_____ September 14-18, 2025 _____ Berlin, Germany

INCOME 2025

11th International Conference on Mechanochemistry and Mechanical Alloying

BAM

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Dear distinguished scholars and colleagues,

On behalf of *International Mechanochemical Association (IMA)* – one of the organisations associated with IUPAC – it is with great pleasure and anticipation that I extend to you a warm welcome to the 11th International Conference on Mechanochemistry and Mechanical Alloying (INCOME 2025).

Series of INCOME conferences is a flagship initiative of IMA. The first edition of INCOME took place in Košice (Slovakia) in 1993. Since then, there has been 10 editions of the conference, that grew in numbers and impact over the years. The 2025 edition of INCOME aspires to foster a robust exchange of scientific knowledge, facilitate interdisciplinary dialogue, and catalyse innovations that will shape the future trajectory of mechanochemistry. On behalf of IMA, I would like to sincerely thank the conference chairperson *Professor Franziska Emmerling* and the local organizers, namely, *Humboldt-Universität zu Berlin*, *Federal Institute for Materials Research and Testing – BAM*, and S*ALSA – School of Analytical Sciences Adlershof*, for organizing and hosting the 11th INCOME as well as for a unique opportunity to meet at a historic site where pioneers of mechanochemistry such as *Gerhard Heinicke* and *Peter-Adolf Thiessen* worked.

As mechanochemical platforms continue to evolve, their practical applicability and broader adoption are expected to increase. This, in turn, will support the development of more standardised protocols, parameter databases, and predictive tools (such as machine learning and artificial intelligence), improving reproducibility and accelerating discoveries across the diverse landscape of chemistry. Realising this potential will require close collaboration among researchers, technologists, and engineers. In that respect, the *International Mechanochemical Association* has emerged as a crucial player that can catalyse the implementation of the international cooperation needed to enhance and harmonise research in mechanochemistry worldwide. The newly formed *EuChemS "Working Party on Mechanochemistry"*, which builds upon the legacy of the *European COST Action CA18112 "Mechanochemistry for Sustainable Industry"*, represents an important step forward for the mechanochemistry community in Europe. These initiatives are particularly important at a time when mechanochemical processes are being investigated at both laboratory and large scales, targeting potential applications across various industrial market sectors.

Currently, several global initiatives are underway aimed at further promoting mechanochemistry to realise its full potential for science and technology. Examples are the *project IMPACTIVE (Innovative Mechanochemical Processes to*

synthesise green ACTIVE pharmaceutical ingredients), funded by the European Union, the *NSF Center for the Mechanical Control of Chemistry (CMCC)*, funded by the National Science Foundation, the *IUPAC Task Group on Terminology and Symbolism for Mechanochemistry*, and the *Round Robin project on mechanochemical transformations*. We hope these efforts will inspire the further creation of dedicated research centres and funded initiatives worldwide, especially where mechanochemical research is just emerging or underrepresented.

Furthermore, a very important milestone for mechanochemical community was the establishment of *RSC Mechanochemistry* in 2023, the first *fully peer-reviewed* international journal dedicated to this transformative field, which offers an inclusive and dedicated home for the ideas, scientific language and approaches that cut across the many disciplines mechanochemistry touches.

A high-calibre scientific programme of INCOME 2025 features a diverse and forward-looking agenda, encompassing oral and poster presentations by renowned experts and emerging talents from around the world. The conference will cover the full spectrum of mechanochemistry – from fundamental mechanisms to novel synthetic methods, sustainable chemistry, and industrial applications. The conference will further provide an enriching platform for scholarly exchange, professional development, and the formation of enduring academic and industrial collaborations.

All the conferences in INCOME series have been characterized not only by a high scientific level but also by a friendly atmosphere, made possible by the dedication of the local committee to ensuring your satisfaction. I am confident that your active participation will greatly enhance the intellectual richness and success of this event. We look forward to your contributions and to engaging with you in what promises to be an inspiring and impactful scientific occasion.

Yours sincerely,

Vladimír Šepelák
President of the International Mechanochemical Association
(http://imamechanochemical.com/)

Dear Mechanochemists,

It is a great pleasure to welcome you to INCOME2025. This year we are meeting at Humboldt University's Adlershof campus in Berlin, an epicentre of scientific innovation that is steeped in the history of mechanochemistry. Our venue carries the legacy of pioneering mechanochemistry, such as Peter Adolf Thiessen and Karsten Peter Thiessen, who helped lay the foundations for solid-state chemistry and mechanochemistry. For generations, Adlershof has been a place where scientists have pushed boundaries, developed theories and instrumentation, and engaged in spirited scientific debate. It is against this history that the breadth of research here at INCOME2025 reminds me how far mechanochemistry has come. Once dismissed as a niche curiosity, our community now plays a central role in reshaping chemistry and materials science, breaking barriers between physics, chemistry, engineering, and the quest for a more sustainable world.

This year, INCOME brings together a truly global community of seasoned veterans and adventurous newcomers. We will hear about cutting-edge mechanistic insights, ambitious projects to scale-up mechanochemical reactions, and how mechanochemistry is being used for applications that were imaginable only a few years ago. But the networks we make and the conversations that spark between sessions, at the coffee tables, and among the posters are just as important. Mechanochemistry thrives on diversity, not just of methods and molecules, but also of the backgrounds, cultures, and perspectives of those who shape it.

As we gather in these halls, let us draw inspiration from Adlershof's tradition of bold scientific inquiry and from the pioneers who believed that mixing, friction, and bold ideas could lead to significant discoveries. Let us celebrate the new ideas, that are flourishing in our community and be inspired to take steps in new directions with the knowledge that the best discoveries are still to come.

Whether you are passionate about theory, laboratory work, applications, or simply the joy of learning, we invite you to celebrate, connect and enjoy these days at INCOME2025.

Welcome everyone!

Franziska Emmerling, Team INCOME2025, Humboldt University, Federal Institute for Materials Research and Testing

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GENERAL INFORMATION

WEBPAGE

https://bit.ly/INCOME2025

WI-FI

Eduroam is available throughout all Humboldt University buildings, providing secure and convenient wireless connectivity for participants affiliated with institutions that support eduroam.

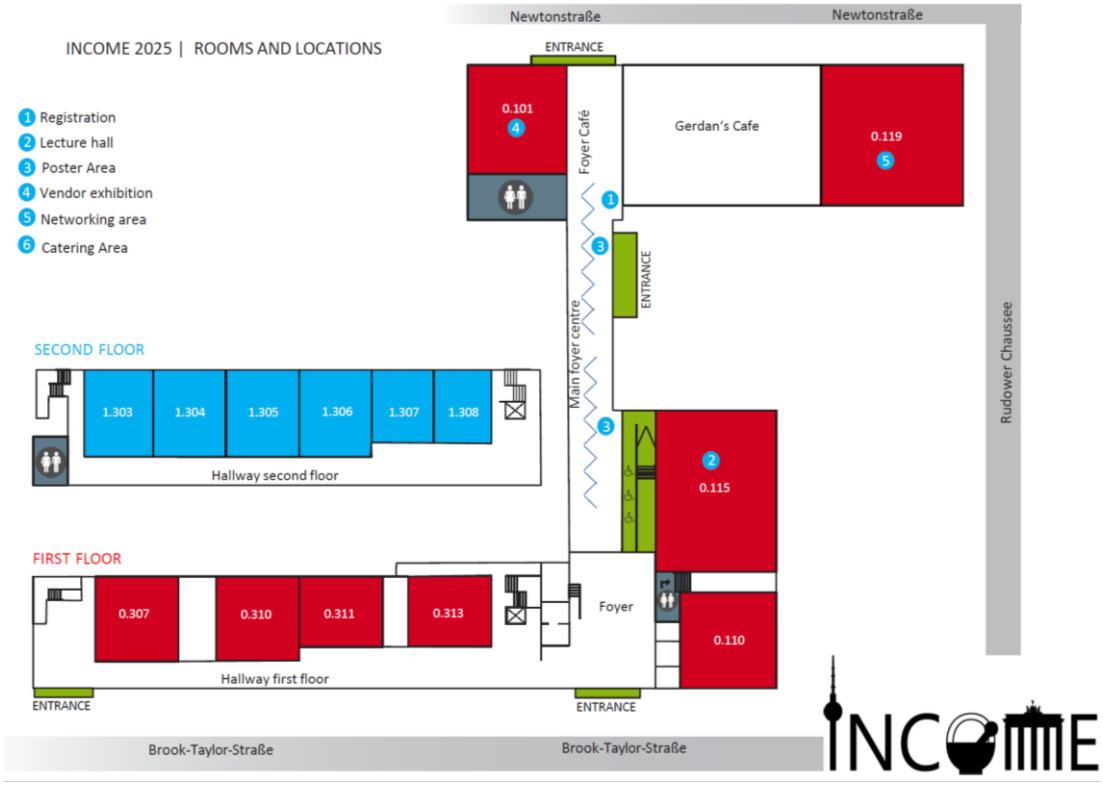
In addition, complimentary Wi-Fi will be provided for all delegates. Access details will be available upon registration.

http://bit.ly/3Ue7XU0

LOCATION

Humboldt-Universität zu Berlin
Erwin-Schrödinger-Zentrum
Rudower Chaussee 26
12489 Berlin

FLOORPLAN



PRESENTATIONS

Presentations will take place in Lecture Hall 0.115 (the conference hall on the ground floor). The presentation computers are equipped with Microsoft PowerPoint. Presenters are kindly requested to upload their files either in person at the registration desk or online (see details below) in the morning or at least two hours prior to their scheduled lecture.

Files should be named in the format Lastname.pptx. On-site technical support will be available if needed.

Invited presentations are allotted a maximum of 25 minutes for the talk, followed by five minutes for discussion. Oral presentations are allotted a maximum of 15 minutes, with an additional five minutes for discussion. All speakers are asked to strictly observe the allotted time to maintain the conference schedule.

Upload presentations:
https://tinyurl.com/25znhhk3
Password: INCOME2025

POSTERS

The poster sessions will take place in the designated area on the ground floor. Delegates are kindly requested to display their posters in a timely manner. Each poster has been assigned a number, as indicated in the conference program, and should be mounted on the corresponding board. Posters must be prepared in portrait orientation and must not exceed dimensions of 70 x 115 cm. All necessary mounting materials will be provided on site. To facilitate interaction, presenters are expected to stand by their posters during the assigned poster session.

Posters with odd numbers will be presented on Monday.

Posters with even numbers will be presented on Tuesday.

LUNCH

Lunch will be provided onsite.

DINNER

The conference dinner will take place on Wednesday, 17 September 2025 at the unique DEEP event location on the Bötzow Areal (Prenzlauer Allee 242-247, 10405 Berlin). This venue combines a modern design with a historic setting, and its centrepiece is a spectacular underground hall with seven-metre-high columns, designed by Chipperfield Architects.

PICTURES

Pictures will be taken throughout the conference. (Password INCOME2025)

Pictures can be downloaded here:
https://tinyurl.com/2yfha7sp
Password: INCOME2025

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Sunday, September 14, 2025

16:00	Registration / Prebadging
17:00	**Lucia Maini** Chosen by Mechanochemistry: How Grinding Has Been Shaping My Research Path
18:00	Welcome Beer & Pretzels

Monday, September 15, 2025

	Session Chair: Franziska Emmerling
8:30	I-00 – Opening remarks
9:00	I-01 – **Peter Baláž** Advances in Mineral Mechanochemistry: From Metals Extraction to Energy Materials and Anticancer Drugs
9:30	I-02 – **Ina Vollmer** Modelling and Mechanistic Study of Polyethylene Chain Cleavage During Ball Milling
10:00	I-03 – **Wilfred T. Tysoe** Understanding the Effect of Mechanical Stress on Chemical Reaction Rates: Tuning Reactivity and Selectivity
10:30	Coffee Break
	Session Chair: Matej Baláž
11:00	I-04 – **Guido Kickelbick** Mechanochemical Pathways for Synthesis and Activation of Metal Oxides
11:30	O-01 – **Ankita Das** Oriented External Electric Field Controls the Rupture Forces in Mechanophores
11:50	O-02 – **Erli Lu** Mechanochemistry-Enabled Electride and Alkalide Chemistry
12:10	O-03 – **Chenhao Wu** Mechanochemical Stabilisation of Arsenic and Uranium in Mine Tailings: An Approach for Sustainable Remediation
12:30	O-04 – **Philippe M. C. Roth** Scalability Parameters in API Synthesis Using Continuous Mechanochemistry

12:50	Lunch Break
	Session Chair: Ina Vollmer
13:50	I-05 – **Krunoslav Užarević** Mechanochemistry in Chemical Evolution
14:20	O-05 – **Dzmitry Kananovich** Breaking Barriers in Organometallic Synthesis with a Ball Mill
14:40	O-06 – **Johanna Templ** A Double Bond Agent on a Mechanochemical Mission – Ball Milling Strategies to Introduce C(sp^2)=C(sp^2) Bonds
15:00	O-07 – **Zara Cherkezova-Zheleva** Mechanochemical Approach Toward Efficient, Green and Sustainable Recycling and Reuse of Waste NdFeB Magnets
15:20	O-08 – **Hui Luo** Process Integration and Life-Cycle Assessment of Moist-Solid Hydrolysis of Polylactic Acid with Lactic Acid Recovery via Electrodialysis
15:40	Coffee Break
	Session Chair: Riina Aav
16:10	O-09 – **Juan José Sáenz De La Torre** Impactive Educational Platform: A Digital Meeting Point Around Mechanochemistry
16:30	O-10 – **Ede Bodoki** Between What We Know and What We Wonder: Revealing Key Experimental Variables Shaping the Mechanochemical Synthesis of Molecularly Imprinted Polymers
16:50	F-01 – Poster Flash Talks
17:30	Poster Session

Tuesday, September 16, 2025

	Session Chair: Frédéric Lamaty
8:30	I-06 – **Deborah Crawford** Pushing the Limits: Evolving Twin-Screw Extrusion for Next-Gen Mechanochemical Processing
9:00	I-07 – **José G. Hernández** Investigating the Detection and Role of Cocrystals in Metal-Mediated C–H Activation through Mechanochemistry

9:30	I-08– **Clara S. B. Gomes** Mechanochemical Engineering of Novel Praziquantel Cocrystals for Enhanced Solubility
10:00	I-09 – **Daniel P. Tabor** Identifying Promising Mechanochemical Reactions through High-Throughput Virtual Screening, Machine Learning, and Simplified Physical Models
10:30	Coffee Break 
	Session Chair: Adam A. L. Michalchuk
11:00	I-10 – **Dominik Lungerich** Kinematic Modelling of Mechanochemical Reactions
11:30	O-11 – **Adam B. Braunschweig** Kinetics of Mechanochemical Reactions
11:50	O-12 – **Maike Mayer** Gas-Mechanochemistry for Catalytic Reactions of Gaseous and Solid Substrates
12:10	O-13 – **Jae Seong Lee** Defect-Mediated Mechanochemical Ammonia Production Using Silicon Nitride as Physical Promoter
12:30	O-14 – **Tanja Butt** Retsch Supporting Mechanochemistry: Equipment, Applications, and Control Strategies
12:50	Lunch Break 
	Session Chair: Krunoslav Užarević
13:50	I-11 – **Riina Aav** Mechanochemical C-N Bond Formation: From Imatinib to Macrocycles
14:20	O-15 – **Dipak J. Fartade** Mechanochemistry-Driven Borrowing Hydrogen Processes for Ru-Catalysed N-Alkylation: A Green and Sustainable Pathway
14:40	O-16 – **Daniel M. Baier** Mechanochemical Deracemization: A Fast and Solvent-Minimized Approach to Enantiopurity
15:00	O-17 – **Wen-Yang Gao** Overcoming Solution Chemistry Barriers to Metal-Organic Frameworks via Mechanochemistry
15:20	O-18 – **Andrea Sala** Mechanochemical Synthesis of Novel Protocatechuate-Based Coordination Polymers and Their Characterization Through 3D Electron Diffraction

15:40	Coffee Break
	Session Chair: Deborah Crawford
16:10	O-19 – **Nikita Gugin** Reactive Extrusion of ZIF-8-Based Biocomposites: Scale-Up Enabled by in situ Monitoring Advances
16:30	O-20 – **Santiago Garrido** Effect of Tangential-to-Normal Stress Distribution on the Mechanochemical Regeneration of NaBH4
16:50	F-02 – Poster Flash Talks
17:30	Poster Session
19:00	IMA Meeting

Wednesday, September 17, 2025

	Session Chair: Clara S. B. Gomes
8:30	I-12 – **Lars Borchardt** How Much "Mechano" is in Mechanochemistry?
9:00	I-13 – **Adam A. L. Michalchuk** Phonon Dynamics and Mechanochemical Reactivity
9:30	I-14 – **Orla S. A. Williams** Shaping Chemistry by Force: Using Advances in Milling Knowledge from the Power Generation Sector to Progress Mechanochemical Extraction
10:00	I-15 – **Martin Fabián** Mechanochemistry-Driven Synthesis of Spinel and High-Entropy Oxide Materials as Candidates for Lithium-Ion Battery Components
10:30	Coffee Break
	Session Chair: Tomislav Friščić
11:00	I-16 – **Frédéric Lamaty** Mechanochemistry Triggers a Change of Paradigm in Peptide Synthesis
11:30	O-21 – **Mateusz Mojsak** Time-Resolved Simulation of Shock-Driven Reactions in Solids: A Step Towards Predictive Mechanochemistry
11:50	O-22 – **Adrien Gallego** A Journey through Optimization: Applying OFAT, DoE and Machine Learning to Sustainable Amidation

12:10	O-23 – **Michael Felderhoff** Mechanochemical Up-Scaling of Co-Crystal Syntheses Successful Procedures and Unexpected Problems
12:30	O-24 – **Ilda Tole** Mechanochemical Activation: Revolutionizing the Production of Green Cements
12:50	Lunch Break
	Session Chair: Paulo F. M. de Oliveira
13:50	I-17 – **James Mack** Beyond Reagents: Exploring the Impact of Reaction Vials and Milling Media in Mechanochemical Synthesis
14:20	O-25 – **Seunghyeon Kim** Mechanically Driven N_2O Decomposition over NiO at Near-Room Temperature
14:40	O-26 – **Jikai Ye** Mechanochemical Reduction of Nickel Oxide with Continuous H_2 Flow
15:00	O-27 – **Marcela Achimovičová** Mechanochemical/Thermal Synthesis of Ternary Chromium Selenospinels
15:20	Coffee Break
	Session Chair: Bilge Baytekin
15:50	I-18 – **Ana Belenguer** Round Robin Project: Reproducibility of Ball Mill Reactions at Steady State
16:20	I-19 – **Tom Leyssens** The importance of the Nature of the Solid State for Mechanochemical Reactivity
19:00	Dinner

Thursday, September 18, 2025

	Session Chair: José G. Hernández
8:30	I-20 – **Hajime Ito** Mechanochemical Transformation of Commodity Polymers into Reactive Platforms for Organic Synthesis Utilizing Mechanical Energy
9:00	I-21 – **James Batteas** From the Molecular to the Macroscale: A Look Inside the Mechanics of Mechanochemical Reactions

9:30	I-22 – **Matej Baláž** Transformation of Mechanochemistry from Materials Science into Chemical Science and an Overview on Mechanically Induced Self-Propagating Reactions
10:00	I-23 – **Bilge Baytekin** Static Electricity as a Mechanochemical Event
10:30	Coffee Break
	Session Chair: Tomislav Stolar
11:00	I-24 – **Paulo F. M. de Oliveira** Mechanochemistry Beyond Material-Making: From Nanoparticles to Defective Solids and (Meta)Stable Phases
11:30	O-28– **Leeroy Hendrickx** Formation of Racemic Phases of Amino Acids by Liquid-Assisted Resonant-Acoustic Mixing Monitored by Solid-State NMR Spectroscopy
11:50	O-29 – **Paolo P. Mazzeo** Prediction of Low Melting Eutectic Formation as an Intermediate for Mechanochemical Synthesis
12:10	I-25 – **Tomislav Friščić** Looking in the Crystal Ball: The Future of Mechanochemistry
12:40	Concluding Remarks

Invited Talks

Sunday, September 14

17:00

Lucia Maini

Department of Chemistry "Giacomo Ciamician", University of Bologna, Bologna, Italy

Monday, September 15

9:00

I–01 Peter Baláž

Department of Mechanochemistry, Institute of Geotechnics, Slovak Academy of Sciences, Košice, Slovakia

9:30

I–02 Ina Vollmer

Department of Chemistry, Institute for Sustainable and Circular Chemistry, Inorganic Chemistry and Catalysis, Utrecht University, Utrecht, Netherlands

10:00

I–03 Wilfred T. Tysoe

Department of Chemistry and Biochemistry, University of Wisconsin-Milwaukee, Milwaukee, USA

11:00

I–04 Guido Kickelbick

Inorganic Solid-State Chemistry, Saarland University, Saarbrücken, Germany;
Saarene – Saarland Center for Energy Materials and Sustainability, Saarbrücken, Germany

13:50

I–05 Krunoslav Užarević

Division of Physical Chemistry, Ruđer Bošković Institute, Zagreb, Croatia

Tuesday, September 16

8:30

I–06 Deborah E. Crawford

School of Chemistry, University of Birmingham, Birmingham, UK

9:00

I–07 José G. Hernández

Instituto de Química, Facultad de Ciencias Exactas y Naturales, Grupo Ciencia de los Materiales (CIENMATE), Universidad de Antioquia, Medellín, Colombia

9:30

I–08 Clara S. B. Gomes

LAQV-REQUIMTE, Department of Chemistry, NOVA FCT, NOVA University of Lisbon, Caparica, Portugal

10:00

I–09 Daniel P. Tabor

Department of Chemistry, Texas A&M University, College Station, TX, USA

11:00

I–10 Dominik Lungerich

Center for Nanomedicine, Institute for Basic Science (IBS), Seoul, South Korea; Department of Nano Biomedical Engineering (NanoBME), Advanced Science Institute, Yonsei University, Seoul, South Korea

