structure

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Monetite (DCPA, CaHPO_4) is a calcium phosphate characterized by higher solubility and resorption than more stable calcium phosphates [1]. Ionic substitution can affect phase stability, crystal structure and morphology in calcium phosphates; manganese is a biologically relevant trace element [2]. While Mn-substituted hydroxyapatite and β -tricalcium phosphate have been extensively investigated, Mn incorporation in monetite has not yet been reported [3]. Furthermore, the influence of the synthesis pathway on Mn incorporation efficiency, phase evolution and structural modifications remain unexplored.

In this work, Mn-containing monetite was prepared by direct precipitation and a brushite-mediated route involving the synthesis of Mn-substituted brushite (DCPD) [4]. Nominal Mn substitution levels were selected to obtain comparable Mn levels in the final monetite phases. Phase composition, crystal structure and morphology were analysed by X-ray powder diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), inductively coupled plasma atomic emission spectroscopy (ICP-AES) and scanning electron microscopy (SEM).

XRD analysis revealed that direct synthesis did not yield phase-pure monetite, leading instead to biphasic DCPA/ β -TCP systems. In contrast, Mn-substituted DCPD was obtained as single-phase brushite and converted into phase-pure Mn-containing monetite after thermal treatment. Profile fitting confirmed a corresponding decrease in lattice parameters and unit-cell volume consistent with the substitution of Ca^{2+} by the smaller Mn^{2+} ion. FTIR results supported the structural modifications observed by XRD, while SEM highlighted significant differences in crystal morphology (**Figure 1**).

These results highlight the key role of the synthesis pathway in controlling phase stability, Mn incorporation and crystal structure. The brushite-mediated route provides phase-pure Mn-substituted monetite and highlights the influence of the synthesis pathway on phase evolution and crystal structure.

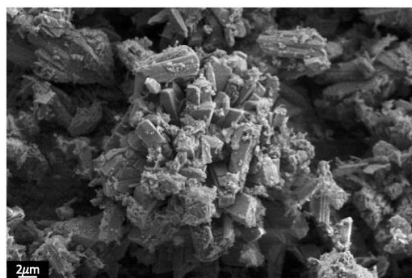


Figure 1. SEM of Mn-substituted monetite crystals.

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From Benzene to Triphenylene: Atomistic Mechanisms of Early Graphene Nucleation on Cu(111) During Low-Temperature CVD

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Graphene is a key material for advanced technologies owing to its exceptional electrical, optical, and mechanical properties. These properties make it highly attractive for applications including energy devices, flexible electronics, and optoelectronic systems. Achieving high-quality chemical vapour deposition (CVD) graphene at reduced temperatures with the aid of liquid aromatic hydrocarbons (e.g., benzene, toluene, etc.) as carbon precursors is crucial to enable scalable integration on temperature-sensitive substrates [1-4].

Density functional theory (DFT) calculations were employed to elucidate the elementary mechanisms governing the initial stages of graphene nucleation during low-temperature CVD (LT-CVD) on Cu(111). Attention is devoted to the evolution of benzene-derived species, from precursor activation to radical condensation and nucleation of extended aromatic structures. Following benzene dehydrogenation, phenyl radicals represent the dominant surface intermediates. Their evolution is dictated by the competition between multiple dehydrogenation and radical coupling, while the progressive depletion of surface hydrogen shifts the reaction environment toward carbon-rich conditions that favour irreversible C–C bond formation.

A complete reaction network leading to triphenylene ($C_{18}H_{12}$), proposed as a representative graphene nucleus, is resolved. Nucleation proceeds through sequential formation of biphenyl and ortho-terphenyl intermediates followed by dehydrogenation-assisted ring closure. Radical recombination is generally favored over extensive dehydrogenation, indicating that C–C coupling drives growth under LT-CVD conditions. Steric effects, molecular topology, and adsorption geometry strongly influence pathway selectivity and reaction energetics. These results provide atomistic insights into the mechanisms governing early graphene nucleation, and highlight the central role of aromatic intermediates in enabling controlled low-temperature growth of graphene during CVD.

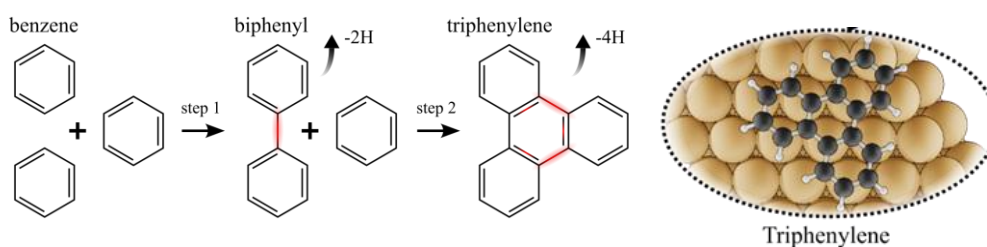


Figure 1. Schematic representation of triphenylene ($C_{18}H_{12}$) formation on Cu(111) from three adsorbed benzene molecules.

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Co-crystals formation from low melting eutectic mixtures

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Some ingredients relevant to human health, environment protection, and nutrition are liquid at ambient conditions; widely known examples are terpenoids and phenolic derivatives used as natural antioxidants such as carvacrol, eugenol, eucalyptol, and valproic acid. Recently, cocrystallization has proven to be a powerful method to control the release of these liquid ingredients in the environment, by engineering of ad hoc intermolecular interactions which strengthen or weaken the tendency of the ingredient to be retained inside the solid matrix [1]. Recent investigations have demonstrated that low-melting mixtures formed upon contact of solid or low melting components can play an active role in the formation of multicomponent crystalline products, enabling the preparation of such materials by direct mechanochemical synthesis [2]: a crucial aspect of some of these transformations in cocrystal formation, is the occurrence of eutectic liquid phases. These liquid intermediates enhance mass transport and molecular mobility, overcoming limitations inherent in solid–solid reactivity. To support the rational design of such processes, PoEM (Predictor of Eutectic Mixtures) has been introduced as a novel predictive tool for estimating melting-point variations in binary mixtures [3], and is freely available online. Complementary to predictive modelling, time-resolved in situ monitoring has offered direct experimental evidence of liquid-phase involvement during cocrystallization of binary mixtures. Such studies reveal that the emergence of eutectic liquids can initiate and accelerate cocrystal formation, supporting mechanistic hypotheses and enabling a deeper understanding of reaction pathways. By integrating predictive tools such as PoEM [4] with advanced in situ techniques, researchers can more effectively design, control, and optimize new functional materials.

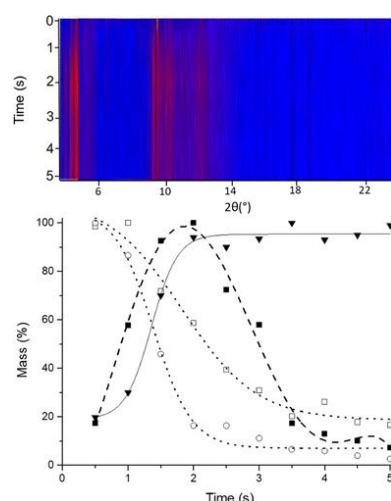


Figure 1. Time resolved in situ monitoring of the formation of a co-crystal through a liquid intermediate.

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Growth of strain-free InGaAs/InAlAs QDs via LDE as C-band single photon emitter

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Strain-free semiconductor quantum dots (QDs) have attracted a significant interest for telecom quantum photonics. In this study, In_{0.7}Ga_{0.3}As QDs emitting in telecom C-band, were fabricated on GaAs(111) substrate via local droplet etching on thin In_{0.7}Al_{0.3}As metamorphic buffer layer (MMBL). The (111)A orientation enables efficient strain relaxation by using thin MMBL while preserving natural C_{3v} symmetry to produce highly symmetric QDs [1]. AFM analysis confirmed triangular pyramidal nanoholes (fig.1a), which were subsequently filled with In_{0.7}Ga_{0.3}As to form telecom-emitting QDs. The QD geometry and electronic structure were optimized using k·p and envelope-function calculations. Photoluminescence measurements demonstrated emission in the range of 1388 to 1553 nm (fig.1b), in consistent with theoretical simulations. The fabricated QDs exhibited single-photon emission with a measured $g^{(2)}(0)$ value of 0.141 ± 0.027 . In addition, time-resolved photoluminescence measurements revealed excitonic lifetimes in the range of 1.3–1.9 ns, demonstrating the potential of strain-free InGaAs/InAlAs QDs as telecom C-band single-photon emitters in quantum telecommunication applications.

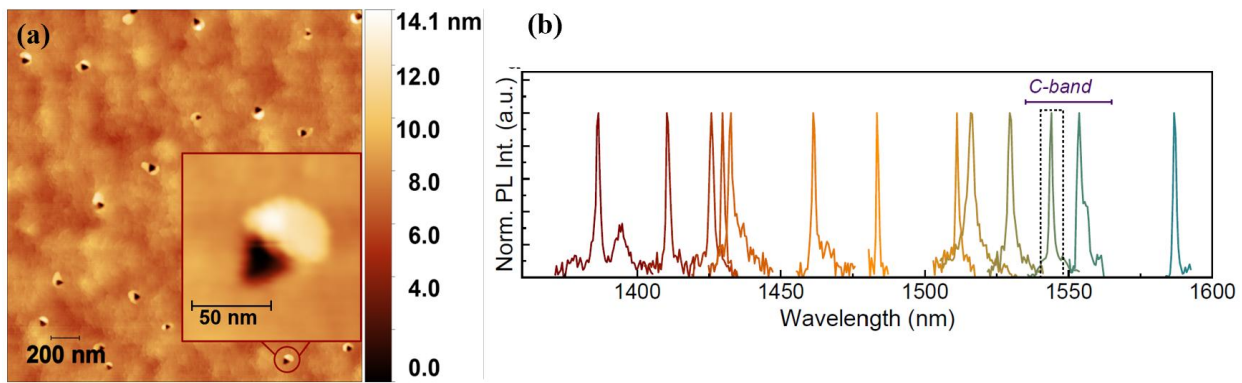


Figure 1: (a) AFM image of nanoholes with 1024×1024 resolution at 2.5×2.5 μm² scale with Inset of nanhole having C_{3v} symmetry
(b) μ-PL measurements of the fabricated QDs exhibiting the emission in C-band

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MS7

**The role of applied mineralogy in bridging resources,
materials, environment and health**

Unlocking the potential of 2:1 clays as supplementary cementitious materials

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The transition to low-carbon binders like Limestone Calcined Clay Cements (LC³) is increasingly focused on expanding beyond kaolinitic clays to include widely available 2:1 phyllosilicates, such as smectites and micas. From a crystallographic perspective, these clay minerals are defined by a TOT layer structure, consisting of one octahedral sheet (typically containing Al³⁺, Mg²⁺, or Fe^{2+/3+}) sandwiched between two tetrahedral silicate sheets. This arrangement differs significantly from the 1:1 (TO) structure of kaolinite, influencing how these minerals respond to activation [1].

Thermal activation (TA) is the primary method to induce pozzolanic reactivity by triggering the dehydroxylation of the octahedral sheet. This process results in structural disorder and partial amorphization; however, unlike kaolinite, 2:1 structures often do not reach a state of total collapse after heating, which can limit the availability of reactive aluminium and silicon. Alternatively, mechanochemical activation (MCA) has emerged as a powerful tool for these minerals. MCA uses high-energy milling to cause severe structural disruption, create reactive sites, and achieve nearly complete amorphization, which is particularly effective for micas like muscovite [2].

Research indicates that smectitic clays (e.g. bentonites) can exhibit "moderate" to "high" pozzolanic reactivity after calcination, with R³ test heat release values exceeding 190 J/g [3]. Furthermore, mechanochemically activated muscovite outperforms its thermally activated counterparts, reaching significantly higher 28-day compressive strengths.

By leveraging an understanding of phyllosilicate crystallography and optimized activation strategies, 2:1 clays can be successfully unlocked as high-performance, sustainable constituents for the cement industry.

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Understanding Carcinogenic Mineral Fibers through a Mineralogical-Crystallographic Lens

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The mechanisms of carcinogenesis induced by mineral fibres remain poorly understood. To fully elucidate these in vivo toxicity pathways, an interdisciplinary approach integrating crystal-chemical, biochemical and toxicology data is essential. This presentation demonstrates the pivotal role of mineralogy-crystallography in properly assessing the parameters that dictate the fibres' toxicity and carcinogenicity potential. Crucially, this work emphasizes the distinct behavior exhibited by amphibole asbestos, serpentine asbestos, and zeolitic erionite within biological systems, highlighting how their specific structural features govern diverse surface reactivities and cellular responses [1-6]. Adopting a mineralogical-crystallographic lens to link atomic-scale structures to biological reactivity provides key data to refine toxicological models, ultimately supporting the development of targeted cancer prevention strategies and innovative therapies worldwide.

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Structural characterization of the aluminosilicate network in alkali-activated binders from thermally treated mineral wool with ssNMR

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Mineral wool (MW), as the most widely used insulating material, is a relevant fraction of construction and demolition waste (2.5 million t/year in EU countries in 2020). Because of the potential risks for health, MW waste is classified as possibly carcinogenic in many EU countries; moreover, landfill disposal is problematic because of the large volumes involved, due to its low apparent density. A recycling opportunity is offered by a patented innovative process [1] consisting in melting packages of exhausted MW in a furnace to obtain an inert glass material. The valorization of this material is crucial for the sustainability of the process. To this aim, in this work the glass is recycled as solid precursor in alkali-activated binders (AABs). The advantages are manifold: the MW waste is converted into a non-hazardous material; landfill disposal is avoided; the exploitation of natural resources is reduced by producing a binder that can partly substitute Portland cement, and possesses lower environmental footprint. Two AABs formulations have been produced with NaOH and sodium silicate activating solutions. Two distinct solid precursors derived from the thermal treatment of MW were employed: one derived from stone wool and one derived from glass wool, given their different reactivity in alkaline environments [2,3]. Samples were formulated with the same water-to-binder ratio of 0.35, sonicated and cured for 3 days at 50 °C after mixing. ²⁷Al solid state NMR spectroscopy allowed for the crystal-chemical and structural characterization of the formed amorphous aluminosilicate network, quantify the participation to the reaction of the solid precursor (of the order of 50-60%) and understanding the very properties of the material. Results were supported by point chemical analysis of the matrix, which also pointed to a Ca-rich aluminosilicate network, given the high Ca content of the glass. Although the network and its degree of polymerization seemed more developed in case of AABs obtained from the stone wool, a high compressive strength (> 60 MPa) and low porosity (< 10%) resulted similar in all samples. This was ascribed to the advantage of using the more isometric particles of the precursor, providing better mechanical support and pore refinement effect, reduced particle clustering (observed in case of fibers) and absence of the hydrophobic coatings commonly applied to fibers, in comparison with samples produced by recycling in similar matrices untreated MW fibers [2,3].

Acknowledgements

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Resolving symmetry in complex clinker systems: the crystallographer's role in cement plant decision-making

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Applied mineralogy plays a central role in modern cement production, where crystallographic information is increasingly used to support real-time and economically relevant decisions. In Portland cement clinker, the major phases C_3S , C_2S and C_3A occur in multiple polymorphic forms whose identification provides insight into kiln conditions, cooling history, hydration reactivity, early strength development and sulfate demand. The ability to distinguish these polymorphs is therefore directly connected to process control, material performance and environmental sustainability.

This work focuses on the crystallographic challenge of resolving different symmetries in the crowded diffraction patterns of commercial clinker samples. C_2S exhibits several temperature-dependent polymorphs, including α , $\alpha'H$, $\alpha'L$, β and γ forms, while C_3S occurs mainly as monoclinic M1 and M3 polymorphs [1] stabilized by minor element substitution. C_3A polymorphism follows a different logic, being largely controlled by alkali incorporation, which stabilizes cubic or orthorhombic variants relative to pristine monoclinic C_3A . Although these distinctions are structurally meaningful and industrially relevant, their detection by routine laboratory X-ray diffraction is complicated by pseudosymmetry, peak overlap and the coexistence of multiple clinker phases.

We compare high-resolution synchrotron powder diffraction data from commercial clinker samples with laboratory XRD data collected under both industrially representative and optimized high-resolution conditions. This approach allows us to assess the theoretical and practical limits of polymorph discrimination and quantification in routine cement analysis.

By drawing a line between what is crystallographically observable and what is industrially actionable, this study demonstrates how laboratory diffraction can be used to inform cement-plant decisions. The results emphasize the role of the crystallographer as an interface between structural science and production strategy, where improved phase and polymorph analysis can contribute to better resource efficiency, reduced environmental impact and more sustainable material manufacturing.

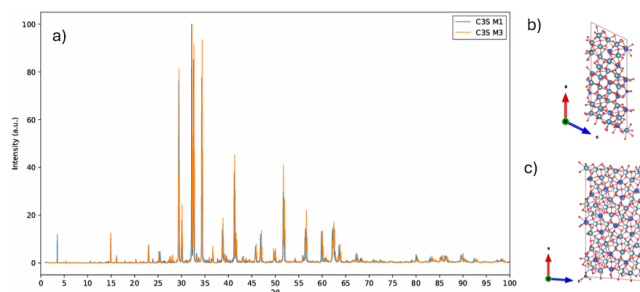


Figure 1. (a) Powder diffraction patterns of the C_3S M1 and M3 phases, showing a high degree of similarity between the two patterns. (b) C_3S M1 crystal structure viewed along the **b** axis. (c) C_3S M3 crystal structure viewed along the **b** axis.

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Nano Ferrites via Sol-Gel synthesis for Pb and Ni adsorption: analysis on Ion-Structure relationships

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Nano iron oxides are known for their promising adsorption capacity toward heavy metals. This property has driven interest in their application for water remediation and environmental protection [1]. However, the fate of metals after they become incorporated into the crystalline lattice is not well understood. To address this, the present study uses nano-sized cubic ferrites (spinel structure) to understand how heavy metals interact with and are retained by these materials.

Because natural ferrites typically contain pre-existing foreign cations that reduce their adsorption capacity, synthesized materials were preferred. The SOL-GEL method was chosen because it produces nanosized particles ideal for cation capture in a cost-effective way. Heavy metals were introduced in two ways: 1) by adsorbing them from solution onto synthesized powders, and 2) by adding the metal ions directly to the SOL-GEL reagents so they become incorporated during synthesis. For the adsorption experiments, a calcination step (200 °C for 2 hours) was applied to better lock cations into the structure.

Results showed high adsorption efficiency from aqueous solutions for lead (see Fig.1), while uptake was lower for other cations such as nickel, despite this latter having an ionic radius comparable to Fe. When heavy metals were present among the synthesis reagents, cation segregation increased. To determine the location of these cations within the structure, pair distribution function (PDF) analyses from both X-ray and neutron diffraction were performed. Because some questions remained, X-ray absorption spectroscopy (XAS) was also used to track heavy-metal behaviour during spinel nano-powder synthesis.

Future work will examine how incorporation efficiency depends on synthesis conditions and whether competitive effects arise when powders are exposed to mixed heavy-metal solutions.

