Session 1 – CO_2 Capture, Utilization, and Storage

C-P1: Highly Effective Separation of Drug Compounds using Greener Methodologies

C-P2: Evaluate Carbon Capture Efficiency with TOC: the Base of Everything

C-P3: Atomically Precise Silver Cluster Enables CO_2-to-CO Electroreduction at 1 A/cm2² with up to 70% Energy Efficiency in a Zero-gap Electrolyser

C-P4: CO_2 Mineralization from Desalination Brine using Electrochemical pH Swing

C-P5: Integrated Direct Ocean Capture System for Carbon Sequestration, Hydrogen Generation, and Energy Recovery

Highly Effective Separation of Drug Compounds using Greener Methodologies

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High-throughput purification of drug compounds is essential in various fields, particularly in pharmaceutical development. Traditional preparative liquid chromatography (HPLC) methods often involve lengthy purification times and substantial organic solvent consumption, leading to increased operational costs and environmental concerns. This study introduces the Nexera™ UC Prep, a semi-preparative supercritical fluid chromatograph (SFC), which utilizes supercritical carbon dioxide as a mobile phase, offering a more efficient alternative for the purification of drug compounds. Thanks to mobile phase's low viscosity, semi-preparative SFC allows fast purification and delivers low volume concentrated fractions in a greener way.

The purification process was evaluated using ketoprofen and indomethacin as model compounds. Both semi-preparative HPLC and semi-preparative SFC were compared. The SFC method was further developed by using stacked injection mode. The performance metrics assessed included fraction drying time, organic solvent consumption and analysis time.

The results demonstrated that preparative SFC significantly reduced fraction drying times by over 20 times compared to conventional preparative HPLC. The stacked injection mode allowed for consecutive sample processing, maximizing the use of waiting times, thereby enhancing purification efficiency. In terms of solvent consumption, preparative SFC utilized 3.5 time less organic solvent than HPLC, translating to a substantial reduction in running costs.

The use of supercritical carbon dioxide not only minimizes organic solvent usage but also accelerates the purification process, making it a more sustainable and cost-effective solution for pharmaceutical applications. The findings indicate that preparative SFC can enhance the efficiency of drug compound purification workflows, positioning it as a valuable and greener methodology.

Evaluate Carbon Capture Efficiency with TOC: the Base of Everything

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Reaching carbon neutrality in 2050 is one major challenge and goal wherever around the world. Even with the development of news technologies like green Hydrogen use for mobility applications or the transformation of industrials processes into greener ones, still several Giga Tons of Carbon Dioxide CO_2 will still be produced and released in the atmosphere in 2050. So, development of various technologies to remove carbon dioxide from the atmosphere to store (CCS) or reuse (CCUS) is mandatory.

Many ways are considered and studied, from Direct Air Capture (DAC) with ammonia solutions to the use of microalgae / microbial fixation passing by CO_2 absorption in concrete.

Whatever the selected way for carbon capture, efficiency of this process must be checked in order to optimize the different parameters and also testify the capacity of the system to catch and retain CO_2. Total Organic Carbon analyzer is thus the perfect answer to this question, capable of performing a fast and accurate analysis of liquids and solids samples.

Atomically Precise Silver Cluster Enables CO_2-to-CO Electroreduction at 1 A/cm² with up to 70% Energy Efficiency in a Zero-gap Electrolyser

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The electrochemical reduction of CO_2 to feedstocks is a promising alternative for offsetting greenhouse gas emissions. While higher hydrocarbon products (socalled C2+ products) are highly revered in academic research, carbon monoxide is arguably one of the most interesting feedstocks from an industrial perspective. Indeed, phosgene, isocyanates, urea, methyl methacrylate, and acetic acid, to name only a few, are directly derived from CO, industrially. Moreover, the electroreduction of CO_2 to CO is the least energetically demanding process with the highest theoretical energy efficiency. As such, herein we present the conversion of CO_2 to CO in a MEA electrolyser, achieving 70% energy efficiency and 40% single-pass conversion at 200 mA/cm², enabled by an all-alkynyl silver cluster and a low-cost treated stainless-steel anode. The system operates at a cell voltage as low as 1.9V at 200mA/cm^2 using KOH as anolyte, while the selectivity is maintained above 98% up to an electrolysis current density of 600 mA/cm2, and 79% at 1A/cm^2. We demonstrated that the silver cluster is able to sustain continuous electrolysis at 200mA/cm^2 for over 160h, with virtually no decrease in faradaic efficiency for CO. The ease of synthesis of the silver cluster catalyst on a large scale, together with the potential of producing anodes from low-cost non-precious metals, demonstrates a scalable path to CO production.

CO_2 Mineralization from Desalination Brine using Electrochemical pH Swing

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Electrochemical Direct Ocean Capture (eDOC) is a process designed to capture carbon dioxide (CO_2) from ocean water. Oceans absorb approximately 25 % of atmospheric CO_2 resulting from human activities, predominantly in the form of bicarbonate. Electrochemical pH swing allows for the extraction of bicarbonate (HCO_3^-) either as carbon dioxide gas ($CO_2(g)$) by lowering the pH, or as a precipitate ($MgCO_3$ or $CaCO_3$) by complexing it in the carbonate form (CO_3^{2-}) with divalent ions such as Ca^{2+} or Mg^{2+} at an elevated pH. The eDOC process which employs electrodialysis with bipolar membranes to dissociate H_2O into H_3O^+ and OH^-, has received attention as a potential Negative Emission Technology (NET) for oceanic carbon removal.[1] This method avoids the production of chemical waste, operates using electrical energy that may be sourced renewably, and typically requires less energy compared to electrochemical water splitting at electrodes. Start-ups have already developed this technology evolving from pilot plant installations to future systems targeting the annual removal of hundreds of tons of CO_2.[2] Nevertheless, significant challenges remain regarding large-scale implementation, particularly membrane fouling caused by divalent ions present in the feed water, which can adversely affect operational stability.[3] This study examines an eDOC flow process for capturing CO_2 from desalination brine, utilizing its high concentrations of bicarbonates and divalent ions.[4] Electrodialysis with a bipolar membrane is employed as a source of NaOH. The objective is to capture Total Inorganic Carbon (TIC) while concurrently converting brine into valuable products, including high-purity $MgCO_3$ and $CaCO_3$ precipitates.[5] Additionally, the softened filtrate will be reinjected into the electrodialysis system to facilitate a closed-loop base production process.To this end, a laboratory-scale electrodialysis device (10 x 10 cm²) is evaluated for hydroxide ion (OH^-) generation. Our findings indicate that, in closed-loop systems, optimizing the configuration of membranes is essential to ensure adequate delivery of OH^- for effective brine softening and prevention of membrane fouling.

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Integrated Direct Ocean Capture System for Carbon Sequestration, Hydrogen Generation, and Energy Recovery

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Carbon dioxide levels are rising significantly, driving climate change and intensifying the greenhouse effect. While current carbon capture technologies primarily target industrial emissions, addressing legacy CO_2 already in the atmosphere requires negative emissions technologies (NETs). Considering the fact that CO2 concentrations are approximately 140 times higher than in the atmosphere, an ocean based emerging ENTs known as Direct Ocean capture (DOC) has gained increasing attention over the past few years [1]. It takes ocean as an electrolyte to capture and concentrate CO2 under the natural chemical equilibrium between CO_2 gas and dissolved inorganic carbon. The removal of the inorganic carbon by electrochemical methods will continuously renew the ocean's capacity to further capture the CO_2 from the atmosphere. To realize its potential, DOC systems must urgently improve energy efficiency and reduce energy costs to become viable at scale.

In this work, we propose the integration of hydrogen generation and energy harvesting process inside a DOC system, which is composed of 3 modules.

Ocean acidification module: It is an electrodialysis cell unit composed of 2 electrodes of Pt/Ir-MMO coated Ti-stretched metal and 2 cation exchange membranes (CEMs), which divide the entire cell unit into 3 separate compartments. When a fixed electrical current is applied, H^+ ions are generated at the electrode along with the generation of O2 in the anode compartment. In the cathode compartment, a reduction process leads to the production of H2, along with the production of alkaline solution of NaOH. Seawater is continuously pumped into the middle compartment. As the excess H^+ ions pass through the CEM to replace the Na^+ in the middle chamber, seawater is locally acidified to a pH lower than 6 to recover the dissolved inorganic carbon [2]. A significant pH difference of the effluents forms an osmotic energy source.

Energy harvesting module: This module is well engineered to convert the pH difference based osmotic energy into electricity. It is a reverse electrodialysis cell composed of 2 water chambers separated by a non-selective membrane. Two solutions of different pH are continuously pumped inside the module within a closed loop, creating an electrical potential difference between the MnO_2 composite electrodes. Under a systematic solution switch realized by an automatic valve, the module delivers a periodic and alternating electrical signal. A boosting strategy could be applied to further ameliorate the power density output to be 4.5 $W.m^{-2}$ for a pH difference of 3.9.

pH transferring module: Experimental results showed that for the pH gradient module, the presence of buffer is mandatory to avoid kinetic problems. As the use of additional chemical components raise environmental concerns, closed loop electrolytes with sufficient quantities of buffers are used inside the Energy harvesting module. It is thus mandatory to realize the pH transfer between Energy harvesting module and the Ocean acidification module.

This is realized by columns of weak acid ion exchange resins for the acidity transfer and the direct injection of alkaline electrolyte in the cathode compartment for alkalinity transfer.

In the present setup, we successfully integrate these 3 modules into a complete DOC system. A preliminary test demonstrates a stable output of an open circuit voltage of 220 mV over 2.5 hours. In the process, we acidify seawater of a quantity 1.5 L, suggesting a theoretical maximum amount of CO_2 sequestration of 3.45 mmol. Meanwhile, we estimate a H_2 production of 0.05 mol and a O_2 production of 0.025 mol.

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Session 2 – Recycling and end-of-life of plastics

R-P1: MALDI for Discrimination between the Recycled PET and the Virgin PET

R-P2: Comprehensive Two-dimensional Gas Chromatography Coupled with Mass Spectrometry for Recycled Plastic Characterization

R-P3: Thermomechanical Recycling of Rubber Waste: Network Topology Investigated by Solid-State NMR

R-P4: Active Aggregation: a Tool for Nano-plastics Recovery?

R-P5: Characterization of the Volatile Fraction of Used Wind Turbine Blade Pyrolysis Oil by Two-Dimensional Gas Chromatography

R-P6: From Waste to Resources: Sample Preparation Strategies Coupled with GCxGC-TOFMS for Characterizing Wind Turbine Blade Recycling Products

R-P7: How HT-GC/MS and HT-GCxGC/MS can Help for the Detailed Characterization of Heavy Distillation Volatile Fractions from Pyrolysis Oils

R-P8: Polymer Biodegradability and the Link Between Abiotic and Biotic Degradation

R-P9: PyC2MC: A Framework for FTMS Data Processing and Molecular Attribution using Mass Difference Analysis

R-P10: Detailed Characterization of Polymers and Microplastics by KMD Plots of Complex MS Spectra

R-P11: Microplastic Characterization and Screening by Combining DART and High-Resolution Mass Spectrometry

R-P12: A gTIMS-MRMS Instrument to Decipher Isomeric Content of Complex Organic Mixtures

MALDI for Discrimination Between the Recycled PET and the Virgin PET

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Due to recent social demands to achieve a sustainable society, the recycling of polymers used in consumable products is a significant issue. Polyethylene terephthalate (PET) is a polymer that is regularly recycled and reproduced into many products such as bottles and clothing fibers. As this circular economy of PET recycling expands, there is a growing need for proper analytical methods to confirm the correct labelling of clothing using recycled fibers. Although some analytical studies on PET oligomers for discriminating recycled PET from virgin one have been reported, their practical capabilities remain unclear. Here, we report the successful discrimination using linear mode MALDI-TOF MS and statistical analysis. Moreover, we will discuss the reason of the discrimination based on chemical structures of oligomers obtained with MS/MS.

Comprehensive Two-dimensional Gas Chromatography Coupled with Mass Spectrometry for Recycled Plastic Characterization

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Global plastic production exceeds 400 million tons annually and continues to rise, making recycling and waste valorization urgent challenges. Chemical recycling offers a promising route to transform plastic waste into valuable feedstocks for the petrochemical industry, supporting the circular economy. In this contribution, we demonstrate the use of comprehensive two-dimensional gas chromatography (GC×GC) coupled with mass spectrometry (MS) as a powerful platform for characterizing complex plastic-derived matrices.