13:50

I–11 Riina Aav

Department of Chemistry and Biotechnology, Tallinn University of Technology, Tallinn, Estonia

Wednesday, September 17

8:30

I–12 Lars Borchardt

Ruhr-University Bochum, Bochum, Germany

9:00

I–13 Adam Michalchuk

School of Chemistry, University of Birmingham, Birmingham, UK

9:30

I–14 Orla S. A. Williams

Department of Mechanical, Materials and Manufacturing Engineering, Faculty of Engineering, University of Nottingham, Nottingham, UK

10:00

I–15 Martin Fabián

Institute of Geotechnics, Slovak Academy of Sciences, v.v.i., Košice, Slovak Republic

11:00

I–16 Frédéric Lamaty

Green Chemistry and Enabling Technologies, Institut des Biomolécules Max Mousseron (IBMM), Université de Montpellier, CNRS, ENSCM, Montpellier, France

13:50

I–17 James Mack

University of Cincinnati, OH, USA

15:50

I–18 Ana M. Belenguer

Yusuf Hamied Department of Chemistry, University of Cambridge, Cambridge, UK

16:20

I–19 Tom Leyssens

UCLouvain, Institute of Condensed Matter and Nanosciences, Louvain-la-Neuve, Belgium

Thursday, September 18

8:30

I–20 Hajime Ito

Institute for Chemical Reaction Design and Discovery (WPI-ICReDD), Hokkaido University, Japan; Division of Applied Chemistry, Faculty of Engineering, Hokkaido University, Sapporo, Hokkaido, Japan

9:00

I–21 James Batteas

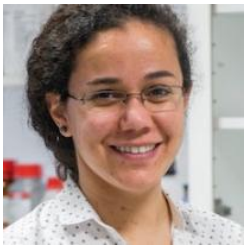
Department of Chemistry, Texas A&M University, College Station, TX USA

9:30

I–22 Matej Baláž

Department of Mechanochemistry, Institute of Geotechnics, Slovak Academy of Sciences, Košice, Slovak Republic

10:00



I–23 Bilge Baytekin

Chemistry Department, Bilkent University, Ankara, Turkey; UNAM, Bilkent University, Ankara, Turkey

11:00

I–24 Paulo F. M. de Oliveira

Institute of Chemistry, University of São Paulo, São Paulo, Brazil

12:10

I–25 Tomislav Friščić

School of Chemistry, University of Birmingham, UK

Oral Presentations

O–01	Oriented external electric field controls the rupture forces in mechanophores *Ankita Das and Ayan Datta*
O–02	Mechanochemistry-enabled electride and alkalide chemistry *Erli Lu and Nathan Davison*
O–03	Mechanochemical stabilisation of arsenic and uranium in mine tailings: an approach for sustainable remediation *Chenhao Wu and Caroline Kirk*
O–04	Scalability parameters in API synthesis using continuous mechanochemistry *Philippe M. C. Roth and Kenneth Banderob*
O–05	Breaking barriers in organometallic synthesis with a ball mill *Dzmitry Kananovich, Jagadeesh Varma Nallaparaju, Suman Sahoo, and Riina Aav*
O–06	A double bond agent on a mechanochemical mission - ball milling strategies to introduce $C(sp^2)=C(sp^2)$ bonds *Johanna Templ and Michael Schnürch*
O–07	Mechanochemical approach toward efficient, green and sustainable recycling and reuse of waste NdFeB magnets *Zara Cherkezova-Zheleva, Daniela Paneva, Radu Robert Piticescu, Setareh Gorji Ghalamestani and Olivier Jay*
O–08	Process integration and life-cycle assessment of moist-solid hydrolysis of polylactic acid with lactic acid recovery via electrodialysis *Hui Luo, Dingchang Yang, Jhuma Sadhukhan, Verdeluz Costica, Robert Dorey, Qilei Song, and Maria-Magdalena Titirici*
O–09	IMPACTIVE Educational Platform: a digital meeting point around mechanochemistry *Leyre Flamarique, Fernando Gomollón-Bel, and Juan José Sáenz De La Torre*
O–10	Between what we know and what we wonder: revealing key experimental variables shaping the mechanochemical synthesis of molecularly imprinted polymers *Ede Bodoki, Andreea Elena Bodoki, and Bogdan-Cezar Lacob*
O–11	Kinetics of mechanochemical reactions *Adam B. Braunschweig, Senad Skejovic, Ryan Kwokb, and Mateusz Marianski*

O–12	Gas-mechanochemistry for catalytic reactions of gaseous and solid substrates *Maike Mayer, Maximilian Wohlgemuth, and Lars Borchardt*
O–13	Defect-mediated mechanochemical ammonia production using silicon nitride as physical promoter *Jae Seong Lee, and Jong-Beom Baek*
O–14	Retsch supporting mechanochemistry: equipment, applications, and control strategies *Tanja Butt*
O–15	Mechanochemistry-Driven Borrowing Hydrogen Processes for Ru-Catalysed N-Alkylation: A Green and Sustainable Pathway *Dipak J. Fartade*
O–16	Mechanochemical deracemization: a fast and solvent-minimized approach to enantiopurity *Job Gieling, Guillaume Wéry, Chrystal Lopes, Joséphine De Meester, Clément Brandel, Yohann Cartigny, Tom Leyssens, and Daniel M. Baier*
O–17	Overcoming solution chemistry barriers to metal-organic frameworks via mechanochemistry *Wen-Yang Gao, Zhuorigebatu Tegudeer, and Terry Plant-Collins*
O–18	Mechanochemical Synthesis of novel protocatechuate- based coordination polymers and their characterization through 3d electron diffraction *Andrea Sala, Danilo Marchetti, Moussa D. F. Diouf, A. Ken Inge and Mauro Gemmi*
O–19	Reactive extrusion of ZIF-8-based biocomposites: scale-up enabled by *in situ* monitoring advances *Nikita Gugin, Alexander Schwab, Francesco Carraro, Isabella Tavernaro, Jana Falkenhagen, José A. Villajos, Paolo Falcaro, and Franziska Emmerling*
O–20	Effect of tangential-to-normal stress distribution on the mechanochemical regeneration of NaBH4 *Santiago Garrido, Dingena Schott, and Johan Padding*
O–21	Time-resolved simulation of shock-driven reactions in solids: a step towards predictive mechanochemistry *Mateusz Mojsak and Adam A. L. Michalchuk*

O–22	A journey through optimization: applying OFAT, DoE and machine learning to sustainable amidation *Adrien Gallego, Matthieu Lavayssiere, Xavier Bantreil, Nicolas Petry, Julien Pinaud, Olivia Giani, and Frédéric Lamaty*
O–23	Mechanochemical up-scaling of co-crystal syntheses successful procedures and unexpected problems *Michael Felderhoff, Jan-Hendrik Schöbel, and Dhyanesh Gopinath*
O–24	Mechanochemical activation: revolutionizing the production of green cements *Ilda Tole and Susan A. Bernal*
O–25	Mechanically driven N2O decomposition over NiO at near-room temperature *Seunghyeon Kim and Jong-Beom Baek*
O–26	Mechanochemical reduction of nickel oxide with continuous H2 flow *Jikai Ye, Gang Liu, Christian H. Liebscher, and Michael Felderhoff*
O–27	Mechanochemical/thermal synthesis of ternary chromium selenospinels *Marcela Achimovičová, Vladimír Kucek, Matej Baláž, Róbert Tarasenko, Martin Orendáč*
O–28	Formation of racemic phases of amino acids by liquid-assisted resonant-acoustic mixing monitored by solid-state NMR spectroscopy *Leeroy Hendrickx, Calogero Quaranta, Emilian Fuchs, Maksim Plekhanov, Mirijam Zobel, Carsten Bolm, and Thomas Wiegand*
O–29	Prediction of low melting eutectic formation as intermediate for mechanochemical synthesis *Paolo P. Mazzeo, Michele Prencipe, Andrea Daolio, Remie M. Sundermann, Paolo Pelagatti, and Alessia Bacchi*

Poster Session 1
Monday, 15th September 2025, 17:30-19:00

Poster presentation (P) Flash talk (F-01, Monday, 15th September, 16:50)	
P–01	Mechanochemical deracemization: a solvent-minimized approach to enantiopure isoindolinones *Guillaume Wery, Daniel Baier, Tom Leyssens*
P–03	Mechanochemical deracemization of a naproxen ester *Job Gieling, Tom Leyssens, and Daniel M. Baier*
P–05 F1	Harnessing extrusion forces for the thermo-mechanochemical synthesis of amides through machine learning *Matthieu Lavayssiere, Xavier Bantreil, and Frédéric Lamaty*
P–07	CZA catalysts sythesis by one-pot mechanochemical route applied to carbon dioxide to methanol conversion *Mattheus H. M. Mendes and Liane M. Rossi*
P–09	The mechanochemical modification of biomaterials *Chengji Yang, Hossein Baniasadi, Tatsiana Nikonovich, Janak Sapkota, Monika Österberg, and Sandra Kaabel*
P–11	Understanding the propensity of calcium carbide towards mechanochemical bis-alkynylation over mono-alkynylation: isatin as a model electrophile *Alejandro Cortés-Lobo, Karen J. Ardila-Fierro, Cacier Z. Hadad, and José G. Hernández*
P–13	Mechanochemically synthesized Ni-based metal-organic framework excels in electrocatalytic alcohol oxidation reaction *Arkendu Roy, Biswajit Bhattacharya, and Franziska Emmerling*
P–15	Maleimide–anthracene mechanophores for reusable enzyme supports *Emmanouil Broumidis and Francesca Paradisi*
P–17	Mechanochemically synthesized COF for PFAS adsorption *Maroof Arshadul Hoque, Thomas Sommerfeld, Jan Lisec, Prasenjit Das, Carsten Prinz, Christian Heinekamp, Tomislav Stolar, Martin Etter, Biswajit Bhattacharya, and Franziska Emmerling*
P–19	Mechanically induced sequential one-pot Wittig olefination – Diels-Alder reaction: a solvent-free approach to complex bicyclic scaffolds *Nika Podlesnik, Nina Biedermann, and Michael Schnürch*
P–21	Mechanisitc insights into co-crystal formation via mechanochemistry: in-situ monitoring and kinetic modelling *Lucia Casali, Maria Carta, Francesco Delogu, and Franziska Emmerling*

P–23	Ball milling for better binding: mechanochemical molecular imprinting of febuxostat *Bogdan-Cezar Iacob, Anna-Mariia Hryb, Andreea Elena Bodoki, and Ede Bodoki*
P–25	Adding value to the forest through mechanochemistry *Elijah Mark Garcia, Paula Nousiainen, Uula Hyväkkö, and Monika Österberg*
P–27	Mechanochemical synthesis of pristine and Li-doped zinc orthostannate from separate and blended binary oxide precursors *Wojciech Marynowski, Marcin Saski, Krzysztof Niciński, Marcin Pisarek, Witold Wałecki, and Janusz Lewiński*
P–29 F2	Scratching beneath the surface: catalyst evolution and reusability in the direct mechanocatalytic Sonogashira reaction *Sheeniza Shah, Mennatullah M. Mokhtar, Thinh Tran, Kathleen Floyd, Lizette Mella, Tim Dao, Alexandria Garza, James Batteas, and James Mack*
P–31 F3	Toward circularity with mechanochemistry: alternative pathways for sustainable materials and processes *Mohammed Mazen Hariri, Ioannis Zuburtikudis, and Lorie Hamelin*
P–33 F4	Direct mechanocatalysis using electroplated reactors - the opportunities and limitations *Maximilian Wohlgemuth and Lars Borchardt*
P–35	Mechanochemical route to OPA-MOF: structural control from milling to extrusion *Alisson L. R. Balbino, Dagoberto O. Silva, Giovanna P. Correia, Priscila J. Zambiazi, Gustavo H. C. dos Santos, Paulo F. M. de Oliveira, and Liane M. Rossi*
P–37 F5	Playing with racemic phases in mechanochemistry: Insights from solid-state NMR spectroscopy *Sven Moos, Calogero Quaranta, Igor d'Anciães Almeida Silva, Ettore Bartalucci, Leeroy Hendrickx Fabio Manzoni, Manu Lahtinen, Mirijam Zobel, Kari Rissanen, Carsten Bolm, and Thomas Wiegand*
P–39 F6	The role of temperature in the mechanical impact sensitivity of energetic materials *Tahlia M. Palmer and Adam A. L. Michalchuk*
P–41 F7	Time-resolved tandem *in situ* raman and X-Ray diffraction monitoring of mechanically induced self-propagating reactions in the nickel-copper-sulfur system *Imelda O. Tampubolon, Ralf Bienert, Dominik Al-Sabbagh, Franziska Emmerling, and Matej Baláž*

P–43	Mechanochemical synthesis of perspective composites for li-ion batteries *Olena Porodko, Martin Fabián, Ladislav Kavan, Barbora Pitňa Lásková, Vladimír Šepelák, Hristo Kolev, Klebson Lucenildo da Silva, Maksym Lisnichuk, and Markéta Zukalová*
P–45	Butyllithium-Free Access to Lithium Amide Bases via Mechanochemistry *Suman Sahoo, Martin TRebunski, Riina Aav, and Dzmitry Kananovich*
P–47	Synthesis and characterization of $SrMoO_4$ and $SrMoO_4$@C powders as lithium storage anode materials *Maria Gancheva, Reni Iordanova, and Maria Shipochka*
P–49 F8	Direct mechanoorganocatalysis via surface functionalization of milling reactors *Joao Tanepau, Maxime Provost, Marie Gressier, Marie-Joëlle Menu, Sandrine Duluard, Frédéric Lamaty, Julien Pinaud, and Xavier Bantreil*
P–51 F9	Building fire safety in the solid state: scaling mechanochemistry for coating applications *Ana Mangas Roca, Giacomo Marra, and Blai López Rius*
P–53	Routes to direct mechano-organocatalysis (DMOC) *Cameron D. Webster, Tristan H. Borchers, and Deborah E. Crawford*
P–55	Photoextrusion: twin-screw extrusion for continuous solid-state photochemistry *Andreas F. Giglio, Julia H. Lehman, and Deborah E. Crawford*
P–57	Efficient and eco-friendly recovery of PGMs from spent automotive catalysts by mechanochemical approach *Zara Cherkezova-Zheleva, Daniela Paneva, Anna Vasileva, Kaloyan Ivanov, Maria Luisa Grilli, and Lakovos Yakoumis*
P–59	Mechanochemical synthesis of framework materials for the electrocatalytic reduction of nitrate to ammonia *Alina Schmalz, Anjana Ros, Biswajit Bhattacharya, and Franziska Emmerling*
P–61	Soft magnetic composites prepared by resonant acoustic mixing and self-milling methods *Vladyslav Kostiuk, Mária Fáberová, Róbert Džunda, and Radovan Bureš*
P–63	One-pot mechanochemical synthesis of spin crossover materials *Yolanda Sabater Algarra, Catherine Dunsford, Lewis Jackson, and Helena J. Shepherd*

P–67	Sustainable mechanochemical synthesis of gallate-based BioMOFs for aqueous chromium removal *Giovanna Pereira Correia, Alisson Luiz Rocha Balbino, Gustavo Henrique Correia dos Santos, Priscilla Jussiane Zambiazi, Bryan Alberto Laura Larico, Dagoberto Oliveira da Silva, Paulo Filho Marques de Oliveira, and Liane Marcia Rossi*
P–69	Mechanochemical aerobic activation of metallic copper for the synthesis of 1,4-allenynes *Ana M. Constantin, Francesco Mele, Matteo Lanzi, Giovanni Maestri, Raimondo Maggi, Nicola Della Ca', and Luca Capaldo*
P–71 F10	Ball mill reaction of ZnO and imidazole: mechanistic aspects inferred from its kinetics *Silvina Pagola, Maria Dolores Masso Ramirez, Jeremy Schwingel, Hamna Hafeez, and Orlando Ayala*

Poster Session 2
Tuesday, 16th September 2025, 17:30-19:00

Poster presentation (P) Flash talk (F-02, Tuesday, 16th September, 16:50)	
P–02	Green mechanochemical synthesis of a novel drug-drug eutectic mixture of acetylsalicylic acid and pyrazinamide for enhanced drug delivery *Luis H.S. Queiroz, Mateus R. Lages, Clenilton C. dos Santos, Mafalda C. Sarraguça, and Paulo R. da Silva Ribeiro*
P–04	Mechanochemical design of high-performance battery electrodes: a route toward scalable and solvent-free synthesis *Ali Karimi and Zohreh Shapoori*
P–06	Mechanochemical synthesis of Cr_3C_2: investigating the role of pressure and temperature *Meet Koshiya and Özgül Agbaba*
P–08	Supported palladium NPs over oxide zinc: structure analysis of potencial catalysts obtained from one-pot planetary and vibratory milling *Mattheus H. M. Mendes, Wilker K. T. De Melo, and Paulo F. M. De Oliveira*
P–10	An open reactions database for mechanochemical reactions *Mark Boyer, Tzu-Hsuan Chao, Yeu-Shiuan Ho, Daniel Tabor, and James Batteas*
P–12	Mechanochemically induced loss of stereochemistry in atropisomers *Leon Poljanic, Daniel M. Baier, and Tom Leyssens*
P–14	Analyzing molecular-recognition processes exploiting the power of solid-state NMR spectroscopy *Calogero Quaranta, Sven Moos, Igor d'Anciães Almeida Silva, Ettore Bartalucci, Leeroy Hendrickx, Benjamin M. D. Fahl, Claudia Pasqualini, Francesco Puccetti, Mirijam Zobel, Thomas Wiegand, and Carsten Bolm*
P–16	Local structure in situ monitoring of mechanochemical reactions *Jacob N. Wilson, Anthony Beauvois, Ana Guilherme Buzanich, Phoebe K. Allan, and Adam A. L. Michalchuk*
P–18	Using a radical scavenger to track the kinetics of mechanochemical depolymerization *Simone Mosk, Claire L. Seitzinger, and Ina Vollmer*
P–20	Scalable mechanochemical synthesis of biotin[6]uril *Elina Suut-Tuule, Eve Schults, Tatsiana Jarg, Dzmitry Kananovich and Riina Aav*
P–22	High entropy oxide support for Ag and Au nanoparticles *Wilker K. T. de Melo and Paulo F. M. de Oliveira*

P–24	The mechanochemical story of the sydnone family: from APIs to coordination complexes *Nicolas Petry, Xavier Bantreil, Florian Luttringer, and Frédéric Lamaty*
P–26	Pyrazinamide–pimelic acid cocrystals: a mechanochemical and thermal study *Anastasia May, Lucia Casali, Inês O. Feliciano, Carlos E.S. Bernardes, and Franziska Emmerling*
P–28 F11	Fast, clean, pillared: mechanochemical routes to interpenetrated MOFs *Giorgio Cagossi, Dario Giovanardi, Andrea Daolio, Iryna Andrusenko, Enrico Mugnaioli , Paolo Pio Mazzeo, Alessia Bacchi, Paolo Pelagatti*
P–30 F12	Shaken, not stirred! Phase diagrams of pharmaceutical solvates from mechanochemistry *Fragkoulis Theodosiou, Toby J. Blundell, John S.O. Evans, Patricia Basford, Noalle Fellah, and Aurora J. Cruz Cabeza*
P–32 F13	The solid-state aspects of metal-TEMPO chemistry *Krzysztof Budny-Godlewski, Michał Leszczyński, Iwona Justyniak, Dariusz G. Piekarski, Arkadiusz Kornowicz, and Janusz Lewińskia*
P–34	Breaking down forever chemicals with mechanochemistry *Jasna Alić and Franziska Emmerling*
P–36 F14	Mechanochemical synthesis of CuAgSe, study of its physicochemical and thermoelectric properties *Dáša Drenčaková, Marcela Achimovičová, Jiří Navrátil, Matej Baláž, Erika Tóthová, Mária Bali Hudáková, Viktor Puchý, Tomáš Plecháček, Stanislav Šlang*
P–38 F15	Kinetic studies of ball mill reactions: a Diels–Alder cycloaddition under second order overall kinetic models *Silvina Pagola, Maria Dolores Masso Ramirez, and Orlando Ayala*
P–40 F16	Tracking the evolution of the periodic and local structure noble metal nanoparticles prepared by milling *Ismael P. L. Xavier and Paulo F. M. de Oliveira*
P–42	Mechanochemical synthesis of essential oil cocrystals *Remie M Sundermann, Andrea Dalio, Michele Prencipe, Alessia Bacchi, Paolo P. Mazzeo*
P–44	Solid-state synthesis of cationic cellulose fibers from low-processed cotton for efficient virus capture *Tatsiana Nikonovich, Yao Yu, Mikko Korkiakoski, Chengji Yang, Iris Seitz, Daniel Langerreiter, Mauri A. Kostiainen, Eduardo Anaya-Plaza, and Sandra Kaabel*

P–46	Oxidation reactions catalyzed by Me3TACN-Mn catalysts under mechanochemical conditions *Michael E. Korfmacher, Hannah Busch, Thomas Wiegand, and Carsten Bolm*
P–48 F17	In situ production of high entropy rare-earth hexaboride and tetraboride by mechanochemical synthesis *Burçak Boztemur and Duygu Ağaoğullari*
P–50	Advancing mechanochemistry's impact: education, innovation, professional development, and outreach programs in the NSF center for the mechanical control of chemistry (CMCC) *Sophia Antillon, Alison Altman, Adam B. Braunschweig, Robert Carpick, Jonathan Felts, Danna Freedman, Timothy Hanusa, Hemamala Karunadasa, James Mack, Mateusz Marianski, Ashlie Martini, Andrew Rappe, James Rondinelli, Isaiah Speight, Daniel Tabor, and James Batteas*
P–52 F18	Asymmetric [5,5] sigmatropic rearrangement: excellent chirality transfer enabled by mechanochemistry *Johanna Breinsperger, Maximilian Kaiser, Michael Schnürch, and Peter Gärtner*
P–54	Ball milling enabled high-throughput mechanochemistry for solvent to solventless organic reaction translation *Cihang Yu, Philipp Kollmus, Christopher Waudby, Marco Santagostino, Frank Roschangar, and Duncan L. Browne*
P–56	Upscaling mechanochemical lignin modification: small ball vs. planetary milling *Friedrich Fink, Anna Theresa Schmitt, Jana Falkenhagen, and Franziska Emmerling*
P–58	Green synthesis and solubility profile of new lenalidomide:quercetin cocrystal *Ana C. S. Carvalho, Vânia André Clara S.B. Gomes, Mafalda C. Sarraguça, Alessia Bacchi, Michele Prencipe, Job Gieling, Natalia Shemchuk, Daniel M. Baier, Koen Robeyns, Tom Leyssens, Hector Polyzois, Lidia Tajber, Christos M. Chatzigiannis, Evelina Colacino, and M. Teresa Duarte*
P–60	Mechanochemical activation of spent lithium cobalt oxide cathodes: a sustainable approach for metals recovery *Lyazzat Mussapyrova, Rashid Nadirov, Matej Baláž, Kaster Kamunur, Aisulu Batkal, and Bagdatgul Milikhat*

P–64	Tailoring accessible surface area of activated carbons via mechanochemical processing *Anna Sturm, Paul Appel, Jonas Krug von Nidda, and Tim-Patrick Fellinger*
P–66	Native mechanochemical ligation *Florian F. Ort, Lukas J.K. Joost, Daniël Blanco Ania, Kevin Neumann, and Floris P. J. T. Rutjes*
P–68	Mechanochemical approach for perovskite-based catalysts: a rapid, one-pot synthesis towards greener materials *Rodolfo L. Coppo, Priscilla J. Zambiazi, Gustavo H. C. Santos, Rafael P. Borges, and Paulo F.*
P–70 F19	Mechanoenzymatic oligomerization and depolymerization transformations *Alexandra Rios-Echeverri, Carlos E. Puerto Galvis, Karen J. Ardila-Fierro, and José G. Hernández*
P-72	From Prediction to Milling: Periodic DFT-Guided Mechanochemistry of Halogen-Bonded Materials L. Kumar, S. G. Dash, K. Leko, N. Bregović, b D. Cinić, M. Arhangelskis
F20	MechSyn: where mechanochemistry meets industry for high-performance material production *Elaheh Bayat and Özgül Agbaba*

INVITED

I-00

Chosen by mechanochemistry: How grinding has been shaping my research path

Lucia MAINI

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In the realm of powder handling and X-ray powder diffraction, grinding is often viewed as a routine operation aimed merely at particle size reduction. Yet this process is far from innocent. Unexpected transformations—hydration, dehydration, amorphization, and phase transitions—frequently emerge, often labeled as "undesired." For the open-minded researcher, however, such events can spark entirely new directions of inquiry.