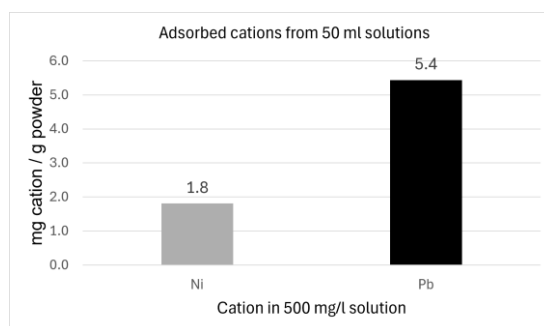


Figure 1. Differences in Ni and Pb adsorption from water solution of 500 mg/l

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Using three-dimensional electron diffraction and TEM to address environmental issues

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It is not obvious to realize how a complex analytical technique such as three-dimensional electron diffraction (3DED), usually applied for the characterization of high-tech nanomaterials and organic compounds, can contribute to answering environmental issues. The greatest difficulty in studying environmental matrices lies in the fact that they are composed of a multitude of different phases, characterized by different chemistries and morphologies. Grain size is often at the limit of resolution for light microscopy, and sometimes even for scanning electron microscopy (SEM). Bulk analyses can reveal the presence of hazardous elements or minerals, but they cannot tell where these elements are hosted or what the morphology of the hazardous phases is.

The transmission electron microscope (TEM) is not just a powerful magnifying glass, but a versatile instrument capable of providing morphological, structural and chemical information from a nanometer sample volume. By combining the chemical information obtained through energy dispersive spectroscopy (EDS) and the structural information obtained through 3DED, it is possible to unequivocally identify all components of any matrix, regardless of the size of the individual grains. Here we illustrate two case studies related to the presence of potentially pathogenic phases in industrial by-products and in the natural environment.

The first case concerns the localization of chromium in industrial waste deriving from tanning processes (KEU) [2]. KEU is a polyphasic micro-crystalline compound obtained through a pyrolysis process that aims to inert Cr in the spinel-type phases. The use of KEU as a filler, however, showed that this material releases Cr⁶⁺ over time, even though this ion is not present in the initial material. TEM analyses showed that chromium is mainly concentrated in chromium oxyhydroxide nanoparticles/nanoflakes, potentially more susceptible to weathering alterations than the expected spinel-type phases.

The second case study concerns the characterization of fibrous minerals in rocks and soils from different locations in New Zealand [3]. These matrices host a variable content of aluminosilicate fibers, with length and diameter potentially dangerous for the respiratory system. X-ray powder diffraction and SEM revealed the presence of erionite, a zeolite listed among carcinogenic agents to humans. TEM and 3DED analysis confirmed the presence of erionite in most samples, but also detected other fibrous minerals, actually not listed as carcinogenic but with morphologies and chemistry not dissimilar from those of erionite (e.g., mordenite, natrolite and mesolite). The analysis also revealed that fibrous erionite is depauperated in the overall cation content and sometimes unusually enriched in Mg, and that, especially in soils, the zeolite framework can be extremely distorted. These characteristics can hinder mineral recognition via bulk analyses or SEM. In general, the combination of 3DED and other TEM-related methods provides in-depth analysis of complex environmental matrices, where unrecognized or modified carcinogens can hide from standard and quick analyses.

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MS8

Protein structures for modeling, function and drug design

Cryo-EM of coagulation factor V

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Coagulation factor V is a key component of the response to vascular injury and the onset of thrombotic complications, acting both as a procoagulant and anticoagulant protein. Recent cryo-EM structures have revealed most of the architecture of this large (2196 residues) protein free [1] and bound to physiological targets [2-4], thereby rationalizing biochemical and clinical data [5]. However, the structure of the large (836 residues) B domain that maintains factor V in its inactive form remains unresolved and constitutes the focus of current cryo-EM studies.

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Structure-guided inhibition of the bacterial SOS response as an anti-virulence strategy

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The bacterial SOS response is a conserved DNA-damage stress pathway regulated by the sensor RecA and the transcriptional repressor LexA. Upon genotoxic stress, RecA oligomerizes on single-stranded DNA and stimulates LexA autoproteolysis, thereby derepressing SOS genes encoding error-prone polymerases, recombinases, virulence factors, and antibiotic resistance determinants. Our work aims to elucidate the molecular mechanisms governing RecA–LexA interplay and to develop potent inhibitors of SOS signaling as a strategy to counter bacterial adaptation and virulence. Using X-ray crystallography and cryo-EM, we solved the atomic structures of the *Pseudomonas aeruginosa* LexA autoproteolytic domain, the oligomeric RecA filament, and their complex, revealing key structural determinants of LexA proteolysis [1]. Leveraging these insights, we generated anti-LexA nanobodies via llama immunization and phage display. These nanobodies potently inhibit LexA autoproteolysis in vitro, as demonstrated by biochemical assays and surface plasmon resonance binding studies. SOS suppression was functionally validated in antibiotic-treated bacterial cultures using RT-PCR and GFP-reporter assays [2]. Building on this structural and biophysical framework, we further engineered nanobody variants with enhanced affinity and inhibitory activity, currently under characterization, collectively establishing a promising anti-SOS strategy as a novel antimicrobial approach.

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Ring-Interface Plasticity and Client Recognition in a Stress-Activated Peroxiredoxin Revealed by Cryo-EM

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Typical 2-Cys peroxiredoxins (Prxs) moonlight as peroxidases and stress-activated holdases through conformational and oligomeric remodeling [1,2]. Although high-molecular-weight assemblies are linked to holdase activity, the structural basis of client recognition remains unclear. We investigated *Schistosoma mansoni* peroxiredoxin 1 (SmPrx1), a protein involved in parasite adaptation to the human host environment during infection [3,4]. A C48D variant, mimicking oxidative perturbation of the peroxidatic cysteine, was characterized by cryo-EM, light scattering, and thermal-shift assays. SmPrx1C48D reduced heat-induced aggregation of citrate synthase (CS) and partially stabilized the client protein. Cryo-EM identified a single ring-shaped decamer, a stacked double decamer, and a CS-bound complex. The decamer adopts a locally unfolded active-site conformation with increased flexibility of catalytic regions. In the double decamer, ring stacking is preserved through remodeling of the ring–ring (R) interface with respect to the X-ray structure obtained under acidic stress [3], revealing interface plasticity while maintaining the overall architecture under conditions mimicking oxidative stress. The SmPrx1C48D–CS complex shows putative client density at peripheral rim/end-cap regions, supporting a binding mode distinct from the luminal sequestration reported for other Prxs [5]. These findings suggest that double-decamer formation expands client-binding surfaces and contributes to holdase activity. We propose that order-to-disorder transitions coupled to R-interface remodeling drive the peroxidase-to-holdase conversion of SmPrx1 (Figure 1). These results provide insights into the structure–function relationships underlying Prx moonlighting. Moreover, oligomerization and client-binding interfaces emerge as potential targets for structure-guided inhibitor design, providing an alternative to conventional active-site targeting.

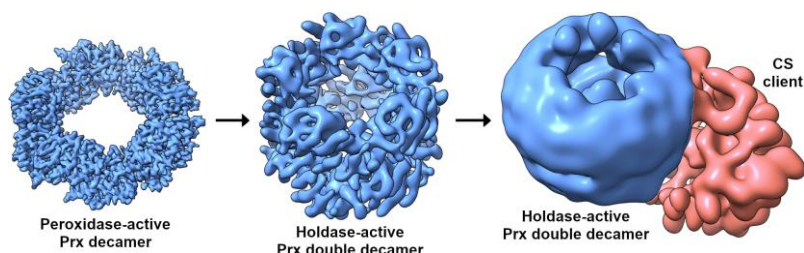


Figure 1. Order-to-disorder transition coupled to holdase activation in SmPrx1C48D

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Comparative Molecular Dynamics Study of MPS I-Associated Mutations in α -L-Iduronidase

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Mucopolysaccharidosis type I (MPS I) is a rare autosomal recessive lysosomal storage disorder caused by mutations in the **IDUA** gene, which encodes α -L-iduronidase.¹ This is a **glycosylated** enzyme that degrades glycosaminoglycans (GAGs).² Enzymatic deficiency leads to GAG accumulation in lysosomes, disrupting cellular function and causing multisystemic damage with clinical severity ranging from mild (Scheie) to severe (Hurler).³

Among pathogenic IDUA variants, **L490P** and **P533R** are commonly linked to attenuated forms. These mutations retain partial α -L-iduronidase activity, reducing systemic severity but still causing subtle neurological impairments, such as cognitive delays and attention deficits, due to GAG accumulation in the central nervous system (CNS).³

We employed unbiased molecular dynamics (MD) simulations to compare the wild-type (WT) protein with the **L490P** and **P533R** mutants, aiming to uncover potential microswitches that lead to protein misfolding. The simulations were performed at neutral and acidic pH to mimic the microenvironment of the endoplasmic reticulum (ER) and of the lysosomes, where the enzyme exerts its function.

We performed three replicates of 1.6 μ s (400 ns equilibration and 1.2 μ s production) for the WT and the two mutants at **pH 7.5** and **4.5** (6 systems, 18 simulations). The root mean square deviation (**RMSD**) and fluctuation (**RMSF**) analyses performed to evaluate the protein's secondary-structure stability did not reveal major differences between the WT and the two mutants. We also employed **RMSF** analyses of the **N372-linked glycan** for all systems at both pH values.

Interestingly, the glycan of **L490P** and **P533R** showed lower fluctuation only at acidic pH compared to the WT. We, therefore, proceeded with **cluster analyses** of the WT and mutants at acidic pH to characterize the different conformational states experienced by the glycan in the mutants compared to the WT (**Figure 1; representative frames**). Finally, the **hydrogen bond** analysis of the **N372-linked glycan** with the enzyme in the simulation of the **P533R** mutant revealed interactions with specific amino acidic sites that are not present in the other systems, responsible for the lower fluctuation of the glycan. This may be an indication of the enzyme's inability to degrade GAGs.

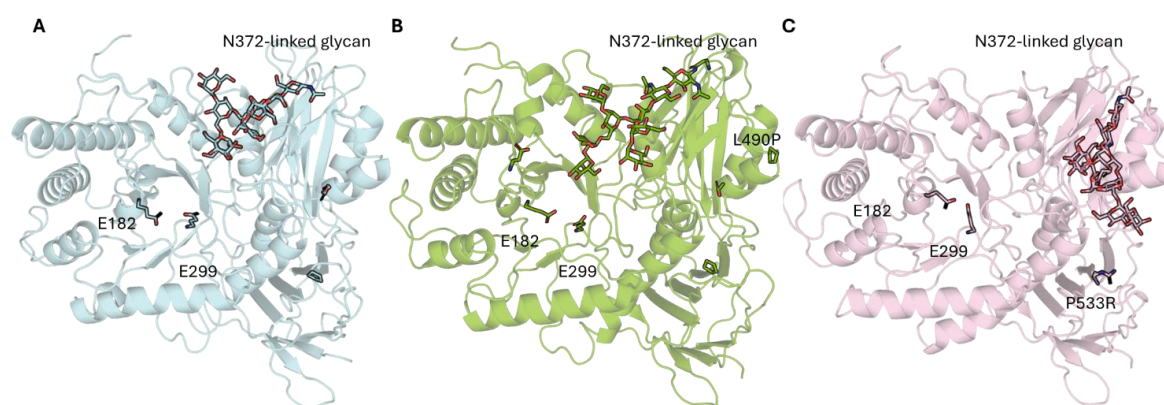


Figure 1. Representative frames of the most populated cluster of each system trajectory at pH 4.5 showing the different conformational space occupied by the N372-linked glycan, namely **A**) WT (in cyan), **B**) L490P (in green), and **C**) P533R (in pink). The catalytic residues and mutated residues are labelled and shown in sticks.

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Structural basis of selectivity in small molecules targeting carbohydrate binding site in galectins

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Galectins are a family of β -galactoside-binding proteins involved in a wide range of physiological processes, including apoptosis, immune regulation, tissue homeostasis, and embryonic vascular development. Sixteen galectins have been identified in mammals, functioning in both intracellular and extracellular compartments. Owing to their involvement in numerous pathological conditions, including inflammation, infection, fibrosis, and cancer, galectins have emerged as attractive therapeutic targets [1,2]. Among them, Galectin-3 (Gal-3) has attracted particular attention because of its central role in tumor progression and fibrotic diseases. Gal-3 consists of an N-terminal domain responsible for oligomerization and a C-terminal carbohydrate recognition domain (CRD), which mediates glycan recognition and regulates diverse biological processes, including apoptosis, cell proliferation, pre-mRNA splicing, cell adhesion, migration, and inflammatory signaling [3,4]. Most Gal-3 inhibitors developed to date are carbohydrate-based or glycomimetic ligands targeting the conserved CRD [5,6]. Although these compounds can achieve high affinity, the conserved architecture of the binding site has limited the development of selective inhibitors, while the carbohydrate scaffold itself restricts the accessible chemical space and often compromises drug-like properties. Consequently, there is a pressing need to identify both molecular determinants of Gal-3 selectivity and novel non-carbohydrate chemotypes capable of selectively targeting this lectin. Here, we combined high-resolution X-ray crystallography, isothermal titration calorimetry (ITC), fragment-based crystallographic screening, and virtual screening to investigate the structural basis of Gal-3 recognition and discover new ligand scaffolds. In parallel, mono- and multivalent carbohydrate-based ligands were used as structural probes to dissect the molecular determinants of Gal-3 recognition by comparison with Galectin-1 (Gal-1) and Galectin-7 (Gal-7). Our structural and thermodynamic analyses identify a Gal-3-specific cation- π interaction between aromatic ligands and residue R144, an interaction that cannot be established in Gal-1 because the corresponding residue is absent or in Gal-7 because the equivalent arginine is conformationally constrained by a stable electrostatic interaction. While previous studies associated R144 primarily with enhanced ligand affinity, our crystallographic, mutagenesis, and binding data demonstrate that this interaction is also a key determinant of Gal-3 selectivity. Moreover, the strength of the cation- π interaction correlates with the electronic properties of the aromatic substituents, providing a structural explanation for the observed structure-activity relationships.

Collectively, these findings establish R144 as a unique selectivity hotspot within the Gal-3 carbohydrate-binding site and provide the first direct experimental evidence that R144-mediated cation- π interactions govern both ligand potency and galectin selectivity. In addition, our fragment- and virtual-screening campaigns expand the chemical space available for Gal-3 inhibition by identifying non-carbohydrate starting points for future optimization. Together, this work provides a structural and mechanistic framework for the rational design of next-generation, selective, drug-like Gal-3 inhibitors.

Acknowledgements

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Structure-function insights into phage-derived depolymerases targeting multidrug-resistant *Klebsiella pneumoniae*: potential antibiotic adjuvants

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Antimicrobial-resistance (AMR) stands nowadays as a pressing concern in public health. A group of six pathogens is named with the acronym ESKAPE because of their ability to evade the biocidal action of traditional clinically used antibiotics [1,2]. In many of these pathogens, cell-envelope polysaccharides constitute a major determinant of antimicrobial resistance, providing a protective barrier against environmental stressors, host immune responses, and antimicrobial compounds. Structural characterization of the molecular determinants underlying these protective mechanisms is essential for understanding the basis of AMR and for developing new therapeutic strategies.

Phage-associated depolymerases are large trimeric multi-domain proteins that enable bacteriophages to overcome the polysaccharide barriers surrounding their bacterial hosts. By degrading these protective structures, depolymerases can enhance the susceptibility of antibiotic-resistant bacteria to conventional antimicrobial treatments [3]. Consequently, recombinant depolymerases have emerged as promising tools in biotechnology and medicine, particularly as potential antibiotic adjuvants [4].

Using X-ray crystallography, we have determined the structures of several phage spike depolymerases targeting the clinically relevant nosocomial pathogen *Klebsiella pneumoniae*. In collaboration with the University of Wrocław (Poland), we further investigated their catalytic mechanisms through mutational analyses and identified truncated variants that retain enzymatic activity while exhibiting a reduced molecular size [5,6]. These studies provide valuable insights into the structure–function relationships governing depolymerases activity. A detailed understanding of their structural organization and catalytic mechanisms will be instrumental for the rational engineering of recombinant depolymerases with improved properties, including reduced size and broader substrate specificity, ultimately supporting the development of innovative strategies to combat multidrug-resistant bacterial infections.

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MS9

Advanced materials for Sustainable Energy

Deciphering Selective Oxidation Chemistry in Copper-Based Porous Catalysts through X-ray Spectroscopy: From Zeolites to MOFs

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Valorizing light alkanes to oxygenates remains a central challenge in heterogeneous catalysis with major implications for the chemical industry and the energy sector. In particular, a direct, mild-condition route for converting methane to methanol (DMTM) would be a breakthrough, delivering substantial energy savings and advancing sustainable development [1]. Notably, nature accomplishes DMTM at ambient conditions via copper-dependent methane monooxygenase (MMO) enzymes. Inspired by MMOs, the past decade has seen intensive efforts to incorporate Cu sites into zeolites. As an encouraging proof of concept, methane activation to the corresponding alcohols has been demonstrated, albeit predominantly through stepwise protocols [2, 3], underscoring that high specificity and selectivity in synthetic catalysts have yet to be fully realized.

In this context, Cu-functionalized MOFs are emerging as promising candidates, offering greater flexibility than conventional zeolites for tuning Cu ligation, cavity confinement, and the structural motif around the active site [3]. Continued progress requires a rigorous understanding of the local structure and electronic/chemical properties of Cu ions embedded, often without long-range order, within zeolitic or MOF frameworks.

In this contribution, I will highlight how in situ/operando X-ray spectroscopy elucidates structure-activity relationships for stepwise DMTM over Cu-zeolites, with emphasis on the MOR topology [4]. I will also present recent findings on Cu-UiO-66-his as a MOF-based case study [5]. These materials, which are active for the selective liquid-phase oxidation of cyclohexene, were comprehensively characterized by in situ XAS under model gas-phase redox conditions: thermal treatment in inert atmosphere up to 150 °C, isothermal reduction in H₂, and reoxidation in O₂. This workflow reveals the nature, local structure, and redox behavior of Cu species formed at different histidine loadings within the MOF scaffold. For both systems, I will emphasize advanced data-analytic approaches that quantitatively determine Cu speciation and resolve the local coordination environment, including statistical analysis/multivariate curve resolution (MCR) and EXAFS wavelet transforms (WT).

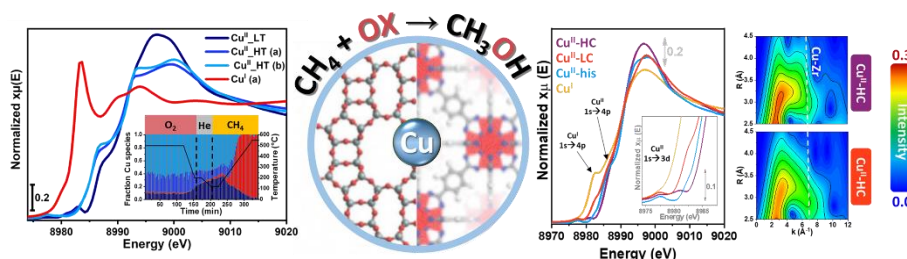


Figure 1. Cu K-edge pure XAS spectra of different Cu^{II} and Cu^I species identified by MCR analysis of a Cu-MOR zeolite (left) and a Cu-UiO-66-his MOF, also including EXAFS-WT maps of selected Cu^{II} species (right), monitored under model redox conditions, relevant to the DMTM conversion (middle).