In the first part, GC×GC with low- and high-resolution MS, supported by photoionization, was applied to pyrolysis oils from municipal plastic waste, enabling molecular-level discrimination of overlapping chemical classes. In the second part, a static head space (SHS) solid-phase microextraction (SPME)--GC×GC-qMS was employed to investigate volatile and semi-volatile organic compounds (mainly C2–C14) released from virgin and recycled plastics. Comparative evaluation of extraction strategies showed that multiple vial–multiple cumulative trap (MV-MCT) at –30 °C enhanced sensitivity and recovery of semi-volatiles, while extraction temperature screening (30–60 °C) confirmed 30 °C as the most relevant condition to simulate real-world off-flavour release.

This work highlights the potential of advanced multidimensional chromatography and MS to support chemical recycling strategies and improve the sustainable management of plastic materials.

Thermomechanical Recycling of Rubber Waste: Network Topology Investigated by Solid-State NMR

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Hutchinson, a subsidiary of TotalEnergies, is a world-class company specializing in the transformation of rubber and high-performance polymer materials. Leveraging its expertise, Hutchinson delivers sustainable, multi-material solutions that meet critical challenges in safety, comfort, and energy efficiency, even under the most demanding conditions. These solutions serve key markets such as acoustics and vibration control, sealing, fluid and thermal management, structural components, and power transmission.

With over one hundred sites worldwide, Hutchinson combines industrial excellence with a strong culture of innovation, placing environmental responsibility at the heart of its operations. As part of its commitment to aligning performance with sustainability, the company is pursuing a path toward carbon neutrality. In this context, the integration of recycled materials—particularly recycled rubber—represents a strategic and essential avenue for development.

Among the various approaches to elastomer recycling, thermomechanical process is currently under review for recycling rubber waste[1-3]. This process involves breaking crosslink bonds using a twin-screw extruder, making it well-suited for industrial-scale applications. Research indicates that thermomechanical recycling primarily cleaves polysulfide bridges but also induces chain scission (C–C bond breakage) due to the high shear stress and localized heat generated during extrusion. Consequently, the term *regenerated* is more appropriate than *devulcanized* when referring to the resulting elastomers. The objective is to study how process parameters—such as temperature, screw speed, and screw profile—affect the properties of regenerated rubber, with the aim of achieving performance levels comparable to virgin materials. From a chemical perspective, chain scission alters the network topology and increases the proportion of shorter macromolecules, which may enhance molecular mobility but potentially reduce elasticity.

To better understand the link between these structural changes and the mechanical behavior of regenerated rubber, solid-state NMR experiments are being considered. In particular, the measurement of double-quantum (DQ) 1H coherences build-up under static conditions has emerged over the past two decades as a reliable method for assessing crosslink density in elastomer networks[4-5]. The Baum and Pines pulse sequence, later optimized by Saalwächter[5], is widely used for this purpose. While this technique has been applied to study rubber aging[6-7], its application to regenerated rubbers remains unexplored.

NMR relaxation signals are typically fitted using mono- or bi-exponential models to describe the reorientational motion of isotropic species such as free or dangling chain segments. However, in the absence of an extraction step, regenerated rubber requires a tri-exponential fit, which increases the risk of parameter interdependence and operator bias. Therefore, the extraction step—based on ISO 1407:2023, with a suitable solvent to the polymer matrix—is essential for reliable analysis. Interpretation of NMR data obtained for the extracted

regenerated rubber has shown that process parameters significantly influence the topology of the regenerated rubber network by modulating the relative contributions of chain scission, devulcanization, and overcrosslinking mechanisms.

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Active aggregation : a Tool for Nano-Plastics Recovery?

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Nano- and microplastics have emerged as a pervasive pollutant, fragmenting from larger debris or manufactured at the nanoscale and now contaminating water, air, soil, and even entering food chains. Their sub-micron dimensions render conventional treatment methods (e.g., filtration, sedimentation) largely ineffective, allowing these particles to evade detection and resist removal. This persistence poses serious risks to aquatic ecosystems and human health, from physical blockages in organisms to the potential for toxic chemical release[1]. Recent research has shown that algae and other microorganisms can naturally trap nano-plastics, either by forming biofilms on their surfaces or by incorporating them into dense biological aggregates[2,3]. Our approach builds on this observation: rather than co-cultivating algae in already-contaminated environments, we propose introducing algal cultures into polluted water post-contamination. The goal is to induce aggregation rapidly, minimizing the exposure of algae to nano-plastics and preserving their biological activity. We aim to take advantage of spontaneous aggregation mechanisms—potentially enhanced through biological or physicochemical means—to facilitate nano-plastic capture.
The use of plankton and algal cultures offers a dual benefit: not only can they fix atmospheric CO_2 and produce valuable biomass, but they also show promise in trapping and aggregating nano-plastics[4,5]. This approach opens the door to a holistic environmental strategy that is both low-energy and bio-based. It also raises important scientific questions: How can this natural aggregation be optimized? Can we enhance or engineer these interactions to scale up nano-plastic recovery? Addressing this issue requires a cross-disciplinary approach at the intersection of physics, biology, and environmental science.

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Characterization of the Volatile Fraction of Used Wind Turbine Blade Pyrolysis Oil by Two-Dimensional Gas Chromatography

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With the global expansion of wind energy sector, finding sustainable solutions for the disposal of wind turbine blades (WTBs) has become increasingly critical. Indeed, most parts of the wind turbines are made of concrete and steel, which are easily recyclable. However, the blades are a complex assembly of different materials (wood, resins, coatings, glass fiber and unsaturated polyester matrix). It is therefore necessary to develop a recycling process to divert the blades from landfill and recover valuable materials.

This study investigates the volatile fraction produced through one of the most promising recycling methods (pyrolysis) using comprehensive two-dimensional gas chromatography (GC × GC) coupled with time-of-flight mass spectrometry (TOFMS) and a flame ionization detector (FID). Two column configurations, normal (non-polar × mid-polar) and reverse (mid-polar × non-polar), were compared to enhance the separation and semi-quantification of chemical families with a particular emphasis on oxygenated compounds. Indeed, oxygenated molecules can significantly impact the macromolecular properties of the recovered materials and act as a catalyst inhibitor therefore a focus has been made to have a clear understanding of oxygen-bearing compounds present in the WTB pyrolysis oil.

Both column configurations revealed a dominance of aromatic compounds, alongside smaller amounts of normal and iso-paraffins, naphthenes and olefins. Over 800 compounds were detected including 80 oxygenated species such as ketones, aldehydes, esters, alcohols and carboxylic acid. The findings highlight the complementary strengths of both chromatographic configurations. This approach not only allows for the semi-quantification of various chemical classes with a focus on oxygenated molecules but also successfully links these identified oxygenated species to their origin within the unsaturated polyester resin. This molecular-level insight into WTB pyrolysis oil supports the development of more effective recycling strategies for composite materials.

From Waste to Resources: Sample Preparation Strategies Coupled with GC×GC-TOFMS for Characterizing Wind Turbine Blade Recycling Products

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The recycling of End-of-life wind turbine blades represents a growing challenge in the context of sustainable materials management. In this work, two recycling processes—solvolysis and pyrolysis—were investigated, each supported by a dedicated sample preparation strategy prior to the detailed characterization of the resulting products.

For solvolysis products, microwave-assisted extraction (MAE) was carried out using a hexane–methanol mixture (10:3 ratio), followed by water addition (2.5 ratio) and centrifugation to separate the phases. Pyrolysis products, in parallel, were subjected to solid-phase extraction (SPE), enabling the isolation of different chemical classes.

The resulting MAE extracts and SPE fractions were subsequently analyzed using comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry (GC×GC-TOFMS). Compound identification was based on mass spectral electron ionization (EI) database matching at 70 eV (≥800/1000) and the Linear Retention Index (LRI) (±20 range). The location of the investigated molecules on the 2D-GC plane was also considered.

Overall, around 120 compounds belonging to different chemical classes such as hydrocarbons, aromatics, and heteroatom-containing molecules were identified in the solvolysis products. For the SPE fractions of pyrolysis-derived products, the use of a normal-phase sorbent with eluting solvents of increasing polarity allowed for the separation of hydrocarbon-rich fraction from one enriched in oxygen- and nitrogen-containing compounds.

How HT-GC/MS and HT-GCxGC/MS Can Help for the Detailed Characterization of Heavy Distillation Volatile Fractions from Pyrolysis Oils

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The continuous growth of the human population, coupled with the rising demand for transportation, has resulted in higher tire consumption and, consequently, greater tire waste generation. In response, various tire waste management strategies have been explored in recent decades, particularly focusing on two main forms of valorization: material and energy recovery. Among these, tire pyrolysis has emerged as a well-established treatment method, enabling the production of value-added raw materials such as pyrolysis oils. According to literature, pyrolysis oils are a complex matrix containing high amounts of aliphatic, aromatic and heterocyclic compounds (usually more than thousands of compounds and some of them have similar boiling points). For this reason, distillation of pyrolysis oils is commonly performed to simplify the matrix and to obtain different fractions for targeted valorization processes including reuse as materials or fuels. However, previous studies have shown that the presence of heteroatomic compounds in these oils can hinder their effective utilization. For example, sulfur and nitrogen compounds are sources of toxic and corrosive gases such as SOx and NOx1. Therefore, detailed characterization of heteroatomic compounds within the distillation fractions of pyrolysis oils is essential for optimizing their downstream applications. The characterization of the volatile fraction of pyrolysis oils has been previously conducted by two-dimensional gas chromatography combined with time-of-flight mass spectrometry (GCxGC/TOFMS, Leco). In this approach, distillation fractions with boiling points up to 369 °C were successfully eluted using our developed analytical protocol. However, due to the limited upper temperature tolerance of the polar column, heavier fractions could not be eluted. Moreover, the use of a nitrogen cryogenic modulator restricts the analysis of high-molecular weight compounds in these fractions. To characterize the heavy fraction (369 °C–509 °C) of pyrolysis oils, the first step of this study involved developing a high-temperature gas chromatography–mass spectrometry (HT-GC/MS) protocol. Specific experimental conditions were optimized, including the selection of the column, as well as the temperatures of the oven, transfer line, and MS source. This protocol was then adapted to establish a high-temperature two-dimensional gas chromatography–mass spectrometry (HT-GC×GC/MS) method using a fluidic modulator (INSIGHT flow modulator, SepSolve) to prevent the trapping of high-molecular-weight compounds. This approach enabled the acquisition of two-dimensional chromatograms of the heavy fractions, which complement the previous GCxGC/TOFMS results.

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Polymer Biodegradability and the Link Between Abiotic and Biotic Degradation

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Polymer persistence is a growing environmental concern, and current biodegradability tests are often designed for small molecules and may not adequately capture the complexities of polymer degradation. This study evaluates the impact of hydrothermal pre-treatment on polyester biodegradability using a novel assessment method combining pre-treatment and advanced physicochemical and chemical analyses in parallel to biodegradability testing, enabling the identification and monitoring of specific degradation products.

Polyhydroxybutyrate (PHB) and polylactic acid (PLA) served as model biodegradable and non-biodegradable polymers, respectively. Hydrothermal ageing (130°C) was performed for varying durations (1-120 hours). Following hydrothermal ageing, water-soluble degradation products were analyzed directly by Liquid Chromatography-High Resolution Mass Spectrometry (LC-HRMS) to characterize the initial breakdown products. Subsequently, OECD 301F biodegradability tests were conducted on pristine and aged PHB and PLA particles, extracted water-soluble oligomers, and the corresponding monomers. To analyze the degradation products formed during these biodegradability tests, Solid Phase Extraction (SPE) cartridges were employed for efficient extraction from the complex biodegradability medium before LC-HRMS analysis.

The analysis confirmed the presence of PHB oligomers in hydrothermally aged samples. These same oligomers were identified at a strategically chosen 5th day of the biodegradation test. Hydrothermally aged PHB showed enhanced biodegradability (shorter lag phase, faster 60% threshold achievement), while PLA remained non-biodegradable. The contrasting responses of PHB and PLA suggest a possible correlation between the extent of hydrothermal degradation and biodegradability. This finding indicates a potential rapid screening test methodology for polyesters, which will be further investigated with additional biodegradable polymers upon solubilization/improved bioavailability such as polycaprolactone (PCL). This improved assessment approach advances our understanding of polymer environmental fate.

PyC2MC: A Framework for FTMS Data Processing and Molecular Attribution using Mass Difference Analysis

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The characterization of complex mixtures (e.g., bio-oils, battery electrolytes, recycled plastics, and many more) requires the resolving power and mass accuracy provided by Fourier Transform Mass Spectrometry (FTMS). Coupling FTMS with separation techniques or doing imaging further enhances the depth of molecular characterization. However, as data complexity increases, so does the need for efficient processing solutions.