I will present a series of serendipitous observations in which simple grinding led to the formation of hydrated polymorphs and co-crystals—events that marked my entry into the world of mechanochemistry. For example, grinding enabled the formation of hydrated polymorphs in organometallic systems purely through mechanical activation, without solvents.[1] I subsequently demonstrated the efficiency of solid-state co-grinding in forming hybrid organic–organometallic hydrogen-bonded assemblies, enabling the selective design of supramolecular architectures.

Mechanochemical routes have also proven powerful for tuning polymorphic outcomes. In the case of barbituric acid, prolonged grinding (24 h) yielded the thermodynamically stable enolate form.[2] In the domain of ionic co-crystals, pressing barbituric acid with alkali halides such as KBr unexpectedly produced new crystalline phases (e.g.,BA·KBr·$2H_2O$), showcasing the potential of mechanochemistry to harness ionic interactions for structural innovation.[3]

I extended this methodology to the synthesis of luminescent copper(I)-based coordination polymers, such as $[Cu_2I_2(L)_2]_n$ with DABCO, piperazine or pyrazine ligands, which exhibit remarkable solid-state photophysical properties, demonstrating how mechanical energy enables exploration of otherwise inaccessible regions of the structural landscape. [4] [5]

The importance of mechanochemistry has gained global traction. Yet the foundational value of grinding predates modern science. The ancient alchemist Zosimus of Panopolis (3rd century CE) emphasized the centrality of grinding to material transformation. In collaboration with historians, I helped reconstruct ancient recipes—first reproducing the cold extraction of mercury from cinnabar, and later exploring the mechanosynthesis of cinnabar itself from mercury and sulfur. These practices, deeply symbolic and chemically significant, resonate with the alchemical concept of the ouroboros: a continuous cycle of destruction and rebirth.[6]

My journey through powders and paradoxes has shown that what begins as accident can become philosophy—and what seems ancient can become newly essential.

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[1] Braga, D.; Maini, L.; Grepioni, F. *Chem. Commun.* **1999**, No. 10, 937–938.

[2] Schmidt, M. U.; Brüning, J.; Glinnemann, J.; Hützler, M. W.; Mörschel, P.; Ivashevskaya, S. N.; van de Streek, J.; Braga, D.; Maini, L.; Chierotti, M. R.; Gobetto, R. *Angew. Chemie Int. Ed.* **2011**, *50* (34), 7924–7926.

[3] Braga, D.; Grepioni, F.; Maini, L.; Prosperi, S.; Gobetto, R.; Chierotti, M. R. *Chem. Commun.* **2010**, *46*, 7715–7717.

[4] Braga, D.; Grepioni, F.; Maini, L.; Mazzeo, P. P.; Ventura, B. *New J. Chem.* **2011**, *35* (2), 339–344.

[5] Cappuccino, C.; Farinella, F.; Braga, D.; Maini, L. Mechanochemistry, an Easy Technique to Boost the Synthesis of CuI Pyrazine Coordination Polymers. *Cryst. Growth Des.* **2019**, *19* (8), 4395–4403.

[6] Marchini, M., Montanari, G., Casali, L., Martelli, M., Raggetti, L., Baláž, M., Baláž, P., & Maini, L. **2024** *RSCMechanochem.*, *1*, 123-129

I-01

Advances in Mineral Mechanochemistry: From Metals Extraction to Energy Materials and Anticancer Drugs

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In this lecture the overall view on activities in mechanochemistry performed under umbrella of Slovak Academy of Sciences will be presented. Selected results from science and technology of extractive metallurgy, materials science and medicine will be illustrated. The golden thread in these activities is the mechanochemical treatment using high-energy mills. The knowledge obtained from treatment of natural minerals to extract metals (I) was later expanded to the application of natural minerals for cancer treatment (II). Recent activities are focused on the preparation of synthetic minerals for application as energy materials (III). The resultant examples will be illustrated:

- Elaboration of mechanochemical technology (MELT) for the treatment of polymetallic (Cu,Sb, Au,Ag) ores (I)[1-3]
- *In vitro* and *in vivo* testing of mechanically activated arsenic minerals for the treatment of various cancer cell lines (II) [4-6]
- Application of mechanochemically prepared synthetic minerals as prospective thermoelectric materials (III) [7-9]

All the mentioned examples represent contribution to the common goal of mechanochemists – to prepare new materials with the desired properties in a reproducible way under easy-operating, environmentally friendly and essentially waste-free conditions.

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[1] Peter Baláž, Roland Kammel, Marcela Achimovičová, *Metall,* **1994,** 48, 217-220; [2] Peter Baláž, Roland Kammel, Félix Sekula, Štefan Jakabský, in: *Proc. XX Intern. Miner. Process. Congress* (Heinz Hoberg, Ed.), Aachen **1997,** Vol. 4, 149-159; [3] Peter Baláž, Mechanochemistry in Nanoscience and Minerals Engineering, Springer-Verlag Berlin Heidelberg **2008**; [4] Peter Baláž, Martin Fabián, Michal Pastorek, Danka Cholujová, Ján Sedlák, *Materials Letters,* **2009**, 63, 1542-1544; [5] Peter Baláž, Ján Sedlák, *Toxins,* **2010**, 2, 1568-1581; [6] Danka Cholujová, Zdenka Bujňáková, Erika Dutková, Teru Hideshima, Richard W. Groen, Constantine S. Mitsiades, Paul G.Richardson, David M.Dorfman, Peter Baláž, Kenneth C.Anderson, Jana Jakubíková, *Brit. J. Haematology*, **2017**, 179,756-771; [7] Peter Baláž, Michal Hegedus, Matej Baláž, Nina Daneu, Peter Siffalovič, Zdenka Bujňáková, Erika Tóthová, Matej Tešinský, Marcela Achimovičová, Jaroslav Briančin, Erika Dutková, Mária Kaňuchová, Martin Fabián, Satoshi Kitazono, Oleksander Dobrozhan, *Progress in Photovoltaics, Research and Application* **2019**, 27, 798-811; [8] Peter Baláž, Marcela Achimovičová,, Matej Baláž, Ken Chen, Oleksander Dobrozhan, Emmanuel Guilmeau, Jiří Hejtmánek, Karel Knížek, Lenka Kubíčková, Petr Levinský, Viktor Puchý, Michael Reece, Peter Varga, Ruizhi Zhang, *ACS Sustainable Chemistry and Engineering* **2021**, 9, 2003-2016; [9] Peter Baláž, Erika Dutková, Nina Daneu, Michal Hegedus, Matej Baláž, Emmanuel Guilmeau, Robert Džunda, Mária Bali Hudáková, Veronika Garbárová, Jiangzhong Jiang, Marcela Achimovičová*, RSC Mechanochemistry*, **2025**, 2, 91-99

I-02

Modelling and Mechanistic Study of Polyethylene Chain Cleavage During Ball Milling

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Mechanochemical conversion of polyethylene (PE) and polypropylene produces monomers and is thus interesting for chemical polymer recycling. However, fundamental knowledge on the effect of crystallinity, degree of polymerization, entanglement, and temperature during milling is lacking due to the difficulty in producing polyolefins with controlled chain length and molar mass distribution. As these polymers make up more than 50% of the worldwide polymer production, studying their conversion during ball milling is especially relevant. Here we synthesize PE by a controlled chain growth polymerization and study its degradation during ball milling at cryogenic conditions, room temperature (RT), with and without air, and using either steel or zirconia grinding spheres.
Resulting molecular weight distributions are fitted using a statistical chain cleavage model suggesting a Gaussian distribution of cleavage probability around the middle of the chain (Figure 1a). With a 95% chance of cleavage in the middle 60% of the chain, the distribution is rather wide suggesting a statistical effect. Cleavage requires that the chain is fixed at two locations in either crystals or entanglements and a minimum length is needed. Cleavage deep in the chain is most likely, because the likelihood that the chain is fixed at each side of the cleavage point is the highest (Figure 1b). Chain cleavage preferably occurs in entangled domains. A micelle grown single crystal ultra-high molecular weight PE without entanglements was milled and its molar mass decreased much less compared to the same sample that was annealed to create entanglements.
Chain cleavage was faster at cryogenic conditions compared to RT (Figure 1c). However, this effect was not due to the higher brittleness of the material below its glass transition temperature (~-120 ˚C) but due to a suppression of radical recombination. In addition, the number of permanent scissions increases by up to 2.6 times under air compared to nitrogen atmosphere. Nuclear magnetic resonance spectra of the milled samples suggest that this is due to reaction of mechanochemically formed chains with air which prevents recombination. In addition, steel milling spheres were shown to stabilize radicals and lead to more permanent chain cleavages (Figure 1c).

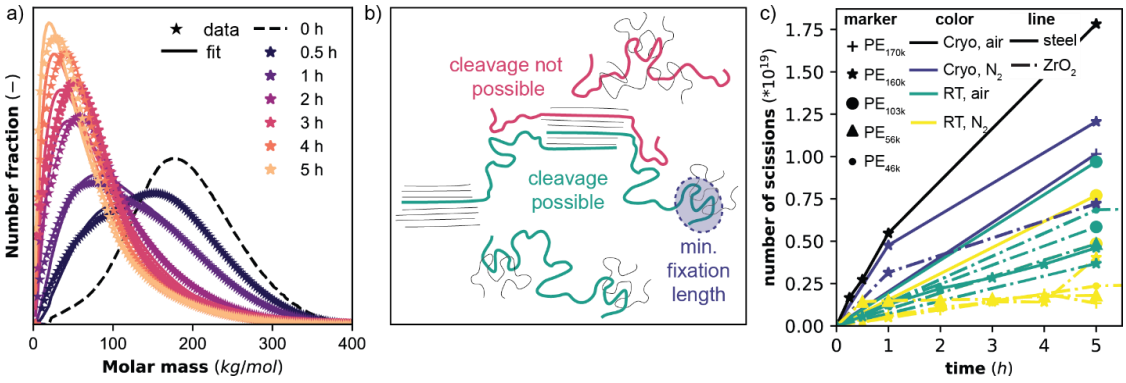

Figure 1: a) Molar mass distributions (star symbols) obtained from HT-SEC before and after milling of 300 mg of PE for 0.5, 1, 2, 3, 4 and 5 h at 30 Hz at RT under air in a 25 ml steel container, using 5 steel grinding spheres (10 mm) together with fits (solid lines) using a statistical chain cleavage model suggesting a Gaussian distribution of chain cleavage probability centered around the middle of the chain. b) Illustration of fixture of polymer chains due to entanglements and crystalline sections, which makes cleavage in the center of the chain most likely. c) number of permanent scissions, before and after milling under various conditions.

Understanding the Effect of Mechanical Stress on Chemical Reaction Rates: Tuning Reactivity and Selectivity

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Mechanochemical reactions are initiated by forces acting on the reaction mixture, for example, in a ball mill, where the stresses (contact pressures) can reach several GPa. Mechanochemical processes are induced by the forces/stresses modifying the potential-energy surface (PES). This then affects the rate at which the system transforms from one state to another to modify the kinetics and selectivities of chemical reactions. Thus, mechanochemistry functions in a fundamentally different way from other sub-branches of chemistry such as thermo-, electro- or photo-chemistry, which change the way in which energy is delivered to the reactant. In principle, all these stimuli can be used in combination with mechanical modification.

This talk describes a method for analyzing mechanochemical reaction rates based on transition-state theory (TST). Evans and Polanyi developed a perturbation method for chemical reactions [1] that allows the influence of a perturbation on the rate of a chemical reaction to be calculated. The theory is tested for the decomposition of methyl thiolate species adsorbed on copper, where the rate is measured under ultrahigh vacuum conditions using an atomic force microscope tip were good agreement is found between experiment and theory. This model predicts that the change in reaction rate depends on the direction of the force relative to a vector from the initial- to transition-state and provides one possible approach to modify reaction selectivity.

This method was extended to studying Diels-Alder cycloaddition reactions in solution,[2] and the results provided excellent agreement with experiment for non-polar reactants, but less good agreement for polar ones, suggesting that solvent-reactant interactions could be used to tune mechanochemical reactivity. It is also found that reactions with larger activation volumes become dominant as the force increases.

Finally, final-state effects are also found for the mechanically induced decomposition of carboxylate self-assembled monolayers on copper. They react by C-COO cleavage to form a hydrocarbon species and evolve carbon dioxide. Mechanochemically reducing the reaction temperature lowers the energy at which the resulting hydrocarbon products is formed and thus modifies the pathway of their subsequent reaction.

Mechanochemical Pathways for Synthesis and Activation of Metal Oxides

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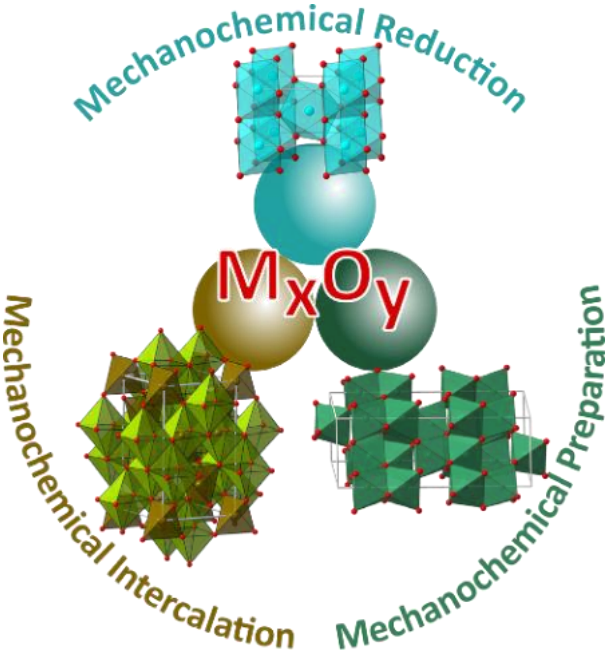
Metal oxides play a crucial role in applications ranging from catalysis and sensing to energy storage. Traditionally synthesized via high-temperature solid-state methods, these materials often settle into their thermodynamic ground states, limiting their reactivity. In this presentation, we explore how mechanochemistry offers a powerful alternative to activate and even synthesize metal oxides under ambient conditions, unlocking new reaction pathways, new structural and functional possibilities.
We present several case studies demonstrating how mechanochemical treatment can stabilize metastable high-pressure phases through in-situ surface functionalization, [1] and how lithium titanates prepared this way exhibit enhanced lithium-ion conductivity and uptake.[2] These findings extend to a broader class of materials, including solid-state phosphates and sodium-ion conductors. Furthermore, we show that mechanochemical reduction of metal oxides using alkali metal hydrides enables access to unconventional oxidation states, as exemplified by TiO_2 and Nb_2O_5, yielding photocatalytically active materials not attainable via conventional synthesis.[3,4] Overall, this work highlights the transformative potential of mechanochemistry in tailoring the structure and reactivity of metal oxides for advanced applications.



[1] Annika Betke, Guido Kickelbick; *New J. Chem.* **2014**, *38*, 1264-1270.
[2] Dennis. Becker, Michael Klos, Guido Kickelbick, *Inorg. Chem.* **2019**, *58*, 15021-15024, Dennis Becker, Robert Haberkorn, Guido Kickelbick, *Eur. J. Inorg. Chem.* **2019**, 4835-4845.
[3] Anna Michaely, Oliver Janka, Elias C. J. Gießelmann, Robert Haberkorn, Hakon T. A. Wiedemann, Christopher W. M. Kay, Guido Kickelbick, *Chem. Eur. J.* **2023**, e202300223.
[4] Anna Michaely, Elias C. J. Gießelmann and Guido Kickelbick, *Solids* **2025**, 6, 26.

I-05

Mechanochemistry in Chemical Evolution

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The prebiotic chemical evolution and the emergence of life on our planet are complex topics that draw a lot of attention, experimental and computational work, and lively debates among scientists from different fields. Among them, prebiotic chemists are dedicated to developing the "prebiotically plausible" procedures for synthesis of more complex organic compounds, such as amino acids, peptides, nucleosides, sugars and similar, from simple precursors that are considered by consensus to have been present on Earth before the life emerged, and primarily in water medium. [1] The vast majority of chemical reactions, however, are hindered by the presence of water due to solubility, solvation, hydrolysis and chemical stability, which, together with the findings of complex organic molecules in extraterrestrial bodies, expands the prebiotic plausibility to solid-state, for example, the reactions on mineral surfaces, and to reactivity in the gas-solid phase.

Surprisingly, unlike the extensive use of photo-, thermo-, and electrochemistry in the existing solution-based prebiotic experimental setups, the use of mechanical force on the bulk solid substrates to induce the reactivity in prebiotically plausible manner is almost unrepresented.[2] This is particularly surprising since the seismological and lithospheric movements, together with the meteoritic impacts and volcanic activity may have provided sufficient friction and impact between the mineral layers or defected surfaces, especially over a long period of time.[3]
We will discuss here a potential of mechanochemical reactivity in prebiotically plausible chemical evolution for the synthesis of complex molecules and molecular aggregates,[4],[5] and it's transfer to modern production of added-value chemicals.[6]

[1] R. Krishnamurthy, N. V. Hud, *Chem. Rev.* **2020***, 120,* 4613–4615.
[2] C. Bolm, R. Mocci, C. Schumacher, M. Turberg, F. Puccetti, J. G. Hernandez*, Angew. Chem. Int. Ed*. **2018**, *57*, 2423 – 2426; S. Lamour, S. Pallmann, M. Haas, O. Trapp, *Life*, **2019**, 9, 52.
[3] H. G. Hansma, *J. Biomol. Struct. Dyn*. **2013**, *31*, 888-895.
[4] T. Stolar, S. Grubešić, N. Cindro, E. Meštrović, K. Užarević, J. G. Hernandez, *Angew. Chem. Int. Ed*. **2021**, *60*, 12727-12731.
[5] T. Stolar, B. K.D. Pearce, M. Etter, K.-N. Truong, T. Ostojić, A. Krajnc, G. Mali, B. Rossi, K. Molčanov, I. Lončarić, E. Meštrović, K.Užarević, L. Grisanti, *Iscience*, **2024**, *27*, 109894.
[6] T. Stolar, J. Alić, G. Talajić, N. Cindro, M. Rubčić, K.Molčanov, K. Užarević, J. G. Hernández, *Chem.Commun.*, **2023**, *59*, 13490-13493.

I-06

Pushing the Limits: Evolving Twin-Screw Extrusion for Next-Gen Mechanochemical Processing

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Our previous work has established twin-screw extrusion (TSE) as an effective approach for scaling mechanochemical reactions into continuous, large-scale, solvent-free processes. Such has been the impact of this technology that it has seen commercial adoption for the synthesis of metal-organic frameworks,[1] with our work cited by IUPAC as one of the top ten emerging technologies set to transform the world.[2] Having expanded our research to encompass a diverse range of materials, including fine organic chemicals [3] and multi-layered graphene, [4] we are now focused on further improving the versatility of twin-screw extrusion. To this end, we are adapting the twin-screw extruder through the integration of complementary technologies, thereby enabling access to advanced chemical manufacturing and the synthesis of exotic compounds typically beyond the reach of conventional TSE methods.