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Advanced X-ray characterization into Next-Generation Energy Materials: Tracking Structural Evolution in Lithium and Sodium-Ion Cathodes

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The urgent global demand for sustainable energy storage solutions relies heavily on the development of high-performance, cobalt/nickel-free, or earth-abundant cathode materials. However, achieving the optimal compromise between sustainability and functional performance requires navigating complex solid-state phenomena and overcoming critical structural degradation pathways. In this context, advanced X-ray characterization methodologies, specifically synchrotron-based *operando* and *ex situ* techniques, are established as the ultimate enabling tool to track real-time structural evolution, thereby unlocking the fundamental design principles required to realize next-generation energy materials.[1]

The deployment of highly sustainable Lithium-ion batteries (LIBs) can be effectively addressed by target-engineering state-of-the-art manganese spinels (LiMn_2O_4). While economically and environmentally ideal, these materials are plagued by severe capacity fading caused by the Jahn-Teller (J-T) distortion. By implementing innovative multi-doping strategies, spanning from dual Fe/Ti substitution to multi-component $\text{LiCr}_x\text{Fe}_y\text{Mn}_{2-x-y-z}\text{Ti}_z\text{O}_4$ systems, it is possible to demonstrate how advanced X-ray tools can directly guide material optimization. Through *ex situ* and *operando* X-ray Diffraction (XRD) and X-ray Absorption Spectroscopy (XAS) at the Mn, Fe, Cr, and Ti K-edges, the active suppression of the cubic-to-tetragonal phase transition is successfully correlated with specific cation disorder and localized domain configurations, establishing a long-lasting lattice framework across extended potential windows.[2]

Concurrently, sodium-ion batteries (SIBs) represent a cornerstone technology for large-scale grid storage, requiring a dual strategic approach to stabilize layered oxide cathodes. On one hand, the performance of layered $\text{P2-Na}_{0.67}\text{Mn}_{0.5}\text{Fe}_{0.5}\text{O}_2$ architectures is deeply dictated by processing and composition: the combination of strategic Mg^{2+} doping to prevent detrimental high-voltage phase transitions with advanced spray-drying synthesis achieves superior sodium diffusion kinetics and cycle life compared to conventional solid-state routes.[3] On the other hand, the sustainability frontier can be pushed toward novel high-entropy oxide (HEO) cathodes, such as $\text{Na}_{0.55}\text{Fe}_{0.2}\text{Mn}_{0.2}\text{Ni}_{0.2}\text{Co}_{0.2}\text{Ti}_{0.2}\text{O}_2$, where tailoring a coarse single-crystal microstructure delivers an outstanding capacity of 180 mAh g^{-1} . To decode the structural stability of these multi-element configurations, high-resolution *operando* synchrotron XRD was deployed. Utilizing an optimized fast 2D setup to prevent cell radiation damage, the dynamic phase fractions and structural evolution were successfully tracked during complete desodiation, mapping the continuous conversion between the pristine O3 framework and the emerging P3 polymorph.

Ultimately, this work highlights that the path toward realizing next-generation energy materials cannot rely on empirical trial-and-error synthesis alone. By bridging tailored compositional engineering and advanced powder processing with state-of-the-art *operando* X-ray diagnostics, a holistic methodology is demonstrated to decode, predict, and master structural transformations, paving the way for the scalable deployment of truly sustainable energy storage systems

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Engineering metal-organic frameworks for highly nonlinear photonics

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Upconversion is a process where two or more low-energy photons are converted into a single higher-energy photon. Among upconversion mechanisms, photon avalanche (PA) is the most nonlinear one, producing a sharp burst of emission once the excitation power exceeds a critical threshold. To date, PA has been observed only in lanthanide-doped inorganic crystals, where emission originates from electron accumulation in the 4f levels of lanthanide ions. [1]

Metal-organic frameworks (MOFs) have been widely studied for their adsorption and catalytic activities. Nonlinear optical properties of MOFs are more recent areas of research, so far mainly focused on second harmonic generation. Here, we show that lanthanide-based MOFs actively enable PA upon IR irradiation, unexpectedly generating a white emission. (Figure) The PA mechanism is different from previously reported ones: the PA develops within the organic ligands. In this process, IR photons are first absorbed and upconverted through the cooperative action of lanthanide ions. The excitation energy is then funneled into a long-lived ligand state that acts as a reservoir, enabling electron accumulation and subsequent avalanche-like re-emission. [2] The observed nonlinearity is comparable to that of state-of-the-art inorganic PA crystals. The synthetic strategy is highly versatile and generalizable, allowing the PA process to be coupled with diverse MOF. [3-5] This work vastly expands the family of materials capable of nonlinear optical responses. Moreover IR-triggered photon upconversion in porous materials presents intriguing prospects for combined functionalities such as molecular sponge, energy harvesting and conversion.

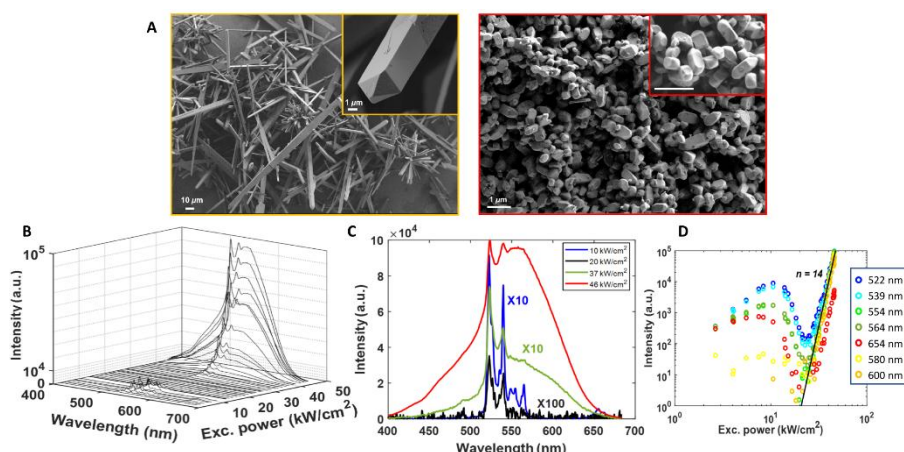


Figure: SEM images of PA MOFs(A); emission spectra upon IR irradiation (B,C); Intensity vs density power plot showing nonlinear emission

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Selective Complex Dissociation of REEs using Photoswitchable Ligands

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Rare Earth Elements (REEs) are essential for modern high-tech applications owing to their unique optical and magnetic properties. However, the rapidly growing demand for these critical metals has increasingly exceeded their availability. In the context of the circular economy, developing more efficient strategies for recovering REEs from waste electrical and electronic equipment (WEEE) and end-of-life (EOL) devices is therefore crucial. [1,2]

In this study, we explored whether the distinctive optical properties of REEs could offer an alternative to conventional recovery methods based on selective precipitation or solvent extraction. Complexes of REE nitrates (REE = La–Lu, except Pm, and Y) were prepared using a photoswitchable acylhydrazone ligand that undergoes quantitative E-to-Z isomerization upon irradiation with 390 nm LED light. Because the Z isomer exhibits lower affinity for REE(III) ions, irradiation induced photodissociation of the complexes, releasing REE nitrates into solution. The dissociation rate depended on the optical properties of the REE(III) ions: non-emissive complexes lacking accessible 4f excited states dissociated faster than emissive complexes possessing accessible 4f excited states.[3]

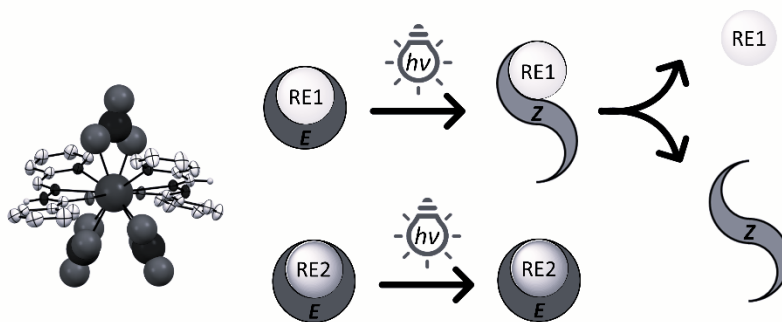


Figure 1. Selective release of RE upon light stimulus.

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Exploring Metal Exsolution in Titanate-Based Perovskites

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Nowadays, conventional electrodes for solid oxide fuel cells suffer from rapid degradation due to unavoidable sintering and coking [1]. La-doped titanates decorated with exsolved metal nanoparticles socketed into the ceramic surface are emerging as promising alternatives owing to their enhanced catalytic activity, improved nanoparticle dispersion, and increased resistance to degradation [2]. In particular, transition metal-doped titanate perovskites are gaining interest in this sector [3]. Nevertheless, in-situ investigations of the exsolution process are still limited, but they're crucial to achieve a deeper understanding of nanoparticle nucleation and growth, as well as of the evolution of the ceramic matrix at both the long-range structural and local distortion scales during exsolution.

To address these aspects La-doped titanates were investigated and compared with the undoped matrix. Samples with composition $\text{La}_{0.45}\text{Sr}_{0.45}\text{Ti}_{0.9}\text{M}_{0.1}\text{O}_3$ ($\text{M} = \text{Ni}, \text{Cu}$) were characterized by X-ray diffraction (XRD). Measurements were performed at ESRF from room temperature up to 900 °C under a 5% H_2/Ar atmosphere, including a 4-hour dwell step, with patterns collected every 3 minutes to monitor the evolution of the exsolution process in real time. The long-range crystalline structure was analysed through Rietveld refinement, while short-range structural features were investigated using Pair Distribution Function (PDF) analysis. Rietveld refinement enabled tracking of the exsolution process by monitoring changes in perovskite and metal lattice parameters and quantifying the amount of exsolved metal as a function of temperature and time. In parallel, PDF analysis provided deeper insight into local structural distortions and precursor states, allowing the probing of distortions around Cu, Ti, and O sites. Finally, the results obtained for the doped titanates were compared with the undoped specimen to provide a comprehensive understanding of such systems, helped with the combination of ex-situ investigations providing a multiscale description of the exsolution process.

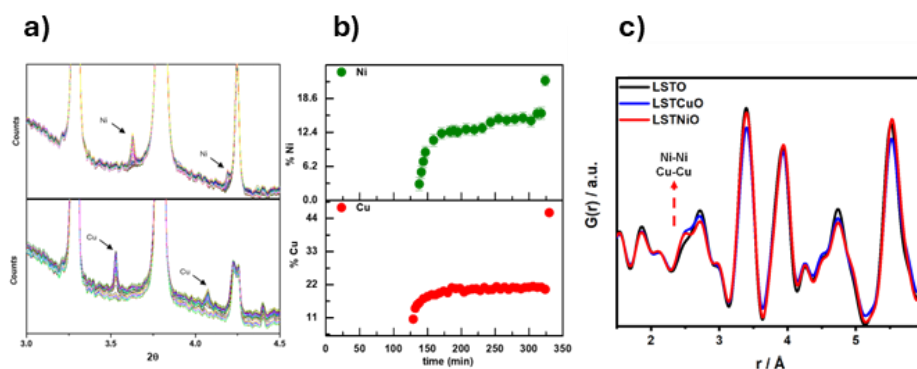


Figure 1. a) XRD overlap of LSTMO (Cu and Ni), displaying the increasing Cu and Ni peaks in time at 900 °C; b) % of exsolved Cu and Ni calculated at 900 °C during the dwell and ex-situ after cooling to RT; c) comparison between PDF curves of the undoped and doped specimens.

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Reordering Frustration in Enantiomer-Enriched Solid Solutions of Ionic Plastic Crystals for Proton-Conducting Materials Design

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The Ionic plastic crystals (IPCs) represent an emerging class of molecular solids characterized by long-range translational order combined with pronounced orientational disorder, resulting in a unique interplay between crystal structure and dynamics. These features make IPCs particularly attractive for the design of solid-state proton conductors under anhydrous conditions.[1] In this work, we investigate a series of triflate (TFO⁻) salts based on closely related globular cations: the achiral 3-quinuclidonium [QHco]⁺, the racemic (3)-hydroxyquinuclidinium S/R-[QH]⁺, and the enantiopure (3)-hydroxyquinuclidinium R-[QH]⁺, focusing on how subtle structural variations influence phase behavior and transport properties. Single-crystal and powder X-ray diffraction reveal that the achiral compound crystallizes in an ordered monoclinic phase, whereas both racemic and enantiopure salts adopt highly symmetric cubic lattices at room temperature, consistent with plastic crystalline phases. Upon cooling, the enantiopure system undergoes a reversible symmetry-lowering transition to a semi-ordered phase, while the racemic counterpart retains a dynamically disordered cubic structure down to low temperature. This striking difference highlights the key role of crystallographic disorder and symmetry in governing phase stability. Variable-temperature Raman spectroscopy further confirms the evolution from dynamic to static disorder through the suppression of the Rayleigh wing and the emergence of lattice phonon features. Solid-solution formation in enantiomer-enriched systems generates phases that remain orientationally disordered over a wide temperature range. This behavior is interpreted as a case of reordering frustration, where substitutional disorder and configurational entropy prevent the establishment of long-range order.[2] Electrochemical impedance spectroscopy demonstrates that such structurally frustrated phases exhibit enhanced proton conductivity, particularly at high temperature, without changes in the underlying Grotthuss transport mechanism.[3] These results establish a clear structure–dynamics–property relationship, where crystallographic symmetry, disorder, and lattice dynamics directly control the accessibility and connectivity of proton transport pathways.

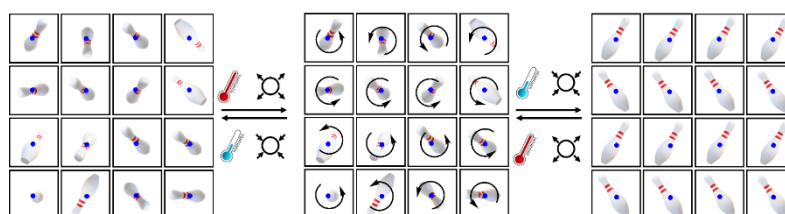


Figure 1. Representation of a statically disordered glassy crystal (left), a dynamically disordered plastic crystal (middle), and an ordered crystal (right).

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MS10

Sharing and disseminating Crystallography

STEAM in action: from educational robotics to materials science

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Robotics can be used as a powerful educational tool to introduce scientific concepts and to develop interdisciplinary activities for secondary school pupils in STEAM subjects [1-3]. The proposed educational activity leverages on the playful nature of LEGO bricks to develop a learning pathway spanning physics, chemistry, computer science and robotics [4]. 4th-5th year secondary school students (9 male and 13 female students) were involved in the building of a working replica of a diffractometer using programmable Lego® robotics kits, a red laser as a light source and a diffraction grating as a sample. The key scientific concepts, as the atomic structure of matter, electromagnetic waves optics, are generally covered in standard 4th-year physics and chemistry syllabuses of secondary school in Italy [5].

Typically the educational proposals include four phases: 1) introductory lectures, 2) visits to a research laboratory, 3) model building and software testing, 4) final presentation of the results.

Introductory lectures by CNR experts focused on the importance of X-ray crystallography and the basic principles of diffraction, highlighting the differences and similarities between a real X-ray diffractometer and the model to be built-up (3 hours). In the second stage, the students visited the research laboratories at the Dept. of Chemistry and Biology "A. Zambelli" and met experts in the field (3 hours). The building of the model diffractometers, code development, testing and improvements took place at the Physic Lab of the secondary school (18 hours). Two different diffractometer configurations were built using the light sensors and motors available in Lego kits and diffraction patterns were collected and displayed. Finally, the students presented their work at a public outreach event, operating their robots and explaining their results to the audience (3 hours).

The lecture will discuss the final outcome of the project, future developments and its general implementation as a learning pathway, where *Science, Technology, Engineering, Arts and Mathematics* come together to build the future generation of scientists.



Figure 1. Examples of student set-up for the detector and laser source.

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Crystallography as a Vehicle for Stimulating Research and Education on a Global Scale

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The relationship between structure and properties lies at the heart of chemistry, physics, biology, materials science, geology, engineering, and many other fields. This places crystallography in a unique position to bridge scientific communities across disciplines. Its inherently interdisciplinary nature makes it a particularly powerful vehicle for stimulating research, education, and collaboration, especially in developing regions where scientific infrastructures and opportunities are still emerging.

In this lecture, I will retrace the evolution of this vision through a personal journey beginning with activities initiated within the International Union of Crystallography, particularly through the International Year of Crystallography 2014 and the IUCr-UNESCO OpenLab programme. These experiences demonstrated how education, access to instrumentation, and direct interaction among scientists, students, institutions, and industry partners can create sustainable local impact while attracting researchers from many different disciplines.

The lecture will describe how these early experiences progressively evolved into broader and more structured international programmes, including the development of *LAAAMP*, where the focus expanded from crystallography alone to advanced light-source techniques and multidisciplinary research infrastructures. Within this framework, regional hubs such as X-TechLab in Benin and CRXSTAL in Jamaica were established as distributed centres for training, scientific networking, technology transfer, and collaborative research.

More recently, these activities have converged into wider initiatives such as BRIDGE Africa (Boosting Research Infrastructure for Development, Growth, and Empowerment in Africa), funded by Italian MUR through the Piano Mattei and the G7 Research Capacity Building with Africa initiative, as well as the UNESCO-endorsed programme *Advanced Light Source Facilities to Empower Global-South Scientists for Sustainable Development*, developed within the framework of the International Decade of Sciences for Sustainable Development 2024-2033. These programmes seek to reinforce the role of science as a driver for sustainable development, inclusion, innovation, and international cooperation. Through this personal perspective, the lecture will reflect on both the opportunities and the challenges involved in transforming scientific outreach activities into enduring collaborative ecosystems. Above all, it will highlight how crystallography, precisely because of its ability to connect disciplines and communities, can become a powerful catalyst for sharing knowledge, empowering regions, and fostering truly global scientific collaboration.

Crystallography behind the button: dissemination, automation and decision-making in industrial environments

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In many industrial environments, X-ray diffraction is not perceived as crystallography, but as a routine quality-control step embedded in a broader production process. A sample is prepared, a measurement is launched, and a result is generated. The final user may be a plant operator, technician or process engineer with limited crystallographic training, yet the output of the analysis can influence decisions in cement production, mining, direct reduced iron processing and other materials-related industries.

Behind this apparent simplicity lies significant crystallographic complexity. Phase identification, polymorph discrimination, peak overlap, solid solutions, preferred orientation, detection limits and Rietveld refinement strategies all affect the reliability and meaning of the final result. In academic and research contexts, this complexity is usually handled by users with specialized training. In industrial contexts, however, crystallographic knowledge must often be translated into robust workflows usable by non-specialists.

