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Posters with examples of synchrotron research projects

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PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including Commission Services)	
CO	Confidential, only for members of the consortium (including Commission Services)	

Document History

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26.06.2023	1.0	Initial draft	Dr. Susanne v. Ameln
24.07.2023	1.1	Final version approval	Dr. Susanne v. Ameln

Objectives of the action

The poster session took place during the Industry Workshop at the SOLARIS NSRC premises between 14th and 15th June 2023. Young scientists affiliated to the Sylinda partners prepared scientific posters presenting examples of synchrotron research projects. Specific poster's topics on possible applications of synchrotron based techniques to industry were discussed and agreed with the appropriate Science Management Department at the partners organizations. . The preparation of the posters and participation in the poster session gave young scientists a unique opportunity to expand their knowledge of science management and enable communication with industry. Additionally, young researchers could learn about the specific needs of industry partners.

List of posters presented at the Industry Workshop. Bold topics are presented as a examples at the bottom of the document:

1. *“Dye fabric with structure color”*, authors Di Cai, Boris Mahkltig
2. ***“X-ray absorption spectroscopy using synchrotron radiation for selenium speciation in biofortified plants”***, authors Marcia Viltres- Portales, María-Jesús Sánchez-Martín, Roberto Boada, Mercè Llugany, Manuel Vallente
3. *“Do the dietary conditions of hens influence eggshells quality?”*, authors Iris H. Valido, Maria Angels Subirana, Rosa Mary Lopez-Alvarez, Sandra Mounicou, Florent Penen, Montserrat Lopez-Mesas, Ibraheem Yousef, Stéphane Faucher, Pascale Sénéchal, Peter Moonen, Alessandra Monteiro, Dirk Schaumlöffel
4. ***“Development of thiol-functionalized sponges for the removal of trace levels of cisplatin and carboplatin from hospital wastewater”***, authors Dong Han, Montserrat Lopez-Mesas, Tania Farias, Ana R. Lazo Fraga, Manuel Vallente
5. ***“How can synchrotron help Startups and Industry”***, author Stephanie Bienefeld
6. ***“The soft X-ray spectroscopy beamline Phelix at the SOLARIS Synchrotron”***, authors Edyta Bayer, Magdalena Szczepanik, Tomasz Sobol, Ewa Partyka-Jankowska
7. ***“A powerful new SOLARIS beamline for applied research”***, authors Henning Lichtenberg, Alexey Maximenko, Josef Hormes, Alexander Prange
8. *“Advanced research technique for high tech”*, author Natalia Olszowska
9. *“Thiazoloethiazole derivatives as new materials for memristive and semiconductor devices”*, authors Lulu Alluhaibi, Karolina Gutmańska, Alexey Maximenko, Ewelina Kowalewska, Andrzej Sławek, Konrad Szaciłowski, Anna Dołęga
10. ***“CIRI beamline – chemical infrared imaging – new research opportunities”***, authors Karolina Kosowska, Paulina Koziół, Danuta Liberda, Maciej Roman, Tetiana Stepanenko, Tomasz Wróbel
11. *“Materials and chemical research at the PIRX beamline”*, authors Ewa Partyka-Jankowska, Joanna Stępień, Marcin Zajac

12. ***“Sample holders and in situ cell construction for ASTRA beamline”***, authors Marcin Brzyski, Dora Zalka, Alexey Maximenko
13. ***“Synchrotron techniques to unravel ARF DNA Binding Domain function”***, authors Isidro Crespo, Willy van den Berg, Dolf Weijers, Roeland Boer
14. ***“POLYX at SOLARIS: layout, specification & first results”***, authors Katarzyna Sowa, Paweł Wróbel, Tomasz Kołodziej, Wojciech Błachucki, Paweł Korecki
15. ***“SOLARIS Cryo-EM Facility”***, authors Paulina Indyka, Michał Rawski, Grzegorz Ważny, Marcin Jaciuk, Sebastian Glatt



Figure 1: Poster session of the Sylinda Industry Workshop

X-Ray Absorption Spectroscopy using synchrotron radiation for selenium selection analysis in biofortified plants

SYLINDA

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SUMMARY

Problematic: Selenium deficiency in soils and foods leads to various health issues: thyroid health, inflammation, brain health, cancer, fertility, immunity, and arthritis. Selenium amino acids are important.

Solution: Biofortification of plants using different strategies. Synchrotron radiation application for speciation analysis (simple, direct, high selectivity).

SYNCHROTRON EXPERIMENTS

Experiment No. 1: Wheat plants biofortified with Se by foliar application of Se. Experiment No. 2: Microgreens biofortified with Se added directly to the hydroponic solution.