To address this challenge, we developed PyC2MC, a framework designed to streamline FTMS data processing. It offers advanced tools for data handling, ranging from script-based and batch-processing modes to an intuitive graphical user interface (GUI). By integrating multiple processing layers, PyC2MC enhances workflow efficiency, ensuring reproducible and high-confidence molecular assignments.

PyC2MC is a Python-based framework supported by extensive documentation and testing to ensure software robustness. It follows a workflow-based approach, allowing users to combine tasks, perform batch processing, and handle large datasets while maintaining analysis reproducibility. Additionally, PyC2MC includes data visualization tools that provide users with real-time visual feedback during data processing. The framework is modular, enabling the processing of FTMS data from transient signals to peak centroid attribution. Each processing step can be configured and incorporated into a workflow that runs simultaneously on multiple datasets. Workflows can be executed via the GUI or the command line, leveraging larger computational resources for scalability.

PyC2MC provides a comprehensive suite of signal processing features, including Fourier transformation, baseline correction, and peak picking, resulting in a structured peak list. Co-addition of transients is implemented to improve the signal-to-noise (S/N) ratio over time-dependent runs, ensuring enhanced spectral quality. Baseline correction is dynamically adjusted as a function of m/z, allowing for more precise noise estimation and improved peak detection across the full mass range.

The software uses mass difference-based algorithms for calibration and molecular formula attribution. Mass difference analysis (MDA) -i.e., computing the differences between each peak in the spectrum- allows the identification of repeated molecular spacings corresponding to building blocks or isotopic patterns. Unlike traditional calibration lists provided by the user or generated using Kendrick series, PyC2MC can use targeted mass differences (e.g., CH_2 or $^{13}C–^{12}C$) to perform recalibration, enabling an unsupervised calibration process. Regarding molecular formula attribution, MDA enables linking nearly all peaks in the spectrum through identifiable mass differences. Thus, the attribution of a single molecular formula can lead to confident assignment of the entire spectrum through propagation and cross-validation using graph theory.

Beyond standard FTMS processing, PyC2MC supports hyphenated high-resolution mass spectrometry (HRMS) techniques such as liquid chromatography (LC), ion mobility spectrometry (IMS), and even imaging MS. The ability to execute workflows scan-by-scan (or pixel-by-pixel) enhances processing accuracy and reinforces confidence in molecular attributions across complex datasets.

By integrating these advanced processing capabilities, PyC2MC provides an efficient, scalable, and reproducible solution for complex mixture analysis, addressing the growing demands of FTMS-based research.

Detailed Characterization of Polymers and Microplastics by KMD Plots of Complex MS Spectra

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A detailed material characterization can be essential to identify the origin, fate and potential toxicity of microplastics. Most of these plastic samples comprise of a complex mixture of several polymers or co-polymers with various end groups, so the MS analysis can be quite complicated. Here, we introduce MALDI-TOF in combination with Kendrick Mass Defect (KMD) plots as a powerful tool for assessing direct information from polymers. KMD plots highly simplify the interpretation of complex spectra. By plotting signals based on their KMD values, the repeat units of the individual polymers generate linear slopes and facilitate the interpretation of composite samples. This makes KMD plots invaluable for intricate mixtures in plastic material.

Microplastic Characterization and Screening by Combining DART and High-Resolution Mass Spectrometry

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Microplastics:

- Small plastic particles (≤5mm)
- Originate from commercial products and plastics breakdown
- Varioussources:e.g.,cosmetics, textiles, packaging material, water bottles degraded by irradiation
- Highly abundant in the environment, detected at an alarming level in our marine life and drinking water

Conventional GC/MS methods
Often extensive sample preparation
Long analysis times :up to 2h (doubles hot method).

Here: DART-HRMS (Direct Analysis in Real Time coupled to High Resolution Mass Spectrometry)Rapid fingerprinting of environmental microplastics and screening of additives for origin identification.

A *g*TIMS-MRMS Instrument to Decipher Isomeric Content of Complex Organic Mixtures

Christopher A. Wootton,[1*] Julien Maillard, [2+3] Alina Theisen, [1] Gregory F. Brabeck, [1] Carlos L. Schat, [1] Christopher P. Rüger, [3] Carlos Afonso, [2+4*] and Pierre Giusti [2+3], Yann Hébert [1]

FT-ICR MS (commercially sold at MRMS) is known as not only the highest performance mass spectrometry technique in terms of raw metrics for resolving power and mass accuracy, but also as the most flexible platform in terms of MS/MS, isolation, and ion manipulation techniques.

However conspicuously missing from current MRMS systems is the ability to natively and effectively conduct ion mobility (IMS) separations.

Thus a new MRMS platform was created as a successor to the SolariX and SciMaX systems, codenamed MATCH, which was designed to effectively incorporate state of the art dual accumulation+analysis TIMS hardware, updated ion optics, improved SB3D MALDI laser, and micro grid MALDI stage hardware.

The new platform was then energy and transmission optimised for wide m/z range, sensitivity, and high flux limits for large ion ensemble analysis to feed the high dynamic range Paracell analyser – up to 15,000,000 ions per scan possible to date

Applications areas include complex mixture analysis – such as environmental extracts, green fuel analysis (bio-oils etc), conformer selective MS/MS using ExD techniques and MALDI imaging (shown separately).

The work presented herein was recently published in Analytical Chemistry, please see full details here:

Christopher A. Wootton*, Julien Maillard, Alina Theisen, Gregory F Brabeck, Carlos L Schat, Christopher P Rüger, Carlos Afonso*, Pierre Giusti.A Gated TIMS FTICR MS instrument to decipher isomeric content of complex organic mixtures. Analytical chemistry 2024, 96, 11343-11352, https://doi.org/10.1021/acs.analchem.4c01370

Session 3 – Photovoltaic solar energy

P-P1: Case Study on the Evaluation of Film Thickness and Chemical States of Perovskite Solar Cell Materials Using Various Analytical Techniques

P-P2: ThermoFisher XPS Family & Revolutionising depth Profiling with Femto-Second Laser Ablation

P-P3: In-situ and in-operando characterization of emerging photovoltaic materials by magnetic resonance spectroscopy and imaging

P-P4: Chemical engineering for the control of CIGS surface topography and chemistry: towards tandem solar cell applications

P-P5: Photoemission Spectroscopy: a Powerful Tool for Understanding the Ageing of $Cu(In,Ga)Se_2$ Solar Absorbers

Case Study on the Evaluation of Film Thickness and Chemical States of Perovskite Solar Cell Materials Using Various Analytical Techniques

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Perovskite solar cells are one of the next-generation solar cell technologies. They are manufactured by forming multilayer films - such as perovskite crystals and transparent conductive oxide (TCO) - on a substrate. Since the thickness and chemical state of these layers significantly affect the performance of the solar cell, accurate evaluation of these properties is essential in cell development. This document presents case studies on the evaluation of perovskite crystals using various analytical instruments, with a particular focus on unique film thickness evaluation examples utilizing EDXRF.

ThermoFisher XPS Family & Revolutionising Depth Profiling with Femto-Second Laser Ablation

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XPS is a very surface sensitive technique, so to investigate changes to the chemistry on the layers below the surface the experiment method involves depth profiling the material by interleaving analysis with removal steps, commonly using ion beams. However, ion beam methods induce changes in the material chemistry, affecting the validity of the results.

Here we are presenting the most recent developments offering the capability of performing depth profiling with a femtosecond laser with a 1030 nm peak wavelength and a pulse duration of 160 fs. Depth profiling has been carried out using traditional monatomic and gas cluster ion beam (GCIB) bombardment and compared to profiles recorded using a new femtosecond laser ablation method.

In the examples shown in this poster, the monatomic and cluster ion sputtering depth profiles exhibited chemical damage due to preferential sputtering of certain atomic species and preferential sputtering artefacts.

Femto-seconds depth profiles fully retained the true chemical composition of the large thick layers over hundreds of nanometers and even dizains of microns.

This revolutionary technique has been implemented in our newly developed system Hypulse, the newest member of the ThermoFisher XPS family based on a customized version of the most widely used new generation ThermoFisher XPS system in the field, the Nexsa G2.

This system benefits of the CISA workflow compatibility allowing to perform XPS with Femto-second depth profiling analysis and SEM on the very same region of interest.

In-situ and in-operando characterization of emerging photovoltaic materials by magnetic resonance spectroscopy and imaging

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In situ and in operando characterization techniques have transformed research in energy and environmental sciences by providing real-time insights into structure–property relationships. These approaches allow direct observation of phase transitions and functional changes such as chemical transformations, photo-responses, charge transport, and energy harvesting and storage. Therefore, the key learnings obtaining from these approaches have far-reaching implications on the emerging technologies: batteries, fuel cells, and solar energy systems. In this context, our work focuses on emerging organic and hybrid photovoltaic materials using magnetic resonance capabilities, including solid-state NMR and EPR spectroscopy and imaging.[1,2] We present findings that highlight how photovoltaic performance can be elucidated by probing the local structure and molecular dynamics in semiconductor layers and at device interfaces in photovoltaic cells.[4]

[1] Nature Reviews Materials, 2020, 5, 910-930
[2] Science, 2024, 384, 1227-1235
[3] Chemistry of Materials, 2024, 36, 3, 1214-1227
[4] Advanced Functional Materials, 2024, 34, 2308616

Chemical Engineering for the Control of CIGS Surface Topography and Chemistry: Towards Tandem Solar Cell Applications

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The CIGS solar cell technology, $Cu(In_xGa_{1-x})Se_2$, remains among the most competitive and robust, making this absorber highly promising for integration into new generations of tandem solar cells [1], particularly in combination with perovskite active layers. In this type of architecture, interface quality is crucial as it directly impacts the efficiency and reliability of photovoltaic devices. Here, we focus specifically on the optimization of the CIGS sub-cell.

To address this issue, we have developed a methodological approach aimed at controlled modification of the CIGS surface topography and/or chemistry. We demonstrated, on a standard sample (glass/Mo/2 µm CIGS deposited by co-evaporation), that the use of bromine-based solutions enables non-selective etching (confirmed by ICP measurements), uniform etching (observed via SEM imaging), and has the particularity of also smoothing the initially rough surface (around 210 nm Rms) to the point of achieving a mirror-polished finish if desired (confirmed by AFM mapping) [2].

Recently, we developed a specific reactor for better control of hydrodynamic conditions, allowing homogeneous etching on a larger scale than 1 cm², with proof of concept on surfaces up to 5 x 5 cm². We also developed a procedure for the in-situ generation of active bromine species to introduce these species at highly precise concentrations, resulting in finer control over the etch rate and therefore the amount of material removed, while enabling neutralization of the solution at the end of the experiment.

This bromine-based chemical etching step results in a specific surface chemistry, which can then be adjusted through chemical engineering] to improve the interfacial properties between the CIGS and the subsequently deposited buffer layer. To this end, we employed X-ray Photoelectron Spectroscopy (XPS) and developed a rigorous quantification procedure for the II-III-VI alloy elements. Thanks to the establishment of a dedicated database, chemical diagnostics can be complemented by clear identification of the species present (oxides, selenide phases, etc.) [3,4].

1. M. A. Ruiz-Preciado et al., ACS Energy Letters 7,7, 2273–2281 (2022).
2. M. Bouttemy et al., Thin Solid Films 519, 7207–7211 (2011).
3. A. Loubat et al., Journal of Vacuum Science & Technology A 37, 041201 (2019).
4. S. Béchu et al., Thin Solid Films 669, 425–429 (2019).

Photoemission Spectroscopy: a Powerful Tool for Understanding the Ageing of $Cu(In,Ga)Se_2$ Solar Absorbers

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One of the most promising solar absorbers is the quaternary alloy Cu(InxGa1-x)Se2 (CIGS), which can achieve efficiencies of up to 23.6%.[1] These excellent efficiencies are the result of decades of research marked by several key advances. Today, one of the major challenges for this technology is the long-term stability of solar cells. Theleen and Daume[2] have identified the main parameters influencing the efficiency of CIGS cells, highlighting the decisive role of relative humidity, illumination and temperature in their degradation. However, few studies have directly addressed the intrinsic reactivity of CIGS material, and more specifically the reactivity of its surface to these ageing factors.

To better understand these mechanisms, an in-depth characterisation of the CIGS surface is essential. X-ray photoemission spectroscopy (XPS) is the analysis technique of choice for this purpose: it allows the chemical environment of materials to be probed over a range of ten nanometres and its evolution during ageing to be monitored. The use of higher-energy X-rays also makes it possible to investigate deeper layers (up to about forty nanometres), thus providing valuable information on the depth of material degradation.