This contribution reflects on the boundary between dissemination and automation in applied crystallography. Should industrial users be trained to understand the crystallographic basis of the analysis, or should complexity be hidden inside automated procedures? Fully educating every user as a crystallographer is often unrealistic, while a purely black-box approach risks disconnecting numerical outputs from their assumptions, uncertainties and limitations.

We discuss an intermediate approach based on guided enablement, where crystallographic expertise is embedded into predefined and validated Rietveld refinement workflows, for example through Profex-based input strategies designed by application specialists. These workflows allow non-specialist users to access advanced quantitative phase analysis under routine industrial conditions, while preserving a controlled crystallographic framework.

This raises a broader question for the dissemination of crystallography: is dissemination only the explicit teaching of crystallographic concepts, or can it also mean enabling reliable crystallographic decision-making by non-crystallographers? Industrial automation may hide crystallography behind a button, but it can also bring crystallography into environments where it was previously absent. The challenge is to ensure that this hidden crystallography remains sufficiently transparent to be trusted, questioned and responsibly used.

Beyond the Invisible: Stories, Science, and Careers in Crystallography

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Podcasts are flexible and accessible digital audio tools that can be listened to anytime and anywhere, making them particularly effective for dissemination. By combining expert interviews, storytelling, and personal experiences, podcasts can communicate complex scientific concepts in an engaging and approachable way while fostering interaction with diverse audiences.¹

This project aims to enhance the visibility of crystallography in Italy through the creation of an Italian-language podcast series named Crystallography Matters entirely dedicated to this field. The initiative will showcase scientific careers and research experiences through direct testimonies from crystallographers, creating a platform that connects experts, students, and enthusiasts while encouraging discussion and broader public engagement with crystallography. Beyond the crystallographic community, the podcast also seeks to involve students and early-career researchers from related disciplines, promoting accessibility and interdisciplinary exchange.

Each episode, lasting approximately 25-30 minutes, will be hosted by three young researchers with complementary scientific backgrounds. The format will combine scientific discussion with personal storytelling, covering applications of crystallography in chemistry, biology, geology, and materials science, together with practical insights into academic and professional career paths. Primarily targeted at graduate students, PhD candidates, and early-career scientists, the podcast aims to inspire, educate, and strengthen networking opportunities within the crystallographic community. Episodes will be distributed through online hosting platforms and promoted via scientific networks and social media channels to maximize outreach and impact. The podcast will also feature several episodes in English, broadening its international reach and impact. Among the highlights is the unique opportunity to interview two Nobel Prize laureate, offering listeners exclusive insights from very influential voices in science. This prestigious contribution not only enriches the scientific value of the project but also aim at enhancing its visibility and appeal to a wider global audience.

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Words of Crystallography in the Italian Legal Language: A Laboratory for High School Students

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Starting from the words of crystallography recorded last year from our colleagues during the AIC Congress in Florence [1], shown in Figure 1, we develop a laboratory dedicated to crystallography and law for high school students.



Figure 1. Word clouds from the crystallographer answers.

At the beginning of the workshop, we present a short introductory text on crystallography in which students are invited to identify terms that are also commonly used in legal language. For example, *cell* may refer to a small room in which a prisoner is confined, as well as to the unit cell in crystallography; similarly, *disposition* can indicate the transfer of property in a legal context, or the arrangement of atoms and molecules in the solid state. We then introduce the IUCr definition of a crystal as a material that exhibits an essentially sharp diffraction pattern when probed with radiation in the Ångström wavelength range [2]. This provides the basis for engaging students in a discussion on whether everyday materials—such as sugar, table salt, aspirin, chocolate, and glass—can be classified as crystalline. This activity often leads to surprises, as students discover that many common solids are indeed crystalline. In parallel, we incorporate a game-based activity in which participants are asked to locate the term *reticolo* (lattice) within the Italian legal database Normattiva.it. Through this exercise, students gain familiarity with the structure and language of legislative texts, as well as an awareness of the multiple meanings that scientific terms may acquire in different contexts.

The workshop concludes with a formative assessment based on a crossword puzzle featuring terms shared between the domains of crystallography and legal language, introduced at the beginning of the session.

Overall, this laboratory experience not only fosters engagement with STEM disciplines, particularly crystallography, but also contributes to civic education by highlighting the importance of consulting authoritative sources for legal information.

Acknowledgements

The authors are grateful for the support of the CHANGEGAME Project, funded by CNR.

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VISION3D: Augmented and Virtual Reality approach for engaging the public in structural science

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We face the problem of the scarce sense of three-dimensional spatiality that students reveal when they learn the concepts of chemical bonding. In fact, we have noticed that first year university students are somehow losing skills related to the description of three-dimensional molecular structures: confounding the concepts of linear and planar, or not being able to assess angular values are examples. When asked to build a 3D model of hybrid atomic orbitals with plasticine, in a sort of bricolage challenge during the lectures on chemical bonding, many students were unable to properly execute the task. We can track back to, and share with, high school the problem of learning tools and methods, where the concept of structure in chemistry is often compressed in few hours, with no practicals attached to the enumeration of geometrical rules. Moreover, increasing use of electronic devices for teaching and representing models might pose problems in understanding 3D facts. We are developing a project to help students to metabolize 3D concepts, and we have tested Augmented reality and Virtual Reality practicals to engage the public in understanding molecular structures. The VISION3D project aims to establish a dedicated hub for the dissemination of crystallographic and molecular sciences, promoting the Crystal Chemistry and Mineralogy Collections of the University of Parma [1,2]. Through advanced 3D representation technologies, Virtual Reality, and Augmented Reality, the project enables the exploration of structure–property relationships in natural, synthetic, and biological materials, integrating data from major international structural databases (CCDC, ICSD, PDB). The initiative will develop immersive pathways and educational tools tailored to diverse audiences—from school students and the general public to university learners—with the goal of fostering scientific culture and supporting STEM education. VISION3D also introduces an innovative teaching approach, enhancing critical understanding and dynamic interpretation of scientific models compared to traditional physical representations. Examples will be given to the audience.

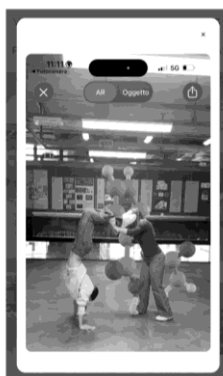


Figure 1. Selfie of high school students performing with an augmented reality molecule partner

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POSTERS

MS1

Perspectives of crystallographic methods and techniques

Advancing Structural Analysis of Nanomaterials: Bridging the Resolution Gap in XRPD through Deep Learning

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High-resolution crystal structure determination from X-ray powder diffraction (XRPD) data is often hindered by poor crystalline quality, limited crystallite size, and instrumental broadening effects. These limitations are particularly critical for nanocrystalline materials, where severe peak overlap significantly reduces the reliability of conventional crystallographic ab initio structure solution methods.

In this work, we present a novel artificial intelligence-based approach to enhance the quality of experimental XRPD data. The method is trained on a large set of simulated diffraction patterns generated from crystal structures extracted from the Crystallography Open Database (COD) [1]. Through this training, the model learns to associate broadened diffraction profiles, representative of nanocrystalline systems, with sharper patterns corresponding to well-ordered crystalline materials.

The goal of the approach is not to replace crystallographic analysis, but to enhance the diffraction data themselves, making them more suitable for standard procedures such as indexing, intensity extraction, and structure solution. In this perspective, the method can be seen as a preprocessing tool that extends the applicability of established crystallographic workflows.

The results show that the enhanced patterns exhibit improved peak definition and reduced overlap, facilitating the extraction of structural information under challenging conditions. The validity of the approach was further assessed through indexing and ab initio structure solution using the EXPO software package [2], with the obtained unit-cell parameters compared against the reference values from the original CIF files. The robustness of the approach was validated across different crystallite sizes and further confirmed through its application to experimental XRPD data.

This approach contributes to extending the applicability of conventional crystallographic methods to nanocrystalline systems affected by severe peak broadening and overlap.

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Beyond Enthalpy: Incorporating Vibrational Entropy into Crystal Structure Prediction through Molecular Dynamics

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Predicting with certainty which polymorphic form a molecule will adopt during crystallization remains an unsolved challenge, although recent advances in crystal structure prediction (CSP) have yielded impressive results, as demonstrated in the latest blind tests organized by the Cambridge Crystallographic Data Centre (CCDC) [1,2]. Both quantum and classical Molecular Dynamics (MD) approaches generally do not provide free energies of cohesion directly, and neglecting entropic effects can significantly bias the relative stability and ranking of polymorphic forms. Recovering entropy terms from quantum simulations is typically very demanding, especially when multiple putative structures must be compared. This burden can be alleviated by using accurate MD trajectories [3], from which entropy differences with respect to the gas or liquid states may be recovered via advanced thermodynamic integration or free-energy perturbation models [4]. Nevertheless, these approaches require simulations of multiple intermediate non-physical states, coupled with extended sampling.

In this context, incorporating entropy calculations into MD-based CSP workflows would represent a substantial advancement. To this end, we developed two different algorithms to estimate vibrational entropy contributions from MD simulations of periodic molecular solids. The first approach is rather traditional, as it consists of analyzing the Fourier modes derived from the velocity autocorrelation function (VACF). The second one leverages the Huang-Widom technique recently proposed for alloys and metals [5], which is based on the variance-covariance matrix of atomic displacements. We applied these methods to trajectories produced by the Milano Chemistry Molecular Simulation (MiCMoS) MD engine [6,7] within the recently developed Symmetry Constrained Monte Carlo (SC-MC) predictor [8]. Preliminary results on urea indicate that the computational cost is substantially lower than that associated with quantum mechanical calculations, and despite the intrinsic limitations of using a classical force field, the results are in good agreement with quantum mechanical reference values. When incorporated into the SC-MC CSP framework, the proposed methodology not only correctly produces the expected polymorph ranking but it also discriminates between the low-pressure and high-pressure polymorphs of urea on the potential energy surface (PES).

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- [6] A. Gavezzotti, L. Lo Presti, S. Rizzato, *CrystEngComm.*, **2022**, 24, 922
- [7] MiCMoS v2.4 User's Manual, **2025**, see https://sites.unimi.it/xtal_chem_group/index.php/research/5-micmos
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MS2

Advances in crystal engineering

Conformational Switch for NIR Emission with Unity Quantum Yield in Thiele-Type Diradicaloids via Mixed Halogenation Patterns

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Luminescent diradicals represent a highly intriguing class of molecules; owing to their unique electronic structures, they are particularly attractive for the development of novel materials in the field of organic electronics.[1] In this framework, Thiele-type diradicaloids based on the *para*-quinodimethane (*p*-QDM) core are gaining increasing attention in recent literature. Halogenation of the parent tetraphenyl *p*-QDM molecule, originally reported by Thiele,[2] provides an effective strategy to achieve outstanding stability against air and moisture. Furthermore, chlorinated and fluorinated Thiele-type compounds have demonstrated exceptional photoemission properties, characterized by high photoluminescence quantum yields (PLQY), large Stokes shifts, and pronounced emission solvatochromism.[3,4]

These photophysical properties are ascribed to a "sudden polarization" mechanism, wherein a highly polar or polarizable emitting state is generated from a substantially apolar ground state. In this work, we investigate the effects of introducing a mixed halogen substitution pattern onto the tetraphenyl *p*-QDM framework.[5,6] The most remarkable results were achieved by combining a tetrachlorinated *p*-QDM core with 2,3,4,5-tetrafluorophenyl exocyclic substituents (Figure 1). This specific substitution pattern induces a pronounced folding of the *p*-QDM core, leading to exceptional photophysical features, including a massive Stokes shift of 2.0 eV. Single-crystal X-ray diffraction (SC-XRD) analysis, complemented by theoretical studies, confirmed that these unique properties are directly traced back to this dramatic structural rearrangement upon excitation.

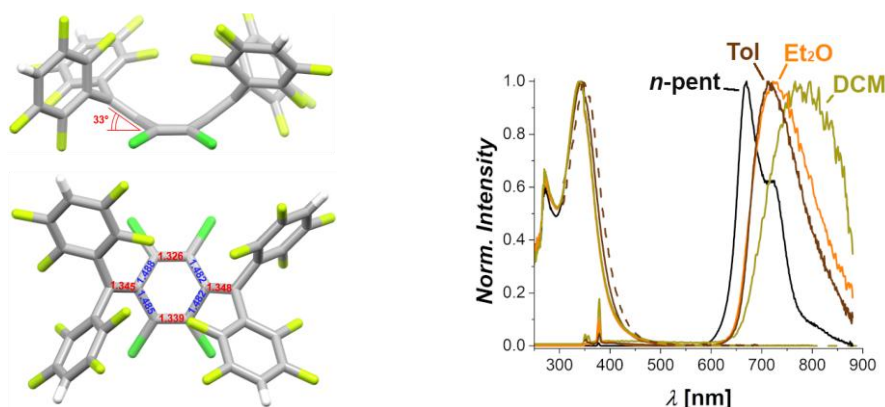


Figure 1. Folded Thiele-type diradical: SC-XRD structure (left) absorption and emission spectra (right).

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Comparison between the crystal structures of fluorinated and not fluorinated enantiopure aryl benzyl sulfoxides

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In our work on the highly enantioselective oxidation of aryl benzyl sulfides by using hydroperoxides in the presence of a complex between titanium and (*S,S*)-hydrobenzoin [1,2], a large chemical library of enantiopure aryl benzyl sulfoxides has been constructed. The crystal structures of many enantiopure and racemic aryl benzyl sulfoxides from this library have been studied with single crystal X-ray diffraction [2-4].

The numerous crystallographic data collected so far have allowed us to identify the main interactions building up the crystal structures of these compounds [2-4]. This comprehension can be of paramount relevance in predicting crystal structures [5].

In sulfoxides where strong hydrogen bonds are absent, we observed that the main interaction building up the crystal structures is a weak hydrogen bond, with the sulfinyl oxygen atom acting as the acceptor [2-4]. Weak interactions connected with the aryl groups complement this weak hydrogen bond and provide a further stability for the crystal structure [2-4].

When comparing the crystal structures of aryl benzyl sulfoxides with those of their fluorinated counterparts, in which a phenyl group is substituted by the pentafluorophenyl group, we often observed the switch from an *anti*-conformation to the more compact *gauche*-conformation (see Figure 1 for a representative example). Energetic calculations, performed with the *CrystalExplorer21* program [6], reveal possible reasons for this behaviour.

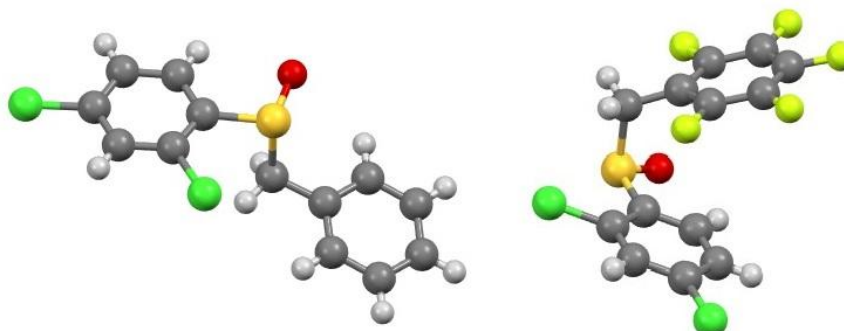


Figure 1. Crystal structures of (*R*)-benzyl 2,4-dichlorophenyl sulfoxide (left) and (*R*)-2,4-dichlorophenyl pentafluorobenzyl sulfoxide (right).

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Chiral Copper-Based Hybrid Perovskites: Structure–Property Relationships in Low-Dimensional systems

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In the recent years, molecular magnetic materials with chiral configurations have emerged as highly promising candidates for advanced applications such as sensing, data storage, and spintronics. Their uniqueness, arising from the combination of magnetic properties and chirality, represents an exciting frontier in the field of materials science. [1] In this perspective, chiral hybrid organic–inorganic perovskites (HOIPs), consisting of an inorganic metal-halide portion interacting with chiral organic ammonium-based cations, have emerged as a platform for developing new multifunctional materials, due to the wide availability of chemical constituents allowing for a variety of compounds where the structural and functional properties can be finely tuned.[2] In such materials, chirality disrupts space inversion and mirror symmetry, leading to intriguing effects like the chiral magnetic effect, chiral-induced spin selectivity, and magneto-chiral dichroism.

To shed light on these aspects, we are designing, synthesizing and characterizing from both a structural and photophysical point of view novel series of lead-free chiral HOIPs, containing Cu²⁺ as the metal center and Cl as the halogen and carefully tuning the chiral molecule. In this manner, we aim to study how the functionalization of the organic ligand influences the crystal structure, dimensionality, and coordination of the system, and what is the impact on the chiro-optical and magnetic properties. Chiral ligands such as X-CIMBA, X-BrMBA, and X-FMBA (MBA = α -methylbenzylamine) have been employed, yielding single crystals with stoichiometry (X-FMBA)₂CuCl₄ (X = 2, 3, 4), (X-CIMBA)₂CuCl₄ (X = 2, 3, 4) and (X-BrMBA)₂CuCl₄ (X = 2, 3, 4) that are now being investigated from the functional viewpoint. We are also exploring other organic cations, in particular chiral diamines, to study their influence on the hydrogen-bond network and on the arrangement of the polyhedra, to study how chiral cations influence the system. By adopting a multidisciplinary approach involving synthesis, structural characterization, photophysical studies, magnetic investigations and theoretical calculations, we aim to correlate the structural features of chiral HOIPs with their functional properties, providing new parameters useful for the design of innovative materials.

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When the Counteranion Chooses the Crystal Packing: Competing Hydrogen-Bonded Slabs in Monoprotonated Diethylenetriamine Salts

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Why do two closely related monoprotated diethylenetriamine salts assemble the same hydrogen-bonded two-dimensional slab, yet pack in different ways in the solid state? In this contribution, we address this question by combining crystallographic characterization and periodic DFT calculations on a family of HDETA salts.

Our starting point is a striking structural observation: the known bromide [1] and nitrate [2] salts of monoprotated diethylenetriamine share the same square-grid hydrogen-bonded slab, but differ in slab stacking (AB vs AA packing of the 2D slabs), revealing two competing packing modes. We used these two arrangements as reference models and investigated their relative stability by solid-state DFT calculations, extending the analysis to halides, trigonal oxoanions, and tetrahedral anions. The results show that the preferred packing is not governed by anion size alone. Instead, it depends on a subtle combination of size, shape, and structural arrangement of the counteranion within the hydrogen-bonded framework.

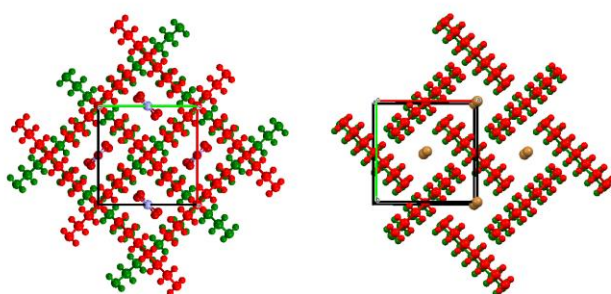


Figure 1. AB and AA packing in HDETA•NO₃ (left) and HDETA•Br salt (right).