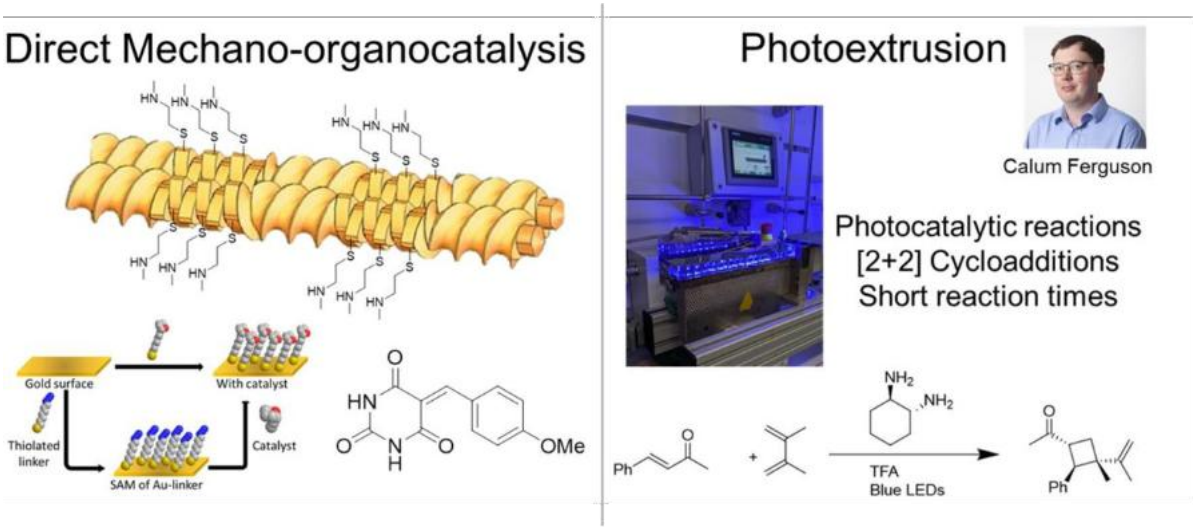

Figure 1: Next-generation mechanochemical processing – advancing twin-screw extrusion through direct mechano-organocatalysis and photoextrusion.

[1] Crawford *et al. Chem. Sci.*, 2015, **6**, 1645.
[2] Gomollón-Bel, *Chem. Int.*, 2019, **41**, 12.
[3] Crawford *et al. Green Chem.*, 2017,**19**, 1507.
[4] Chen *et al*. *Adv. Mater. Technol.,* 2024, **9**, 2301780.

Investigating the Detection and Role of Cocrystals in Metal-Mediated C–H Activation Through Mechanochemistry

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Mechanochemical, metal-mediated activation and functionalization of C–H bonds have demonstrated methodological advantages that surpass those of traditional solution-based protocols.[1] Despite these advancements, the mechanisms governing C–H bond activation under mechanochemical conditions are still not well understood.

This presentation will describe recent findings in the mechanochemical synthesis of metalacycles via ball milling of organic substrates and half-sandwich organometallic complexes including the detection and involvement of cocrystal intermediates between the reactants (Figure).[2,3,4]

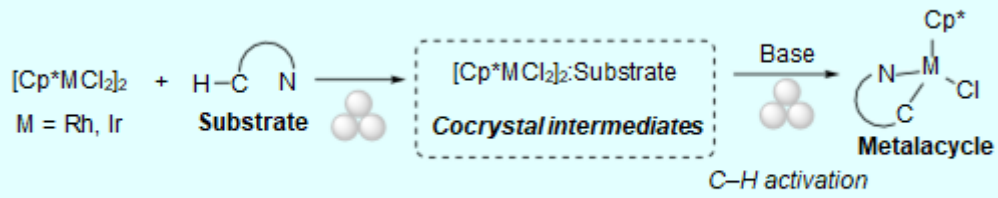

Figure. Cocrystal intermediates in mechanochemical cyclometalation reactions

Understanding these mechanistic details will enable the identification of the underlying reasons for the superiority of certain mechanochemical C–H activation processes and their potential applications.

1 Shengjun Ni, Matic Hribersek, Swarna K. Baddigam, Fredic J. L. Ingner, Andreas Orthaber, Paul J. Gates, Lukasz T. Pilarski, *Angew. Chem. Int. Ed.* **2021**, *60*, 6660–6666.
2 Karen J. Ardila-Fierro, Mirta Rubčić, José G. Hernández, *Chem. Eur. J*. **2022**, *28*, e202200737.
3 José G. Hernández, Karen J. Ardila-Fierro, Sara Gómez, Tomislav Stolar, Mirta Rubčić, Edi Topić, Cacier Z. Hadad, Albeiro Restrepo, *Chem. Eur. J.* **2023**, *29*, e202301290.
4 Sara Gómez, Santiago Gómez, Natalia Rojas-Valencia, José G. Hernández, Karen J. Ardila-Fierro, Tatiana Gómez, Carlos Cárdenas, Cacier Hadad, Chiara Capelli, Albeiro Restrepo. *Phys. Chem. Chem. Phys*. **2024**, *26*, 2228.

I-08

Mechanochemical Engineering of Novel Praziquantel Cocrystals for Enhanced Solubility

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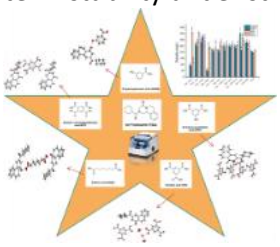
Praziquantel (PZQ) is the main treatment for schistosomiasis, but its unsatisfactory water solubility (BCS class II) restricts oral bioavailability and causes dosage inconsistency across formulations. To address these issues, we employed mechanochemistry as an efficient and sustainable method to synthesize novel multicomponent crystalline forms of PZQ with pharmaceutically acceptable coformers.[1] Liquid-assisted grinding using a Retsch MM400 mill enabled the rapid and reproducible preparation of five new cocrystals or cocrystal solvates: PZQ·3-hydroxybenzoic acid (1:1), PZQ·benzene-1,2,4,5-tetracarboxylic acid (2:1), PZQ·suberic acid (2:1), PZQ·trimesic acid hydrate (1:2:2), and PZQ·5-hydroxyisophthalic acid·MeCN (1:4:2).

The crystalline phases were confirmed by powder X-ray diffraction and fully elucidated via single-crystal X-ray diffraction. Structural analysis showed consistent hydrogen bonding between the PZQ carbonyl groups and the coformers' carboxyl/hydroxyl moieties, stabilizing distinct supramolecular assemblies. The PZQ·suberic acid system received special focus, with DSC/TGA, FTIR, SEM, and XPS confirming the formation of a robust 2:1 cocrystal exhibiting improved thermal properties and long-term stability under stress conditions (40 °C/75% RH, 5 months).



Solubility studies in water and simulated gastrointestinal fluids revealed that most of the new cocrystals exhibit faster dissolution than pure PZQ, outperforming previously reported forms under certain conditions. This work highlights mechanochemistry as a powerful, green platform to enhance API performance through crystal engineering.

Acknowledgements: This work is a contribution to the COST Action CA18112- Mechanochemistry for Sustainable Industry and was supported by EU project IMPACTIVE (no. 101057286). CSBG, VA and MTD thank FCT IP for funding LAQV (UID/50006/2023, LA/P/0008/2020), CQE (UID/00100/2020) and IMS (LA/P/0056/2020) projects.

[1] Marieta Mureşan-Pop, Simion Simon, Ede Bodoki, Viorica Simon, Alexandru Turza, Milica Todea, Adriana Vulpoi, Klara Magyari, Bogdan C. Iacob, Alexandra Iulia Bărăian, Mateusz Gołdyn, Clara S. B. Gomes, Margarida Susana, M. Teresa Duarte, Vânia Andre *Cryst. Growth Des.* **2024**, *24*, 11, 4668–4681

I-09

Identifying Promising Mechanochemical Reactions Through High-Throughput Virtual Screening, Machine Learning, and Simplified Physical Models

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Recent advances in mechanochemistry have demonstrated that external mechanical forces can accelerate chemical reactions (such as Diels-Alder reactions) to a significant degree, providing a potential avenue to greener chemical synthesis of many compounds. However, identifying reactions amenable to such acceleration remains a slow, manual process due to a lack of systematic design principles.

Here, we will discuss our work to understand the fundamental design principles for optimizing chemical reactions under external forces. We utilize a combination of high-throughput screening, optimization methods, and accurate machine-learned interaction potentials to conduct a broad search for reactions that can be significantly accelerated by external forces that are achievable in modern mechanochemical reactors. Our methods use machine learning potentials in combination with reaction path searching protocols (e.g., nudged elastic band and the growing string methods) to find potential transition states. We then explore candidate “activatable” coordinates—specific deformation modes that lead to enhanced reaction rates—by analyzing a mix of localized and normal coordinates. The most promising reactions and degrees of motion are then verified by higher-level calculations.
We will focus on lead-candidate reactions discovered by our framework, the types of realizable forces that most often lead accelerated reaction outcomes (across different types of reactions), and the connections between the explicit modeling of these reactions and the fundamental chemical physics of reaction rate theories. Our results offer a systematic pathway to optimize mechanochemical reactions, contributing to the rational design of force-accelerated synthetic processes.

I-10

Kinematic Modelling of Mechanochemical Reactions

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The rising prospects of mechanochemically assisted syntheses hold promise for both academia and industry, yet they face challenges in understanding and, therefore, anticipating respective reaction kinetics. Particularly, dependencies based on variations in milling equipment remain little understood and globally overlooked.
This talk aims to address this issue by identifying critical parameters through kinematic models, facilitating the reproducibility of mechanochemical reactions across the most prominent mills in laboratory settings, namely planetary and mixer mills.
We found through a series of selected experiments replicating major classes of organic, organometallic, transition metal-catalyzed, and inorganic reactions from literature the independence of kinematic parameters on reaction kinetics when the accumulated energy criterion is met. As a step forward and to facilitate the practicability of our findings, we provide a freely accessible online tool “Ball-Mill Calculator” that allows the calculation of respective energy parameters for different planetary and mixer mills.[1]
Finally, this talk will also discuss the limitations of our model and challenges that require to be addressed in order to advance this rapidly evolving field.

Mechanochemical C–N Bond Formation: From Imatinib to Macrocycles

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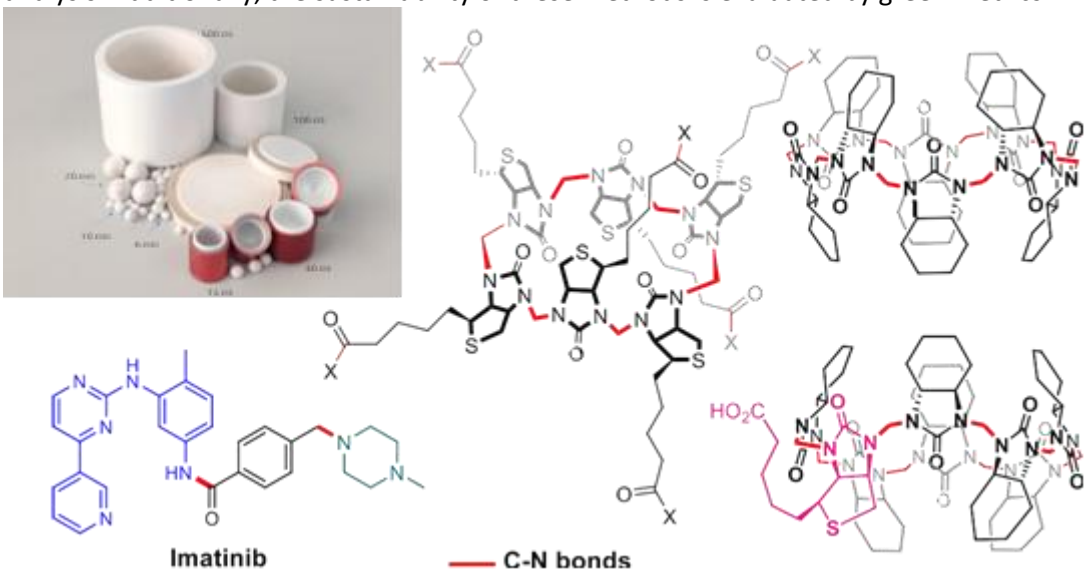
Mechanochemical synthesis maximizes reagent concentrations and overcomes limitations of solution-based synthesis, such as poor solubility of starting materials and reduced reactivity caused by solvation shells. This presentation will focus on the development of mechanochemical techniques for forming C-N bonds[1,2,3] [4,5,6]. Methods for the formation of acylaminal, amide, amines have proven valuable for synthesizing various products, including the active pharmaceutical ingredient Imatinib[4] and as well as a number of different hemicucurbiturils[1,2,5,6]. Synthesis of one the latter has been successfully scaled up to a multidecagram scale using a planetary mill.[6] We have shown that internal standard enhances the accuracy of yield estimation in mechanosynthesis[7] and helps to uncover significance of reaction parameters by using a design-of experiment approach[5]. Special attention will be given to critical chemical and technical parameters, quantitative reaction monitoring, and product analysis. Additionally, the sustainability of these methods is evaluated by green metrics.



[1] Sandra Kaabel, Robin S. Stein, Maria Fomitsenko, Ivar Jarving, Tomislav Friščić, Riina Aav, *Angew. Chem. Int. Ed.*, **2019**, *58*, 6230 –6234.

[2] Tatsiana Dalidovich, Kamini A. Mishra, Tatsiana Shalima, Marina Kudrjašova, Dzmitry G. Kananovich, Riina Aav, *ACS Sustainable Chem. Eng.* **2020**, *8*, 15703−15715.

[3] Tatsiana Dalidovich, Jagadeesh V. Nallaparaju, Tatsiana Shalima, Riina Aav, Dzmitry G. Kananovich *ChemSusChem*, **2022**, *15*, e2021022.

[4] Tatsiana Nikonovich, Tatsiana Jarg, Jevgenija Martõnova, Artjom Kudrjašov, Danylo Merzhyievskyi, Marina Kudrjašova, Fabrice Gallou, Riina Aav, Dzmitry Kananovich. *RSC Mechanochemistry*, **2024**, 1, 189-195.

[5] Elina Suut-Tuule, Tatsiana Jarg, Priit Tikker, Ketren-Marlein Lootus, Jevgenija Martõnova, Rauno Reitalu, Lukas Ustrnul, Jas S. Ward, Vitalijs Rjabovs, Kirill Shubin, Jagadeesh V. Nallaparaju, Marko Vendelin, Sergei Preis, Mario Öeren, Kari Rissanen, Dzmitry Kananovich, Riina Aav, *Cell Reports Physical Science*, **2024** *5* (9), 102161

[6] Elina Suut-Tuule, Eve Schults, Tatsiana Jarg, Jasper Adamson, Dzmitry Kananovich, Riina Aav, *ChemSusChem* **2025**, e202402354.

[7] Tatsiana Jarg, Jevgenija Tamm, Elina Suut-Tuule, Ketren-Marlein Lootus, Dzmitry Kananovich, Riina Aav *RSC Mechanochem*., **2025**, 2, 507-515.

How Much “Mechano” Is in Mechanochemistry?

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The original purpose of ball mills was to utilize mechanical energy from colliding milling balls to grind materials and reduce particle size. It is a natural extension of this concept to assume that the same mechanical energy can overcome activation barriers and drive chemical reactivity. This assumption aligns neatly with the IUPAC definition of mechanochemistry as “chemical reactions initiated by the absorption of mechanical energy.” Indeed, this definition underpins much of the mechanochemical community’s work, where reactions are regularly transferred from solution-based protocols to ball mill setups.
However, a growing number of counterexamples challenge this mechanistic view and question the validity of the current definition. This talk centers around a provocative but necessary question: **How much “mechano” is truly in mechanochemistry?** Or more precisely: **Is mechanical energy from ball collisions genuinely responsible for chemical transformations?** Or do many reactions proceed via more conventional thermal or mixing pathways, merely disguised as mechanochemical?
To address these questions, we adopt a multi-faceted approach. We experimentally determine the apparent activation energies of mechanochemical reactions (Fig. 1), both in ball mills and in media- free setups such as resonant acoustic mixers. We then investigate the influence of liquid-assisted grinding (LAG) on these reactions and observe a transition from solid-state to liquid-phase behavior, where the reactions exhibit similar characteristics. These findings additionally offer new insights into the role of LAG—an aspect of mechanochemistry that remains underexplored.
In parallel, we introduce a novel technique to probe the actual temperatures experienced during milling—not merely the macroscopic temperature of the milling vessel, but the microscopic temperature that directly affects the reacting molecules. To achieve this, we propose the use of Raman thermometry, a contactless method traditionally applied in fields such as semiconductor research, catalysis, and biological systems, where precise local temperature measurements are critical. This technique enables us to simultaneously monitor both the temperature of the reactants and the chemical transformations occurring during milling. By doing so, we gain deeper insights into the thermal conditions that govern mechanochemical reactions, ultimately advancing our understanding of their underlying mechanisms and addressing the central questions posed earlier.

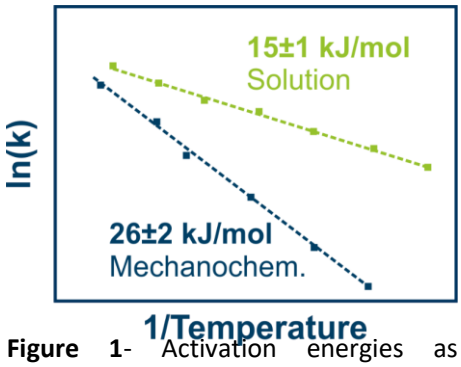

Figure 1- Activation energies as determined for the Diels-Alder reaction in solution and in a mechanochemical system

Overall, this talk aims to critically examine the foundational assumptions of mechanochemistry and spark a deeper discussion about its underlying principles. As the field gains traction in both academia and industry, refining its conceptual framework is pivotal. Only by challenging its core definitions can we transform mechanochemistry from a "black-box" phenomenon into a robust and predictable synthetic methodology.

Michalchuk, A. A., Boldyreva, E. V., Belenguer, A. M., Emmerling, F., & Boldyrev, V. V. (2021). Frontiers in chemistry, 9, 685789.
Martinez, V., Stolar, T., Karadeniz, B., Brekalo, I., & Užarević, K. (2023). Nature Reviews Chemistry, 7(1), 51-65.
Speight, I. R., Ardila-Fierro, K. J., Hernández, J. G., Emmerling, F., Michalchuk, A. A., García, F., ... & Mack, J. (2025). Nature Reviews Methods Primers, 5(1), 1-18.
Templ, J., Hwang, S., Schwemin, T., Baltaci, H., & Borchardt, L. (2025). RSC Mechanochemistry.

I-13

Phonon Dynamics and Mechanochemical Reactivity

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Mechanical energy can induce physical or chemical transformations through a variety of different mechanisms, effectively giving rise to different 'types' of mechanochemical reaction. [1] To study those reactions that are *directly* initiated by dynamic mechanical stress, energetic materials (explosives, propellants, pyrotechnics; EMS) offer an exciting playground. EMs are a prototypical example of the direct coupling between mechanical stress and a (violent) chemical reaction, which ultimately leads to the rapid release of energy. Understanding the mechanochemistry of EMs is not only of fundamental importance, but underpins the safe handling, deployment, and design of these important functional materials.

In this talk we will highlight our recent efforts to understand how mechanical energy can drive the initiation of EMs.[2] We will focus on our understanding of how the kinetic energy that is mechanically 'injected' into a material can localize into molecular bonds to ultimately incite a chemical reaction.[3] Using this framework we will highlight how the mechanochemistry of EMs can be predicted from first principles, and demonstrate how this reactivity depends on the crystal form (polymorph or cocrystal).[4] We will finally discuss how these predictions extend towards elementary models of mechanically-induced reactions. Our work ultimately paves the way towards understanding the non-conventional reactivity that is observed in mechanochemical transformations that are directly induced by dynamically applied mechanical stress.[5]

[1] AAL Michalchuk, *Chem Commun.* **2024**, *60*, 14750-14761.
[2] AAL Michalchuk, J Hemingway, CA Morrison, *J Chem. Phys.* **2021**, *154*, 064105.
[3] JM Hemingway, HM Quayle, C Byrne, CR Pulham, S Mondal, AAL Michalchuk, CA Morrison, *PhysChemChemPhys.*, **2025**, *27*, 11640-11648.
[4] IL Christopher, X Liu, HJ Lloyd, CL Bull, NP Funnell, P Portius, AAL Michalchuk, SR Kennedy, CR Pulham, CA Morrison, *PhysChemChemPhys.* **2024**, *26*, 16859-16870.
[5] AAL Michalchuk, *Faraday Discuss.* **2023**, *241*, 230-249.

Shaping Chemistry by Force: Using Advances in Milling Knowledge from the Power Generation Sector to Progress Mechanochemical Extraction

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Milling is one of the biggest consumers of energy globally and is commonly used in material processing processes. This presentation shows the potential of taking recent advances in milling knowledge in the power sector and applying them in mechanochemical extraction. Over the last two decades, solid fuel combustion has changed from using coal to biomass in several countries around the world. Most large-scale coal fired power station conversions have adapted coal mills, which use impact and abrasion to break down brittle coal, to mill biomass, which favours shear fracture. This mismatch between milling fracture mechanisms and material properties has led to numerous issues, including mill choking and mill explosions[1]. This has led to advancements in milling knowledge, including novel milling metrics and greater awareness of the limit of impact milling for ductile materials.

Mechanochemical extraction has the potential to synergistically combine particle size reduction and targeted extraction of high value products into a single method. Ball milling uses impact fracture mechanisms, but the effective milling of all materials using impact fracture mechanisms is limited by their critical particle size for comminution via brittle fracture[2]. This particularly limits the effectiveness and efficiency of milling brittle materials like biomass, resulting in increased milling energy and poor particle size reduction. Mechanochemistry often combines dissimilar materials, and thus understanding co-milling materials with different fracture materials is essential for successful reactions. This presentation will report recent work in co-milling brittle and ductile materials which showed synergistic benefits for the ductile fraction of materials, enabling a greater particle size reduction and eliminating mill choking potential which can happen when ductile materials are milled separately.