RESULTS AND INDUSTRIAL APPLICATIONS

Table 1: Selenium (Se) concentration in different plant parts (mg/kg).

Plant Part	Control	Se-1	Se-2
Grain	0.01	0.02	0.03
Straw	0.01	0.02	0.03
Root	0.01	0.02	0.03
Shoot	0.01	0.02	0.03

Table 2: Selenium (Se) concentration in different plant parts (mg/kg) for microgreens.

Plant Part	Control	Se-1	Se-2
Grain	0.01	0.02	0.03
Straw	0.01	0.02	0.03
Root	0.01	0.02	0.03
Shoot	0.01	0.02	0.03

CONCLUSIONS

The speciation analysis revealed that selenium in biofortified plants is in the form of selenomethionine and selenocysteine. This confirms that plants are able to bioaccumulate selenium into the amino acids selenomethionine and selenocysteine. The results show that the biofortification of plants with selenium is a viable strategy to increase the selenium content of food crops. The results also show that the biofortification of plants with selenium is a viable strategy to increase the selenium content of food crops.

ACKNOWLEDGMENTS

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 952148.

Development of thiol-functionalized sponges for the removal of trace levels of cisplatin and carboplatin from hospital wastewater

Dong Han¹, Montserrat López-Mesa¹, Tania Parra-Añel¹, Luis Fraga¹, and Manuel Valera¹
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Backgrounds

- Occurrence of platinum-based cytostatic drugs in hospital wastewater
- Toxicity to exposed humans and aquatic organisms
- Recalcitrance to conventional wastewater treatment technologies

Materials

Cellulose Substrate, MPA, Cys, Cisplatin, Carboplatin

Adsorption

Chemical reaction: HS-CH2-CH2-SH + H2SO4 + H2O2 -> HS-CH2-CH2-S-S-CH2-CH2-SH + H2O

Mechanism elucidation by synchrotron XAS

4 Pt-S coordination environment was confirmed by EXAFS fitting.

Industrial application

Fixed-bed column: Hospital wastewater → Sponge → Clean water

ACKNOWLEDGMENTS

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How can Synchrotron help Startups and Industry?

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HNX Your way to start up

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www.hsnr.de/HNX

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 952148.

Available Measurement Techniques:

- X-ray Photoelectron Spectroscopy (XPS)
- Angle Resolved Photoelectron Spectroscopy (ARPES)
- Spin-resolved ARPES
- Circular dichroism ARPES
- Resonant Photoelectron Spectroscopy (ResPES)
- X-ray Absorption Spectroscopy
- Total Fluorescence Yield (TFY)
- Total Electron Yield (TEY)

X-RAY PHOTOELECTRON SPECTROSCOPY

Electron detection mode analyzing angle-resolved photoelectron spectroscopy measurements is used for XPS.

RESONANT PHOTOEMISSION SPECTROSCOPY

Resonance photoelectron spectroscopy (ResPES) studies were performed. The map for the Fe 2p resonance was collected for NiO(Cr-MWCNT) in situ as received composite as the first one in the history of the PHEX beamline.

X-RAY ABSORPTION SPECTROSCOPY

Absorption spectroscopy (XAS) measurements in the sample current (TEY) and total fluorescence (TFY) mode.

THE SOFT X-RAY SPECTROSCOPY BEAMLINE PHEX AT THE SOLARIS SYNCHROTRON

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JAGIELLONIAN UNIVERSITY IN KRAKOW

PHEX: SOFT X-RAY SPECTROSCOPY BEAMLINE

Radiation source: elliptically polarizing undulator (EPU), APPLE II type with permanent magnets

Energy range: 40-1500 eV for horizontal polarization, 50-1500 eV for vertical polarization, 40-350 eV for circular polarization (100%), 50-3035 eV for elliptical (50%)

Resolving power: >10,000

Beam size on sample: 30x80 μm² @ 50 eV, 10x60 μm² @ 1500 eV

Photon flux on sample: 8.2x10¹⁰ ph/s @ 50 eV, 1.4x10¹⁰ ph/s @ 1500 eV, RP = 10,000, electron ring current = 500 mA

END STATION

Available Measurement Techniques: X-ray Photoelectron Spectroscopy (XPS), Angle Resolved Photoelectron Spectroscopy (ARPES), Spin-resolved ARPES, Circular dichroism ARPES, Resonant Photoelectron Spectroscopy (ResPES), X-ray Absorption Spectroscopy, Total Fluorescence Yield (TFY), Total Electron Yield (TEY)

X-RAY PHOTOELECTRON SPECTROSCOPY

Electron detection mode analyzing angle-resolved photoelectron spectroscopy measurements is used for XPS.