To test these predictions experimentally, DETA was reacted with HCl, HI, and HBF₄ under strongly amine-rich conditions designed to favor monoprotection. Powder X-ray diffraction, solution NMR, and single-crystal X-ray diffraction were used to characterize the resulting solids. Chloride provides the clearest validation of the model, yielding a monoprotated salt isomorphous with the bromide analogue from single crystal. For HDETA•I it was not possible to obtain single crystal. Powder diffraction spectra only support the formation of a monoprotated phase, but the packing modes could not be distinguished unambiguously. In contrast, HBF₄ leads to a diprotonated product with a combination of F and BF₄ counteranions, highlighting the experimental difficulty of controlling protonation equilibria and monoanion during the crystallization. Overall, the study shows that the counteranion is not a passive spectator, but a genuine structure-directing element in HDETA salts.

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Unravelling solvent-dependent crystallization in triphenylsulfonium lead-halide perovskitoids through *in situ* studies

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Organometal halide perovskitoids (OMHPs) based on sulfonium cations have recently attracted increasing attention due to their structural versatility and the presence of charge-assisted chalcogen bonding interactions. In particular, the bulky triphenylsulfonium cation $[(\text{Ph})_3\text{S}^+]$ promotes the formation of low-dimensional hybrid architectures,[1] where subtle changes in crystallization conditions can strongly influence the resulting framework topology.

In this work, we investigate the role of solvent-mediated crystallization in directing the assembly of lead-halide compounds derived from a common triphenylsulfonium precursor. Recrystallization experiments performed in different solvent systems yielded a variety of crystalline phases characterized by distinct structural arrangements. Depending on the solvent employed and on the halide composition (Br^- or I^-), both common and unique crystal structures were obtained. Single-crystal and powder X-ray diffraction analyses reveal that all compounds adopt low-dimensional organizations of corner- or edge-connected PbX_6 octahedra, generating 0D and 1D inorganic frameworks stabilized by a network of weak intermolecular interactions involving the sulfonium cation.

Particular attention was devoted to the crystallization pathways leading to solvent-containing phases. *In situ* experiments were performed in a custom-made chamber [2] to demonstrate the rapid formation, within a few minutes, of the DMF-solvated compounds $(\text{Ph})_3\text{S}[\text{PbBr}_3(\text{DMF})]$ and $(\text{Ph})_3\text{S}[\text{PbI}_3(\text{DMF})]$. Analogous investigations performed in ethanol and acetonitrile did not lead to the corresponding recrystallized products, highlighting the crucial role of solvent coordination and solvation effects in determining phase selectivity. Furthermore, all isolated phases exhibit remarkable stability under ambient conditions, retaining their crystallinity after more than one year of air exposure.

These results provide insight into the solvent-dependent formation mechanisms of low-dimensional sulfonium-based hybrid halides and establish crystallization conditions as a key parameter for controlling structural diversity in this emerging family of materials.

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Co-Crystal Discovery in the era of AI and Mechanochemistry

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Co-crystals are crystalline materials composed of two or more neutral building blocks in a defined stoichiometric ratio.[1] They have attracted considerable interest due to their potential ability to modify key physicochemical properties of pharmaceuticals, agrochemicals and functional materials without altering the molecular structure of the active component.[2] Among the various synthetic approaches available, mechanochemistry has emerged as a sustainable, quick and cost-effective method for co-crystal synthesis, minimizing or completely avoiding the use of solvents.[3] Three dihydroxybenzoic acid isomers (2,4-, 2,5-, and 2,6-dihydroxybenzoic acid) were investigated as co-crystal formers. Eight co-formers were selected through a combined literature-based and AI-assisted strategy. Mechanochemical screening was initially carried out by neat grinding the reagents in a 1:1 molar ratio, followed by powder X-ray diffraction (PXRD) analysis of the obtained products. Fourteen samples showing new diffraction peaks were selected for further investigation. Crystallization experiments were subsequently performed using different solvents and conditions in order to obtain single crystals. The crystal structures and stoichiometric ratios of the obtained phases were determined through single-crystal x-ray diffraction (SCXRD). Mechanochemical syntheses were then repeated using the experimentally determined stoichiometric ratios and the resulting powders were reanalysed by PXRD. SCXRD analysis led to the identification of four new co-crystals. In some cases, agreement between PXRD patterns and SCXRD-derived phases was observed, while in other systems discrepancies suggested the formation of different phases depending on the preparation method. The combination of AI-assisted coformer selection, mechanochemical reactions and crystallographic characterization is effective for the investigation of co-crystal formation. Moreover, the differences observed between phases obtained by mechanochemical and solution-based approaches suggest that the synthetic pathway plays a key role in directing the formation of crystalline phases.

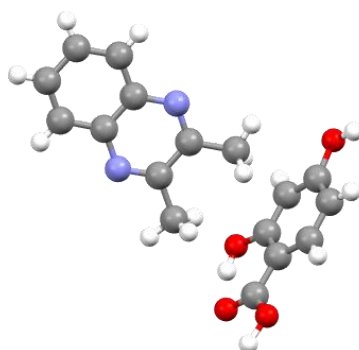


Figure1. Crystal structure of the 2,4-dihydroxybenzoic acid – 2,3-dimethyl quinoxaline co-crystal.

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Antimicrobial Molecular Materials Combined with Polymeric Matrices for the Mitigation of Biodeterioration in Cultural Heritage Substrates

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Biodeterioration of cultural heritage materials represents a widespread challenge that increasingly demands conservation strategies that are both highly effective and environmentally sustainable while minimizing risks to operators. This project aims to develop innovative and sustainable antimicrobial hybrid materials for mitigating biodeterioration in cultural heritage substrates and conservation supports. Crystal engineering principles and mechanochemical synthesis are employed to design antimicrobial metal-based compounds with reduced toxicity, as well as to tailor the physicochemical properties of natural bioactive molecules. In parallel, the versatility of electrospun polymeric matrices is exploited for the delivery of controlled antimicrobial activity. Regarding the active molecular materials, two main approaches are pursued. The first involves light-coloured zinc-based coordination complexes incorporating *trans*-cinnamic acid derivatives as antifungal ligands, which can subsequently be immobilized onto the nanofibers by either *in situ* precipitation or co-electrospinning. The second approach, developed in collaboration with the Academy of Fine Arts of Bologna, consists of the encapsulation of bioactive essential oils in β -cyclodextrin, followed by their incorporation into electrospun fibers. Both systems could be suitable for different applications, such as paper conservation and canvas relining procedures. Although the project is still at an early stage, promising preliminary results have already been obtained. Several known zinc-based complexes were successfully synthesized by mechanochemistry, together with two novel coordination compounds, namely $\text{Zn}_2(\text{cinnamate})_4(\text{nicotinamide})_2$ and $[\text{Zn}(\text{p-coumarate})_2(\text{H}_2\text{O}) \cdot 3\text{H}_2\text{O}]_n$. Their immobilization within the polymeric matrices is currently under investigation. In parallel, β -cyclodextrin inclusion complexes with thymol and carvacrol were repurposed to reduce the volatility of the active molecules [1,2]. After confirming the retention of their crystallinity in different electrospinning solvents and polymer systems by PXRD and TGA analyses, they were successfully incorporated into polystyrene nanofibers by doping the electrospinning solution, yielding continuous, bead-free microfibers. Ongoing and future work will focus on completing the microbiological assays to validate the effectiveness of the developed systems and assessing their compatibility with real conservation scenarios, with the ultimate goal of transferring these laboratory findings into practical conservation tools.

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Obtaining and structural studies of novel genistein crystalline phases with improved solubility

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Genistein is a natural isoflavonoid that exhibits antioxidant, anti-inflammatory, antitumor and antibacterial properties. Consequently, it is considered a promising agent for the treatment of cancer, diabetes, obesity, genetic disorders and skin diseases [1-3]. However, poor aqueous solubility which results in low bioavailability severely limits its therapeutic efficacy. One of the most common strategies applied to enhance the bioavailability of active pharmaceutical ingredients (APIs) and biologically active compounds is the formation of new crystalline phases, such as cocrystals, solvates or salts [4]. This method does not require modification of the chemical structure of the active substance, so it does not reduce its biological activity. It is a very promising and effective approach in drug design.

The aim of this study was to obtain new genistein cocrystals exhibiting better water solubility and, consequently, increased bioavailability. The design was based on the structural analysis of the compounds, which allowed predicting which synthons were likely to form. The crystal structures of seven new genistein cocrystals with 4-methylpyridine and lutidine isomers were successfully obtained and determined. The systems were investigated using differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), as well as FTIR and Raman spectroscopy methods. Using the QTAIM Theory (Quantum Theory of Atoms in Molecules), hydrogen bond binding energies at the bond critical points were estimated.

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MS3

High-throughput and dynamic studies on biosystems

Dimensionality-dependent Binding Affinity of Silver(I)–Fluoroquinolone Systems toward Bacterial Enzymatic Targets

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Fluoroquinolones (FQs) are a well-known class of broad-spectrum antibacterial agents that have been used for decades to treat several bacterial infections [1], including respiratory, urinary tract, intra-abdominal, skin, and soft-tissue infections. Their clinical relevance is mainly associated with inhibition of bacterial type II topoisomerases, particularly DNA gyrase and topoisomerase IV. The increase in microbial resistance to FQ antibiotics has reduced their effectiveness and prompted the search for alternative strategies to enhance or modulate their antibacterial potential [2,3].

One strategy that has received growing attention is the association of antibiotics with metal ions to create metal-based antibacterial systems, often referred to as metalloantibiotics or metallodrugs [4]. In the case of FQs, metal coordination is particularly relevant because they contain suitable N- and O-donor groups that can coordinate with transition metals, thereby influencing their structural organization, physicochemical properties, and potentially enhancing interactions with biological targets [5]. Within this framework, we investigated the self-assembly and crystallization of silver(I) with a selected FQ antibiotic ligand under different experimental conditions, including variation of pH and solvents. Three new Ag(I)–FQ compounds were obtained and characterized in the solid state. Structural analysis revealed distinct molecular and extended architectures, corresponding to 0D, 1D and 2D topologies (see **Figure 1**), in which Ag(I) and the FQ ligand exhibit various coordination and chelation modes. The compounds were further characterized by FTIR spectroscopy, solid-state UV–Vis absorption and emission spectroscopy, and their thermal stability was evaluated by DSC analysis. The antibacterial activity of Ag(I)–FQ systems against selected bacterial enzymatic targets was explored *in silico* through molecular docking and compared with that of the free FQ antibiotic. The binding affinity and interactions with the targets suggest that the potential relevance of these systems is markedly influenced by their dimensionality, highlighting dimensionality as a key parameter to consider in future experimental studies.

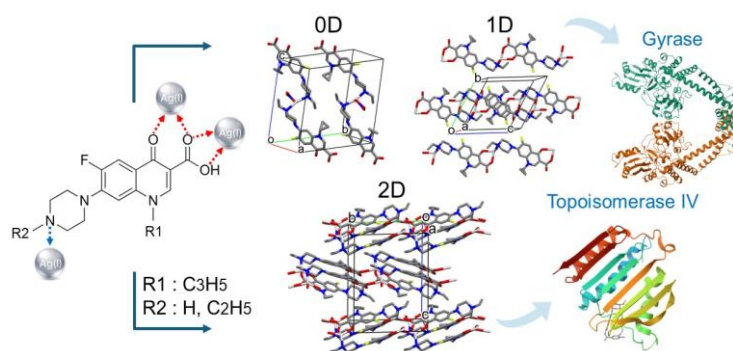


Figure 1. (left) Coordination modes the used fluoroquinolone ligands of with silver(I); (center) dimensionality of the resulting compounds; (right) *Escherichia coli* enzyme targets.

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MS4

**From ambient to non-ambient conditions:
exploring mineral structures and transformations**

High pressure behavior and phase transitions in benitoite

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Benitoite (ideally $\text{BaTiSi}_3\text{O}_9$) is a rare cyclosilicate characterized by a framework of three-membered silicate rings, which represents a significant mineralogical analogue for several high-pressure silicate and carbonate systems. Interestingly, benitoite has $(\text{Si}_3\text{O}_9)^{6-}$ silicate-ring units, which is a relatively uncommon feature in silicates. Only CaSiO_3 pseudowollastonite present a structure with 3-fold silicate ring units with the same topology as benitoite [1]. Considering the interest of the CaSiO_3 system for the Earth's inner mineralogy, high pressure study on benitoite is a rather relevant comparative system to be investigated.

The main aim of this contribution is to show the high-pressure (HP) behaviour and structural evolution of benitoite through synchrotron single-crystal X-ray diffraction experiments up to 22.3 GPa. Our results show the occurrence of three phase transitions: between 3.93(5) and 4.40(5) GPa from benitoite to benitoite -II, between 12.44(5) and 13.05(5) GPa, to benitoite-III and between 16.36(5) and 17.08(5) GPa to benitoite-IV. The crystal structures of the two new HP-polymorphs, benitoite-III (space group $Pbn2_1$) and benitoite-IV (space group $R32$), were successfully solved and refined. The structural evolution is driven by a progressive deformation of the Si_3O_9 rings and the Ti-octahedra, coupled with an increase in the barium coordination number. In benitoite-IV, the topology of the silicate rings becomes distinct, with the tetrahedral bases becoming almost parallel, a modification that correlates with a significant increase in overall density. A remarkably similar behaviour is observed in the CaSiO_3 system, where the high-pressure polymorphs breyite and parabreyite, which feature distorted three-membered rings, are significantly denser compared to the low-pressure pseudowollastonite. The findings on benitoite provide valuable insights into the stabilization mechanisms of three-fold rings under extreme conditions, offering a structural model that may also explain the behaviour of high-pressure carbonates with sp^3 -hybridized carbon.

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Average and local structural response of cryptomelane to Tl, Pb and Zn incorporation

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Hollandite-supergroup minerals are tunnel oxides with a deceptively simple structural topology, based on double chains of edge-sharing octahedra defining large cavities, commonly occupied by mono- or divalent extra-framework cations. Their general formula can be expressed as $A_xX_8O_{16}$, where $A = K, Ba, Pb, Sr, Tl$ and $X = Mn, Ti, Fe, Cr, V, Al$. Despite their apparent simplicity, hollandites are often challenging to describe from a structural point of view, owing to symmetry lowering, tunnel-cation displacement and positional disorder within both the tunnel and octahedral framework [1,2].

Cryptomelane-type materials, ideally KMn_8O_{16} , where the tunnel site is mainly occupied by K, can undergo ion-exchange processes involving environmentally relevant heavy metals. Here, we investigate synthetic cryptomelane powders treated with aqueous Tl-, Pb- and Zn-bearing solutions, combining chemical analysis using particle-induced X-ray emission (PIXE), powder X-ray diffraction (PXRD) and X-ray absorption spectroscopy (XAS). The comparison between treatments highlights the different ability of hollandite to accommodate monovalent and divalent cations, with contrasting size, charge and stereochemical behaviour, within the tunnels.

The average structure of the untreated material is better described in the monoclinic space group $I2/m$ rather than in the ideal tetragonal $I4/m$ model. Ion exchange produces distinct structural responses depending on the employed cation. Tl incorporation yields an expansion of the unit cell and is associated with marked positional disorder, characterized by an additional tunnel site displaced from the conventional A position along the tunnel axis. In contrast, Pb- and Zn-treated samples show only minor unit-cell contraction and can be modelled using the conventional tunnel site. At high concentration, Tl shows the most pronounced incorporation among the investigated elements, consistent with its monovalent character and its close crystal-chemical similarity to K. On the other hand, the behaviour of Pb^{2+} and Zn^{2+} is broadly consistent with a divalent exchange process, inducing only minor changes in the average structure. A local structural approach, however, reveals a more complex picture. EXAFS data agree with PXRD results in indicating that Tl is mainly hosted within the tunnels, with Tl–O and Tl–Mn distances consistent with a displaced tunnel-cation configuration. Pb and Zn, however, display first-shell environments incompatible with a simple location at the centre of the tunnel. Pb appears to occupy an asymmetric local environment, likely controlled by both cation size and lone-pair activity, whereas Zn is strongly displaced toward the tunnel walls, where it adopts a short Zn–O coordination. Thus, the three cations do not simply replace K in the same way: Tl mainly promotes axial disorder within the tunnel, Pb develops an asymmetric off-centre environment, and Zn approaches the tunnel walls. Overall, cryptomelane responds to heavy-metal incorporation through cation-specific mechanisms, ranging from tunnel-axis displacement to wall-directed coordination, rather than through a unique ion-exchange model.

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Structural response of leucite and kalsilite under non-ambient conditions: new insights from *in situ* synchrotron XRD

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Leucite (KAlSi₂O₆) and kalsilite (KAlSiO₄) are key framework silicates critical for modeling alkaline, ultrapotassic magmatic systems and mantle metasomatism [1-3]. Although their polymorphic behaviors under independent high-pressure (*P*) or high-temperature (*T*) regimes have been extensively documented [4-6], their structural responses and stability limits under simultaneous high *P-T* conditions remain poorly constrained. To address this gap, this study investigates the joint *P-T* behavior of both feldspathoids through *in situ* synchrotron X-ray diffraction, combining single-crystal and powder techniques with Large Volume Press (LVP) experiments.

Synchrotron-based X-ray diffraction experiments under extreme environments have revealed that a trend toward high structural complexity is a shared response among alkali feldspathoids. In the case of leucite, initial single-crystal data collected at the P02.2 (PETRA-III) and ID15B (ESRF) beamlines uncovered an unprecedented phase transformation above 2.2–2.5 GPa, where the tetragonal (pseudo-cubic) *I*4₁/*a* structure evolves into a highly complex trigonal symmetry (*R*-3), featuring four distinct tetrahedral positions and two independent K sites. The reproducibility of this transformation and the definitive mapping of its stability field, under simultaneous high *P-T* conditions, were subsequently achieved through Large Volume Press (LVP) setups at the ID06 beamline (ESRF). Mimicking this behavior, parallel high-pressure investigations on kalsilite have also brought to light new polymorphic configurations. Preliminary single-crystal XRD at ambient temperature and high pressure indicates a transition toward a complex supercell architecture, whereas combined *P-T* environments induce a rearrangement into a phase metrically analogous to nepheline (*c'* = 2*c*). Collectively, these experimental observations emphasize how high-resolution, *in situ* crystallographic techniques are indispensable for decoding the sophisticated atomic rearrangements that control framework silicate stability deep within the Earth.

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Seeding in mechanochemical reactions

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Mechanochemistry is an old but reemerging field of chemistry. Indeed, it does not require solvents, or high temperatures to perform fast and efficient chemical transformations and syntheses, making it an atom and energy efficient technique. To increase industrial use and research efficiency however, the community is greatly hampered by the lack of knowledge of how solid state reactions proceed at nanoparticle level [1]. Herein, we demonstrate that nucleation is a fundamental process happening in most solid state reactions. Through seeding various reactions, which until now were known for their unexplained long induction times, we reduced in many instances the induction times to the time required for particle comminution [2]. For some reactions, this allows unleashing of reaction rates where previously long milling minutes or hours had to pass before any meaningful product conversion could be achieved [3]. Seeding is already a widely known technique for modifying anorganic reaction paths and modifying product polymorphs, however, the scarce instances of reported reduction of induction times went until now mostly ignored [4–6]. This is the first attempt, to the authors knowledge, to systematically report on induction times seeding effects in mechanochemical reactions.