Extraction of compounds from biomasses has been one of the main focus areas of mechanochemical extraction research but can be limited by issues such as caking. This presentation will show how the same issues have been experienced with transporting olive residues through screw feeders in power stations, which were overcome by understanding the role of moisture and temperature in the glass transition of olives[3]. Furthermore, this presentation will show how novel milling metrics can rank the milling performance of any material at laboratory scale and correlate this performance to full scale mill[4]. Mechanochemical extraction requires similar metrics to link energy, particle size reduction and yield, and this presentation will show ongoing developments in this area.

[1]Williams, O. et al., 2016. Influence of mill type on densified biomass comminution. Appl. Energy, 182, 219-231.

[2]Kendall, K., 1978. The impossibility of comminuting small particles by compression. Nature 272, 710-711.

[3] Williams, O. et al., 2017. Overcoming the caking phenomenon in olive mill wastes. *Ind. Crops Prod.*, 101, 92–102.

[4] Williams, O. et al., 2025. A proposed novel combined milling and combustion performance model for fuel selection. J. Energy Inst., 120, p.102046.

I-15

Mechanochemistry-Driven Synthesis of Spinel and High-Entropy Oxide Materials as Candidates for Lithium-Ion Battery Components

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Lithium-ion batteries are popular for their high power, low self-discharge, and no memory effect, but challenges such as flammability, high costs, material degradation, and performance issues with components limit their wider use. In this context, state-of-the-art oxides and sulfides with high Li- ion conductivity and good electrochemical stability are among the most promising candidates for solid-state electrolytes in secondary batteries. As their electrochemical properties are closely linked to elemental composition, structure, and morphology, mechanochemistry has emerged as a transformative tool in this domain, offering a solvent-free, energy-efficient approach to material synthesis and processing, including structure and morphology modifications. Hence, we report on several structures of mechanosynthesized complex oxides with spinel structure, including lithium titanate (LTO) and high-entropy spinel oxides, i.e., $(M)Fe_2O_4$ (HEOFe), as well as their lithiated oxyhalide derivatives $Li_{0.5}(M)_{0.5}Fe_2O_{3.5}F_{0.5}$ (LiHEOFeF) and $Li_{0.5}(M)_{0.5}Fe_2O_{3.5}Cl_{0.5}$ (LiHEOFeCl), ($M=Zn_{0.25}Mg_{0.25}Co_{0.25}Cu_{0.25}$). The LTO spinel nanoparticles synthesized via ball milling and thermal treatment, using a precursor of TiO_2 nanoparticles and ethanolic Li acetate, showed minimal impurity formation influenced by calcination conditions. The as-prepared LTO achieved a charge capacity of 142 mAh/g at 0.1 mV/s, outperforming commercial LTO despite a lower surface area, as confirmed by galvanostatic testing. Furthermore, LTO spinel powders were modified by mechanical disintegration. X-ray diffraction confirmed the main LTO phase with minor rutile and WC impurities. TEM revealed two morphologies: larger crystals surrounded by nanocrystals, with their ratio depending on milling time. The sample with a 21 m^2/g surface area showed the highest charge capacity, excellent cycling stability, and discharge capacities of 170, 167, and 160 mAh/g at 1, 2, and 5C, respectively, without carbon additives. Its capacity retention was above 95% over multiple cycles, outperforming commercial LTO. On top of that, high-entropy oxides (HEOs) are gaining attention as promising materials for lithium-ion batteries because of their potential to improve stability and electrochemical performance, recently. These materials, defined by a single-phase solid solution containing multiple metal cations, allow for adjustable compositions and possess distinctive qualities such as structural robustness and high ionic conductivity. In this context, we have successfully synthesized (HEOFe), (LiHEOFeF), and (LiHEOFeCl). The particles, 50–200 nm with uniform atomic distribution, matched the nominal compositions. Electrochemical tests on 2032 coin cells with lithium metal anodes showed voltammetric capacities of 450 mAh/g (HEOFe), 694 mAh/g (LiHEOFeCl), and 593 mAh/g (LiHEOFeF), with LiHEOFeCl performing best due to smaller particle size. Galvanostatic cycling confirmed high initial capacities but some fading over 100 cycles. Raman analysis of LiHEOFeF indicated reversible processes. Impedance measurements revealed the lowest initial charge transfer resistance for LiHEOFeCl, which decreased during cycling; HEOFe's resistance increased slightly, likely due to different reduction mechanisms.

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I-16

Mechanochemistry Triggers a Change of Paradigm in Peptide Synthesis

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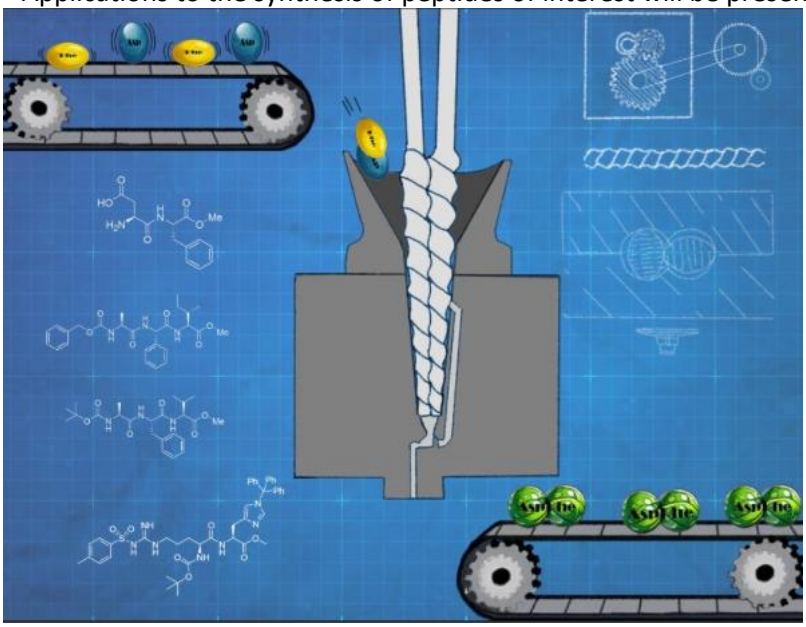
Peptides play a central role in biological mechanisms and therefore are becoming a must-sought therapeutic solutions. It has been reported that a large number of peptides are in clinical trials, and some of them will join the more and more developed peptide therapeutic armory.[1]
However current peptide production suffers from major environmental issues, including the very large amount of organic solvent required in the synthesis and purification. Research efforts are on-going to find alternatives to the use of these organic solvents. The challenging approach that we have chosen is to develop reactions in the absence of solvent, by mechanochemistry.
Since our pioneering work using ball-mills in peptide synthesis, we have contributed to the field in further developing this mechanochemical approach including fragment-coupling strategy to synthesize larger peptides[2,3] and continuous solvent-free scalable process by reactive extrusion,[4] resulting in a sharp decrease of the Process Mass Intensity.
Applications to the synthesis of peptides of interest will be presented.



Peptide factory of the future

1 Lei Wang, Nanxi Wang, Wenping Zhang, Xurui Cheng, Zhibin Yan, Gang Shao, Xi Wang, Rui Wang, Caiyun Fu *Sig. Transduct. Target Ther.* **2022**, *7*, 48.
2 Yves Yeboue, Marion Jean, Gilles Subra, Jean Martinez, Frédéric Lamaty, Thomas-Xavier Métro, *Org. Lett.* **2021**, *23*, 631-635.
3 Yves Yeboue, Nadia Rguioueg, Gilles Subra, Jean Martinez, Frédéric Lamaty, Thomas-Xavier Métro, *Eur. J. Org. Chem.* **2022,** e202100839.
4 Tharwat Mohy El-Dine, Matthieu Lavayssiere, Hélène Adihou, Gilles Subra, Thomas-Xavier Métro, Olivier Ludemann-Hombourger, Frédéric Lamaty, *ChemistryEurope* **2024**, *2*, e202400007.

Beyond Reagents: Exploring the Impact of Reaction Vials and Milling Media in Mechanochemical Synthesis

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Mechanochemistry, a field with historical roots dating back centuries, is gaining increasing attention and becoming more mainstream. The surge in research articles highlights its growing importance. As we delve deeper into the fundamentals driving these reactions, the roles of the reactor vial and milling material become more apparent. Traditionally, mechanochemical reactions were primarily considered in terms of the reagents involved. However, recent studies have shed light on the significant impact of the milling vial and milling media, particularly concerning organic synthesis.

Our recent advancements in performing organic mechanochemical reactions under diverse conditions have opened new avenues for understanding the energetics of these systems. It has become evident that the reaction vial and milling media can serve various functions depending on the chemistry involved. The vial's versatility in mechanochemistry is demonstrated by its ability to integrate electrochemical techniques, enabling precise control over reactions under minimal solvent conditions. Additionally, its role as a metal catalyst in solvent-free Sonogashira coupling reactions, using palladium and copper co-catalysts, facilitates in situ generation of active catalysts and allows multiple reaction cycles without additional palladium, enhancing efficiency and selectivity. The vial also functions as a regioselective base, which can be used to alter the regioselectivity of reactions, enabling desired product pathways.

These findings highlight mechanochemistry's transformative potential in organic synthesis. By leveraging the multifunctional roles of the reaction vial, we can explore sustainable pathways for chemical transformations, offering new opportunities for efficiency, selectivity, and sustainability. These multifunctional roles of the vial enhance our understanding of when these reactions mirror traditional solution-based reactions and when they are wildly different.

I-18

Round Robin Project: Reproducibility of Ball Mill Reactions at Steady State

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We report the experimental design and outcome of a Round Robin project on the reproducibility of 5 representative mechanochemical reactions or transformations over different fields (organic, inorganic and MOFs reactions, cocrystals formation and even a polymorph transformation) performed by 32 scientists over Europe, UK, and one from Brazil. This project was restricted to oscillatory milling equipment, moving either in the vertical or horizontal direction. The project allowed the experimentalists to use any type of oscillatory milling equipment, new or old, as well as any commercially available or home-made milling jars manufactured from stainless steel, zirconia, agate or tungsten carbide, using any design for the jar closure, the internal volume ranging from 5 mL to 50 mL, and any internal shape design. The only requirement was that the starting materials had to be weighed accurately and stoichiometrically, transferred quantitatively into milling jars, and the mixture had to be milled for long enough to reach maximum transformation at steady state. All experimentalists performed all 5 reactions in duplicate (2 under neat grinding and 3 under liquid assisted grinding conditions).

All ball neat and liquid assisted milling reactions were found to be reproducible across all 32 experimentalists at steady state conditions, each experimentalist having to adapt the milling experimental conditions to the specific parameters of their milling equipment and milling jar volume and design selected.

I-19

The Importance of the Nature of the Solid State for Mechanochemical Reactivity

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This presentation looks at how mechanochemical conditions can impact the stability of solid forms, and how transitions from one form to another can easily occur during milling.
Pharmaceutical industry has long ago recognized the importance of the solid-state of drug compounds. The nature of the solid-state indeed impacts properties such as solubility, dissolution rate, bioavailability, hardness, ….
But if the nature of the solid-state has such a strong impact on these properties, it is very likely it will also impact mechanochemical reactivity. This factor is often ignored, or almost never studied, in mechanochemical studies. In this talk, I will look at how easily solid-state transitions can be indused during mechanochemical processes, looking at the impact of temperature, pressure, particle size, and additives on the stability of the solid form in the milling vessel.
My talk will be based on a book chapter, of the educational book[1] 'An introduction to mechanochemistry of solids' that will be published in the coming year. Specific examples will be shown based on litterature examples, as well as research by my own group. The goal of this talk is to sensibilize the mechanochemist to the importance of studying (and controlling) the solid-state nature of their reactants during mechanochemical processes, to achieve robust and reproducible processes.

Mechanochemical Transformation of Commodity Polymers into Reactive Platforms for Organic Synthesis Utilizing Mechanical Energy

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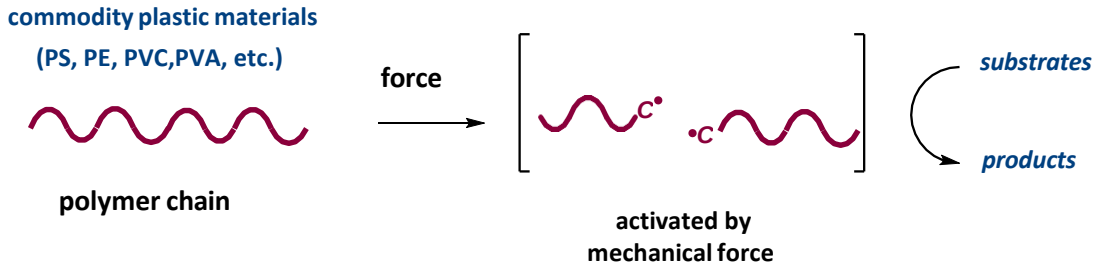
Direct activation of small molecules by mechanical stimulation is generally considered challenging under mechanochemical conditions, primarily because the mechanical energy is dissipated as thermal energy, rather than activating the small molecules. In contrast, polymers are known to undergo mechanical activation through methods such as sonication, ball milling, and extrusion, etc. Homolytic cleavage of the main chain of many commodity polymers is a well-documented phenomenon under mechanical agitation. Recent studies have extensively explored structural and functional changes in polymers containing so-called mechanophores, molecular units that respond to mechanical force, under various forms of mechanical stimulation. However, the application of these phenomena to organic synthesis remains underdeveloped.

We hypothesize that mechanically activated polymers can serve as mediators in the mechanochemical transformation of small molecules. In this context, we first reported the transformation of commodity polymers into fluorescent polymers by trapping radical species generated during ball milling, using a pre-fluorescent probe. A variety of polymers, including polystyrene, PMMA, PVMK, PE, PPS, cellulose, and polysulfone, were successfully converted into luminescent materials.[1] Based on these findings, we also demonstrated real-time visualization of mechanochemical damage in double-network hydrogels in collaboration with Gong and colleagues.[2]

To further explore synthetic applications, we investigated the use of commodity polymers as radical initiators for the reduction of organic halides.[3] Various organic halides were converted to the corresponding hydrocarbons in the presence of hydrosilanes, with radical initiation promoted by polyethylene and polyvinyl acetate under ball-mill reaction conditions. Interestingly, a polyethylene plastic bag obtained from a local supermarket was found to promote the radical reaction, offering a compelling example of upcycling plastic waste for use in organic synthesis. In this lecture, I will also present some of our latest achievements.



[1] Kubota, K.*; Toyoshima, N.; Miura, D.; Jiang, J.; Maeda, S.; Jin, M.*; Ito, H.* *Angew. Chem. Int. Ed.* **2021**, *60*, 16003–16008.
[2] Zheng, Y.; Jiang, J.; Jin, M.*; Miura, D.; Lu, F. X.; Kubota, K.; Nakajima, T.; Maeda, S.*; Ito, H.*; Gong, J. P.* *J. Am. Chem. Soc.* **2023**, *145*, 7376–7389.
[3] Kubota, K.*; Jiang, J.; Kamakura, Y.; Hisazumi, R.; Endo, T.; Miura, D.; Kubo, S.; Maeda, S.; Ito, H.* *J. Am. Chem. Soc.* **2024**, *146*, 1062–1070.

From the Molecular to the Macroscale: A Look Inside the Mechanics of Mechanochemical Reactions

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Mechanochemistry is fast becoming a leading methodology for greener and more sustainable chemical synthesis and manufacturing. Central to all types of reaction systems, whether they be vibratory mills, extruders, or acoustic mixers, is the input of mechanical energy, whereby applied forces not only drive reactions, but also facilitate particle fracture, as well as diffusion and mixing of reagents. As such, developing a clear understanding of the complex mechanical environments at play is key to advancing these methodologies, and affording the design of predictable and scalable methods. Recently we have focused on three main elements related to reactions run in ball mills: (1) examining the impact of the mechanics of the milling jar materials on reactions, (2) the role of auxiliary grinding agents, and how to optimize their use, and (3) how the surface properties of the milling jar materials can play a role in facilitating reactions. To this end, two model systems have been explored. In the first, we have examined the kinetics of the Knoevenagel condensation reaction of vanillin and barbituric acid in stainless steel, Teflon, zirconia, and aluminum reaction vessels using different milling frequencies to determine the role of reagent mechanics under a range of different mechanical loading environments. In addition, we introduce reaction vessels with interchangeable mid-sections and end caps of different materials to explore the role of jar/ball material surface energy and localized shear vs. normal loading forces on the reaction kinetics. In the second, we have examined the mechanochemical synthesis of sulfonylurea via direct mechanocatalysis in Cu milling jars to elucidate the complex changes of the Cu surface during reaction and the role it plays for in situ catalyst generation to help drive reaction. We exploit the unique sensitivities of this reaction to probe the chemically relevant transformations of the copper surface under a variety of atmospheric conditions, [1] to obtain critical knowledge for mechanochemists hoping to employ direct mechanocatalysis with copper.

[1] Kathleen Floyd, Lori Gonnet, Tomislav Friščić, James Batteas, *RSC Mechanochem.* **2024**, *1*, 289-295.

I-22

Static Electricity as a Mechanochemical Event

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When two insulator materials come into physical contact, their surfaces acquire electrical charges. The accumulation and retention of these charges cause problems in various industries, e.g., sheet sticking, jamming, agglomeration, particle segregation, and particle adhesion to surfaces. A sudden release of the stored charges can raise the possibility of major issues, such as fire or explosions. A systematic understanding of the electrification phenomenon is crucial for its prevention and mitigation. Nevertheless, despite over 2600 years of research, even the fundamental issues in the electrification event are still debated, due to its inherent complexity at all scales. Yet, we and others have shown that the electrification of polymer materials is directly related to their mechanochemistry; specifically, the covalent bond breaking leads to mechanospecies which include charges and charge stabilizers on polymer surfaces.[1] These species can be detected by AFM-KFM, chemical methods, and EPR. We have also demonstrated that it is possible to mitigate charges from surfaces by using additives, such as radical scavengers,[1] organic dyes, [2] charge-transfer complexes,[3] and quantum dots,[4] each with distinct chemical mechanisms. These findings can help prevent the above-mentioned problems in industry, as well as provide insight into why, in some mechanochemical reactions with organic materials, charging (and, e.g., sticking to the ball mill walls) is observed, and why it is not in others.

1 Baytekin H. T., Baytekin B., Hermans T., Kowalczy B., Grzybowski B.A. Science 2013, 341, 1368-1371.
2 Cezan D., Nalbant A, Buyuktemiz M., Dede Y., Baytekin H. T., Baytekin B. Nature Comm. 2019, 10, 276.
3 Ekim S.D., Kaya G.E., Daştemir M., Yildirim E., Baytekin H. T., Baytekin B. ACS Appl. Mat. & Int. 2022, 14, 56018-56026.
4 Ekim S.D., Aydın F. Kaya G.E., Baytekin H. T., Alptekin D. A., Baytekin B.. ACS Appl. Nano Mat. 2023, 6, 22693-22700.

Transformation of Mechanochemistry From Materials Science Into Chemical Science and an Overview on Mechanically Induced Self-Propagating Reactions

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Mechanochemistry has transformed from a raw materials- and material science-oriented field into an established synthetic chemical pathway in the last 15 years. [1,2] The first part of the lecture will be devoted to the demonstration of this change by providing a time-resolved overview on the published papers number in different research areas (mainly materials science vs. chemistry) and also according to data from few selected previous editions of the INCOME conference. In the second part, the overview on the mechanically induced self-propagating reactions (MSRs) will be provided. Despite the fact that mechanochemistry has witnessed a rapid development in the last years, the research in MSRs remains scarce. This unique reaction pathway makes it possible to prepare the desired products in shorter and energy-saving manner in comparison with classical gradual reactions.[3] This lecture will provide a brief peak into MSR history, types of compounds that can be prepared in this way, *in situ* monitoring techniques particularly interesting for MSRs and the actual trends in the area. Examples from our lab on the synthesis of metal chalcogendies via MSR will be given.[4] Also the self-made transferrable device for monitoring of temperature during the planetary ball milling in Pulverisette 7 premium line mill developed in our laboratory with specific aim to monitor the MSRs' ignition will be presented.[5]

This work was supported by the Slovak Research and Development Agency (APVV-23-0372) and Slovak Grant Agency VEGA (project 2/0112/22).

1 Joseph L. Howard, Qun Cao, Duncan L. Browne, *Chem. Sci.* **2018**, 9, 3080-3094.

2 Matej Baláž, *Environmental Mechanochemistry: Recycling Waste into Materials Using High-Energy Ball Milling,* Springer. **2021**, 619 p.

3 Laszlo Takacs, *Prog. Mater. Sci.* **2002**, 47, 355-414.

4 Matej Baláž, Róbert Džunda Radovan Bureš, Tibor Sopčák, Tamás Csanádi, *RSC Mechanochem*. **2024**, 1, 94-105.

5 Imelda Octa Tampubolon, Patrik Jacko, Matej Bereš, Peter Baláž, Matej Baláž, *J. Visualized Exp.* **2024**, 210, e67249.