RESONANT PHOTOEMISSION SPECTROSCOPY

Resonance photoelectron spectroscopy (ResPES) studies were performed. The map for the Fe 2p resonance was collected for NiO(Cr-MWCNT) in situ as received composite as the first one in the history of the PHEX beamline.

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Absorption spectroscopy (XAS) measurements in the sample current (TEY) and total fluorescence (TFY) mode.

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SOLARIS National Synchrotron Radiation Centre
SYLINDA Hochschule Niederrhein University of Applied Sciences

A powerful new SOLARIS beamline for applied research

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ASTRA: Absorption Spectroscopy beamline for Tender energy Range and Above

Frontend designed and built by FMB (Berlin)

Energy Range: Unique: measure at absorption edges of low Z, chem. elements → shine a light where most other beamlines cannot reach!

Basic Setup for X-ray Absorption Spectroscopy: Absorption edge scans, X-ray fluorescence, EXAFS, etc.

Examples from ongoing projects: Energy Resolution, Surface / Interface Sensitive Methods, On-Site Infrastructure, Automated Procedures, Complementary Methods Combination, Spatial Resolution, Advanced Data Analysis.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 952148.

CIRI BEAMLINE
- CHEMICAL INFRARED IMAGING - NEW RESEARCH OPPORTUNITIES

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JAGIELLONIAN UNIVERSITY IN KRAKÓW

CIRI (Chemical InfraRed Imaging) is an upcoming infrared synchrotron beamline in Poland and Central and Eastern Europe with advanced imaging capabilities, including micro- and nanometric spatial resolution. This development represents a significant milestone in IR technology, enabling noninvasive and nondestructive chemical characterization and imaging of nanomaterials using advanced techniques such as s-SNOM/AFM-IR and O-PTIR microscopies.

FT-IR MICROSCOPY **O-PTIR MICROSCOPY** **S-SNOM MICROSCOPY** **AFM-IR MICROSCOPY**

The noninvasive and nondestructive chemical characterization and imaging of range spectrum of materials are possible thanks to infrared microscopy.

The O-PTIR and s-SNOM/AFM-IR microscopies break the resolution limit of infrared light with spectral resolutions of 400-500 nm and 20-50 nm, respectively.

EXEMPLARY APPLICATIONS IN POLYMERS AND LIFE SCIENCE

MACHINE LEARNING: CANCER DETECTION
 Histopathology based on infrared microscopy is a new tool for obtaining complementary information for classical medicine. The histopathological annotations are marked on the IR image → spectra and used for model creation. The pancreatic cancer classification model was prepared based on spectral data from 600 biopsies. 600 biopsies gave almost 700 million spectra of different tissue types. Decision trees are methods that use trees to solve classification and regression problems based on binary split data.

3D MACROMOLECULAR ORIENTATION IN POLYMERS
 Simultaneous analysis of two bands of roughly perpendicular transition moment orientations was proposed as a method of determining the orientation of the molecule in three-dimensional space. The 'concurrent analysis' (4P-3D) was applied infrared spectromicroscopy data for obtaining orientation angles of a model polycaprolactone spherulite.

AFM-IR AND S-SNOM: HUMAN ERYTHROCYTE MEMBRANES
 By employing s-SNOM, the composition of red blood cell (RBC) membranes has been examined, revealing the presence of various vital biomolecules: lipids, which constitute a significant proportion of the membrane's mass, integral and peripheral proteins. Carbohydrates and cholesterol acids are also present.

Visualization of Alpha-helix and Phosphate groups.

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 952148.

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SAMPLE HOLDERS AND IN SITU CELL CONSTRUCTION FOR ASTRA BEAMLINE

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INTRODUCTION
 Beamline experiments require prior preparation the reaction plate and the technical equipment of the line. These two branches should work closely together to achieve the intended objective. One of the challenges that beamline engineers could face is that the scientist has an idea, but the technical side of the experiment is not clear, which measurement works to conduct research, but does not have a properly prepared sample holder for the sample.

when the sample is in powder or pellet form and a required in-situ during current measurements, one solution is to use a CR2032 battery case. The SOLARIS Team has designed a holder that allows easy replacement of such samples. The holder was made in various variants based on the most common electronic components.

Many beamlines have standardised holders, such as long style. However, for some beamline techniques and samples, an individual approach is required. This situation can be encountered especially when the beamline is upgraded and the ability to adapt to a new sample type is required. Some technical changes seem easy to implement, but it is worth making sure that they are not related to other restrictions on the beamline. Therefore, a useful discussion with the beamline staff, who also have technical support in the team, gives a much better chance that the experiment will be properly prepared for the beamline.