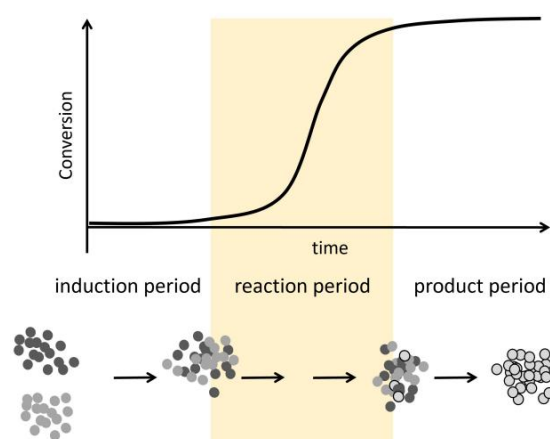


Figure 1. Kinetic profile of a typical mechanochemical reaction.

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Mineralogical and Phase Transformations in Prehistoric Ceramics from Colombare di Villa (Verona, Italy): Constraining Firing Technologies

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The prehistoric site of Colombare di Villa (Negrar, Verona, Italy) represents a multi-phase settlement occupied from the Late Neolithic (4200 cal BC) to the Late Bronze Age (10th century BC). Due to its proximity to the Lessini flint sources, the site's role as a specialized knapping area during the Neolithic and Copper Age transitions has been widely discussed [1]. Within the P:ANIS project, this study explores whether ceramic data can clarify settlement seasonality and exchange networks with Alpine sites. Furthermore, we tentatively evaluate manufacturing techniques as potential indicators of local craft traditions and interactions with different archaeological facies characterized by distinct technological practices.

A representative dataset of 200 non-diagnostic body sherds, equally split between the Late Neolithic and Copper Age, was investigated using a multi-analytical archaeometric approach. Mineralogical assemblages and microstructures were investigated via thin-section petrography, X-ray powder diffraction (XRPD), and thermal analysis (TG/DTA). Furthermore, an experimental refiring protocol (500–800 °C) coupled with SEM-EDS was designed to constrain firing dynamics, assessing potential temperature thresholds of irreversible phase transformations, such as carbonate decomposition, matrix-temperature reactions, and incipient sintering.

Preliminary macroscopic and petrographic sorting tentatively identified six fabric groups for the Late Neolithic and five for the Copper Age. Both assemblages display generally homogeneous mineralogical features, characterized by silicate-rich matrices with what appear to be deliberately added calcite and carbonate rock tempers. The widespread preservation of these carbonate inclusions, observed via XRPD and microscopy, suggests that extensive decarbonation reactions did not occur during manufacture. TG/DTA results and preliminary refiring observations seem to constrain the equivalent firing temperatures to a relatively low range, which might point to the use of simple pyrotechnological structures with limited control over temperature and atmosphere. The close technological and compositional similarities observed between the two datasets tentatively suggest a substantial continuity in raw material procurement and manufacturing choices between the Late Neolithic and Copper Age communities, though further data integration remains necessary to confirm these trends.

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MS5

New frontiers in X-ray sources for materials science

Structural characterization of bioactive antibacterial nanostructured surfaces by combined transmission and reflection X-ray scattering with advanced lab-scale setups

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X-ray scattering techniques, in the Small and Wide-Angle regimes (SAXS and WAXS), are suitable for investigating nano/bio-materials in different physical states, as liquid, gel and solid. Two complementary state-of-the-art laboratory setups (Rigaku [1] and Xenocs [2]) equipped with high brilliance X-ray micro-sources (Fr-E+ SuperBright and Excillum Metal jet) are available at Institute of Crystallography-CNR in Bari, enabling advanced structural investigations, typical of synchrotron facilities. The in-house developed software SUNBIM [3] is exploited to support data collection, reduction and processing.

Transmission and reflection (Grazing Incidence, GI) geometries are here successfully employed to achieve lateral spatial resolution and high surface sensitivity (WAXS and GIWAXS options, respectively). A combination of WAXS and GIWAXS was indeed used to study bioinspired Antibacterial Nanostructured Surfaces (ANSs) composed of calcium phosphate (CaP) nanoneedles. The measurements probed structure and texture across all material layers, from the amorphous CaP substrate (ACP tablet), through the intermediate hydroxyapatite crystallization layer, up to the crystalline nanoneedles at the top surface. The high degree of preferred orientation and crystallinity observed in the control sample was significantly reduced upon addition of a citrate stabilizer to the ACP tablet [4]. The modulation of nanoneedles structure and morphology was further investigated as a function of different stabilizers (citrate and hydroxycitrate) directly added to the growth solution, at varying concentrations, revealing strong inhibiting effects, including concentration-dependent changes in nanoneedles orientation and crystallinity.

Acknowledgements

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MS6

Crystal growth, from fundamentals to applications

Seashell Waste-derived Biogenic Calcium Carbonate powders as Sustainable Carriers for Crop Nutrients

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The valorisation of biogenic calcium carbonate derived from aquaculture shell waste represents a promising strategy for the development of sustainable multifunctional materials within a circular economy framework [1,2]. This work is part of the European PataFEST project [3] and aims to develop sustainable strategies for improving potato health and resilience through innovative and sustainable approaches. Within this framework, clam- and oyster-derived powders composed of two different calcium carbonate polymorphs, namely aragonite and magnesium calcite respectively, were investigated as potential matrices for the development of innovative nutrient-delivery systems in potato cultivation. These biofertilizer were designed to provide a controlled and slow-release source of essential nutrients while simultaneously supporting plant health and enhancing crop resilience against biotic and environmental stresses. With this aim, the biogenic CaCO_3 powders were loaded with macro and micro-nutrients such as P, Fe, Zn, Mo and B through treatment with their inorganic precursors, exploiting different preparation strategies, namely aqueous impregnation (M1), wet milling (M2) and dry mechanochemical milling (M3) (Figure 1). A multidisciplinary approach combining ICP-MS, PXRD, FTIR, TGA and SEM-EDS analyses was adopted to characterize the resulting materials, in term of nutrient incorporation, elemental distribution, structural transformations and thermal stability. Our results indicate a significant influence of both the processing method and nutrient nature on incorporation efficiency and phase evolution. Mechanochemical treatments generally enhance nutrient incorporation, particularly for phosphorus, promoting the formation of phosphate-based phases with increased thermal stability. Nutrient incorporation was also found to be precursor-dependent, with iron exhibiting the highest sensitivity type. Lastly, also the polymorphic nature of the biogenic calcium carbonate substrates significantly affected reactivity and transformation pathways.

In conclusion, this study explores the potential of seashell waste as versatile crystalline platforms for nutrient incorporation relevant to agriculture, highlighting how processing routes and precursor chemistry can be leveraged to tailor incorporation efficiency and polymorphic transformations.

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Synthesis of ink-ready PbO nanopowders for large-area printed radiation detectors

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Lead oxide (PbO) is a promising material for direct-conversion X-ray detectors owing to its high atomic number and large X-ray attenuation coefficient, which enable thinner detection layers and improved performance at higher photon energies. In this work, we present a novel, cost-effective synthesis and deposition route for PbO-based detectors for X-ray and soft γ -ray applications. Starting from β -PbO powder, a liquid-assisted ball milling process was employed to promote partial transformation into the α -phase, yielding a nanostructured ink suitable for printing technologies. The ink was deposited onto FTO substrates by the Doctor Blade technique, producing homogeneous and compact PbO films.

SEM and XRD analyses confirmed the crystallinity of the material, with an α -PbO phase purity exceeding 97%.

Detectors obtained from these films showed stable and reproducible current responses under X-ray irradiation at various energies.

These results demonstrate that a simple, scalable, solution-based fabrication route can deliver PbO layers suitable for flexible, large-area radiation detection systems, particularly in the low-energy domain (<200 keV).

Nitrogen incorporation in MOVPE-grown planar and core-shell nanowire III-V hetero-structures for intermediate-band solar cells application

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Intermediate band (IB) III-N-V semiconducting alloys (such as GaNAs or GaNP) within a free-standing III-V nanowires (NWs) represent promising candidates for fabrication of next-generation high-efficiency solar cells (SCs) [1,2,3,4]: while IB materials ensures multiple-band absorption within a single-junction SC [5], core-(multi)shell NW heterostructures provide enhanced solar radiation absorption thanks to nano-antenna and light wave-guiding effects [6].

We report for the first time on low-pressure MOVPE of GaAs-GaNAs-AlGaAs core-multishell NWs using tertiarybutylhydrazine, trimethyl-gallium, trimethyl-aluminum and tertiarybutyl-arsine. Dense arrays of GaAs NWs were grown at 400°C on (111)B-GaAs by the Au-catalyzed method, and overgrown by multiple shells, initiating with a GaNAs shell (grown at 540-560°C), and followed by a second AlGaAs shell for passivation. Different V:III and N:As ratios in the vapor and nominal planar growth rates during GaNAs shell growth were used to study the degree of N-incorporation into the GaNAs shell. Planar GaNAs epilayers were also grown on (100)-, (111)B-, (110)-oriented GaAs to compare their N incorporation. Experiments were performed on (110)-GaAs, as this orientation coincides with that of the NW sidewall facets.

Photoluminescence (PL) and high-resolution X-ray diffraction (HRXRD) analyses were carried out on GaNAs epilayers to evaluate N incorporation. All investigated samples exhibited a near-infrared (NIR) broad emission band, ascribed to charge carrier radiative recombination across the E₋ band gap of the GaNAs alloy, the N concentration in planar GaNAs alloys reaching values as high as 0.6% at high growth rates (2.32 μm/hr). A significantly lower N-content was detected by PL for the as-grown NWs when compared to planar samples grown under the same condition, the N concentration in the GaNAs shells further decreasing with increasing the nanowire array density onto the GaAs substrate (observed by SEM). The results are interpreted in terms of N-content variations within the GaNAs shells with their actual growth rate, as estimated based on a previously reported multi-shell nanowire growth model [7].

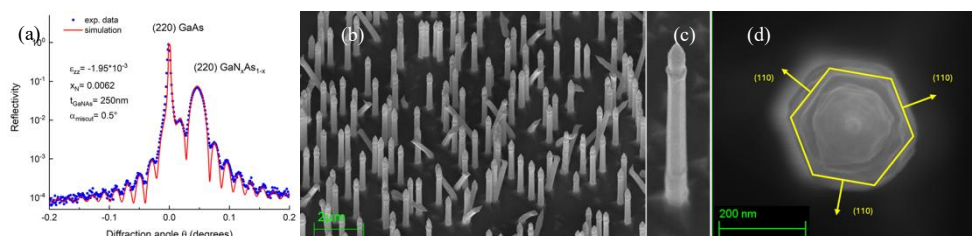


Figure 1. (a) Experimental (blue dots) and simulated (red curve) HRXRD patterns of a planar GaN_xAs_{1-x}/(110)GaAs sample. Tilted-view of (b) an ensemble of core-shell NW array (density: 2.2–2.4·10⁸ cm⁻²), and (c) a single-NW. (d) Plan-view of a single NW showing six sidewall facets aligned normal to the <110> in-plane substrate directions.

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Growth and characterization of freestanding $\text{LaAlO}_3/\text{Sr}_2\text{RuO}_4$ oxide micro membranes

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Functional oxide materials are a rich platform for exploring emergent phenomena arising from the interplay of lattice, charge, orbital, and spin degrees of freedom. The recent development of freestanding oxide membranes has enabled the investigation of low-dimensional physics beyond substrate-induced constraints [1,2]. In particular, oxide heterostructures offer unique opportunities for engineering electronic properties, as demonstrated by the fabrication of freestanding $\text{LaAlO}_3/\text{SrTiO}_3$ (LAO/STO) micromembranes using a strain-engineering approach combined with RHEED-assisted pulsed laser deposition [3,4]. Extending this strategy, we have fabricated freestanding $\text{LaAlO}_3/\text{Sr}_2\text{RuO}_4$ (LAO/SRO) micromembranes to explore strain-dependent superconductivity in SRO. The LAO layer was deposited by RHEED-assisted pulsed laser deposition on high-quality SRO single crystals grown by the optical floating-zone method. Structural and compositional analyses, including SEM and EDX, confirm the successful realization of the LAO/SRO bilayer and provide insight into its morphology and crystallographic properties. Preliminary scanning TEM observations, together with possible correlations with magnetic measurements, are also presented. We also demonstrate membrane manipulation and transfer onto pre-patterned Si substrates. These freestanding LAO/SRO micromembranes offer a versatile platform for studying strain-tunable superconductivity and emergent interfacial phenomena without substrate-induced constraints.

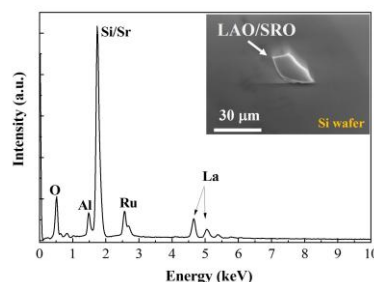


Figure 1. EDX spectrum of the LAO/SRO micro-membrane on a silicon wafer, shown in the inset SEM image. The data confirm that the sample contains elements from both the film and the substrate.

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The complex polymorph landscape generated by a [2+2] photocycloaddition of (E)-2-furyliden-oxindole: the crystal structures of the elusive intermediate states

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Once upon a time I attended my first congress together with P. Redana (XXVI Congresso AIC, Alessandria 1-4 Settembre 1996) and he presented a poster describing his master thesis, i.e the crystal structures of the reactant and of the recrystallized product of the [2+2] photocycloaddition of (E)-2-furyliden-oxindole (Figure 1 left). Moreover, in that poster the existence of one or two intermediate polymorphs was envisaged. The abstract ended with the sentence “some more work is needed”. “Only” 30 years after, the crystal structures of the two intermediate dimer polymorphs were solved. It was thus possible understanding at the molecular level the photosensitive effect observed exposing the reactant to UV light. The procedure to obtain the two intermediate phases, named α and β in a form suitable to solve their crystal structure was optimized. The complex polymorph landscape was elucidated (Figure 1 right), individuating the effect of the morphology of the starting reactant, its grinding, UV light power, time of illumination, exposition of solvents, humidity, mild thermal treatments, and ageing in dark in determining the reaction evolution. The crystal structure of the first intermediate (α) just after UV illumination of the monomer (2-3 hours with occasional shaking) was finally solved and refined by 2D X-ray powder diffraction data. The dimer is in the same molecular conformation of the recrystallized phase γ (solved by single crystal in P. Redana thesis in 1996) but with a packing similar to the reactant (confirming the minimal motion principle, often governing solid-state reactions) and a larger $P-1$ unit cell with two molecules in the asymmetric unit. Wetting of pure α with ethanol causes its complete conversion to β , a relaxed version of α with an increased density (but still smaller than γ) and a different molecular conformation, with a 120° rotation of the furyl moieties. Hirshfeld and energy framework analysis confirmed an unstable packing of α with the transformation toward β if the stimulus is applied at the solid state. Noteworthy the re-crystallization of solutions obtained dissolving β produces again β . IR data confirmed that α and β have different local environments, in agreement with the different molecular conformation (furyl rotation) observed in the solid state. SEM images demonstrated that the reactant crystallites undergo an elongation at the micron scale along the b-axis in the first 4 minutes of UV illumination, followed by their cracking in smaller crystallites. A very small UV dose with a negligible number of molecular dimers is thus able to induce a marked macroscopic strain. This contribution is dedicated to the memory of Prof. D. Viterbo, who pursued and guided us toward the solution of this intriguing polymorph puzzle.

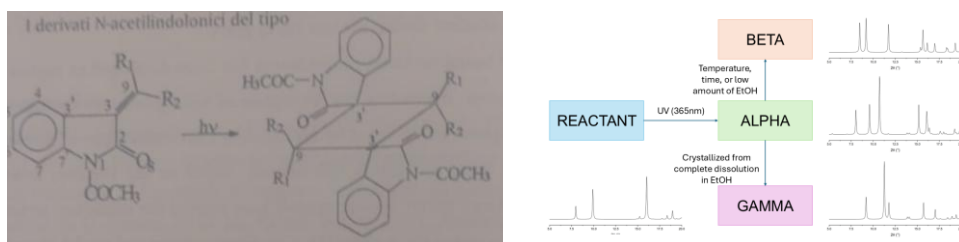


Figure 1. Monomeric reactant and recrystallized dimer (R₁=furyl, R₂=H) reproduced from the original 1996 AIC abstract (left), and the polymorph landscape finally elucidated (right), 30 years later.

Sol–Gel-Assisted In Situ Nucleation of Carbonated Hydroxyapatite in Chitosan/ β -Glycerophosphate Thin Films for Bone Tissue Regeneration

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Hydroxyapatite (Hap), the principal inorganic constituent of bone, is increasingly used in biomimetic composites that replicate the organic–inorganic architecture of native hard tissue [1–4].

In this work, thin films were prepared by casting, comprising a chitosan matrix crosslinked with β -glycerophosphate and a Hap-incorporated composite in which the inorganic phase was nucleated and grown directly within the organic matrix via a sol–gel approach. The inorganic-to-organic ratio was systematically varied to assess the quality of the composite material.

Samples were characterized by SEM-EDS, FTIR, μ -Raman spectroscopy, and XRPD, revealing a multilayered microstructure in the composite material. This layered structure was attributed to diffusion-controlled nucleation and growth of calcium phosphate phases. FTIR analysis confirmed the coexistence of two distinct phosphate environments: a crystalline apatite phase, evidenced by characteristic ν_3 PO₄ stretching and ν_4 PO₄ bending bands, and a non-crystalline contribution from β -glycerophosphate. A carbonate band further confirmed the presence of biomimetic carbonated apatite, with this band being surprisingly more prominent in the mineral fraction grown within the chitosan matrix than in the sol–gel-grown mineral fraction used as a reference.

The results demonstrate that the chitosan/ β -glycerophosphate matrix supports the nucleation of nanocrystalline, carbonated, calcium-deficient Hap under physiological temperature and pressure conditions, yielding a composite whose characteristics closely resemble those of bone, underscoring its potential as a biomimetic platform for bone tissue regeneration.

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Structure–Property Relationships in Low-Dimensional Triphenylsulfonium based Organometal Lead Halide Perovskitoids

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Organometal halide perovskitoids (OMHPs) based on small aliphatic sulfonium cations have recently attracted increasing attention owing to their remarkable structural and property versatility, as well as the possible occurrence of charge-assisted chalcogen-bonding interactions. Furthermore, unlike conventional nitrogen-based cations, sulfonium cations lack acidic hydrogen atoms that favor interactions with water, leading to enhanced moisture stability.

In particular, the bulky triphenylsulfonium cation $[(\text{Ph})_3\text{S}^+]$ promotes the formation of low-dimensional hybrid architectures,[1] where subtle variations in the crystallization conditions can strongly influence the resulting framework topology. Low-dimensional anionic frameworks are generally more stable than their higher-dimensional perovskitoid counterparts, although they often exhibit less favorable electronic properties.[2]

In this work, we investigate a family of six polymorphic compounds derived from a common triphenylsulfonium precursor by selectively changing the solvent. Structural characterization by single-crystal X-ray diffraction reveals a remarkable diversity of structural motifs, despite the use of very similar synthetic conditions.