Mechanochemistry Beyond Material-Making: From Nanoparticles to Defective Solids and (Meta)Stable Phases

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Mechanochemical processes have been used for centuries in the transformation of minerals and metals.[1] In the last decades, however, with the increasing demand for advanced materials with enhanced performance and activity in areas such as catalysis and energy, the range and sophistication of materials prepared by mechanochemical means have led to a true revolution in materials design through milling. [2] Metal nanoparticles with plasmonic properties, [3] one-pot synthesis of hybrid systems for solar energy harvesting and chemical transformations,[4] energy materials for both conversion and storage,[5] biomedical applications, and catalysts[6] are just a few examples of areas where the active materials can be prepared by milling. While the application- driven motivation is undeniably important, the fundamentals behind materials formation under mechanochemical conditions are often underestimated or simply neglected, restricting the potential of mechanochemistry to synthesis-only, leaving a large synthesis–material–structure– property landscape unexplored. Milling induces the formation of structural defects in solids not only through particle breakage, but also by driving atomic-scale disorder via impact and shear.[7] During ball milling, mechanical energy is randomly distributed across the particles, and even under such ill-defined stress conditions, a wide range of controlled structural disorder can emerge — including specific defect formation (point, planar, etc.), rearrangement into different crystalline phases, or transitions to structures that completely lack long-range order.

We have explored how mechanochemistry enables not only the synthesis of materials, but also their transformation through defect generation, phase transitions, and how the material can handle such mechanical stress. The bottom-up synthesis of Au[8] and Ag nanoparticles by milling have revealed different kinetic behavior of chemical reductants, while the local structure of the final nanoparticles indicates that the structural disorder can be accommodated in the lattice and tends to be related to the milling time. The mechanochemical reduction of MoO_3 to oxygen-deficient MoO_{3-x} in a planetary mill with increasing milling time illustrates the capacity of mechanical energy to induce the partial reduction of Mo^{6+} to Mo^{5+} via vacancy formation, and how its optical bandgap can be tuned. We have revisited the phase transitions of Nb_2O_5 induced by milling,[9] and we are demonstrating the complexity of phase change - either crystal-to-crystal or crystallization from an amorphous phase. The results emphasize how mechanochemistry allows to access and tune structural features and material properties that are often inaccessible by conventional methods.

[1] C. Suryanarayana, *Prog Mater Sci*, **2001**, *46*, 1–184. P. Baláž, et al., *Chem Soc Rev*, **2013**, *42*, 7571.
[2] A. P. Amrute, et al., *Chem A Eur. J.*, **2021**, *27*, 6819–6847. Z. Chen, et al., *Prog Mater Sci*, **2024**, *145*, 101299.
[3] P.F.M. de Oliveira et al., *J Mater Chem A*, **2020**, *8*, 16114–1614. M.J.Rak et al., *Green Chem*, **2014**, *16*, 86-89.
[4] J. Quiroz, et al., *ACS Sustain Chem Eng*, **2021**, *9*, 9750–9760.
[5] C.Welch et al., *Chem.Mater*, **2024**, *36*, 6748–6764. B.L.Souza et al., *J. Electroanal. Chem.*, **2021**, 896, 115459.
[6] R.T.P. Silva et al., *ChemPlusChem* **2023**, 88, e202300268. L.A.Faustino et al., *Chem. Eng J*, **2025**, 513, 163072.
[7] V. Šepelák et al., *Dalton Trans*, **2012**, *41*, 11927. M. Senna and A. A. L. Michalchuk, *RSC Mechanochemistry*, **2025**, 2, 351–369.
[8] I. P. L. Xavier, et al., *PCCP*, **2024**, *26*, 11436–11444.
[9] T. Ikeya and M. Senna, *J Mater Sci*, **1987**, 22, 2497–2502; *J Non Cryst Solids*, **1988**, 105, 243–250.

I-25

Looking into the Crystal Ball: The Future of Mechanochemistry

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Despite an extremely long history, chemical and materials transformations driven or sustained through mechanical forces (mechanochemistry) have only recently emerged as an exciting and versatile route to chemical and materials synthesis. Over the past 20 years the interest in, and applications of, mechanochemical reactivity have grown with surprising intensity, attracting the attention of chemists, materials scientists, physicists, engineers, industries, and many others, with reports on mechanochemistry captivating the attention of the general public, professional societies, as well as policy makers. The community of mechanochemists and researchers interested in the area has probably never been larger, and we are witnessing the creation and advances of large, collaborative international efforts (such as, for example, the National Science Foundation Centre for Mechanical Control of Chemistry, the EU COST Action Mechanochemistry for Sustainable Industry) that are simultaneously targeting the fundamentals of mechanochemistry, whilst systematically bridging the gap between research and manufacturing. Significant driving forces behind such developments have been the outstanding potential of mechanochemistry for green and sustainable chemistry, as well as the excitement of discovery in synthesis, mechanistic understanding and materials structure. In many ways, the mechanochemical reaction environment represents the exciting, but not (yet) charted land of chemical and materials synthesis, at whose horizon are new discoveries and cleaner, more efficient reactivity needed for large-scale manufacturing on Earth or elsewhere. Such recent progress suggests that the time might now be ripe for the mechanochemistry community, brought together through organisations such as the International Mechanochemical Association (IMA) and the International Union for Pure and Applied Chemistry (IUPAC), to look into the crystal ball and try to help chart a route through this *terra incognita* towards new, improved chemistries and chemical manufacturing processes of the future. As a potential contribution to this process, this presentation will outline some of the exciting developments related to applications and understanding of mechanochemistry and the underlying reaction environment, and also highlight gentler, often easily overlooked or forgotten aspects of mechanochemistry, related to non-covalent, supramolecular structure.

L. Takacs, Chem. Soc. Rev., **2013**, 42, 7649–7659.

J. Batteas, K. G. Blank, E. Colacino, F. Emmerling, T. Friščić, J. Mack, J. Moore, M. E. Rivas, W. Tysoe, *RSC Mechanochemistry*, **2025**, 2, 10–19.

A. A. L. Michalchuk and T. Friščić, *RSC Mechanochemistry*, **2025**, 2, 333–335

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ORALS

O-01

Oriented External Electric Field Controls the Rupture Forces in Mechanophores

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Controlling the reactivity of molecules under a mechanical pull has generated significant interest in organic and polymer chemistry. Inducing mechano-lability for otherwise rigid molecules has been possible through structural alterations like adjusting the pulling group, ring strain, and electron density of the scissile bond. We report that an oriented external electric field (OEEF) can significantly assist in mechanochemical transformations. Using a structurally diverse set of ring- opening reactions, we show that the critical force required for bond-cleavage, F_{rup}, gets appreciably reduced when the OEEF acts *in-phase* with the bond-polarity direction. The primary condition for utilizing OEEF along with mechanochemistry is the requirement of structural asymmetry along the target bond. Effectively therefore, any polar ring-opening reaction might be manipulated by OEEF. The versatility of the strategy of using OEEF and mechanical force together can also be appreciated by the enhanced rupture force when the direction of the OEEF is flipped. We show that mechanical pulling and electric field can act as *entwined twins* toward mechano-lability.

[1]Das, A.; Datta, A. Oriented External Electric Field Controls the Rupture Forces in Mechanophores. *J. Phys. Chem. B* **2024**, *128* (28), 6951–6956.
[2]Das, A.; Datta, A. Designing Site Specificity in the Mechanochemical Cargo Release of Small Molecules. *J. Am. Chem. Soc.* **2023**, *145* (24), 13484–13490.

O-02

Mechanochemistry-Enabled Electride and Alkalide Chemistry

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There are two textbook chemical curiosities in the world of inorganic chemistry. The first is electride [1], which is a class of unique materials featuring unbound electrons as anions. A classic example of electride is the dissolved Na metal in liquid ammonia used in Birch reduction. Another is alkalide, where the least electronegative elements, alkali metals, counterintuitively carry a negative charge via the formation of an ns^2 closed shell structure. Both electride and alkalide appear in every modern inorganic chemistry textbook, but beyond being chemical curiosities, their potential in synthetic chemistry has never been properly explored.

This is due to their extremely challenging synthesis. The electride and alkalide chemistry was first explored by the late James Dye group in the 1970s, spanning over the decades into the 2000s [2]. Undoubtedly, The Dye group's achievements in electride and alkalide are landmarks in the history of inorganic chemistry, but their synthesis are extremely challenging, requiring low boiling point solvents (e.g., ammonia, methylamine), low temperatures, bespoke glassware and high vacuum. As a result, these synthetic methods are not scalable.

To unleash the full potential of electride and alkalide, a scalable and accessible synthetic route is much needed. We recently reported the first such route. By using mechanochemistry, we reported a Li-K heterobimetallic room temperature stable electride (RoSE), which mediated the first solvent-free Birch reduction [3]. Later on, we reported the first scalable and accessible synthesis of a sodide, i.e., a sodium anion complex, which mediated the first alkali metal single-metal-two-electron redox chemistry [4].

[1] F. Sun et al., *Fundamental Research* **2025**, 10.1016/j.fmre.2024.11.026
[2] J. L. Dye, *Acc. Chem. Res.* **2009**, *42,*1564-1572
[3] N. Davison et al., *Chem* **2023**, *9*, 576.
[4] N. Davison et al., *Inorg. Chem.* **2024**, *63*, 15247.

O-03

Mechanochemical Stabilisation of Arsenic and Uranium in Mine Tailings: An Approach for Sustainable Remediation

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Mechanochemistry—an emerging solvent-free synthetic approach that harnesses mechanical forces such as grinding, shearing, and friction—has gained traction as a green alternative in materials synthesis. Its applications span pharmaceuticals, MOFs, battery materials, and increasingly, environmental remediation. Owing to its low energy requirements, scalability, and elimination of harmful solvents, mechanochemistry is particularly promising for recycling and stabilising hazardous waste.

Our research explores the mechanochemical remediation of mine tailings containing potentially toxic elements, specifically arsenic (As) and uranium (U). Current remediation methods for these elements often rely on energy-intensive thermal treatments or solvent-based extraction, both of which generate secondary waste streams. Uranium tailings, in particular, are typically stored as slurry in tailing ponds, lacking effective long-term stabilisation strategies.

We propose a novel, scalable mechanochemical method to immobilise soluble and unstable As and U species by converting them into stable mineral phases suitable for long-term storage. In our preliminary studies, we successfully synthesised meta-zeunerite [$Cu(UO_2)_2(AsO_4)_2{\cdot}8H_2O$] and geminite [$CuHAsO_4{\cdot}H_2O$]—minerals known for their capacity to immobilise uranium and arsenic—using simple grinding or ball milling at room temperature. These minerals were formed from reagent-grade As_2O_3/As_2O_5, the dominant arsenic species in tailings. Stability tests in deionised water showed that over 96.5 wt% of arsenic and nearly 100 wt% of uranium were immobilised in meta-zeunerite after one week, while geminite stabilised over 88 wt% of arsenic after one month. Importantly, we demonstrated the method's real-world applicability by transforming a tailing sample containing 70 wt% As_2O_3 into meta-zeunerite.

Further investigations involved synthesising a range of metal arsenate phases by grinding arsenic-containing waste with abundant, low-cost reagents such as calcium nitrate, iron(III) nitrate, and sodium hydroxide. These studies are ongoing, with a focus on evaluating the long-term stability of the resulting phases.

Our findings highlight the potential of mechanochemistry as a sustainable, scalable solution for remediating arsenic- and uranium-contaminated mine tailings. This approach could be extended to other hazardous solid wastes, offering a practical pathway to reduce environmental and public health risks.

O-04

Scalability Parameters in API Synthesis Using Continuous Mechanochemistry

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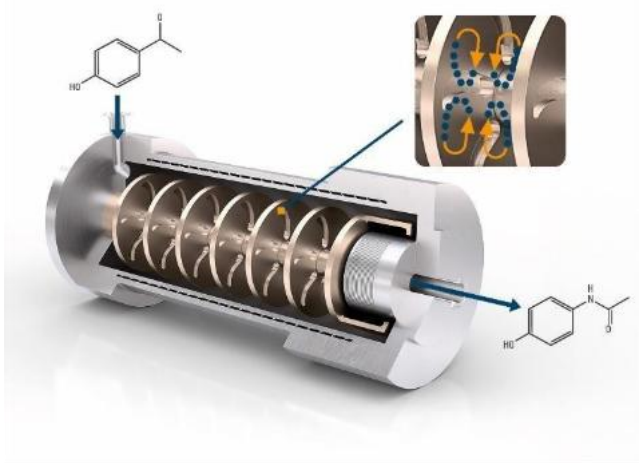
Mechanochemistry, the study of chemical reactions driven by mechanical force, has emerged as a promising approach for more sustainable chemical processes. Unlike conventional methods that rely on heat, solvents, or other energy-intensive techniques, mechanochemistry utilizes mechanical energy to initiate or accelerate reactions, often under milder conditions. This offers significant advantages, including reduced energy consumption, minimized waste, and the replacement of hazardous solvents with greener alternatives.
Pharmaceutical research requires some reliable equipment to ensure that the patients' lives are safe, thus equipment must be made safely.

The goal of WAB-GROUP® is to develop cutting-edge technologies to provide state-of-the-art solutions (the picture shows the principles of a continuous flow reactor). To assess the potential of the existing equipment for mechanochemical processes, a thorough evaluation of key process parameters was conducted, including heat transfer coefficients for various product flows, cooling media, and process settings, as well as residence time distributions under multiple operating conditions, and micro mixing efficiency. These studies aimed to fully characterize WAB mills and identify their properties towards mechanochemical applications.
A particular focus was placed on the **WAB** *IMPA***°C***T REACTOR®*, which integrates both conventional flow chemistry and bead milling technology.
Heat transfer was found to be highly dependent on cooling flow characteristics, and optimization strategies were analytically defined to address these challenges. The residence time is influenced by geometry, flow characteristics, tip speed, and bead filling degree. Our findings show that the mills operate within a range similar to traditional reactors, from near-ideal CSTR behavior to plug- flow characteristics, depending on the chosen process settings.



This presentation will provide an overview of characterization results and proposed optimization strategies, highlighting the benefits of bead mills for synthetic applications. Applications in the synthesis of pharmaceutically active material such as paracetamol will be discussed.

O-05

Breaking Barriers in Organometallic Synthesis with a Ball Mill

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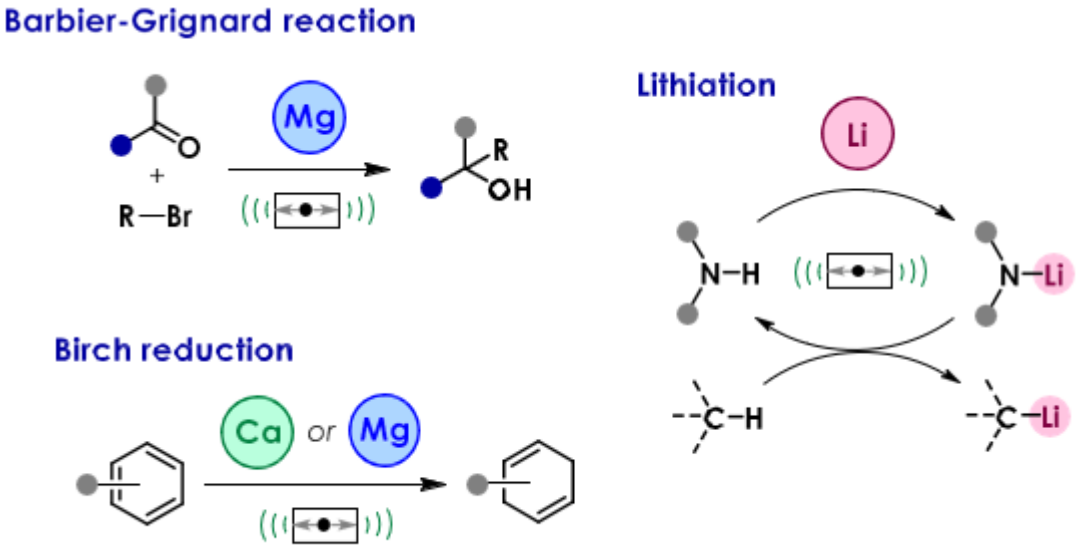
Alkali and alkaline earth metals are essential reagents in organic synthesis, enabling versatile reductive transformations and serving as indispensable tools for the generation of synthetically powerful polar organometallic species, such as Grignard reagents. These transformations are traditionally performed in hazardous organic solvents, and their broader applicability is constrained by air and moisture sensitivity, pyrophoricity, cryogenic conditions, the need to activate bulk metals, and side reactions that compromise yields.

Currently, mechanochemistry is reshaping these century-old practices and overcoming many of their long-standing limitations. Here, we present the developments from our laboratory illustrating this shift. We show that mechanochemical activation of magnesium significantly improves yields in the rarely used Barbier-Grignard reaction, providing a compelling alternative to traditional Grignard synthesis.[1] Expanding this concept, we demonstrate that the reducing power of magnesium and calcium can be significantly boosted, enabling their application as efficient reductants in ammonia-free Birch-type reductions. [2] Finally, we present the generation of lithium amide bases under mechanochemical conditions and their application in C–H deprotonations, including directed ortho-lithiation of arenes. Key challenges will also be addressed, including limited mechanistic understanding, scale-up difficulties, and the need for improved practicality to support broader adoption.



[1] Jagadeesh Varma Nallaparaju, Tatsiana Nikonovich, Tatsiana Jarg, Danylo Merzhyievskyi, Riina Aav, Dzmitry G. Kananovich, *Angew. Chem. Int. Ed*. **2023**, *62*, e202305775.

[2] Jagadeesh Varma Nallaparaju, Riin Satsi, Danylo Merzhyievskyi, Tatsiana Jarg, Riina Aav, Dzmitry G. Kananovich, *Angew. Chem. Int. Ed.* **2024**, *63*, e202319449.

O-06

A Double Bond Agent on a Mechanochemical Mission - Ball Milling Strategies to Introduce C(sp^2)=C(sp^2) Bonds

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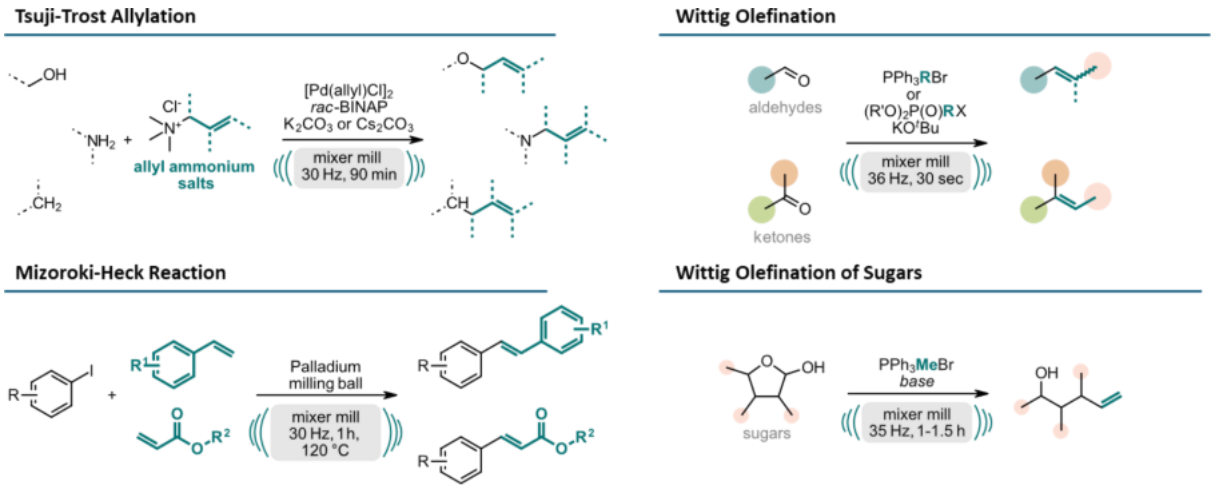
Introducing double bonds into chemical compounds - through vinylation, allylation, or olefination - is a fundamental strategy in organic synthesis, providing reactive handles for further functionalization such as epoxidation, dihydroxylation, metathesis, or radical reactions. Although these transformations are well-established in conventional solution-based chemistry, the introduction of C(sp^2)=C(sp^2) bonds remains underdeveloped under solvent-free conditions…

…at least until our double bond agent took on the mission.

We demonstrate how these key transformations can be performed under mechanochemical conditions, focusing on three reactions: the Tsuji-Trost allylation,[1] the Wittig olefination,[2] and the Heck coupling under direct mechanocatalysis.[3] Beyond eliminating solvents, these protocols offer notable advantages: reaction times as short as 30 seconds (Wittig olefination), drastically reduced catalyst loadings (0.5 mol% Pd for the Tsuji-Trost allylation), and efficient catalyst recovery - using a palladium milling ball as the active catalyst in the Heck reaction.



Along the way, we tackled what should be close to impossible: the olefination of sugars under neat conditions.[4] Given their low open-chain content, sugars are typically poor substrates for olefination without solvent. The fact that this reaction proceeds cleanly under ball milling conditions challenges conventional expectations in dry-state synthesis.

Finally, we highlight the applicability of these methods to pharmaceutically relevant compounds, demonstrating their practical value beyond proof-of-concept.[5]

1 Johanna Templ, Michael Schnürch, *Angew. Chem. Int. Ed.* **2024**, 63, e202314637.

2 Johanna Templ, Michael Schnürch, *Angew. Chem. Int. Ed.* **2024**, 63, e202411536.

3 Johanna Templ, Suhmi Hwang, Tino Schwemin, Hakan Baltaci, Lars Borchardt, *RSC Mechanochem.*, **2025**, *2*, 598-602.

4 Francesco Mele, Nina Biedermann, Christoph Suster, Johanna Templ, Christian Stanetty, Michael Schnürch, *Manuscript in preparation*

5 Johanna Templ, Lars Borchardt, *Angew. Chem. Int. Ed.* **2025**, e202503061.

O-07

Mechanochemical Approach Toward Efficient, Green and Sustainable Recycling and Reuse of Waste NdFeB Magnets

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The strategic position of NdFeB permanent magnets as the highest energy product BHmax at room temperature makes them vital for numerous emerging technologies in key sectors of industrial evolution, defense and space, but also in manufacturing of small devices for everyday life. The future demand of NdFeB permanent magnets is expected to rise additionally in regard to the green energy transition and new technologies such as renewable energy production, e-mobility, automation, etc. Consequently, the demand for rare earth elements (REEs) dramatically increases in parallel to a significant uncertainty in their supply due to geopolitical and economic factors. The research community is very active in searching for alternative materials with competitive performance containing less or zero REEs. Nevertheless, no better magnetic material has been introduced since the discovery of NdFeB magnets at the early 1980s. Thus, the long-term sustainable solution of the problem is to close the value chain with the recovery of end-of-life (EoL) permanent NdFeB magnets in a sustainable, resource- and cost-efficient way.