In this work, we seek to elucidate the ageing mechanisms of CIGS by using conventional and high-energy XPS to study the progressive evolution of its chemical structure from the surface to the subsurface, when subjected to humidity and illumination.

1. NREL efficiency chart: www.nrel.gov/pv/interactive-cell-efficiency.html
2. M. Theelen, F. Daume, Solar Energy, 2016, 586-627, 133
3. S. Béchu et al., Appl. Surf. Sci., 2022, 576 ,151898

Session 4 – Decarbonized hydrogen

H-P1: Water Purity Control – the Key for an Efficient Green Hydrogen Production

H-P2: How to Check the Purity of Green Hydrogen?

H-P3: Multiscale Mapping of Crystallographic Phase and Chemical State in Industrial Catalysts

H-P4: Operando Surface-Sensitive X-ray Scattering of Pd Nanoparticles for Selective NH3 Oxidation

H-P5: Implementation of a New Alkaline Water Electrolysis Test Setup for Evaluation of Electrolyzer Flow Field Geometries for Efficient Bubble Removal: Electrochemical Performance

H-P6: Organic Semi-conducting Nanoparticles Dispersed in Water for Photocatalysed Hydrogen Generation

H-P7: Sampling and Analysis of Hydrogen and Carbon Dioxide Matrices for Trace Compound Monitoring in the Context of Renewable Gas Integration

Water Purity Control – the Key for an Efficient Green Hydrogen Production

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Reliable green hydrogen hinges on rigorous control of both feed water and electrolyte quality across electrolysis technologies.

PEM electrolysis stacks require ultrapure water to prevent membrane fouling, catalyst damage, corrosion, and H_2 impurity. A robust treatment train must thus be paired with continuous, high-sensitivity monitoring to prevent any electrolyzer malfunctioning.

Even for alkaline electrolysis, KOH/NaOH electrolytes absorb CO_2, elevating TIC (carbonate/bicarbonate) and potentially TOC which can cause several troubles and accelerate aging of the electrolyzer. Regular analysis of these electrolytes are thus nonskippable in order to maximize hydrogen production efficiency and stack lifetime.

Together, continuous UPW monitoring and rigorous TOC/TIC control of hydroxide electrolytes protect efficiency, extend stack lifetime, and ensure high-purity green H_2 which is critical for economical and a scalable decarbonization.

How to Check the Purity of Green Hydrogen?

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Green hydrogen is, up to now, mainly produced for industrial applications as a feedstock for many different processes such as green steel production or green methanol or ammonia production. Moreover, in the coming decades the use of this green fuel will increase significantly with transportation applications, mainly based on Proton Exchange Membrane Fuel Cell (PEMFC).

In all these applications, impurities are a constant challenge and headache. In fact, presence of Sulfur based products, Carbon dioxide / monoxide or others will have a strong impact on the final process efficiency, the final product properties (ex. Carbon level in the steel) or even the lifetime of the PEMFC for mobility applications.

In this poster, discover how, based on ISO-14687 requirements, a Gas chromatography (GC) can be equipped with a large variety of detectors (FID, BID, SCD…) to realize a complete analysis of the green Hydrogen purity.

Multiscale Mapping of Crystallographic Phase and Chemical State in Industrial Catalysts

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The performance of heterogeneous catalysts in industrial processes is governed by their structural and chemical properties across multiple length scales. In industrial settings, catalysts are often shaped into extrudates, forming porous media that influence mass transport and reaction kinetics. To optimize catalyst design and longevity, it is essential to map the crystallographic phases (e.g., FCC/BCC, amorphous/crystalline), chemical states (e.g., oxidation states), and their spatial inhomogeneities across scales ranging from nanometers to centimeters. This study presents Shell's integrated multiscale microscopic workflow for diagnosing and visualizing these critical features in catalyst extrudates.

We employ a tiered imaging strategy to capture structural and chemical information across scales: (i) Millimeter to micrometer scale: Synchrotron-based micro XRD-CT enables non-destructive mapping of crystallographic phases and elemental distributions. (ii) Micrometer to nanometer scale: FIB-SEM tomography provides high-resolution 3D imaging of pore structures and local morphology. (iii) Nanometer and sub-nanometer scale: 4D-STEM techniques allow precise mapping of nanoscale inhomogeneities in phase and chemical state. (iiii) This multiscale approach bridges seven orders of magnitude in spatial resolution, offering a comprehensive view of catalyst architecture.

We applied the workflow to the Fisher Tropsch catalysts. XRD-CT revealed significant inhomogeneities in elemental and phase distributions within extrudates. FIB-SEM tomography enabled detailed reconstruction of pore networks, essential for modeling mass transport. 4D-STEM exposed nanoscale variations in crystallographic and chemical states, highlighting regions of potential catalytic activity or degradation.

Shell's multiscale and multimodal imaging workflow provides a powerful toolset for characterizing porous solids in energy transition technologies. By mapping phase, chemical state, and inhomogeneity across scales, this approach supports catalyst optimization for applications including hydrogen production, biomass conversion, and direct air capture but the field of application likely much wider.

Operando Surface-Sensitive X-ray Scattering of Pd Nanoparticles for Selective NH_3 Oxidation

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In the transition towards decarbonized energy solutions, ammonia (NH_3) is emerging as a promising carbon-free fuel and hydrogen carrier. It offers the advantage of large-scale production using well-established infrastructure and can be efficiently stored and transported. Beyond its traditional role in fertilizer production, NH_3 is being explored for direct combustion, fuel cells, and hydrogen storage/release applications. In this context, controlling its oxidation to maximize N_2 selectivity is crucial, as this minimizes NO_x emissions. To this end, we have investigated oxide-supported arrays of Pd NPs, prepared via plasma-assisted micelle nanolithography, as catalysts for NH_3 oxidation, focusing on the role of subsurface species in governing activity and selectivity. Simultaneous operando grazing incidence X-ray diffraction (GIXRD) and grazing incidence small-angle X-ray scattering (GISAXS) were performed at the SixS beamline of Synchrotron SOLEIL (France). We demonstrate that plasma-assisted synthesis allows for tuning the reactivity of Pd NPs and their activity in oxidation reactions. Our experimental results highlight the crucial role of plasma-assisted synthesis in the architectural design of nanomaterials: by precisely altering the synthesis procedure, one can effectively tailor metal-support interactions, ultimately impacting the structure, morphology, and catalytic performance of the nanoparticles.

Implementation of a New Alkaline Water Electrolysis Test Setup for Evaluation of Electrolyzer Flow Field Geometries for Efficient Bubble Removal: Electrochemical Performance and Gas Quality Analysis

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Recent estimates of the global production of Green Hydrogen are ~ 40 kt (2021), significantly less than total production (100 Mt). The primary reason is cost, with Grey Hydrogen costing only 1-2.4 USD / kg [International Renewable Energy Agency, IRENA] compared to 3.6-7.5 USD / kg (50 kW electrolyzer). A breakdown of average installation costs of an electrolyzer are shown in Figure 2(a). Decreasing costs would require both a lower price of installation (CAPEX) and operation (OPEX). Lower OPEX can be achieved at a lower electricity cost or at higher efficiencies – the EU target for 2050 is system efficiency < 45 kWh / kg H_2. Lower CAPEX may be achieved through economies of scale, or simplification of balance of plant (BOP) – the EU target for 2050 is < 200 USD / kW for (minimum) 10 MW installations [IRENA]. In alkaline water electrolysis (AWE), gases (H_2 & O_2) are generated at both electrodes. Presence of bubbles on the electrodes and in the electrolyte influence the electrochemical performance, and efficiency, of the electrolyzer in three ways: (i) increased charge-transfer overpotential (ηCT) due to decreased electrochemical surface area, (ii) increased Ohmic overpotential (ηO) due lower electrolyte conductivity and electrolyte-electrode contact, and (iii) decreased transport overpotential (ηT) due to disruption of supersaturation in the vicinity of the electrode. The most significant impact is the increase in Ohmic resistance seen at higher cell voltages. Minimization of the influence of bubbles at both electrodes can improve the operational efficiency of an AWE, beneficially lowering the OPEX. The evacuation of bubbles can be enhanced through forced convection of electrolyte past the electrodes. The AWE housing contains a flow field geometry which flows the electrolyte and removes the two-phase fluid from each electrode chamber. The roles of the flowing electrolyte in the electrolyzer include supply of H_2O, ionic connectivity (OH- conductivity), heat management, and bubble removal. H_2O(cathode) and OH- depletion (anode), and bubble accumulation may lead to increased cell voltage / decreased current density. AWE BOP system optimization includes minimizing the power consumption by auxiliaries, include the electrolyte pump. Heat management requirements will depend on the tolerable temperature gradient throughout the balance of plant (BOP). To evaluate different flow field geometries for AWE, an experimental test setup is required. This should allow: circulation of corrosive KOH electrolyte, application of electrical power, and safe generation and analysis of H2 and O2. Here, we describe the assembly of such a test rig. The design and 3D printing of flow field geometries for efficient bubble removal from single zero-gap architecture AWE. Performances are evaluated through electrochemical and gas quality measurements under various flow rates and back-pressure regulation (26 wt% KOH, 20°C). The goal is to select an optimum flow geometry for greater electrochemical performance (lower Ohmic overpotential, higher efficiency) and enhanced H_2 purity, all while operating under safe conditions (low H_2-in-O_2 crossover).

Organic Semi-conducting Nanoparticles Dispersed in Water for Photocatalysed Hydrogen Generation

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The sun's photons provide the largest source of energy on Earth and harnessing them could help solve the energy crisis without harming the environment. p-conjugated organic semiconductors (OSCs), have recently been dispersed in water as nanoparticles to provide human and environmentally friendly solutions for processing organic photovoltaics (OPV) as an alternative to classical toxic organic solvents.[1,2] These dispersions have been also thoroughly studied and developed by us for OPV, demonstrating that binary donor-acceptor nanoparticles allowed to obtained solar cells with an efficiency of 10% (state of the art for water inks). But, the main limitation of photovoltaics is that the electricity production is intermittent.

Solar fuels, which result from the conversion of photon energy into chemical bonds, are being developed as energy storage systems. In this context, Hydrogen (H_2) production by photocatalytic water splitting has emerged as a fantastic solution to store solar energy in a distributable fuel that does not produce CO_2, neither during production nor consumption. Pinaud et al. have shown, using life cycle analysis, that a colloidal system, where the photocatalysts are dispersed as nanoparticles (NPs) in water would be the cheapest technology for H2 production.[3] H2 is produced (and oxygen) by H2O splitting with an energy barrier of 1.23 eV, which can be overcome by using sunlight energy to drive the reaction.

In this report, we will present two systems of NPs based on polymers either P3HT or PTQ10, with the acceptor PCBM or Y6, changing also the surfactant used during the nanoprecipitation process.[4]

The standard P3HT/PCBM couple exhibited a lower hydrogen evolution rate (HER) than that of a more efficient couple PTQ10/Y6 (as observed for OPV). Moreover, by playing in both systems with the surfactant nature (mandatory to ensure dispersion stability) different HER were obtained. The study considers sodium dodecyl sulfate (SDS) and sodium 2-(3-thienyl)ethyloxybutylsulfonate (TEBS), both anionic surfactants. After a deep characterization of the NP morphology, that can be intermixed, Janus or core/shell, we found out that P3HT:PC61BM NPs were synthesized with Janus and core-shell morphologies, with SDS and TEBS, respectively.

As a result, the superior photocatalytic performance was attributed to the more favorable Janus morphology. This same photocatalytic trend was observed when using the PTQ10:Y6 couple. We show that this enhanced performance is attributed to a more favorable D:A blend morphology, which positively influences exciton dissociation in the case of P3HT:PC61BM, and platinum cocatalyst loading for PTQ10:Y6 nanoparticles. These findings were supported by advanced analytical characterizations as cryo-TEM, steady-state fluorescence, TAS measurements for morphology study, and SP-ICP-MS for platinum loading on NPs.

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Sampling and Analysis of Hydrogen and Carbon Dioxide Matrices for Trace Compound Monitoring in the Context of Renewable Gas Integration

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The energy transition requires the deployment of sustainable and technically reliable solutions to replace fossil fuels and achieve carbon neutrality by 2050, as defined by the European Union (1) and France's National Low-Carbon Strategy (2). In this context, renewable and low-carbon gases such as biomethane, synthetic methane, or hydrogen (H_2) play a key role. Carbon capture is essential for reducing industrial emissions and supporting the path to net-zero.