ASTRA BEAMLINE - GENERAL INFORMATION
 ASTRA (Absorption Spectroscopy) beamline is a compact beamline dedicated to X-ray absorption spectroscopy (XAS) and related techniques. The beamline has a modified monochromator type Double Crystal Monochromator (DCM) working in the energy range from 10 to 10 keV and is designed for quick exchange of the crystal sets.

Measurements in sample chamber can be performed in a high-vacuum (10⁻⁶ mbar), but most often at low vacuum or atmospheric pressure (0.1 mbar, 1 bar) or nitrogen gases is used. In this region and other technical aspects of the construction, beamline is open to customer and does not require time-consuming chamber conditioning period. The ASTRA is designed for basic and applied research in the fields such as biomaterials, materials science, physics, chemistry and environmental protection.

OVERVIEW OF SAMPLE HOLDERS AND CELLS
 For most of the measurements of the ASTRA beamline samples are in the powder or pellet form, in this case it is possible to attach them to special frames with a Kapton strip. The frames are made of stainless steel or plastic using DMJ (DMJ printing) technology.

An interesting issue is the study of electrolyte samples. They require the use of additional electrodes to measure the current during measurement. A properly designed holder should take into account a reliable electrical connection, it is also important the material from which it is made does not react with the sample or interfere with the measurement itself.

MEASUREMENT RESULTS
 An example of the research where holders for liquid samples manufactured by the SOLARIS Team were applied: Impact of biomass-based multifunctional binders on Li-S battery chemistry - analysis of polysulfide phases by in-operando XAS.

In this experiment cells equipped with VMC separator, with liquid and solid electrolyte containing composite conductive carbon (CC) and LiPF₆ were used. The electrochemical (EIS) spectra are showing during the charge-discharge cycles, where the low impedance of the separator doesn't change significantly. This means that the separator can control the polysulfides to the liquid electrolyte and avoid their formation.

CONCLUSION
 Several sample holders were designed and manufactured in the SOLARIS Synchrotron, which were successfully used in the measurements. New scientific challenges bring a cooperation needs to develop research lines in technical terms. New scientific challenges bring a cooperation needs to develop research lines in technical terms. Thanks to new production technologies, the possibilities of more advanced materials thanks to new production technologies, the possibilities of more advanced materials to increase material's extended laser and future device application in the individual needs of research groups.

ALBA National Synchrotron Radiation Centre
SYLINDA

Synchrotron techniques to unravel ARF DNA Binding Domain function

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ABSTRACT
 Plant development is mainly governed by the phytohormone auxin through the nuclear auxin pathway, which ultimately relies on Auxin Response Factors (ARFs) for specific gene selection. The first ARF DNA Binding Domain (ARF-DBD) structures shed light on the DNA recognition mechanism, but the high DNA-contacting residues were proposed to be determined for effective DNA binding, a hypothesis known as molecular caliper. Here we present a multi-technique approach combining Synchrotron X-ray (Macromolecular Crystallography, Small-Angle X-ray Scattering) with Bioinformatics analysis to unravel the mechanism of ARF gene specificity. This data shows that the ARF-DBDs have been preserved throughout evolution, and confirms the general validity of the molecular caliper model. Interestingly, we find that structural similarities and in vitro properties of ARF-DBDs do not match the functional classification of full length ARFs, suggesting that ARF-DBDs were subject to domain swapping during evolution. Therefore DNA recognition by ARF-DBDs is functionally decoupled from the gene response of the ARF orthologs. Thus, an increase in the variability of auxin response is achieved by combinations of DBD-mediated DNA recognition and MR-mediated expression response.

SAXS

Fluorescence anisotropy

Macromolecular Crystallography

CONCLUSIONS
 • Our methodology combining Synchrotron techniques MX and SAXS, with FA allowed us to determine the affinity of ARF DNA binding domains towards different DNAs, including biological and synthetic sequences.
 • ARFs from different functional classes (MpARF2 and AtARF5) are structurally similar and consistently comparable in FA, SEC and SAXS analyses.
 • The ARF-DBD is an ancestral specificity selector of ARF binding to the promoter sequence.
 • Our results add an additional layer of regulation, where the DBD specificity is functionally decoupled from the response elicited by the ARFs.

INDUSTRIAL APPLICATION
 • ARF Plant gene regulation knowledge is essential to ensure future food security, increase yields and reduce losses.
 • The combination of methodologies used here allows to study changes in proteins in presence of ligands/drugs.
 • This approach can be expanded to potential pharmaceutical drug targets.

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www.albasynchrotron.es

Figure 2: Examples of the posters presented during the poster session of the Sylinda Industry Workshop