This study investigates the recycling of NdFeB magnets from electronic and motor waste due to their significant input in the waste flow. Sintered waste magnets were collected, precisely sorted, selected, and pre-treated. Mechanochemistry was applied as a main method for waste processing, which allowed optimization of the recycling protocol: room-temperature working conditions, low waste-emitting, energy-saving methodology in consideration with the green and sustainable principles. The characterization approach was based on the XRD, SEM/EDS, TEM/SAED analysis and Mössbauer spectroscopy. These methods were employed to monitor phase composition, oxidation, and microstructural evolution throughout the processing chain. The properties of treated materials were evaluated with an emphasis on structure–magnetic behavior relationships, as well as in the context of their further reuse for 3D printing of bonded magnets. Mössbauer spectroscopy was used to get deeper in the nature of presented iron-bearing phases and to follow the transformation of the $Nd_2Fe_{14}B$ magnetic phase. Mechanochemical activation, recognized for its solvent-free and resource-efficient nature, was optimized to maintain the structural and magnetic integrity of the Nd-Fe-B magnet phase for a direct reuse of studied materials. The results confirm that the established protocol of resource-efficient recycling of EoL magnets allows preservation of Nd2Fe14B magnetic grains and refining the material's microstructure and particle size. Formation of an anisotropic particle shape and formation of a thin Nd/REE-rich layer on the grain surface were achieved in the recycled material. The benefits of this mechanochemical recycling approach include significant improvement in the processing metrics, such as energy use, ecological impact, technology simplification, and cost reduction.

Acknowledgements: This study is based on the project activities of H2020 ERA-MIN3 Project № КП- 06-ДО02-3/06/06/2022: Microwave enhanced recovery of REEs and plastic from WEEE and re-use in Additive Manufacturing of novel magnetic components (MW4REMAM).

O-08

Process Integration and Life-Cycle Assessment of Moist-Solid Hydrolysis of Polylactic Acid With Lactic Acid Recovery via Electrodialysis

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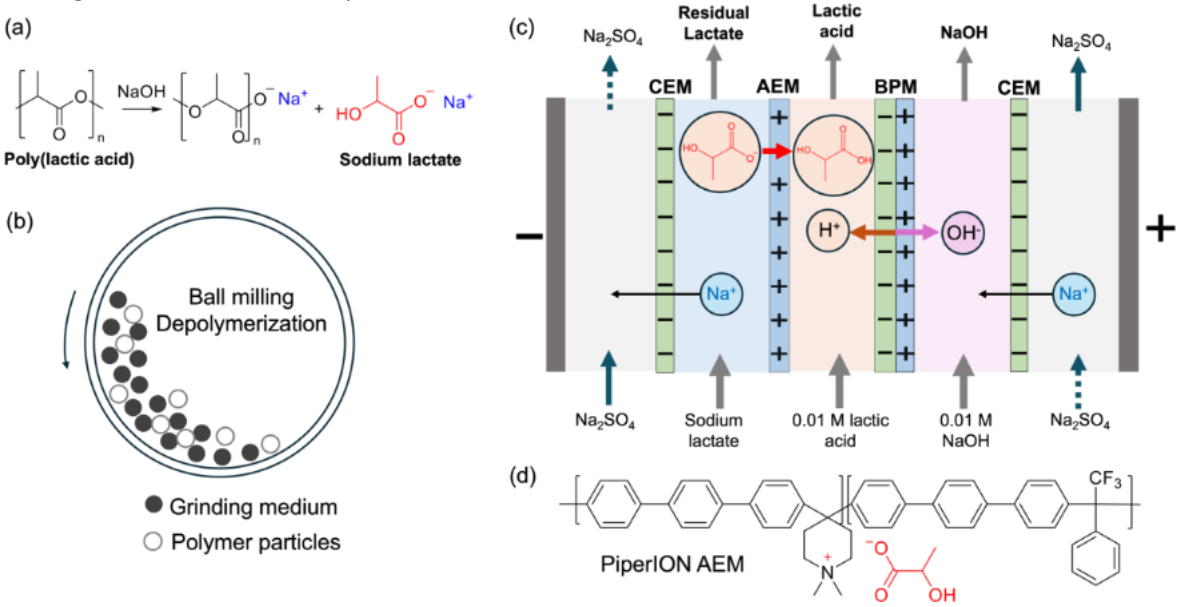
Poly(lactic acid) (PLA), a biodegradable plastic derived from starch, has gained prominence as a sustainable alternative to petroleum-based plastics. However, the slow degradation of PLA in natural environments and contamination of recycling streams due to incompatibility with other plastics, highlight the need for efficient recycling techniques. This study reports a sustainable closed-loop PLA recycling strategy by hydrolysis depolymerisation under moist-solid condition and recovery of the valuable monomer lactic acid. By employing ball milling and resonance acoustic mixing, we investigate the effects of milling speed, time, ball size, alkali type, and ageing on lactate yield, in order to optimise the performance and demonstrate the efficiency of mechanical and moist-solid conditions in polymer chain scission without bulk heating. Furthermore, we integrate a bipolar membrane electrodialysis process for conversion of lactate to lactic acid with simultaneous NaOH production, enhancing the sustainability of the recycling process. Life cycle assessment (LCA) was employed to assess the environmental impact of such PLA recycling approach for lactic acid production and provide recommendations for future improvements. This work demonstrates the effectiveness and environmental benefits of mechanically induced moist-solid chemical recycling and integration with advanced membrane separation processes, offering a promising pathway for sustainable PLA waste management and lactic acid production. [1]



[1] H. Luo, D. Yang, J. Sadhukhan, V. Costica, R. Dorey, Q. Song, M-M Titirici, *ChemSusChem* ***2025***, *00*, *e202500503*.

O-09

IMPACTIVE Educational Platform: a digital meeting point around mechanochemistry

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Mechanochemistry is rapidly gaining attention as it shows great potential in the context of green chemistry and sustainable production, making more people eager to explore its possibilities. Despite this rising interest [1] [2], there is still a lack of clear, high-quality resources on mechanochemistry. In addition, the various stakeholders working in the field often work separately, without easy ways to share knowledge or collaborate with others.
We aim to change this situation by creating an online community around mechanochemistry: the IMPACTIVE Educational Platform. Designed as both a knowledge hub and a social connector, the platform brings together valuable resources and fosters communication between individuals and institutions with a shared interest in mechanochemistry.

The platform is built on two main pillars: first, a rich repository of educational and research materials, including articles, interviews, laboratory protocols, software tools, and multimedia content; and second, a networking space that enables users to discover related initiatives, and stay informed through news and events.
The goal is to build a digital ecosystem around mechanochemistry, where a diverse range of users—from the general public to researchers and industry representative—can actively participate. This will ensure the platform's long-term sustainability and establish it as a cornerstone of the mechanochemistry community, supporting innovation, education, and collaboration. The educational platform can be accessed at www.mechanochemistry.eu

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[1] Sáenz de la Torre J.J.; Flamarique L.; Gomollon-Bel F.; Colacino E. Open Res. Europe 2025, 5, 73. DOI : 10.12688/openreseurope.19848.1
[2] Galant, O.; Cerfeda, G.; McCalmont, A.S.; James, S.L.; Porcheddu, A.; Delogu, F.; Crawford, D.E.; Colacino, E.; Spatari, S. ACS Sustainable Chem. Eng. 2022, 10, 1430-1439.

Between What We Know and What We Wonder: Revealing Key Experimental Variables Shaping the Mechanochemical Synthesis of Molecularly Imprinted Polymers

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Molecularly imprinted polymers (MIPs) are synthetic materials engineered with highly specific recognition sites that mimic natural receptors, enabling selective binding of target molecules—an approach crucial for enhancing drug delivery, diagnostics, and therapeutic monitoring in pharmaceutical and biomedical applications. The conventional synthesis of MIPs using the non-covalent approach is based on a free-radical-mediated chain-growth polymerization of vinyl monomers carried out in an appropriate, often aprotic solvent, of low or medium polarity as reaction media.

Nevertheless, it has been demonstrated by our group that such an imprinted polymer may also be obtained by a solvent-free, mechanochemically-assisted synthesis [1] on an in-house instrumnetal assembly. Therefore, it became obvious that through a better understanding of the underlying reaction pathways and their correlation with common solid-state synthesis process variables a series of novel opportunities may be explored and the experimental constrains related to the conventional solution- based non-covalent MIP synthesis may be circumvented.

Therefore, the next step in the transition towards a scalable solventless molecular imprinting implies method transfer to standardized grinding instrumentation (i.e. ball mills). Using a data-driven experimental design on a model hydrophylic template molecule (atenolol), we sought to progressively reveal currently unresolved correlations with different process variables by identifying hidden patterns and dependencies that impact key performance metrics of such functional polymers, such as reaction yield, imprinting factor, chemoselectivity, rebinding efficiency, and kinetics.

Acknowledgements: This work was supported by a grant of the Ministry of Research, Innovation and Digitization, CNCS-UEFISCDI, project number PN-IV-PI-PCE-2023-0662, within PNCDI IV.

[1] B-C. Iacob, A. E. Bodoki, D. F. Da Costa Carvalho, A. A. Serpa Paulino, L. Barbu-Tudoran, E. Bodoki, *Int. J. Mol. Sci.* **2024**, 25(10), 5504.

O-11

Kinetics of Mechanochemical Reactions

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The use of mechanochemical methods for driving covalent bond formation can reduce solvent waste substantially and lead to products that are not favored under conventional solvothermal conditions, and wider adoption of mechanochemistry could revolutionize how organic chemistry is practiced. A barrier to the wider adoption of mechanochemistry in organic synthesis is the poor understanding of the mechanisms of mechanochemical reactions and how these mechanisms may differ from when the same reagents are reacted solvothermally. In this talk, a new approach to anticipating mechanisms of organic reactions under mechanochemical activation, where rate accelerations are caused by molecular distortions will be discussed[1]. In addition, the concept of "primary" and "secondary" mechanochemical reactions, as a method of examining rates, will be discussed. Examples will be taken from surface studies using nanoscale probes[2], and unpublished work on mechanochemical glycosylations and ionic reactions in solution.

[1] Zholdassov, Y. S.; Kwok, R. W.; Shlain, M. A.; Patel, M.; Marianski, M.; Braunschweig, A. B. "Kinetics of Primary Mechanochemical Covalent-Bond-Forming Reactions" *RSC Mechanochemistry,* **2024** *1*, 11 – 32.

[2] Zholdassov, Y. S.; Luan, Y.; Romero Garcia, S.; Kwok, R. W.; Boscoboinik, A.; Valles, D. J.; Marianski, M.; Martini, A.; Carpick, R. W.; Braunschweig, A. B. "Acceleration of Diels-Alder reactions by mechanical distortion" *Science*, **2023**, *380*, 1053 – 1058.

O-12

Gas-Mechanochemistry for Catalytic Reactions of Gaseous and Solid Substrates

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Gas-mechanochemistry is a promising method for the catalytic reaction of gaseous and solid substrates. Their direct reaction remains challenge in synthetic chemistry, traditionally circumvented by dissolving solid substrates. However, this approach is hampered by significant limitations; dissolution introduces mass transfer barriers and solubility constraints. These limitations are especially pronounced in catalytic processes, where the transport of gaseous substrates to solid catalyst surfaces often becomes rate-limiting and complicates reactor design. Recent advances in mechanochemistry have opened new avenues for overcoming these challenges by enabling the direct and efficient reaction of solid and gaseous substrates under solvent-free conditions. Mechanochemistry facilitates contact between gaseous and solid phases, bypassing solubility and diffusion limitations. Remarkable potential for mechanochemical reactions of gaseous substrates with solid catalysts has been demonstrated in processes such as ammonia synthesis [1], CO_2 hydrogenation[2], and CO oxidation[3].

Building on these advances, we have explored the reactions between solid substrates and various gases. Using direct mechanocatalysis, wherein the catalyst is incorporated in the form of catalytically active milling material, these reactions allow for simple catalyst recovery and reuse as well as also highly efficient transformations. We have established robust protocols for various reduction and oxidation reactions. Hydrogenation of a multitude of functional groups and reductive amination proceed directly with hydrogen under mild conditions. As such, the complete hydrogenation of diphenylacetylene at 0.5 MPa at room temperature is achieved within 20 minutes using a palladium-plated vessel achieving turnover numbers and frequencies several orders of magnitude higher than conventional heterogeneous systems. [4] Furthermore, we demonstrate that atmospheric oxygen suffices as an oxidant for the selective oxidation of alcohols to aldehydes, again with exceptional catalytic performance.[5]

As emphasized by these examples, mechanochemistry provides a versatile tool for gaseous reactions, enabling atom-efficient transformations. Despite these significant advances, the implementation of gaseous reagents in mechanochemical systems introduces new technical challenges, particularly in vessel design to ensure gas containment at elevated pressures. Addressing these engineering hurdles is critical to fully realizing the potential of gas-solid mechanocatalysis.

This presentation will provide an overview of the recent progress and unique opportunities afforded by direct mechanochemical reactions between gaseous and solid substrates, with particular emphasis on their implications for catalysis.

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[1] a) Steffen Reichle, Michael Felderhoff, Ferdi Schüth, *Angew. Chem. Int. Ed.,* **2021**, *60*, 26385-26389. b) Gao-Feng Han, Feng Li, Zhi-Wen Chen, Claude Coppex, Seok-Jin Kim, Hyuk-Jun Noh, Zhengping Fu, Yalin Lu, Chandra Veer Singh, Samira Siahrostami, Qing Jiang, Jong-Beom Baek, *Nat. Nanotechnol.* **2021**, *16*, 325-330.

[2] Runnan Guan, Li Sheng, Changqing Li, Jiwon Gu, Jeong-Min Seo, Boo-Jae Jang, Seung-Hyeon Kim, Jiwon Kim, Hankwon Lim, Qunxiang Li, Jong-Beom Baek, *Nat. Nanotechnol.* **2025**, https://doi.org/10.1038/s41565-025-01949-6

[3] Rene Eckert, Michael Felderhoff, Ferdi Schüth, *Angew.Chem. Int. Ed*., **2017**, *56*, 2445-2448.

[4] Maike Mayer, Maximilian Wohlgemuth, Anastasia Salomé Straub, Sven Grätz, Lars Borchardt, *Angew. Chem. Int. Ed.* **2025**, *64*, e202424139.

[5] Maximilian Wohlgemuth, Sarah Schmidt, Maike Mayer, Wilm Pickhardt, Sven Grätz, Lars Borchardt, *Angew. Chem. Int. Ed.* **2024**, *63*, e202405342.

O-13

Defect-Mediated Mechanochemical Ammonia Production Using Silicon Nitride as Physical Promoter

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The global demand for sustainable ammonia (NH_3) production has intensified research efforts to develop alternatives to the energy-intensive Haber-Bosch process. This conventional approach limits its application to large-scale centralized facilities.[1] With ammonia emerging as a promising hydrogen carrier containing 17.6 wt% hydrogen,[2] there is urgent need for decentralized production technologies that can operate under milder conditions.

Our research introduces a groundbreaking mechanochemical methodology that facilitates ammonia synthesis under near-ambient conditions through mechanical energy input. This paradigm represents a fundamental departure from traditional thermal activation, enabling chemical transformations without harsh requirements. The key innovation lies in employing silicon nitride (Si_3N_4) as a novel physical promoter that induces high-density defects on iron catalyst surfaces through dynamic mechanochemical action.

Silicon nitride was strategically selected due to its exceptional physical properties including superior hardness, minimal sinterability, and outstanding oxidation resistance. [3] Si_3N_4 offers remarkable stability while effectively generating active surface defects on iron (Fe) catalysts. The Si_3N_4-promoted Fe system demonstrates extraordinary catalytic improvement, achieving 5.6-fold higher ammonia yield compared to unpromoted Fe catalyst. Furthermore, extensive stability testing demonstrates sustained catalytic performance across multiple reaction cycles, contrasting with conventional chemical promoters that exhibit significant activity decay.

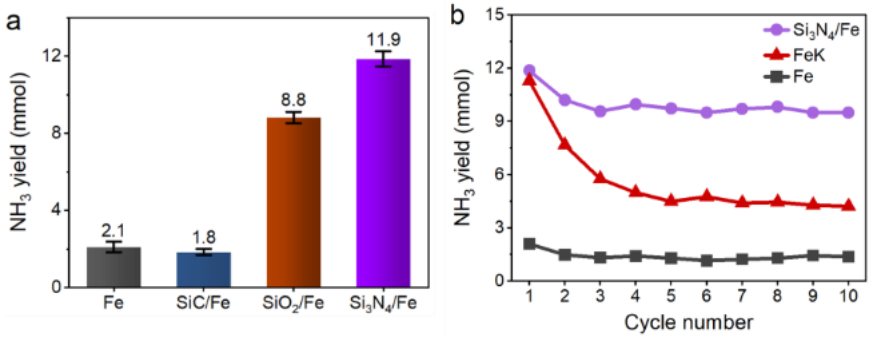

Figure. a, NH_3 Comparative NH_3 yields obtained with various silicon-based promoters. **b,** Stability assessment between physical and chemical promoters.

This mechanochemical methodology opens pathways for decentralized ammonia production. The dynamic defect engineering approach establishes new catalyst design strategies where mechanical activation becomes a controllable parameter for optimizing catalytic performance. This approach also addresses pressing environmental concerns by potentially utilizing silicon waste from photovoltaic (PV) systems for Si_3N_4 production. The methodology represents industrial symbiosis principles, simultaneously addressing ammonia synthesis efficiency and waste management challenges.

[1] Gong, Y. et al. Ternary intermetallic LaCoSi as a catalyst for N_2 activation. *Nat. Catal.* 2018, *1*, 178–185.

[2] Klerke, A., Hviid Christensen, C., K. Nørskov, J. & Vegge, T. Ammonia for hydrogen storage: challenges and opportunities. *J. Mater. Chem.* **2008**, *18*, 2304–2310.

[3] Fukuhara, M., Fukazawa, K. & Fukawa, A. Physical properties and cutting performance of silicon nitride ceramic. *Wear* 1985, *102*, 195–210.

Retsch supporting Mechanochemistry: Equipment, Applications, and Control Strategies

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The presentation covers various aspects of mechanochemistry, including homogenization and quality control, grinding examples of different materials including polymer or lignite materials prior to mechanochemistry trials, and the use of cutting mills and ball mills. Special attention is given to the new and advanced equipment such as the Cutting Mill SM 50, the new Planetary Ball Mill PM 300 and the use of aeration lids or temperature and pressure monitoring, the High Energy Ball Mill Emax or the Mixer Mill MM 500 control which offer enhanced functionality and efficiency. The workhorse MM 400 with new accessories is also highlighted with regards to its outstanding reproducibility and Raman-readiness. The presentation furthermore explores the principles of temperature control in mechanosynthesis and highlights diverse applications in various fields such as recycling, batteries, co-crystallisation scale-up and mechanical alloying.

O-15

Mechanochemistry-Driven Borrowing Hydrogen Processes for Ru-Catalysed N-Alkylation: A Green and Sustainable Pathway

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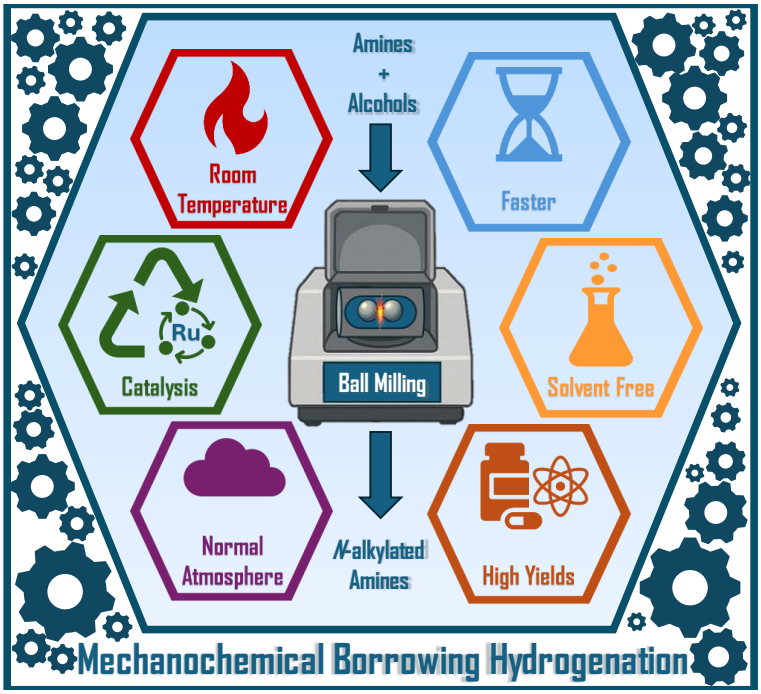
Amines and their derivatives represent a wide range of compounds with important roles in the agrochemical, pharmaceutical, and food sectors. Achieving efficient C–N bond formation is therefore crucial for synthesising these fine chemicals. However, traditional methods of amine alkylation often depend on highly reactive and toxic alkyl halides, raising significant safety and environmental issues.

To overcome these challenges, hydrogen auto-transfer or borrowing hydrogenation (BH) strategies have gained considerable attention for C–N bond formation without external hydrogen gas. In a typical BH process, an amine and an alcohol react with a suitable catalyst at elevated temperature to afford N-alkylated amines. However, BH reactions often require high-boiling solvents (e.g., toluene), temperatures above 100 °C, expensive catalysts, inert conditions, and long reaction times. Although milder protocols have been reported, these constraints largely remain.