Captured carbon dioxide (CO_2) can be utilized as a feedstock in the production of synthetic methane via methanation, synthetic fuels, chemicals, or building materials, or permanently stored in deep geological formations to prevent its release into the atmosphere (CO_2 sequestration). Hydrogen (H_2) can be used as a low-carbon feedstock and energy vector for various sectors such as industry (e.g., steel or ammonia production), transport (hydrogen fuel cell trucks, trains, or buses), or flexible power generation (e.g., co-firing in gas turbines). These uses require a specific gas quality. Although present at very low concentrations, volatile organic compounds (VOCs) may interfere with odorant systems, degrade combustion efficiency, or contribute to infrastructure corrosion. A better understanding of their nature and concentration is essential to support process design and gas quality specifications.

This study aims to transfer, adapt, and validate a complete analytical workflow for the detection and quantification of trace VOCs in H_2 and CO_2 matrices. Gas sampling is carried out using stainless steel adsorption tubes filled with an adsorbent phase (e.g., Tenax TA, a porous polymer adsorbent). Tubes are placed in a sampling bench equipped with flow and pressure control, allowing a defined volume of gas to pass through the adsorbent. Only the target trace compounds are retained on the adsorbent phase, while the main components of the matrix, such as H_2, CO_2, or H_2O, pass through without being trapped. The analytes are then thermally desorbed and analyzed by comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry (TD-GC×GC-TOFMS), using a Pegasus BT 4D system (LECO), with a non-polar Rxi-5ms column in the first dimension and a semi-polar DB-1701 column in the second dimension (3).

Compounds are identified through deconvolution and spectral matching using ChromaTOF software (LECO). Quantification is achieved through external calibration by chemical family using selected representative compounds. This allows the distribution of chemical families to be assessed in each sample. Further comparisons of the gas matrices are performed using the ChromaTOF Tile software (LECO), which highlights statistically significant differences between sample groups by comparing analyses.

Preliminary results, involving CO_2 and H_2 used to load a reference mixture onto adsorption tubes, indicated that the method previously developed at LSABM for biomethane sampling and analysis could be applied to H_2 and CO_2 with minor adaptations. These include adjustments in sampling volumes and flow rates to address matrix-specific behaviors (3).

This analytical strategy contributes to the development of robust and transferable methods for trace VOC monitoring in H_2 and CO_2 and provides technical support for the integration of these gases into existing infrastructures.

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Session 5 – Bioresources

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Support Biofuels and Biogases Synthesis Challenges with Analytical Solutions

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Net zero emission target cannot be reached only with batteries or hydrogen-based fuel cells. Due to previous devices technical limitation and for some applications, such as air and sea transport, need of fuels will remain up to 2050 and even after.

So use of Biofuels and biogases are essential to support these activities field and maintain the required transportation level by our actual world. Bio-ethanol in addition to fossil fuels, e-methanol as ship fuels, Sustainable Aviation Fuels (SAF) and all the Renewable Fuels of Non-Biological Origin (RFNBO according RED III directive) are thus keys in the actual strategy to reduce human impact on climate change.

This new biofuels / biogases synthesis processes need of course to reach an efficiency to be compliant with economic targets and consequently a lot of different parameters need to be carefully checked and controlled during all the synthesis and in the final product. In this poster, discover how various analytical technics like GC, IC, ICP-MS are fundamental to manage synthesis process and rich the expected quality level.

Biofuels - Perfect Quality Control with Leco

Michael Jakob [1]

[1] European field product manager, LECO application & technology center, Berlin

This work presents advanced analytical solutions for quality control in biofuels and sustainable aviation fuels (SAF). LECO instruments enable rapid and precise characterization through CHNS elemental analysis, which quantifies carbon, hydrogen, nitrogen, and sulfur to evaluate combustion properties and emissions. Thermogravimetric analysis (TGA) provides insights into moisture, volatile compounds, and ash content, key factors influencing overall fuel performance. Calorimetry offers accurate determination of heating values, while ash fusion analysis predicts slagging tendencies by examining ash melting behavior. Additionally, comprehensive separation science using GC×GC-TOFMS reveals the complex chemistry of biofuels, supporting detailed characterization and regulatory compliance. Together, these methods ensure reproducible, efficient, and robust results for both research and industrial applications in sustainable energy.

Proximate Analysis of Solid Biofuels with Thermogravimetry

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This study evaluates the use of the LECO TGA 801 for proximate analysis of solid biofuels, focusing on moisture, volatile matter, and ash content. Thermogravimetric methods were compared against ISO standard procedures to assess accuracy and reliability. Results showed strong agreement between the automated TGA and manual ISO methods, with minimal deviations for moisture and ash values across wood pellets, peat, coal, and plastic waste samples. For volatile matter, both methods demonstrated a close correlation, with an R^2 of 0.94 and an average difference of only 0.1%. The automated TGA 801 allows simultaneous analysis of up to 19(38) samples, providing significant advantages in efficiency, throughput, and data security. Overall, the TGA 801 delivers accurate, reproducible results without the need for additional calibration, making it an effective tool for biofuel quality control.

Application of a Novel Reverse-fill-flush Modulator and Splitter for Simultaneous GCxgC-TOFMS/FID Analysis of Synthetic Aviation Fuels

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The certification of sustainable aviation fuels (SAFs) requires rigorous compositional assessment to ensure equivalence with conventional fuels and compliance with regulatory standards. Comprehensive two-dimensional gas chromatography (GC×GC) has proven to be a robust approach for hydrocarbon group-type analysis, providing detailed compositional profiles that support evaluation against ASTM and DEF STAN specifications.

In this study, a reverse fill–flush modulator combined with a flow splitter enabled simultaneous dual detection by flame ionization detection (FID) and time-of-flight mass spectrometry (TOFMS) within a single GC×GC run. This configuration links quantitative FID data with mass spectral identification, thereby improving confidence in both bulk group-type classification and the characterization of trace oxygenated or aromatic species relevant to performance and emissions.

Petroleum-derived fuels and a range of synthetic alternatives, including Fischer–Tropsch synthetic paraffinic kerosene (FT-SPK), FT-SPK with aromatics (FT-SPK/A), hydroprocessed esters and fatty acids (HEFA-SPK), and synthetic aviation turbine fuel (SATF), were analyzed. The workflow provided structured chromatographic separation of paraffins, naphthenes, and aromatics, alongside targeted identification of heteroatom-containing constituents. Classification templates applied to FID data yielded reproducible quantification across fuel types, supporting comparability of results across production pathways.

The demonstrated approach integrates chromatographic resolution, quantitative accuracy, and spectral specificity into a single analysis. By aligning compositional characterization with regulatory testing requirements, this method offers a valuable tool for advancing the approval and adoption of SAFs.

Electrochemical Study of Lignin Derivatives; Protection and Valorization of Biopolymers

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Lignocellulosic biomass has the potential to replace fossil fuels as a source of renewable carbon. It stands out as a rich source of carbonaceous materials, mainly composed of cellulose, hemicellulose and lignin. Unlike cellulose, lignin holds a distinguished reputation due to its aromatic nature. Since lignin is composed of three fundamental monolignols or phenylpropanoids, such as p-hydroxyphenyl (H), Guaiacyl (G), and syringyl (S) units connected via different linkages, the depolymerization of lignin can be regarded as a prime renewable source to obtain platform chemicals. However, this heterogeneous aromatic biopolymer has been undervalued and is burnt to produce cheaper heat and electricity. Lignin is produced as a byproduct during pulp and paper processing via acidic hydrolysis of lignocellulosic biomass. The harsh conditions during acidic hydrolysis induce conformational changes within the lignin polymeric chain, yielding condensed lignin and elevated temperature conditions result in the undesired degradation of lignin into gaseous products. Thus, the lignin obtained is recalcitrant and hard to depolymerize. Developing a protection protocol to avoid the condensation within the lignin so to have nearly native type lignin can greatly help to upgrade the biorefinery process and increase the productivity and profitability. Moreover, conceiving a greener depolymerisation strategy, such as electrochemical methods to produce value-added monomers, can enhance the current valorisation technologies at an industrial scale, thereby leading to the viable and economical production of commodity chemicals.

Advancing GC-Combustion-MS: A Novel Reactor Design for Multiplexed Elemental Detection and Quantification

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A new GC-Combustion-MS system has been recently introduced and extensively studied for the detection of compounds containing C, H, S, N, or O through a combustion interface positioned between the GC and MS instruments.[1],[2],[3] The system employs the online addition of an oxidizing gas flow (0.4 mL/min of 0.3% O_2 in He) and a ceramic reactor with platinum wires acting as a catalyst. Importantly, these targets elements have been analyzed separately rather than simultaneously in a single chromatographic run. This limitation arises because quantification of N- and S-containing compounds is only accurate at high combustion temperatures (>1000 °C), whereas oxygen-containing compounds are more difficult to quantify above 850 °C due to the activation of the oxygen present in the alumina reactor walls. Such activation promotes their exchange with the oxygen travelling along the alumina tube, both the isotopic combustion gas and most importantly, the target O originally present in compounds, hampering its detection. To address this issue, a platinum sleeve was installed to prevent such exchange with the ceramic walls, enabling reliable multi-elemental quantification at high temperatures.

This improvement provides a complete elemental profile of each organic compound without the need of compound-specific standards, combining element-selective detection of C, H, O, N, and S with molecular identification by MS. For unknown samples, O-, N-, and S-containing compounds can be easily discriminated in 3D-plots by calculating the 16:12, 32:(16+68):12, and 68:12 ratios for each detected GC peak. Furthermore, detection limits achieved were as low as 20 pg O and below 0.2 pg for N and S. The approach proposed showed excellent agreement with certified values of various SRMs using only simple, generic internal standards. Finally, it was successfully applied to the simultaneous detection and quantification of O-, N-, and S-compounds naturally present or spiked at low ppm levels in a complex diesel sample.

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Characterization of Bio-oils Derived from Biomass Pyrolysis using Liquid Chromatography Hyphenated with 18 T FTICR Mass Spectrometer

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Lignocellulosic biomass is the most abundant renewable feedstock and can be used to produce bio-oils through fast pyrolysis processes. Depending on the processes and feedstocks used, the bio-oils have very different molecular properties. For this reason, it is important to characterize these bio-oils precisely, in order to optimize upgrading processes. Since they are highly complex mixtures, their characterization at the molecular level is generally done by direct introduction FTICR-MS yielding loss of isomeric information and loss of information through ionization discrimination. For these reasons, RPLC has been used to probe the isomeric level and faces ionization competition.

Chromatographic analyses were performed with an ultra-high performance liquid chromatography (UHPLC) system (Thermo Vanquish), on two different types of reversed-phase columns: an ACQUITY UPLC HSS T3 (100 × 2.1 mm, 1.8 µm). Experiments were carried out on an 18 T FTICR prototype (Bruker timsMRMS) [1], equipped with a electrospray ionization (ESI) in positive mode.

A preliminary RPLC-Orbitrap study optimized the ionization and separation of 37 standards, representative of potential bio-oil compounds and covering a wide polarity range, analyzed to evaluate two different C18 columns and elution gradients. Based on these results, LC-FTICR mass spectrometry was used to increase the dynamic range and enabled a better understanding of bio-oil composition, resolving additional unresolved peaks. Thanks to the power of the 18 T magnet and despite the significant amount of ions in the ICR cell, the mass measurement varied by only about 0.5 ppm, allowing for accurate assignment of molecular formulae. This study allowed the classification of distinct molecular families, including sugars, acids, polyphenols, PAHs, and fatty acids. The coupling enabled the observation of 1,610 molecular formulae compared to direct injection. Among the compounds separated in the bio-oil, potential isomers were observed. In addition, different profiles for the same EIC (Extracted Ion Chromatogram) were observed between two different bio-oils.This work enabled us to determine molecular differences as a function of treatments and feedstocks, despite the isomeric and isobaric complexity.

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Comprehensive Gas Chromatography Combining with Orbitrap Based Mass Spectrometer GCxGC-HRMS for Biofuels Characterization

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Many samples are characterized by several hundreds or thousands of compounds, by one-dimensional gas chromatography, these compounds are frequently co-eluted causing a lot of difficulty for detection and identification, even for the very efficient detector such as high resolution mass spectrometer Orbitrap based technology. It is clearly important to separate these co-eluted compounds from the gas chromatography, and one of the most effective way is the use comprehensive GC. In this case, the co-eluted compounds by the first column may be separated by the second column which facilitates the detection. In this work, we report our results in analyzing alkaline mixture, and a biomass sample, by using two-dimensional gas chromatography (GCxGC) combine with the mass spectrometer Q-Exactive GC. The goal of the present research is to integrate high separation capacity of two-dimensional gas chromatography with high resolution and precision mass spectrometric detection in order to maximize compound identification from complex sample such as biofuels. Our results showed an important gain of intensity by using GCxGC-MS in comparison with GC-MS, the resolution and separation were increasingly improved which facilitated the detection and identification. The untargeted identification confidence was also evaluated with different databases and combined with the elemental composition. Various electron ionization energies, along with different chemical ionizations, were also used to identify of untargeted compounds in biofuels.