The role of chalcogen bonding and other weak intermolecular interactions in directing the assembly of the inorganic framework has been investigated through charge-density analysis within the Quantum Theory of Atoms in Molecules (QTAIM) framework.

Several key properties relevant to photovoltaic applications are also discussed. First, the exceptional stability of these compounds under ambient conditions has been experimentally assessed and rationalized. Second, their electronic properties have been thoroughly investigated by UV–Vis absorption and emission spectroscopy and interpreted in conjunction with periodic electronic structure calculations.

Finally, a correlation between the structural features and the observed physical properties is established. These results provide new insights into previously underexplored aspects of the structural chemistry of this remarkable family of hybrid perovskitoids.

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**The role of applied mineralogy in bridging resources,
materials, environment and health**

Microplastics and Carbonate Precipitation: Implications for Carbon Sequestration in Marine Ecosystems

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Plastic pollution and climate change are among the most pressing environmental issues of our time. Marine ecosystems are particularly vulnerable, accumulating both polymer debris and microplastics formed when these materials break down due to weathering. They also absorb large amounts of atmospheric carbon dioxide. Therefore, the interplay between microplastics and geochemical carbon sequestration is a question worth asking. Yet it remains poorly explored so far.

This study investigates the impact of a variety of microplastics on the capture of carbon through carbonate growth in artificial seawater. Carbonation experiments were carried out by introducing microplastics into supersaturated calcium carbonate solutions of different compositions and monitoring the precipitation that occurred at room temperature. The materials were then characterized mineralogically and morphologically using powder X-ray diffraction, Raman and infrared spectroscopy, zeta potential measurements and scanning electron microscopy.

In general, plastics can actively interact with carbonate precipitation processes by acting as heterogeneous nucleation substrates within aqueous systems, already proven by [1]. The electrostatic charge on the surface of microplastics promotes cation adsorption and may influence carbonate nucleation. Compostable polymers were found to have strong mineral-polymer interactions: carbonate crystals were embedded in the polymer matrix, and polymer fragments were found in the mineral phase. Further findings included the preferential precipitation of gypsum over carbonate phases on some compostable polymers, indicating localized changes in geochemical conditions and a shift in the evaporitic sequence [2].

Overall, these results suggest that microplastics are not merely passive contaminants in marine systems, but can actively take part in mineral precipitation, which could potentially impact the efficiency of natural carbon sequestration and, more widely, the geochemical interactions within natural environments.

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From Crystal Structure and Crystal Chemistry to Predicted Toxicity: The Case of Fibrous Datolite

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Mineral fibres are widespread in both natural and anthropogenic environments, making their characterization and assessment of potential health effects essential. The characterization of newly recognized fibrous minerals is particularly important for the development and validation of predictive models capable of estimating the toxicity and carcinogenicity potential of mineral fibres based on their physical, chemical, and morphological properties. Among the parameters most strongly influencing fibre toxicity are size and morphometric characteristics, biodurability, surface properties, and the presence of iron or other potentially toxic elements.

Within this framework, this study [1] presents the first comprehensive characterization of fibrous datolite, $\text{CaBSiO}_4(\text{OH})$, a calcium boron hydroxide nesosilicate that typically occurs in non-fibrous habits. A rare pearly-white fibrous occurrence was identified within an opicalcite from the Rio Manubiola Valley (Parma, Northern Italy). Although datolite is a relatively common boron-bearing mineral and is locally exploited as a boron ore, its fibrous habit has received little attention in the literature and its potential health effects remain unexplored.

The investigated material was characterized through a combination of mineralogical, crystallographic, and chemical techniques. Fibrous datolite was found to be chemically and structurally equivalent to non-fibrous datolite, crystallizing in the monoclinic space group $P2_1/c$, with $a=4.83679(1)$ Å, $b=7.61346(2)$ Å, $c=9.63754(3)$ Å, $\beta=90.156^\circ$. Morphometric analysis revealed average fibre length, width, and aspect ratio values of 9.80 µm, 1.32 µm, and 9.53, respectively. According to the World Health Organization criteria, a significant proportion of the fibres therefore falls within the respirable size range.

To assess its potential biological hazard, the Fibre Potential Toxicity Index (FPTI) was applied [2]. This quantitative model integrates crystal-chemical, morphological, and surface-related parameters to estimate the relative toxicity and carcinogenicity potential of mineral fibres. Despite the respirable dimensions of the fibres, fibrous datolite yielded an exceptionally low FPTI value of 1.50(1), among the lowest reported for mineral fibres investigated to date. This result is mainly attributable to the absence of iron and other toxic metals in the crystal structure, together with its predicted low biodurability.

The negligible toxicological potential predicted for fibrous datolite suggests that this mineral could represent a suitable negative reference material for in vitro toxicity studies, similarly to other non-hazardous fibrous phases such as wollastonite.

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The role of applied mineralogy-crystallography in developing sustainable environmentally-friendly materials. The case of the green synthesis of diopside from hazardous waste

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Diopside, ideally $\text{MgCaSi}_2\text{O}_6$, is a common rock-forming mineral that belongs to the pyroxene group of silicates characterized by single chains of Si-centred tetrahedra [1,2]. Diopside crystallizes in the monoclinic system with space group $C2/c$ ($Z = 4$) and unit cell parameters $a = 9.746 \text{ \AA}$, $b = 8.899 \text{ \AA}$, $c = 5.251 \text{ \AA}$ and $\beta = 105.63^\circ$ [2,3]. Natural or synthetic diopside based raw materials have several novel high-value industrial or technological applications [4,5]. A green and efficient route to synthesize pure diopside using recycled hazardous mineral wool waste is presented, marking the first successful transformation of this waste into a valuable ceramic material like diopside. Hazardous mineral wool undergoes industrial thermal inertization to form an inert glass (Re.Wo) to be used at up to 40 wt.% in the diopside formulation. Chemical-grade reagents generally used for the diopside synthesis are replaced by waste and natural raw materials. Unlike other waste-based approaches, this method requires no energy-intensive pre-treatments and avoids CO_2 -emitting inputs. The resulting diopside exhibits broad application potential, including biomedical devices, low-expansion dielectric ceramics, photo-catalysis, and environmental remediation—leveraging transition metals within the crystal structure to enhance catalytic performance. Importantly, the process also immobilizes hazardous elements like Cr without generating carcinogenic crystalline silica phases, offering both environmental and functional advantages.

Acknowledgments

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The “Mamma Caura” Geomaterial as a Natural Biofilter: reuse of a Byproduct in Artisanal Salt Production in the Trapani-Marsala Saltworks

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Traditional and small-scale Mediterranean saltworks have gradually declined, giving way to the mechanization of operations and large-scale production [1]. However, certain artisanal practices endure over time, becoming a vital part of the local cultural heritage. Among these, in the Trapani-Marsala saltworks (Sicily, Italy), the reuse of "mamma caura", historically known as “salt yeast”, stands out. This gypsum-based byproduct appears as a dark sediment that accumulates in the warm evaporation ponds. Its centuries-old application, passed down through generations, has consistently ensured the production of clean, high-quality edible sea salt. This study aims to investigate and validate the role of this geomaterial within the production cycle, focusing on the biogeochemical interactions that occur in this hypersaline ecosystem.

Saltwork pond sediments and the "mamma caura" were analyzed via X-ray diffraction (XRD) for mineralogical characterization, and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS) for detailed morphological observations and chemical analyses. Additionally, the pigmented gypsum crystals of the "mamma caura" were analyzed using Raman spectroscopy, enabling the identification of carotenoids produced by the halophilic biomass inhabiting the ecosystem [2].

The analyses clearly demonstrate the interaction and synergy established between the living biomass and gypsum crystallization under specific precipitation conditions. Specifically, solid inclusion processes take place during crystal growth, a phenomenon closely driven by the interplay between supersaturation conditions and crystal growth rates. Not only do gypsum crystals preserve the signature of the living biomass within a specific spatial and temporal framework [3], but in the context of the salt production cycle, this bio-mineral synergy endows the geomaterial with a bio-filtration capacity. This allows the gypsum matrix to act as a natural, selective filter that traps organic matter, ultimately yielding a cleaner edible salt. The findings of this study not only elucidate the biogeochemical processes underlying a centuries-old empirical practice but also promote the valorization of a byproduct, upgrading it to a biotechnological resource. This underscores the efficacy of local artisanal knowledge, which aligns perfectly with modern circular economy principles.

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CO₂-assisted hydrothermal upcycling of inertized rock and glass wool into zeolitic sorbents

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Thermally inertized rock wool (RE.WO) is a non-hazardous glass rich in Si, Al and Ca, but its large-scale reuse still requires high-value outlets [1]. We investigated a low-temperature alkaline hydrothermal route that combines zeolite crystallization with CO₂ retention. RE.WO was treated with 1 M NaOH at 130 °C for 24-96 h either in air or under a CO₂-enriched headspace. The atmosphere strongly controlled the reaction pathway. In air, the Ca-rich system mainly produced 11-A tobermorite and transient CaCO₃ polymorphs, whereas zeolite formation was limited to minor analcime. Under CO₂, carbonate speciation shifted toward stable calcite, which acted as a calcium sink and lowered the effective Ca/Si ratio, thereby favouring Na-P and Na-phillipsite crystallization. After 96 h, the total zeolitic content increased from 2.4 wt% in air to about 9.9 wt% in CO₂, and the cation exchange capacity of the synthesized geomaterial shifted from 12.3 ± 0.9 to 40.7 ± 1.6 cmol(+)/kg. At the same time, CO₂ retention increased from 2.41 ± 0.33 to 4.49 ± 0.36 wt%, with better stability over time in the CO₂ system. These results indicate that CO₂ is not only fixed by mineral carbonation, but also drives phase selection during synthesis [2]. Coupling zeolite production and CO₂ sequestration could therefore provide a circular-economy route for upgrading mineral-wool products through the *End-of-Waste* process.

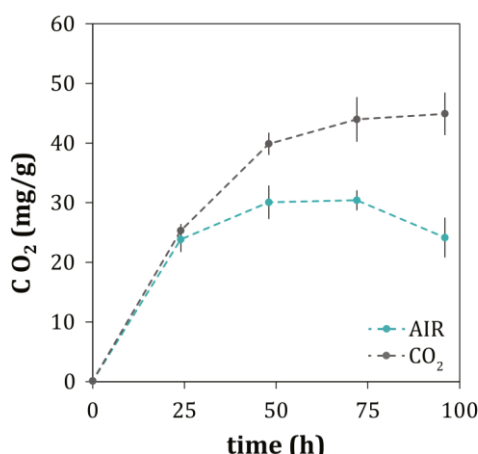


Figure 1. CO₂ converted into carbonates during alkaline hydrothermal treatment in air and under a CO₂-enriched atmosphere.

Acknowledgments

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Non-Equilibrium Sintering Pathways in ceramics: SPS vs Conventional Firing

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Understanding densification mechanisms in ceramic systems under high-temperature conditions is critical for the development of energy-efficient and high-performance processing routes. In this work, Spark Plasma Sintering (SPS) is investigated not only as a rapid consolidation technique but as a methodological tool to probe non-equilibrium sintering pathways, in direct comparison with conventional firing.

An industrial ceramic batch (BRB01) was processed via conventional sintering (1000-1200 °C, 15-45 min dwell) and SPS (900-1100 °C, 5 min dwell, <30 min total cycle). The SPS approach combines rapid heating rates (up to 200 °C min⁻¹), pulsed electric current, and uniaxial pressure, enabling volumetric Joule heating and enhanced mass transport under tightly controlled thermal conditions.

A multi-technique characterization strategy (density and porosity measurements, SEM-EDS, XRPD, FTIR, and colorimetry) reveals a fundamental shift in sintering mechanisms between the two processes. Conventional firing is dominated by thermally activated viscous-flow-assisted densification, driven by liquid-phase formation and progressive dissolution of feldspathic and quartz phases, resulting in pore elimination, mullite crystallization, and microstructural homogenization. In parallel, diffraction analysis indicates that, under SPS conditions, mullite formation and feldspar dissolution proceed effectively and shift to lower temperatures than under conventional firing, indicating an earlier onset of liquid-phase development. Despite this accelerated phase evolution, the glassy phase shows greater chemical and structural heterogeneity, suggesting that rapid heating rates and short dwell times limit melt homogenization under non-equilibrium conditions.

In addition, SPS enables densification at temperatures reduced by 100–200 °C, with significantly lower residual porosity and drastically shorter processing times. This behavior is attributed to the combined action of electric current and applied pressure, which promotes rapid particle rearrangement, accelerated diffusion, and early-stage neck formation. The short dwell time effectively suppresses grain growth, leading to refined and highly compact microstructures.

The comparison highlights a transition from equilibrium, thermally-driven sintering regimes to non-equilibrium conditions in SPS, where densification and phase evolution are partially decoupled. These findings point to distinct kinetic pathways governing microstructural development and phase stability, emphasizing the role of coupled thermo-electric-mechanical effects in controlling sintering at elevated temperatures.

MS8

Protein structures for modeling, function and drug design

Structural basis for mycobacterial HtpG chaperone activation of Toll Like Receptor 4

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HtpG_{Mtb} of *Mycobacterium tuberculosis* is an ATP-dependent heat shock protein that assists the correct folding of nascent and stress-accumulated misfolded proteins, in concert with other chaperones. Besides playing a role in stress response, it is able to elicit an immune response against *M. tuberculosis* infection by activating Dendritic Cells in a Toll Like Receptor 4 (TLR4)-mediated manner [1-3]. However, we lack a full understanding of the molecular determinants of HtpG_{Mtb} catalytic activity and Toll Like Receptor 4 activation, due to the lack of structural and biophysical data. Here, we report the first crystal structure of HtpG_{Mtb}, in complex with the non-hydrolysable form of ATP. The crystal structure shows that the *M. tuberculosis* HtpG_{Mtb} dimer adopts a conformationally silent architecture that prevents the catalytic domains from dimerising, thereby abolishing the ATP-hydrolytic activity typical of active chaperones [1]. Complementary binding studies reveal that HtpG_{Mtb} engages TLR4 with nanomolar affinity and that its dimeric organisation enables simultaneous binding of two receptor molecules. This structural arrangement suggests that HtpG_{Mtb} activates TLR4 by driving receptor dimerisation, an essential prerequisite for initiating downstream signalling.

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Structural characterization of a thermophilic PETase isolated from a geothermal environment

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Driven by the urgent environmental challenge of poly(ethylene)terephthalate (PET) waste accumulation, scientific research has increasingly shifted toward sustainable recycling strategies [1]. In particular, the enzymatic degradation of PET via specialized PETases has become a major focal point. Scaling up this industrial process demands thermally stable hydrolases able to function at the high temperatures needed to increase plastic flexibility near its glass transition point [2,3]. In this study, we present a detailed structural analysis of DIEGO (*Depolymerising Isolated Enzyme from Geothermal Origin*), a thermophilic PETase discovered in geothermal metagenomes. DIEGO enables the one-step bioconversion of PET waste into TPA and EG at 60 °C [4]. Operating at this elevated temperature improves substrate accessibility and triggers the spontaneous breakdown of MHET and BHET intermediates, resulting in the complete degradation of post-consumer PET within 96 hours [4]. Structural characterization in both solution and crystalline states demonstrated that DIEGO adopts a classic α/β -hydrolase fold, comprising a core of nine β -strands flanked by ten α -helices. The enzyme exhibits remarkable intrinsic thermostability, featuring a melting temperature of approximately 89°C. Consistent with established PETase behavior, this stability is further enhanced by adding divalent cations (Mg^{2+} and Ca^{2+})[5]. Crystal structure analysis confirmed the presence of the canonical Ser-Asp-His catalytic triad and a single C-terminal disulfide bond. DIEGO was evaluated in both its free form and bound to PEG, which serves as a partial mimic for the PET substrate. Notably, a specific magnesium-binding site was resolved, which reduces local conformational flexibility. Comparative structural analyses with the mesophilic *Piscinibacter sakaiensis* (formerly *Ideonella sakaiensis*) PETase (IsPETase) and the thermophilic Leaf-Branch Compost Cutinase (LCC) highlighted the distinct conformational traits of this novel enzyme. Combining high-temperature activity with robust scaffold stability, DIEGO represents a highly promising candidate for industrial PET upcycling and circular economy purposes.

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Same Molecule, Different Strategies: Active-Site Dynamics reveal Divergent Structural Adaptation of Alcohol Dehydrogenases to Ethanol Metabolism

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Ethanol is one of the most biologically relevant small organic molecule present in nature and is widespread across living organisms. It is naturally produced through the fermentation of sugars by yeasts or plants and is therefore commonly present in fermenting fruits, plant tissues, and other sugar-rich environments. Ethanol has also acquired major physiological, evolutionary, and cultural significance in animals and humans due to its widespread consumption and metabolism. Despite their different metabolic requirements whether related to ethanol production or detoxification, these organisms share a common molecular component: Alcohol Dehydrogenase (ADH).

Contrary to what its name might suggest, the most evolutionary widespread member of the alcohol dehydrogenase family primarily acts on S-nitrosoglutathione (GSNO), a ubiquitous endogenous S-nitrosothiol that serves as a major reservoir and transporter of nitric oxide in living organism's process [1]. Phylogenetic studies suggest that the alcohol dehydrogenases involved in ethanol metabolism independently evolved from this ancestral GSNO reductase-like enzyme in both plants and animals [2,3].

In this work, we obtained a new structural conformation of ADH1 from *Arabidopsis thaliana* and used it as a starting point to investigate active-site dynamics through analysis of the first principal component derived from experimentally determined conformations. Using structures available in the Protein Data Bank, the same computational approach was applied to ADH1E from *Equus caballus* and to GSNOR enzymes from *Homo sapiens* and *Solanum lycopersicum*. Our analyses revealed both the structural adaptation of the active site toward ethanol-related functions and the conformational differences between ADHs from ethanol-producing organisms and those from ethanol-consuming organisms. These findings provide new insights into the evolutionary relationship between protein structure, conformational dynamics, and biological function.

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Ligand-Driven Allosteric Rearrangements of the Sigma-1 Receptor Revealed by Molecular Dynamics

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The sigma-1 receptor (S1R) is a membrane-embedded chaperone located at the endoplasmic reticulum membrane, where it regulates the cellular stress response by maintaining the Ca^{2+} homeostasis and redox balance. Crystallography data have revealed that the receptor presents a trimeric architecture, yet biochemical studies have identified higher-order oligomeric states that appear to play distinct functional roles [1,2].

Interestingly, S1R ligands, both agonists and antagonists, differently alter the oligomerization pattern. Moreover, changes in assembly have been associated with neurodegenerative diseases, such as amyotrophic lateral sclerosis, suggesting a connection between supramolecular organisation and S1R functional response. All these elements establish the oligomerization as a central regulatory mechanism and promising therapeutic target in S1R-associated clinical conditions. To investigate this mechanism, we examined S1R bound to haloperidol or pentazocine through molecular dynamics simulations. We analysed interface stability, residue-level interactions, and the protein network that may regulate oligomer formation. We found that, although the two ligands occupy the same binding pocket, they remodel communication pathways in a distinct manner. The binding induces changes that propagate through a network of interacting helices and loop elements, defining a continuous allosteric route from the ligand site to monomer-monomer contacts.