Development and Application of Analytical Methods for Monitoring Halogenated Contaminants in Samples from New Energy Sources

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Sustainable fuels are derived from agricultural or industrial waste, tipically in solid form. One method for converting these wastes into a liquid state is pyrolysis, a process that transforms solid material into an organic liquid called pyrolysis oil. This oil can subsequently be upgraded through refining to improve its quality.[1] This highly promising technique enables waste to be recycled and reduced in volume.[2] However, during the life cycle of the waste, compounds may have been added, such as pesticides, flame retardants or other additives... Certain compounds present in these raw materials are then likely to end up as contaminants in the production cycles or even in the finished products. Contaminants include halogenated compounds such as polyfluoroalkylates (PFAS)[3], brominated compounds found in flame retardants[2], and chlorinated compounds, mainly found in pesticides and also present in polyvinyl chloride-based plastics.

The aim of this work is to develop a method using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) to measure the contamination of pyrolysis oils by halogenated compounds. ICP-MS is the instrument of choice for total elemental analysis or speciation by coupling with chromatographic techniques. However, as far as halogens are concerned, their low ionization efficiency in ICP plasma does not allow acceptable detection limits to be reached. Detection is therefore based on the addition of Barium salts, enabling the formation of polyatomic ions (BaF+) that are more easily detected [4], [5]. This methodology takes advantage of the high sensitivity of ICP-MS and its ability to analyze complex matrices, while using speciation techniques already developed for the analysis of complex matrices. The main objective is to improve the monitoring of halogenated contaminants, primarily fluorine, in matrices derived from sustainable fuels, in particular those produced from pyrolysis oil.

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Development Bio Oil Characterization by GC-C-MS and FT-ICR-MS

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The increasing production of lignocellulosic biomass waste presents a dual challenge and opportunity for sustainable resource recovery. Thermochemical conversion appears to be the most promising method for producing bio-oils and other compounds, such as bio-char or bio-gaz. The pyrolysis process has been shown to be an effective technique for enhancing the value of biomass, with the potential to be applied to a wide range of biomass sources. The process entails subjecting biomass polymers to heating in an environment devoid of oxygen, thereby facilitating their depolymerisation while impeding combustion. The biomass pyrolysis process produces three phases of material: ash (solid), bio-oil containing a mixture of water and organic matter (liquid) and non-condensable gases (gas). Subsequent calcination of the ash at 700°C results in the generation of silicon particles.[1]

Corn biomass pyrolysis oils are a complex mixture of organic compounds containing a mass of highly oxygenatedcompounds, including phenols, ketones, carboxylic acids, etc., which can be determined by techniques such as Gas Chromatography coupled to Mass Spectrometry (GG-Combustion-MS).[2], [3] FTIR spectra, on the other hand, indicate the presence of functional groups in bio-oils. By optimising the pyrolysis process, it is possible to produce a material that can be refined into sustainable aviation fuel (SAF), notably through the use of Fourier transform very high-resolution mass spectrometry (FT-ICR-MS).

The integration of pyrolysis into the circular economy has been demonstrated to be a highly effective method of reusing biomass waste, reducing environmental impact and supporting renewable energy systems.

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Pyrolysis of Corn Stover for Decarbonization: Exploring the Potential of a Major Agricultural Residue

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The global transition toward carbon neutrality requires a profound transformation of the energy system, particularly in hard-to-electrify sectors such as industry and transport. According to IEA (2024) data, renewable energy accounted for only 14% and 2% of the heat and transport sectors, respectively, in 2023, while it reached 30% in electricity generation, dominated by solar, wind, and hydropower. By 2030, renewables are projected to represent 46% of electricity production, but only 19% and 6% in heat and transport, respectively, highlighting the need for complementary solutions. In this context, bioenergy plays a crucial role, providing solid, liquid, and gaseous carriers capable of displacing fossil fuels and diversifying the renewable energy portfolio.

Among biomass resources, corn stover stands out as one of the most abundant lignocellulosic materials worldwide. According to FAO's World Food and Agriculture – Statistical Yearbook 2024, maize is the world's second most important primary crop after sugarcane, with a global production exceeding 1.2 billion tonnes annually, compared to 0.8 billion tonnes each for wheat and rice. This enormous production generates a correspondingly vast quantity of residues, which remain largely underutilized.

Corn stover's intrinsic properties—high lignin content and low moisture—make it less suitable for biochemical pathways such as anaerobic digestion, but highly favorable for thermochemical conversion. Within thermochemical routes, pyrolysis emerges as the most versatile, capable of producing a solid energy carrier (biochar), a liquid fuel precursor (bio-oil), and a combustible gas (bio-syngas). These products can contribute simultaneously to carbon sequestration, renewable fuel supply, and energy diversification. Compared to gasification or hydrothermal liquefaction, pyrolysis offers a flexible operating range and compatibility with heterogeneous residues (Awasthi, et al., 2023), making it especially suitable for agricultural byproducts like corn stover.

In the current industrial context, companies involved in decarbonization from agricultural residues generally focus on biogas and biomethane production through anaerobic digestion, using manure, agro-food wastes, or mixed agricultural residues. Similarly, the biochar sector is growing, but primarily with forest biomass rather than maize residues, highlighting an underutilized opportunity.

Within this landscape, the University of Pau (UPPA) is developing a research project to valorize corn stover via pyrolysis, producing biochar, biofuels, and bio-syngas. This initiative aims to explore the technical potential of this major residue for decarbonization, integrating it into renewable energy strategies and addressing sectors that are difficult to electrify.

By deploying pyrolysis-based valorization of corn stover, multiple opportunities arise: (i) Biochar as a stable carbon sink, soil amendment, and solid fuel with high energy density. (ii) Bio-oil as a versatile liquid biofuel, with or without upgrading depending on the intended use. (iii) Bio-syngas as a renewable gaseous fuel, complementing existing bioenergy infrastructures.

In conclusion, corn stover represents a promising lignocellulosic residue for advancing decarbonization. Its abundance, favorable properties for pyrolysis, and underutilization make it a strategic resource. Integrating pyrolysis pathways into renewable energy strategies can significantly contribute to sustainable energy deployment, carbon sequestration, and valorization of agricultural residues.

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Tracing Renewable Oils by Isotope Ratio Measurement and Elemental Characterization

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Around 96% of the fuels used for transportation are derived from crude petroleum reserves, which contributes to the current climate crisis. Renewable fuels provide a lower environmental impact since they can be obtained through chemical, biochemical, and thermochemical processes by employing multiple biomass sources such as lignocellulosic biomass, organic waste or algae [1]. Due to its high oxygen content, they cannot be used as fuel without prior treatment. Co-processing these oils with fossil fuels is a promising opportunity to convert low-quality renewable oils into valuable fuels [2]. To maximize the incorporation of renewable oil in conventional fuels, optimizing the co-processing parameters could be done by tracking the renewable fraction [3]. Accurately measuring renewable fraction in final fuel after co-processing will enable refineries to produce “greener” fuels [4].

The aim of this project is to use Isotope Ratio Mass Spectrometry (IRMS) to trace the renewable fraction in co-processed fuels, but also to explore the isotopic fractionation in recycled plastics, in corn waste and the products obtained after pyrolyzing it, and in recycled and conventional base oils. A methodology will be established using Elemental Analyzer – IRMS (EA-IRMS) technique to quantify the total content of carbon and hydrogen isotopic ratios (δ13C, δ2H). Gas Chromatography – Combustion – IRMS (GC-C-IRMS) technique will be also used to investigate the measurement of the isotope ratios of δ13C, δ2H of individual volatile and semi-volatile compounds extracted and detected by an approach of compound specific isotope analysis (CSIA).

In this work, we present an original approach in the application of isotopic analysis to trace the renewable fraction of complex matrices, which has not been widely investigated in previous research. This novel perspective will provide valuable information that would facilitate the integration of a greater proportion of recycled materials into industrial processes and thereby advancing sustainability efforts.

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Structural Characterization of Lignocellulosic-based Pyrolysis Oil by Two-dimensional Mass Spectrometry

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Pyrolysis oils are the most important product in the process of manufacturing biofuel from lignocellulosic biomass. They are complex organic mixtures composed of thousands of distinct elemental compositions. Because raw materials are varied and complex, their composition can be unpredictable and cause problems that reduce biofuel energy content. Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS) enables high-confidence assignments of elemental compositions of pyrolysis oils, but cannot differentiate between isomers without fragmentation [1]. Ion isolation before fragmentation is not practical for the structural characterization of a sample with thousands of compounds. Here, we use two-dimensional mass spectrometry (2D MS), in which precursor and fragment ions are correlated without isolation, for the structural characterization of pyrolysis oil.

2D mass spectra of lignocellulosic-based biomass pyrolysis oil were recorded on an FT-ICR mass spectrometer with Gäumann's pulse sequence coupled with gated trapped ion mobility spectrometry (gTIMS) capability [2,3]. Ionization was performed in positive mode with ion sources such as electrospray and atmospheric pressure chemical ionization. Fragmentation was performed with electron induced dissociation. Data processing and visualization for 2D mass spectra was performed with the open-source Spectrometry Processing Innovative Kernel (SPIKE) software, developed in 64-bit python language [4]. For peak assignments and data interpretation, the “python tools for complex matrices molecular characterization” (PyC2MC) open-source software was used [5].

In 2D mass spectra, fragment *m/z* are plotted horizontally and precursor *m/z* are plotted vertically. Characteristic lines can be extracted from 2D mass spectra, such as the autocorrelation line, which shows the *m/z* of the precursor ions, fragment ion scans, which show the fragmentation pattern of each precursor, precursor ion scans, which show all the precursors of a given fragment, and neutral loss lines [2]. Extracting precursor ion scans and neutral loss lines enables the quick visual identification of compounds with structural similarities, which is an obvious advantage for the analysis of complex samples. Resolving powers of 120,000 at *m/z* 400 were obtained for fragment ion peaks, and of 2000 at *m/z* 400 for precursor ion peaks. The autocorrelation line was extracted for elemental composition assignments, which were compared to those obtained from one-dimensional mass spectra. The correlation between precursor and fragment ion peaks was obtained for differences of the order of 10 mDa (e.g. CH4 vs. O). Neutral loss lines were extracted and the elemental composition of the neutrals could be assigned with high confidence. Some of them

corresponded to well-known compounds in pyrolysis oils (e.g. levoglucosan at C6H10O5) and others were more unexpected [6]. Here, we show the performance of 2D MS of pyrolysis oil, the workflow that was established in SPIKE and PyC2MC, and we discuss the structural information that was gained for lignocellulosic-based biomass pyrolysis oil

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[2] doi/10.1007/s00249-019-01348-5
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Session 6 – Electrical Storage and Batteries

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Shimadzu Total Solution of the Analysis for Lithium-ion Batteries

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Lithium-ion batteries (LiBs) are vital for mobility and energy applications in a decarbonized society. With the rapid growth of the EV market, demand extends beyond high performance to stable quality and sustainability, driving the need for advanced analytical technologies throughout the battery lifecycle.

Shimadzu provides comprehensive solutions for R&D, material characterization, manufacturing quality assurance, degradation analysis, and recycling. This presentation highlights practical applications of particle evaluation systems and testing machines for quality control, as well as analytical approaches for battery recycling."

Comprehensive Analysis of Lithium-Ion Battery Electrolytes and Internal Gases Using Advanced Systems

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Lithium-ion batteries have become crucial in today's society, powering everything from portable electronics to electric vehicles and energy storage systems. Ensuring their performance, safety, and longevity requires various analyses and evaluations. These include assessing the composition and purity of components like electrolytes and analyzing internal gases to evaluate battery degradation. Gas chromatography (GC) play a vital role in these analytical tasks.

The GC-2050 demonstrated its usefulness by analyzing carbonic esters and additives in the electrolytes with high separation and repeatability, making it effective for routine quality control.

Moreover, we focused on the analysis of internal gases, which is crucial for evaluating lithium-ion battery degradation. In the presentation, we will introduce new methodologies for internal gas analysis using the GC-2050 with a state-of-the-art system.

Black Mass Analysis – Characterize Inorganic and Organic Content for an Efficient Recycling

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Lithium Batteries recycling is a key and growing topic. Due to the increase of the use of lithium batteries and their content in expensive metals such as Cobalt, nickel or even lithium, developing a way to extract and reuse them in a new batteries become economically interesting. Moreover, this recycling also prevents the risk of environmental pollution from old used batteries

Results of the batteries crushing the Black mass (BM) contain a complex mix of cathode oxides, graphite, current-collector foils, salts and residual electrolytes. Accurate, fast, and traceable quantification of major metals and volatiles compound content is essential for process control and for meeting EU 2023/1542 recovery targets. Discovers how EDXRF and HS-GCMS solve this main challenge.

Determination of Lithium-Ion Battery Electrolyte Composition with PolyarcMicroreactor

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Electrolytes composition is key to ensure and maintain performances during all the battery life time and variations in their composition can significantly affect battery performance and safety. Consequently, quality assurance is essential to determine the precise composition of organic carbonates in the electrolyte.

But this analysis facing a major challenge. All the classical analysis technics (GC-FID, GC-MS, LC or NMR) provide a molecule dependent response. An external calibration with specific standard is thus mandatory to obtain accurate results which make the analytical process more complicated, especially for degradation studies.

The Polyarc system has a catalytic microreactor that enhances gas chromatographs with flame ionization detectors((FIDs) by converting all organic compounds to methane molecules prior to their detection by the FID, therefore converting the FID into a quantitative carbon detector (QCD). This approach eliminates thus complex calibrations and enhances significantly the accuracy of quantification.

Correlative XPS & SEM analysis with Cisa workflow

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Across a wide range of application areas, understanding the chemistry and structure of surfaces and interfaces is crucial. Developing in Research and industry in the last fifty years, X-ray photoelectron spectroscopy (XPS) has recently become one of the key techniques for measuring surface and interface chemistry, and advances in instrumentation have enabled it to master with the requirements for both academia and industry. XPS can deliver quantified surface chemistry measurements, and by using depth profiling, an understanding of layer and interfacial chemistry.

Other experimental techniques like SEM are unable to match the surface selectivity of XPS, but the influence of a changing morphology of the surface on the measured composition can be determined using SEM in addition of XPS. Electron microscopy can provide high resolution imaging, with elemental composition provided by energy dispersive X-ray microanalysis, but without the same surface selectivity seen with XPS or Auger electron spectroscopy (AES). While SEM can easily visualize 2D materials, these layers are typically too thin to be easily characterized with the analytics commonly present on the microscope such as energy dispersive X-ray (EDX) analysis. Performing this kind of correlative XPS and SEM experiments is only possible if SEM and XPS analysis can be performed exactly on the same region of interest, this has been made possible thanks to the development of the CISA workflow.

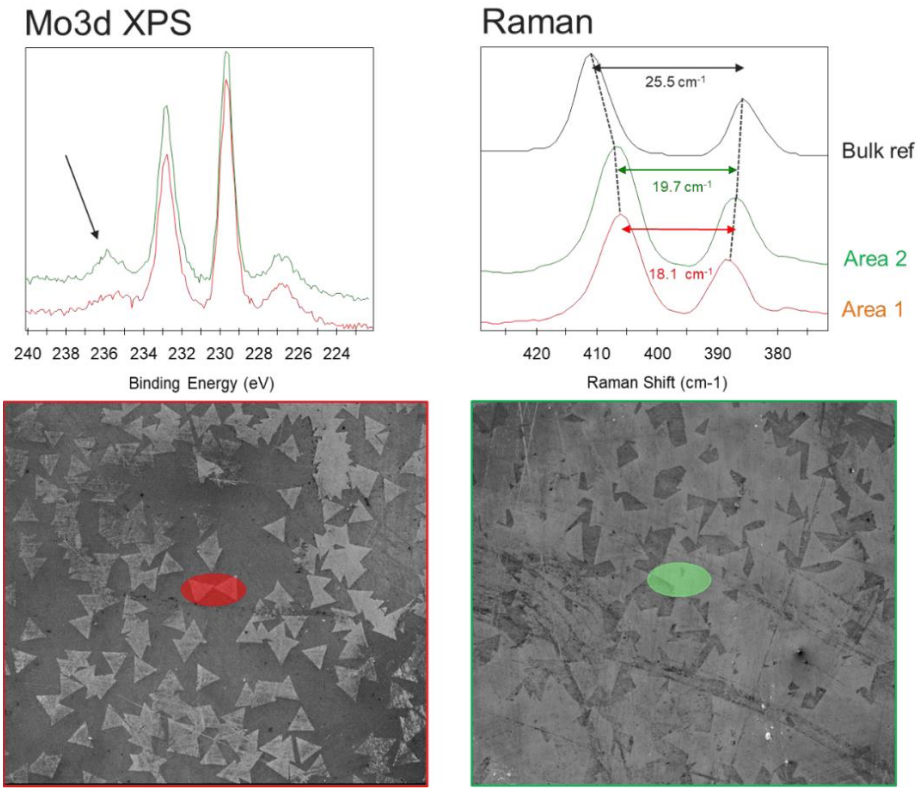

Figure 1. Coincident and correlative XPS & Raman and SEM analysis of single-layer, 2-dimensional materials of MoS2 crystallites on SiO2 substrate. XPS and Raman analysis were performed with the same Nexsa G2 system at two sample locations on the same surface analysis instrument. XPS reveals the stoichiometry and cleanliness of the MoS2, while Raman can determine monolayer vs multilayer structure of the crystallites. The sample was transferred to an SEM using the CISA workflow and VTM2 clean transfer without pollution. The SEM images of the sample areas reveal the crystallite shapes, orientations, and coverage.

Combined Experimental and Theoretical Surface Studies of Anti-wear Additives for E-mobility

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With the rise of electric mobility, new forms of wear are emerging in mechanical contacts. This necessitates the development of new fluids to meet the evolving constraints. Anti-wear additives, in particular, play a crucial role in addressing these issues.[1] Phosphorus-based additives are especially significant due to their unique ability to form protective layers on metal surfaces, thereby reducing friction and wear under high-stress conditions.[2] In our research, we employed a robust approach that combines experimental data from quartz crystal microbalance (QCM) and X-ray photoelectron spectroscopy (XPS) with theoretical data obtained through density functional theory (DFT). This methodology was used to screen several phosphorus-based anti-wear additives in oil formulations. The additives were studied both individually and in combination to determine their synergistic and antagonistic effects. The results were then correlated with tribological tests to classify the performance of the additives. Experimental results from QCM and XPS indicated that the behavior of the additives on the surface varied according to their phosphorus-based chemistry. Some additives degraded, while others reacted to form protective layers. DFT calculations further validated these findings. Notably, bond order analyses revealed that the P–O bond exhibited varying strengths, which influenced the effectiveness of the additives.

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Elucidating via Operando XAS the Hindered Reactivity of High-Mn Content Mixed-Olivine Cathodes for Na-ion Batteries

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Sodium-ion batteries (SIBs) are gaining increasing attention as a sustainable and cost-effective alternative to lithium-ion systems for large-scale energy storage [1]. Among the various cathode candidates, mixed-olivine phosphates are particularly attractive due to their low cost, low toxicity and high stability. Indeed, partial substitution of Fe with Manganese has been demonstrated as an effective strategy to enhance the $NaFePO_4$ performances by increasing the operating voltage [2,3]. However, when the Mn content becomes too high, mixed-olivine shown hindered electrochemical reactivity that were linked to the limited Mn redox activity and structural instabilities [3,4]. Furthermore, another key drawback of Na-based mixed-olivine cathodes lies in their synthesis, as inactive maricite or natrophilite phases are thermodynamically favored, lacking Na^+ diffusion channels and, in the latter case, prone to Na/Mn antisite mixing that hinders Mn reactivity. In this work, we present an in-depth *operando* X-ray absorption spectroscopy (XAS) investigation of the electrochemically converted $NaFe_{0.6}Mn_{0.4}PO_4$ (NFMP) cathode, aiming to elucidate the structural dynamics responsible for its hindered reactivity. By probing both Fe and Mn K-edges under operando cycling conditions, we directly correlate the redox activity with local structural rearrangements. Complementary ex-situ characterizations—including X-ray diffraction, scanning electron microscopy, and X-ray photoelectron spectroscopy—provide further insight into the phase stability and oxidation states of NFMP cathodes obtained via electrochemical de-lithiation/sodiation conversion process. Our results reveal a full and reversible oxidation of the Fe2+ in the NFMP cathode to Fe^{3+} during charge, accompanied by progressive distortions of FeO_6 octahedra. In contrast, Mn shows only partial oxidation. Concurrently, Extended X-ray absorption fine structure analysis highlights significant bond-length elongation and octahedral distortion during de-sodiation, confirming the occurrence of Jahn–Teller distortions at Mn sites. This phenomenon, already known in lithium-based analogues [5], is particularly detrimental here to ion transport and structural integrity. Our findings provide a direct explanation for the limited Mn redox activity and performance degradation of high-Mn sodium olivines. Importantly, they demonstrate how Mn incorporation beyond a certain threshold destabilizes the de-sodiated phase, reducing both capacity and cycling efficiency. By disentangling the distinct roles of Fe and Mn during battery operation, this study pave the way to Mn content optimization strategies in mixed olivine cathodes, to achieve the best compromise in terms of delivered capacity and improved energy density. These insights may guide the rational development of next-generation, sustainable sodium-ion cathodes with improved performance for grid-scale applications.

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Hyperpolarized ^{13}C NMR by Dissolution-DNP Enables Snapshot Detection of Degradation Products in Lithium-ion Battery Electrolytes

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Dissolution Dynamic Nuclear Polarization (dDNP) is a powerful hyperpolarization technique enabling tremendous sensitivity gains in solution Nuclear Magnetic Resonance (NMR). Over the last decades, researchers' efforts have led to an extension of dDNP applications in numerous research fields. Lithium-ion batteries are among the most widespread rechargeable batteries, and a proper understanding of the physicochemical reactions at stake inside them is paramount to make them safer, more efficient, and sustainable. One of the key challenges lies in better understanding and limiting the degradation of the battery electrolyte, which can significantly impact the battery's performance. While NMR has been used in attempts to understand these mechanisms, notably by investigating the degradation products, the intrinsic lack of sensitivity of this technique, combined with the limited accessible volume of such compounds, makes its application often challenging. This work combines several state-of-the-art dDNP methodologies, including the use of recently introduced hyperpolarizing polymers (HYPOP) to efficiently hyperpolarize degraded battery electrolytes and acquire their enhanced natural abundance 13C solution NMR spectra. We show that we can successfully detect 13C signals on formulated, degraded battery electrolyte solutions, in one scan on a 600 MHz spectrometer, with sensitivity gains of up to 3 orders of magnitude. This work paves the way for studying lithium-ion battery electrolyte degradation under usage conditions (cycling, thermal aging, air exposure...) with a 13C detection limit below the micromolar range. This methodology has the potential to provide new insights into degradation mechanisms and the role and effectiveness of additives to mitigate electrolyte degradation.

Loop Currents in Ion-exchange Membranes During Osmotic-energy Harvesting

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The osmotic energy is the energy that can be collected from the environment thanks to osmotic effects between two water solutions of different salinities. These two water feeds can originate from a river and the ocean for instance, or from waste water. A promising method to extract energy is the reverse electrodialysis (RED). This method is based on the transport of ions from the high to the low concentration solutions through ion-selective membranes.

To establish the potential and viability of the RED process, it is necessary to finely understand the complex transport of ions at stake. Moreover, beyond RED, ion exchange membranes are the key element of many strategic processes: water treatment, water desalination and carbon capture for instance.

Such ion-exchange membranes are characterized by their selectivity to cations or anions. To measure this selectivity, a cell is used where membranes are placed between two electrolytes at different concentrations: the diffusion of the cation coupled to the blockage of the anions (or the opposite) by the membrane results in transmembrane voltage. This voltage is thus an indication of the membrane selectivity, which is a key indicator to estimate the power produced by a RED cell.

We show experimentally and theoretically that for membranes with a finite selectivity, the selectivity measurement suffers from strong concentration polarization, even though the circuit is open: in the high-concentration side, the concentration is locally lower close to the membrane, while in the low-concentration side, the concentration is locally higher close to the membrane. Concentration polarization is expected when current is crossing the membrane, but usually neglected when the circuit is open. However, varying the flow rates of the solutions enables us to finely characterize the ion transport through the membranes. Ultimately, this allows us to finely measure the membrane properties.