This arrangement provides a structural basis for ligand-dependent modulation of S1R assembly. By identifying protein hotspots for communication between different regions of the protein, our computational approach potentially enables virtual screening of drug candidates that could selectively stabilize or disrupt specific oligomeric interfaces, redirecting aberrant oligomerization patterns observed in clinical conditions.

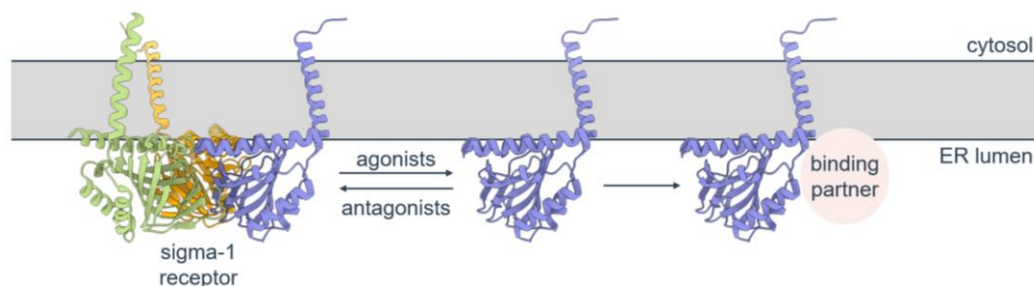


Figure 1. Schematic model of sigma-1 receptor (S1R) ligand-dependent conformational dynamics

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PROTACs vs Flaviviruses: A New Antiviral Frontier

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Flaviviruses are vector-borne RNA viruses causing severe human diseases and recurrent outbreaks [1]. Among them, West Nile virus (WNV) has emerged as a relevant human pathogen and is now endemic in Italy. Its enzootic transmission cycle, involving birds as reservoir and mosquitoes as vectors, enables rapid dissemination and increases the risk of outbreaks. Despite its public health relevance, no approved antivirals or vaccines are currently available [2], highlighting the urgent need for innovative drug-discovery strategies.

In this context, we propose a proteolysis-targeting chimera (PROTAC) strategy to selectively degrade the flaviviral NS2B-NS3 protease, which is essential for the viral replication. PROTACs are heterobifunctional molecules designed to induce ubiquitination and subsequent proteasomal degradation of a protein of interest by bringing it into close proximity with an E3 ubiquitin ligase[4].

Starting from available structural information we have developed a series of PROTAC molecules. Among them, we identified a promising PROTAC candidate with low micromolar IC₅₀ value. MicroscaleThermoforesis (MST) studies confirm the cooperative formation of a ternary complex between WNV NS2B-NS3 and VHL mediated by the PROTAC. Structural studies of this ternary complex are ongoing to elucidate the molecular details of PROTAC-induced substrate recruitment to the E3 ligase machinery. These insights will guide the rational optimization of next-generation PROTACs against emerging flavivirus infections.

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Structural Basis of Human Prothrombin Conformational Modulation by Anti-Thrombin Aptamers

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Disorders of blood coagulation represent a major cause of mortality worldwide, driving the search for anticoagulant agents capable of selectively modulating key steps of the coagulation cascade [1,2]. Among emerging therapeutic strategies, nucleic acid aptamers targeting thrombin have attracted considerable interest due to their high specificity and favorable pharmacological properties, and several candidates have been evaluated in preclinical and clinical studies [3]. Notably, anti-thrombin aptamers exert a dual anticoagulant effect by directly inhibiting thrombin activity and by interfering with the activation of its precursor, prothrombin [4-7].

To elucidate the molecular determinants of this mechanism, we investigated the interaction between anti-thrombin aptamers and human prothrombin using an integrated biophysical and structural approach. Calorimetric and spectroscopic analyses demonstrated that these aptamers recognize thrombin exosite I and the corresponding proexosite I of prothrombin with comparable affinity. Structural characterization by small-angle X-ray scattering (SAXS) of the complex between prothrombin and NU172, the only anti-thrombin aptamer currently in advanced clinical development [8], revealed key features of the recognition mechanism. In particular, aptamer binding to proexosite I was found to shift the open-closed conformational equilibrium of prothrombin [9] toward the open state, a finding further supported by limited proteolysis experiments.

Taken together, these results provide new insight into the structural determinants of anti-thrombin aptamer recognition of prothrombin and their potential impact on prothrombin activation.

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Structure-guided discovery of SARS-CoV-2 PLpro inhibitors within the AVITHRAPID platform

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The EU-funded AVITHRAPID project aims to develop **broad-spectrum antivirals** through an integrated pre-clinical drug discovery workflow combining molecular modelling, biochemical and cell-based assays, macromolecular crystallography, medicinal chemistry and biophysical characterization. Within this framework, **SARS-CoV-2 papain-like protease (PLpro)** is a particularly attractive target because it is essential for viral polyprotein processing and also counteracts innate immune signaling through deubiquitinating and deISGylating activities [1].

To support **structure-based drug design**, multiple PLpro constructs were produced, including the catalytic domain, an N-terminal deleted construct (Δ Ub-PLpro) and a C-terminal extended PLpro-NAB construct containing the nucleic-acid-binding domain. **SAXS** analysis of PLpro-NAB revealed an elongated architecture with a flexible interdomain region, while biochemical assays and grating-coupled interferometry on wild-type and Delta-variant mutants (S466R, T467K) showed reduced catalytic activity and lower affinity for the natural substrate proISG15, highlighting the contribution of the NAB region to substrate recognition.

In parallel, **screening campaigns** based on repurposing collections and the European Lead Factory library identified **CPI-169** as a novel non-covalent PLpro inhibitor. A multi-technique validation pipeline, including fluorescence and MS-coupled enzymatic assays, thermal shift analysis, **STD-NMR** and **tr-NOESY**, showed that CPI-169 is a specific redox-independent inhibitor binding the same S1 region as the reference ligand GRL-0617, and antiviral activity was confirmed in Vero-E6 cells [2, 3]. **Crystals** obtained with Δ Ub-PLpro were collected at the Elettra XRD2 beamline yielding the **first co-crystal structure** with an ELF-derived hit, providing a structural framework for ongoing hit-to-lead optimization within the project.

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MS9

Advanced materials for Sustainable Energy

Unveiling crystal structure, morphology and surface termination of CuInS₂ Quantum Dots by X-ray Total Scattering

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Cu-based ternary quantum dots (QDs) are emerging as a green alternative to conventional Cd- and Pb-based semiconductors, combining reduced toxicity with size-dependent optical tunability. Among them, CuInS₂ is a direct band gap I-III-VI₂ semiconductor, featuring broad UV-visible absorption and tunable emission peaks from 650 to 900 nm. These properties make it a promising candidate for sustainable optoelectronic and energy-harvesting applications, including luminescent solar concentrators and light-emitting diodes [1, 2].

CuInS₂ QDs with selected size and morphology have been synthesized via heat-up method [3]. The present study focused on characterizing the structure and microstructure of samples of different sizes (2-10 nm) to establish correlations with optical properties.

The study addresses a current gap in the literature concerning a comprehensive X-ray-based characterization of phase stability, crystal structure, and defectiveness in colloidal CuInS₂ QDs.

To this end, we apply advanced, synchrotron-based high-resolution wide-angle X-ray Total scattering, at the ID22 beamline of the European Synchrotron Radiation Facility, and small-angle X-ray scattering techniques.

The structural and microstructural characterization of QDs is carried out by reciprocal space X-ray total scattering methods based on the Debye Scattering Equation [4], after a thorough data reduction to subtract the scattered intensity of extra-sample components and to account for absorption effects, according to a well-established protocol.

Atomistic modeling of QDs morphology indicates a tetrahedral shape with an anion-rich (S²⁻) surface termination. Larger particles develop mixed morphologies, with both tetrahedral and prismatic particles observed for sizes above approximately 4 nm.

At the nanoscale, although chalcopyrite and wurtzite structural motifs are commonly reported [5, 6], our reciprocal space data analysis support a zincblende-like structural framework with cation compositional disorder, as evidenced by the absence of diffraction features characteristic of an ordered phase. We do not observe evidence of long-range chalcopyrite ordering; instead, we detect short-range structural correlation consistent with that reported for the wurtzite polymorph [7].

Acknowledgements

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MS10

Sharing and disseminating Crystallography

ISCoC: more than a feeling

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The International School of Crystallography (ISCoC) has been organizing courses on the topic of crystallography and structural sciences since 1974. Throughout all these years thousands of scientists passed through the cobble-stoned streets of Erice: some of them returned several times with different roles within the school: participants, speakers, directors; most of them had successful careers, in several cases becoming Nobel Prize Laureate.

No matter the topic of the course, the "Erice" experience is something that all participants remember forever: it is the magic and history of the village (starting from the Punic and Carthaginian times passing through Roman and Arabs domination to Saracen and Norman era), the science that is taught there but most of all, it is the special atmosphere that days after days grows among the group of people attending the courses. Collaboration and friendships born in Erice last for a long time and would often represent a turning point of a carrier.

Erice is a small and fairly isolated place, and the Courses are usually 9 days long: participants have then the possibility of spending a lot of face-to-face time (days and evenings, lectures and social activities) with each other, with peers as well as with tutors and senior scientists. This can create very deep connections with lasting effects, an experience very different from the many on-line and virtual courses that have been common after the COVID experience.

ISCoC has evolved over the years, and the organization of the courses (hosted at the Ettore Majorana Foundation and Centre for Scientific Culture ets) has been taken over by a new organization called LODOS APS ETS (Legacy of the Organizers of the school for the Diffusion Of the Structural Sciences). But do not worry: this new association is composed by the same people and has the same outlook and enthusiasm and excitement of always.

Why they do that? ... Because they believe in Erice



Figure 1: The author of this abstract during the celebration of the 50th anniversary of the ISCoC

WiC project

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The WiC (Women in Crystallography) project was born a few years ago from a growing awareness of the imbalance in gender representation and diversity management in the scientific community [1]. This topic also came to the attention of the general public after the United Nations adopted the 2030 Agenda for Sustainable Development, in particular Sustainable Development Goal 5: **to achieve gender equality and empower all women and girls** [2].

With this mission in mind, different projects have come to life with the initial nest of ECA (European Crystallographic Association) which supported both the people involved and their actions. The first initiative started in 2018 at the ECM31 in Oviedo, when a bunch of scientists tried to raise awareness by launching a series of T-shirts with a special design, embedding the famous Photo 51 of Rosalind Franklin. The idea took a wider step forward, and now the IUCr is also involved in the project, together with some companies that support it (DECTRIS Ltd. and STOE & CIE GmbH). The funds raised through the T-shirt campaign support the attendance of young female scientists to school and conferences. Under this umbrella, the "Women in Crystallography: a video series" [1,3] was also created within the framework of the Royal Society of Chemistry Inclusion and Diversity Fund. The videos feature ten different scientists (both women and men) who share their experience regarding the importance of women in science and the obstacles they face during their career.



Figure 1. The logos of the T-shirt (left) and of the video series (right)

[1] A. Guerri, C. Massera, *Sapere* **2025**: 10.12919/sapere.2025.05.5

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Engaging young minds through crystal growth: from “The Journey” to the next theme

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The sixth Italian Crystal Growth Competition for students has just concluded. This initiative aligns with the broader goals of the International Union of Crystallography (IUCr), particularly those associated with the International Year of Crystallography (IYCr). The IYCr offers a valuable opportunity to promote and disseminate crystallography at all levels of education and to increase public awareness of the subject and its potential. Since the IYCr began, the crystallographic community has organized outreach activities to educate the public about crystallography and its connections to real-world issues and daily life. These include technological developments in modern society, pharmaceuticals, biology, cultural heritage, and environmental and medical issues.

This edition of the Italian competition underwent a significant overhaul, made possible by collaboration among the AIC's Crystallography and Epitaxy Section, the Regional Museum of Natural Sciences in Turin, Italy, and the IUCr's patronage. This collaboration marks a significant development, as it facilitates interaction between two distinct spheres: the museum world, which is intrinsically oriented toward outreach and the promotion of scientific awareness, and the crystallographic world, which is predominantly focused on research and peer discussion.

The competition concluded in May 2026 and immediately resumed, having changed its frequency from biennial to annual. These changes mean that data is not available for direct comparison with earlier editions.

Further changes and developments were introduced during the project to improve and expand communication and outreach activities and to reach the national school network more broadly.

[1] AAVV Crystallography matters ... more! Events and outcomes from the international year of crystallography, **2014**, <https://www.iycr2014.org/home>

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Advances in detector technology continue to expand the capabilities of laboratory single-crystal X-ray diffraction (SC-XRD). The combination of high-energy X-ray radiation with modern charge-integrating pixel array detectors offers significant opportunities for improving data quality, reducing experimental limitations, and extending the range of samples that can be studied. This presentation explores the benefits of high-energy crystallography enabled by **PHOTON IV** detector technology and highlights applications where shorter-wavelength radiation can deliver substantial advantages. High-energy X-rays provide increased penetration depth and reduced absorption compared with conventional laboratory wavelengths. These characteristics are particularly valuable for strongly absorbing materials, metal-containing compounds, framework materials, and larger crystals where absorption effects can compromise data quality. The use of higher-energy radiation can reduce systematic errors, improve intensity accuracy, and provide more reliable structural information from the entire crystal volume. Additional benefits include reduced extinction effects, lower background scatter, and more efficient data collection.

PHOTON IV detectors employ charge-integrating pixel array detector (CPAD) technology designed to deliver high sensitivity, low noise, and excellent performance across a broad energy range. Advanced detector architecture and inter-pixel processing enable accurate measurement of both strong and weak reflections while maintaining the dynamic range and speed required for modern crystallographic experiments. These characteristics make the detector particularly well suited to high-energy diffraction applications.

Representative applications demonstrate how high-energy data collection can improve structure determination, enhance refinement quality, and support high-resolution crystallographic investigations. Examples include metal–organic frameworks, inorganic materials, and small-molecule systems where reduced absorption and improved data quality contribute to more accurate structural models. The combination of advanced detector technology with high-energy laboratory sources enables researchers to tackle increasingly demanding crystallographic problems while retaining the convenience and accessibility of laboratory instrumentation.

The presentation highlights how modern detector developments are broadening the scientific opportunities available through high-energy SC-XRD and providing new pathways for advanced crystallographic research.



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GNR Analytical Instruments Group designs and manufactures analytical instrumentation for elemental and structural characterization of materials, including Optical Emission Spectrometers (OES), X-ray Diffractometers (XRD) and X-ray Fluorescence Spectrometers (XRF) for both industrial and research applications.

Founded in 1984, GNR has developed extensive expertise in analytical technologies and has established a worldwide network of sales representatives and service centers to support customers across a broad range of markets and applications. The GNR X-ray diffraction portfolio covers numerous applications in materials characterization and quality control, ranging from academic research to industrial process monitoring. Recent developments have focused on improving instrument flexibility, performance and ease of use, while maintaining the reliability required for routine laboratory operations.

X-ray diffraction provides essential information on crystal structure, phase composition and microstructural properties for all materials exhibiting a sufficient degree of crystallinity. GNR has continuously upgraded its diffractometer platforms to provide advanced analytical capabilities for both standard and specialized applications.

During this presentation, several X-ray diffraction solutions developed by GNR for different application fields will be presented. Examples will include quantitative phase analysis, thin film characterization, residual stress measurements and texture analysis.

GNR offers dedicated solutions for research laboratories and universities as well as robust instruments specifically designed for industrial environments. Particular attention has been devoted to the development of systems for residual stress and retained austenite measurements, combining advanced analytical performance with simplified operation, allowing effective use also by personnel without extensive expertise in X-ray diffraction.

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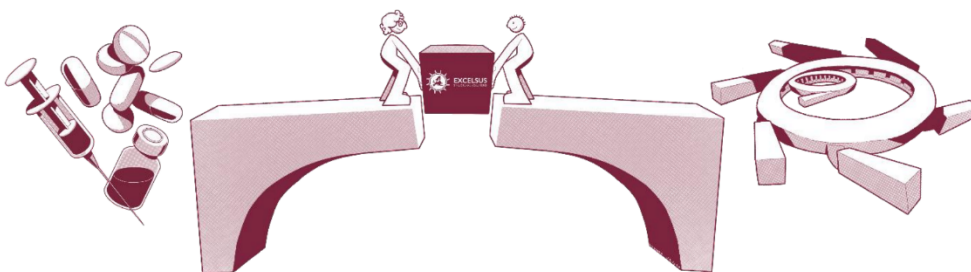
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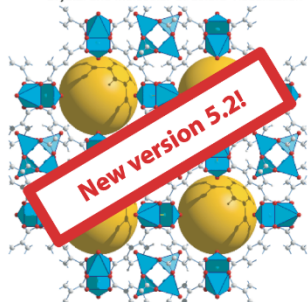
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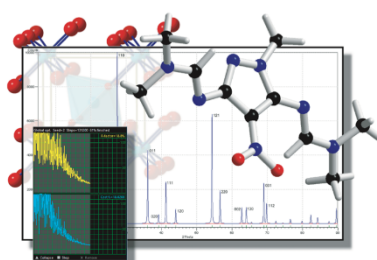
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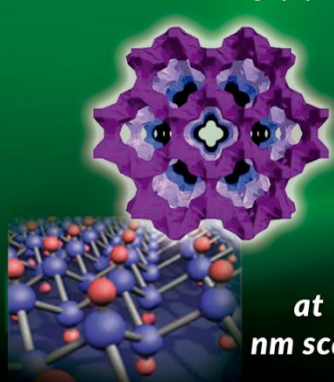
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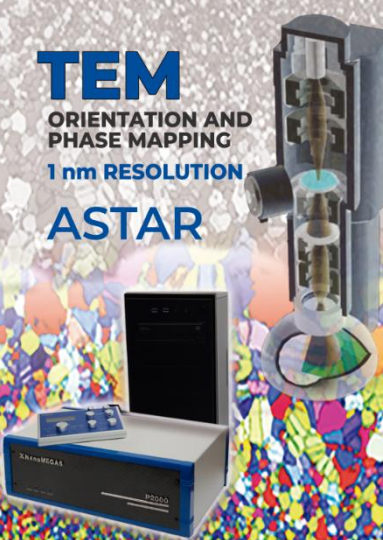


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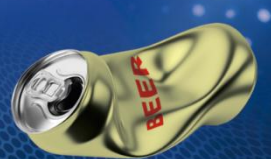
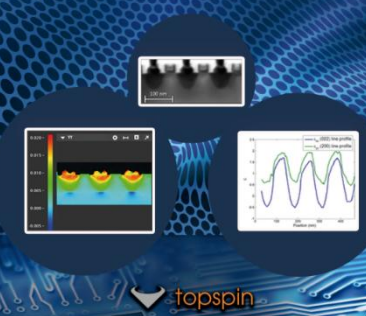


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