We also demonstrate that for low feeding flow-rate, concentration polarization is not homogeneous, and loops of currents appear through the membrane at the scale of the whole cell. This effect can also occur when the cell is connected to a resistor, in the configuration of energy harvesting. Accordingly, both the measured selectivity and the maximum harvested power per unit area can depend on the feeding flow rate, but also on the size of the membrane, and must be interpreted with extra care.

These experiments and modeling allow us to understand how to avoid these detrimental effects. We edit rules on how to scale membrane-processes to adjust the size based on the chosen flow rates, and how to rationalize the design of segmented electrodes.

Upscaling Salinity Gradient CRED Cell for Blue Energy Harvesting

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CRED (Capacitive reverse electrodialysis) cells are a hybrid technology that were originally introduced by Vermaas to extract electrical energy from salinity gradient[1]. They use selective IME (ion membranes exchange) just like RED (Reverse Electrodialysis) systems but borrow long-lasting, non-faradaic and inexpensive capacitive electrodes from Capmix (Capacitive Mixing) systems achieving power densities around 0.9 W/m² of IME, 5 times higher than Capmix systems[2] but 2 times lower than RED systems[3].

Since then, we reviewed this CRED cell in the MIE lab in order to simplify and optimize it. The current system designed by Youcef BRAHMI[4] only uses one IME and carbon felts as capacitive electrodes. The whole cell is equivalent to an ideal generator Ecell, an internal resistance Rcell and an internal capacitor C in series. Due to the internal capacitor, the cell must be used in an alternative regime by switching the concentrated and diluted solutions of sodium chloride over a switching-period T. The uptake of these 3D electrodes increases drastically the capacitance allowing us to achieve around 2.3 W/m² by stabilizing the potential; which can even reach 3.3 W/m² by choosing an appropriate half-period and reducing the internal resistance by employing thinner membranes and higher concentrations[5]. Yet, this still represents about 2/3 of the maximum theoretical power obtained for a square potential generator like in RED systems. The maximal power is obtained for loading resistances Rload slightly higher than Rcell.

Very recently, Nan Wu proposed a very surprising technique to recover around 90% of the theoretical power density[5]. She used an additional power supply in phase with the fluid switching. For a given switching-period, using this boosting technique adds 60% of the power compared to the non-boosting functioning after retrieving the power contribution of the external source. The boosting acts formally like an additional resistance that increases the characteristic time of the RC circuit stabilizing the whole cell potential at its highest value. It also allows to work with higher external resistances.

Currently, our techno-economic analysis estimates that our cell can produce energy at a cost of 200 €/MWh in the best conditions[6]. Given the decrease of the cost of the IME, these systems could become quite soon a profitable renewable energy. This motivates us in building a cell capable of producing 50 W. For that, we will upscale our CRED cell. The initial strategy involves the industrialization of the CRED cell by enlarging the surface area of individual membranes. Particular attention will be given to the impact of increasing the membrane length (along the flow direction) and width. The extension of the length highlights the problem of the filling time that has to be as short as possible in order to reach the maximal potential as quick as possible and approach a square signal while not having too much viscous loss. Regarding the width, as the system displays an invariance regarding the flow, the main issue is to reduce viscous loss.

Increasing the surface of one membrane helps reducing the total inner resistance of the cell resulting in a higher current. We will then associate in series CRED cells to obtain cumulative potential and power while preserving power density building bigger and compact cells.

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Advanced Molecular Characterization of Passivation Layers in Lithium-ion Batteries by 12 T FTICR & Imaging Mass Spectrometry

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Understanding the formation and composition of Solid Electrolytes Interphase (SEI) is a key point to improve performance of lithium ions batteries (LIBs). Its properties (*e.g.* insolubility, ionic conductivity, electron resistivity and mechanical stability), depend on its molecular composition and also on the spatial distribution of its compounds [1]. In this work we demonstrated the interest of Fourier transformed ion cyclotron resonance mass spectrometry (FTICR MS) and imaging mass spectrometry to access the SEI composition and to the monitoring of degradation products on the electrode surface [2-3]. Thanks to its high resolution and sub-ppm mass accuracy, the ions of interest were unambiguously characterized and then tracked over the electrode's entire surfaces enabling a deeper understanding of SEI composition.

The study was carried out on lithium-ion pouch cells filed with electrolyte composed of highly pure LiPF6 and battery grade solvent ethylene carbonate and ethyl methyl carbonate, formulated under controlled atmosphere. After 400 cycles, cells were washed and attached on a MALDI plate using aluminum tape and directly analyzed with laser desorption ionization. Analyses were performed on two Fourier transform ion cyclotron resonance mass spectrometers (FTICR MS): Bruker ScimaX 12 T and Bruker timsMRMS 18 T. The electrode images were recorded with the Bruker ScimaX 12 T.

The first objective was to characterize the SEI after cycling. The mass spectra obtained highlighted an advanced molecular complexity after aging. For a S/N > 4 more than 50000 signals were observed between *m/z* 74 and 500 with more than 50 signals in some 0.5 Da windows. Combination of isobaric (> 150 species per nominal mass) and elemental (C, H, F, O, P, Li, Cu, Al) complexity led to an extremely challenging data treatment. The PyC2MC open-source software was used to recover relevant molecular information. In addition, predictive models derived from Data-driven Massive Reaction Networks were tested and optimized thanks to their comparison with the experimental data. The overall objective was to gain a better understanding of the degradation pathways at interfaces and thus to clarify the nature of the SEI.

Once characterized, the second objective was to obtain information on the spatial repartition of the SEI, to track and observe directly on the electrode surface the aging mechanisms that occur throughout the cell cycling. Imaging analysis was carried out on electrodes surfaces [2-3].

This approach led to the localization and mapping of numerous SEI's compounds. We succeeded in observing different degradation products of various origin such as the lithium salt degradation, solvents degradation ($LiC_3H_6O_4^+$/$Li_2C_4H_5O_4^+$), some reaction between solvent and lithium salts degradation ($LiC_4H_9PO_5^+$) and aliphatic chains.

Analysis of LIB Electrolyte Composition and Aging Indicators Using GCMS-QP2050/Polyarc

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Lithium-ion batteries (LIBs) have emerged as a cornerstone of modern energy storage solutions, driven by their high energy density, compact design, and extended lifespan. The performance and reliability of LIBs are intricately tied to the composition and purity of their electrolytes, as well as the impurities formed during manufacturing, storage, or operation. In this application, two investigations of LIB electrolytes were conducted: 1. the analysis of carbonate esters and additives using GCMS-QP2050 and the evaluation of a phosphate compound as an indicator of electrolyte degradation, 2. the qualitative analysis of phosphate compounds as impurities using GCMS-QP2050 and their quantitative analysis Polyarc technology. Polyarc enhances GC/FID quantification by enabling the flame ionization detector (FID) to provide a uniform response to nearly all organic molecules, thereby improving the accuracy and reliability of quantitative analysis.

The first investigation focused on the qualitative and quantitative analysis of carbonate esters and additives. These compounds significantly influence critical battery attributes, including capacity retention and cycle life. Several common carbonate esters and additives that typically make up electrolytes were selected as target compounds. Chromatograms acquired in scan mode exhibited good separations, enabling reliable identification of all compounds using library searches with high similarity indices. Calibration curves were developed using six concentration points ranging from 10 mg/L to 500 mg/L, demonstrating the high linearity and repeatability. Further analysis was conducted on four commercial LIB electrolytes, two containing Lithium bis(tri-fluoro-methane-sulfonyl)imide (LiFSI) and two containing Lithium hexa-fluorophosphate ($LiPF_6$). Quantitative results revealed that the three carbonate esters were present in a 1:1:1 ratio. For LiPF6-based electrolytes, additional peak corresponding to Dimethyl fluorophosphate (DMFP) was observed by a lower ionization energy (LEI) method with an ionization voltage of 14 eV. This approach enhanced the signal intensity of the molecular ion by approximately four times compared to the standard 70 eV method, providing strong evidence of DMFP's presence.

The second investigation examined phosphate compounds, which are reliable indicators of electrolyte degradation and aging. These compounds form electrochemically during charge–discharge cycles and harm battery performance by raising internal resistance and lowering capacity. To study their behavior, electrolyte samples containing $LiPF_6$ were over-charged at 40 °C using three voltages: 4.0 V, 5.0 V, and 5.6 V.

GC–MS analysis indicated that electrochemical over-charge conditions markedly accelerate the formation of phosphate compounds in the $LiPF_6$-based electrolyte. At 40 °C, the highest applied potential (5.6 V) generated a pronounced phosphate-related chromatographic signal, whereas only trace-level responses were observed at the minimal operating potential of 4.0 V.

Quantitative analysis using Polyarc provided precise measurements of phosphate concentrations, leveraging its ability to normalize sensitivity across a wide range of compounds. The principle behind Polyarc lies in its ability to convert all organic compounds into methane molecules before detection by the FID, thereby standardizing the sensitivity response. This advanced quantitative carbon detector (QCD) enhanced the accuracy of analyzing compounds without available standards and reduced calibration time, making it highly effective for impurity quantification. The findings underscore the critical role of phosphate compounds in monitoring LIB electrolyte aging and the impact of operating conditions on impurity generation

In conclusion, the combined GCMS-QP2050/Polyarc platform advances electrolyte analysis. First, the QP-2050 enabled a reliable, high-resolution method for profiling the principal carbonate esters and additives that dictate the bulk properties of LIB electrolytes. Second, by interfacing the same instrument with a Polyarc, we were able to establish to quantify the phosphate esters without standard samples - critical degradation markers - thereby verifying their pronounced accumulation during cell ageing and quantifying them even in the absence of authentic standards. Together, these methods enhance electrolyte analysis, improve monitoring, and enable safer, more reliable energy-storage systems.

Understanding Interfacial Mechanisms of Li-Ion Batteries Using High-Resolution Mass Spectrometry: Effect of Lithium Difluoro Phosphate as an Electrolyte Additive

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Understanding the formation and composition of the Solid Electrolyte Interphase (SEI) is a key point to improve the performance of lithium-ion batteries (LIBs). Its properties, e.g., insolubility, ionic conductivity, electron resistivity, and mechanical stability, are mainly dictated by the composition of the electrolyte [1]. Such composition is therefore carefully chosen according to the chemical and electrochemical properties of the salts, solvents, and more specifically, additives [2-3]. In this work, the role of lithium difluoro phosphate ($LiPO_2F_2$) as an electrolyte additive is studied thanks to the Fourier Transform Ion Cyclotron Resonance mass spectrometry (FTICR), allowing the access of relevant information about SEI molecular composition and electrolyte degradation pathways. The FTICR high resolution and sub-ppm mass accuracy enable unambiguous characterization of the ions of interest. These ions can subsequently be tracked over the electrode's entire surface thanks to imaging mass spectrometry [4-5], facilitating a deeper understanding of the interfacial mechanisms of LIBs. [5]

Chemical and thermal aging was performed on two electrolytes composed of highly pure lithium salts ($LiPF_6$ and $LiPO_2F_2$) and battery-grade solvents, ethylene carbonate (EC) and ethyl methyl carbonate (EMC). The molecular composition of the SEI, resulting from an electrochemical aging of pouch cells filled with the two electrolytes (1M $LiPF_6$ in EC/EMC and 1M $LiPF_6$ – 1% $LiPO_2F_2$ in EC/EMC) was also investigated. Samples were deposited on a MALDI plate and directly ionized with laser desorption. Analyses were subsequently performed on two FTICR mass spectrometers: Bruker ScimaX 12 T and Bruker timsMRMS 18 T.

Electrochemical results confirmed that the addition of LiPO2F2 improves the performance of LIBs with a gain of about 150 cycles concerning the lifespan of the cell. To further understand this improvement, mass analysis of the electrolyte highlighted a protective effect of $LiPO_2F_2$ on the major salt LiPF6, limiting its degradation and thus preserving the integrity of the electrolyte upon cycling. In addition, imaging analyses of the SEI were carried out to map the various previously identified degradation products of LiPF6 salt ($Li_2PO_2F_2^+$/Li_4PO_4+).

On one hand, without the additive, the SEI no longer contains LiPF6 but predominantly its final degraded form, $Li_4PO_4^+$. On the other hand, the addition of lithium difluoro phosphate in the electrolyte logically leads to the observation of the initial degraded form of $LiPF_6$, $Li_2PO_2F_2^+$, along with the persistent presence of the PF6- ion on the electrode surface. The protective effect of $LiPO_2F_2$ on the major salt is thus confirmed, even during electrochemical activity.

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