Mechanochemistry has become a powerful and sustainable method, allowing solvent-free or minimal-solvent synthesis through liquid-assisted grinding (LAG). Here, we report the first mechanochemical BH reaction catalysed by the commercially available Ru-MACHO complex, achieving N-alkylation of primary amines with aliphatic alcohols under solvent-free, room-temperature, and aerobic conditions. This approach avoids high temperatures, solvents, inert atmospheres, and lengthy reaction times, producing secondary amines efficiently.



[1] Benjamin G. Reed-Berendt, Daniel E. Latham, Mubarak B. Dambatta, Louis C. Morrill, *ACS Central Science*, **2021**, *7(4)*, 570-585.

[2] Sourav Behera, Dipak J. Fartade, Rita Mocci, Michela Matta, Lidia De Luca, Andrea Porcheddu, *Angew. Chem. Int. Ed.* **2025**, *64*, e202508050.

Mechanochemical Deracemization: A Fast and Solvent-Minimized Approach to Enantiopurity

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Today, over 50 % of pharmaceuticals are chiral and ensuring enantiopurity has become a top priority for the pharmaceutical industry.[1] While significant progress has been made in asymmetric synthesis, many compounds are still produced as racemates, necessitating subsequent resolution steps. Traditional resolution methods are inherently wasteful, often discarding the undesired enantiomer. In contrast, deracemization processes—where the undesired enantiomer is racemized and converted to the desired one—offer a sustainable alternative with a theoretical yield and enantiomeric excess (ee) of 100 %.[2]

Deracemizations require an external energy input.[2] Mechanical energy, as first demonstrated by Viedma[3], offers a solvent-based alternative via grinding of a suspension, whereby racemization in solution is followed by deracemization by crystallization; however, these reactions are limited to racemic conglomerates and require large amounts of toxic solvent and long reaction times.[2]

Mechanochemistry has emerged as a sustainable and versatile alternative to traditional solvent-based reactions. Recently, we reported the first examples of mechanochemical deracemization (MCDR), achieving enantiopurity in hours rather than days with minimal solvent use.[4]

In this presentation, I introduce MCDR as a sustainable and generalizable strategy for obtaining enantiopure compounds. I demonstrate its broad applicability via deracemization of several key organic functionalities—ketones, isoindolinones, imines, and esters—as well as an inorganic compound. The critical reaction parameters, including milling material, ball number and size, liquid-assisted grinding (LAG), and the use of a bulk material are discussed. Our findings reveal the pivotal role of these parameters in achieving high ee with reaction times reduced by up to 97 % while solvent use can be reduced by as much as 100 %.

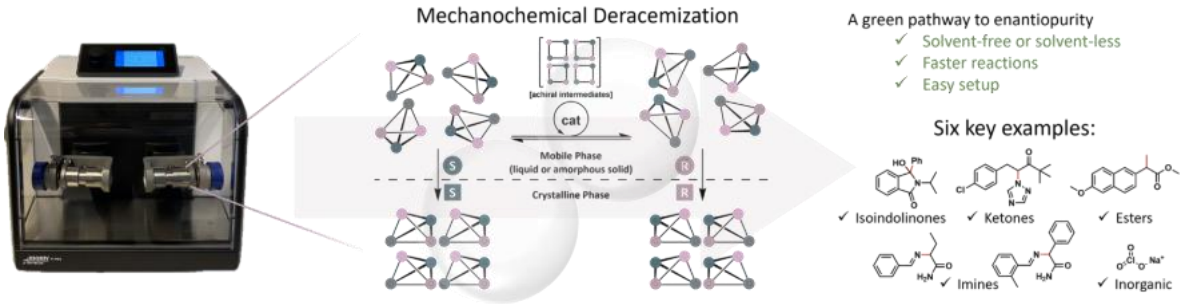

Figure 1: Mechanochemical deracemization (MCDR). MM400 ball mill used for MCDR (left), possible mechanism for MCDR similar to Viedma Ripening (middle), advantages of MCDR and examples studied (right).

[1] E. Sanganyado, Z. Lu, Q. Fu, D. Schlenk, J. Gan, *Water Res.* **2017**, *124*, 527–542.
[2] M. Huang, T. Pan, X. Jiang, S. Luo, *J. Am. Chem. Soc.* **2023**, *145*, 10917–10929.
[3] C. Viedma, Phys. Rev. Lett. **2005**, 94, 065504
[4] J. Gieling, G. Wéry, C. Lopes, J. de Meester, C. Brandel, Y. Cartigny, T. Leyssens, D. M. Baier, *Chem. Eur. J* **2025**, *n/a*, e202404120.

O-17

Overcoming Solution Chemistry Barriers to Metal-Organic Frameworks via Mechanochemistry

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Crystalline metal-organic frameworks (MOFs) have attracted increasing attention as functional porous materials with promising applications in gas storage, gas separation, and catalysis. Their crystallinity is typically achieved through reversible metal–ligand bond formation under solvothermal conditions, which require high temperatures, long reaction times, and excessive amounts of toxic solvents. In this presentation, I will demonstrate how mechanochemistry offers a sustainable and effective alternative for synthesizing MOFs that are challenging to obtain via conventional solvothermal methods. Mechanochemical synthesis harnesses mechanical forces to drive chemical reactions, eliminating the need for high temperatures and solvents. Beyond enabling the preparation of known MOF structures, our work highlights how solid-state mechanochemistry provides access to MOFs that are otherwise difficult to synthesize due to kinetic constraints, ambient temperature requirements, and solid-phase reaction conditions.[1,2,3,4,5]

[1] Tegudeer, Z.; Davenport, L. C.; Kordesch, M. E.; Gao, W.-Y. Harnessing Mechanochemistry for Direct Synthesis of Imine-Based Metal–Organic Frameworks. *J. Am. Chem. Soc.* **2025**, *147*, 13522–13530.

[2] Tegudeer, Z.; Moon, J.; Wright J.; Das, M.; Rubasinghege, G.; Xu, W.; Gao, W.-Y. Generic and facile mechanochemical access to versatile lattice-confined Pd(II)-based heterometallic sites. *Chem. Sci.* **2024**, *15*, 10126–10134.

[3] Salvador, F. E.#; Tegudeer, Z.#; Locke, H.; Gao, W.-Y. Facile mechanochemical synthesis of MIL-53 and its isoreticular analogues with a glance at reaction reversibility. *Dalton Trans.* **2024**, *53*, 4406–4411. (# equally contributed)

[4] Salvador, F. E.; Miller, V.; Shimada, K.; Wang, C.-H.; Wright, J.; Das, M.; Chen, Y.-P.; Chen, Y.-S.; Sheehan, C.; Xu, W.; Rubasinghege, G.; Gao. W.-Y. Mechanochemistry of Group 4 Element-Based Metal−Organic Frameworks. *Inorg. Chem.* **2021**, *60*, 16079–16084.

[5] Plant-Collins, T.; Tegudeer, Z.; Athapaththu, D. V.; Ringle, H.; Chen, J.; Gao, W.-Y. Clustered Defects Enabled by Mechanochemistry for Metal-Organic Frameworks with Enhanced Hierarchical Porosity. *Submitted*, **2025**.

O-18

Mechanochemical Synthesis of Novel Protocatechuate-Based Coordination Polymers and Their Characterization Through 3D Electron Diffraction

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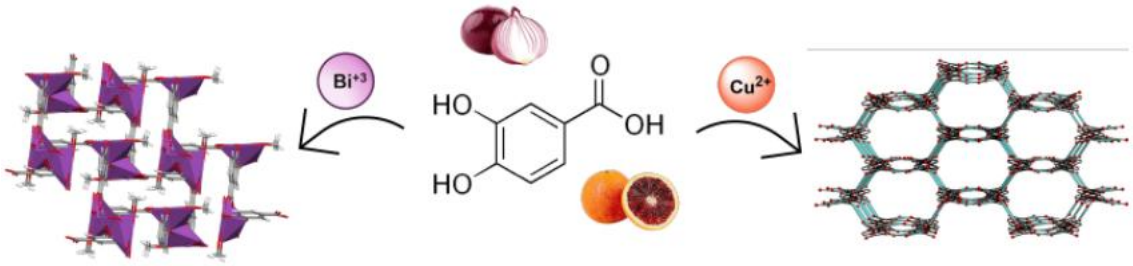
Conventional methodologies for synthesizing coordination polymers (CPs), including MOFs, predominantly involve solvo/hydrothermal techniques. While these methods undeniably yield crystalline products of exceptional quality, their implementation necessitates substantial investments of time and energy, and often overlooks the toxicity or bioavailability of constituent substances. In recent years, mechanochemistry (MC) has garnered considerable attention as a promising alternative. This technique, through localized high-energy solid-state reactions induced by sphere impacts, achieves remarkable performances within significantly compressed timescales. Contemporary advancements in instrumentation even facilitate the precise control of critical parameters, such as the synthesis temperature. Nevertheless, a primary limitation of MC is the structural characterization of the resultant nanocrystalline powders. In this regard, 3D Electron Diffraction (3D ED) has established itself as a powerful tool for the structural elucidation of such materials using a Transmission Electron Microscope (TEM), thereby mirroring the capabilities of a conventional Single-Crystal X-Ray Diffractometer (SCXRD). [1]

In the present investigation, we have elected to integrate the exploration of novel CPs derived from non-toxic, naturally occurring ingredients through the application of MC. Protocatechuic acid, a common antioxidant, has been identified as the primary ligand, while Cu(II), Zn(II), and Bi(III) salts have been selected to form metal nodes due to their distinctive properties. Notably, the utilization of Cu(II) in conjunction with water as a milling aid has facilitated the formation of a novel MOF,[2] and auspicious outcomes have been achieved with Bi(III). In the latter instance, where solvothermal synthesis fails to meet minimum repeatability criteria, MC proves to be an entirely adequate substitute, particularly when employing hot milling. A series of CPs has been synthesized through temperature-controlled milling in various solvents, including water and methanol. Current research efforts are actively pursuing the synthesis of a Bi-based MOF and novel materials incorporating Zn.



[1] M. Gemmi and Arianna E. Lanza, *Acta Cryst*. **2019**, B75, 495-504.
[2] Andrea Sala, Moussa D. Faye Diouf, Danilo Marchetti, Lea Pasquale, and Mauro Gemmi, *Cryst. Growth Des*. **2024**, 24 (8), 3246-3255.

Reactive Extrusion of ZIF-8-Based Biocomposites: Scale-Up Enabled by In Situ Monitoring Advances

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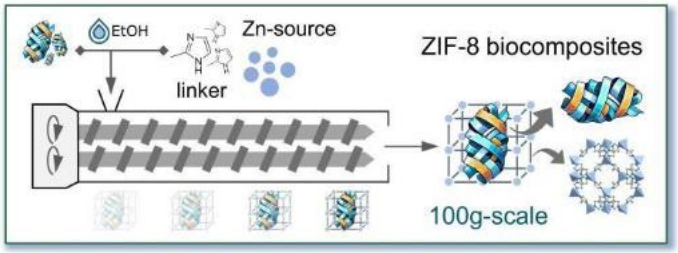
Mechanochemistry offers a solvent-free, sustainable alternative to conventional synthesis of metal-organic framework (MOF) biocomposites, which show great promise for drug delivery, biocatalysis, and biosensing. However, current approaches remain limited to batch-type, gram-scale syntheses that hinder industrial application.

Building on our previous work in *in situ* monitoring of extrusion reactions – including real- time Raman spectroscopy[1] and energy-dispersive X-ray diffraction (EDXRD),[2] which revealed the formation mechanism of zeolitic imidazolate framework-8 (ZIF-8) and enabled process optimization, we developed a scalable solid-state method for producing MOF-based biocomposites via continuous reactive extrusion.

The process begins with rapid model reactions using hand-mixing,[3] allowing encapsulation of diverse biomolecules into ZIF-8, including proteins, carbohydrates, and enzymes, thereby enabling fast screening and optimization of reaction conditions. We then translated the batch protocol to twin-screw extrusion, achieving continuous and scalable synthesis of biocomposites such as bovine serum albumin (BSA)@ZIF-8 with tunable protein content. The resulting materials were highly crystalline and porous, with protein loadings of up to 26 wt% and encapsulation efficiencies as high as 96%. The production rate reached 1.2 kg d^{-1}, surpassing previously reported continuous methods.

To demonstrate industrial viability, we extended the approach to produce shaped ZIF-8 monoliths loaded with hyaluronic acid (HA) in a single-step extrusion. These monoliths maintained their structural integrity during washing and released HA without measurable degradation, as confirmed by size-exclusion chromatography.

This study establishes reactive extrusion as a robust platform for the scalable synthesis and shaping of MOF biocomposites, expanding the toolkit for drug delivery and biocatalytic applications.



[1] Nikita Gugin, José A. Villajos, Olivier Dautain, Michael Maiwald, Franziska Emmerling, *ACS Sustain. Chem. Eng.* **2023**, *11*, 5175-5183.
[2] Nikita Gugin, Kirill Yusenko, Andrew King, Klas Meyer, Dominik Al-Sabbagh, José A. Villajos, Franziska Emmerling, *Chem* **2024**, *10*, 3459-3473.
[3] Nikita Gugin, José A. Villajos, Ines Feldmann, Franziska Emmerling, *RSC Adv.* **2022**, 12, 8940.

EFFECT OF TANGENTIAL-TO-NORMAL STRESS DISTRIBUTION ON THE MECHANOCHEMICAL REGENERATION OF $NaBH_4$

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Although mechanochemistry is maturing as a solvent-free synthesis route, the distinct roles of shear-dominated (tangential) and compression-dominated (normal) stresses inside ball mills remain largely unquantified. Recent analyses point out that most mechanochemical models still collapse the full stress tensor into a single scalar "pressure", leaving the influence of stress anisotropy "surprisingly unexplored"[1]. Insight has begun to emerge from tribological model systems, such as ZDDP tribofilm formation, where shear and normal stresses can be varied independently, but these tests do not replicate the collision statistics of a ball mill and therefore cannot gauge their impact on practical solid-state synthesis [2]. Here we supply an unbiased reactor-scale comparison of tangential- and normal-dominated regimes for the practical solid-state synthesis of NaBH4.

Discrete element method (DEM) mechanical descriptors were leveraged to design experiments with matched constant-speed and constant-power to decouple total power input from the distribution of tangential and normal stressing events in the mechanochemical regeneration of NaBH4. A low fill ratio (6 %), which maximizes tangential dissipation, achieved a record 94% regeneration yield while reducing the ball to powder ratio by 40%, shortening milling time by 38 %, and reducing milling speed by 34% compared to previous literature results.

The specific yield (product per Watt) peaked at 0.28 yield/W, and yield declined linearly with fill ratio ($R^2 > 0.99$); increasing the fill level from 6% to 17% reduced conversion by 40%–50% without any energy benefits (Fig. 1). Because the key DEM descriptors $\{E_n, E_t, f_{col}/n_{ball}\}$ are mill-agnostic, the results provide a transferable blueprint for energy-efficient scale-up and cross-platform benchmarking.

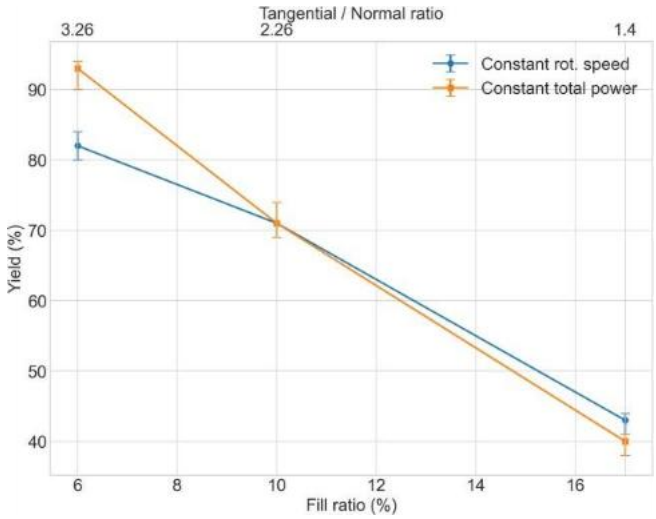

Figure 1. Yield versus fill ratio for experiments run (i) at constant rotational speed (blue) and (ii) at constant total power (orange). The secondary (top) axis converts each fill ratio to the corresponding tangential/normal dissipation ratio inside the jar. Error bars show the variability in yield.

[1] Sergey V. Sukhomlinov, Guido Kickelbick, Martin H. Müser, *Tribol. Lett.* **2022**, *70*, 102.
[2] Lu Fang, Spyridon Korres, William A. Lamberti, Martin N. Webster, Robert W. Carpick, *Faraday Discuss.* **2023**, *241*, 394-412.

O-21

Time-Resolved Simulation of Shock-Driven Reactions in Solids: A Step Towards Predictive Mechanochemistry

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Shock-driven reactions are a subclass of mechanochemical transformations that underpin the functional behaviour of energetic materials, and are proposed to be involved in the origins of life.[1] Yet, the atomic-scale mechanisms that govern how a mechanical shock triggers a physico-chemical transformation remain elusive.[2] This lack in understanding limits our ability to systematically predict and design such reactions, and presents a major obstacle to fully harnessing the advantages of mechanochemistry over traditional chemical processes or designing safer shock-sensitive materials.[3]

In this work, we develop a theoretical framework to simulate the vibrational response of crystalline materials to mechanical shock. Our framework focuses on the dynamics of kinetic energy redistribution among the vibrational degrees of freedom in a crystal via anharmonic phonon scattering (Figure 1). By propagating a time-resolved phonon population model, we track how kinetic energy that is introduced by a high-velocity mechanical impact dissipates throughout the system. Specifically, we identify the vibrational modes into which kinetic energy is preferentially channelled. Using local vibrational mode theory, we quantitatively characterise the chemical nature of the vibrations that become excited and hence identify chemically meaningful internal coordinates that are involved in shock-driven reactions. These simulations open the path to exploring how a mechanical shock affects the electronic structure of crystalline materials and pave the way for the prediction of shock-driven chemical reactions in solids.

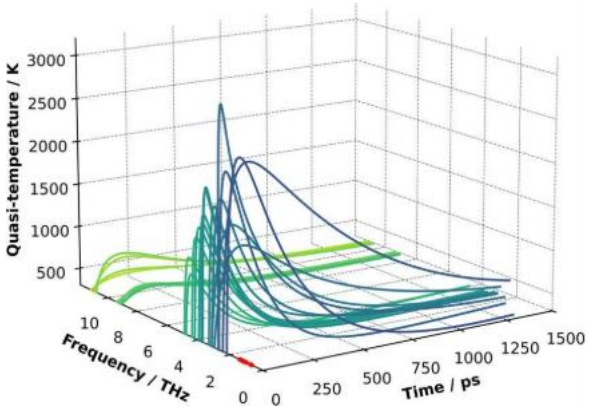

Figure 1. *Evolution of the phonon quasi-temperatures following a simulated mechanical shock into the red phonon modes.*

Our novel methods establish a new mechanistic link between mechanical excitation and chemical reactivity. By resolving the earliest stages of shock-driven transformations at the atomic level, our approach offers a foundation for predictive mechanochemistry, enabling the rational design of mechanically responsive systems, safer energetic materials, and more sustainable solid-state synthetic methods.

1 B.W. Hamilton, M. N. Sakano, C. Li and A. Strachan, *Annu. Rev. Mater. Res.*, 2021, **51**, 101–130.
2 A. A. L. Michalchuk, *Faraday Discuss.*, 2023, **241**, 230–249.
3 A. A. L. Michalchuk, J. Hemingway and C. A. Morrison, *J. Chem. Phys.*, 2021, **154**, 064105.

O-22

A Journey Through Optimization: Applying OFAT, DoE and Machine Learning to Sustainable Amidation

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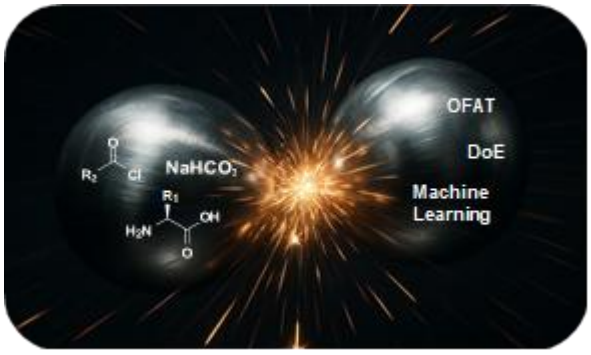
Amide bond formation has been broadly investigated in organic chemistry due to its particular relevance in many fields such as medicinal chemistry.[1] Starting from a carboxylic acid and an amine, this reaction is greatly facilitated when coupling agents are employed to activate the acid. Another possibility is to use an activated carboxylic acid such as an acyl chloride. These approaches, combined with mechanochemistry, have found many developments, since our pioneering works in amidation[2] and peptide synthesis, [3] conducted in a ball-mill, to identify more sustainable ways to form amide bonds.

The optimization of reaction conditions is part of every development of a new synthetic pathway. However, depending on which method is used, the number of experiments to carry out can exponentially increase. The well-known "one factor at a time" method (OFAT) is the perfect example: tens or hundreds of experiments can be necessary to find the best conditions, depending on how many variables are considered. Performing such number of experiments can question the sustainability of the method. As an alternative, statistical approaches have been developed such as Design of Experiment (DoE)[4] or Bayesian-based machine learning optimization (BO)[5].



Inspired by all these recent advances, we herein disclose the optimization of a very simple amidation reaction in a ball-mill, by reacting an amino acid and an acyl chloride, using various available optimization methods. Moreover, to align with the 12 principles of green chemistry, the optimization process was implemented for each molecule within a given scope in a fast and straightforward manner. Such compounds can then be further functionalized to lead to more complex molecules.[6]

1 P. Acosta-Guzmán, A. Ojeda-Porras, D. Gamba-Sánchez, *Adv. Synth. Catal.* **2023**, 365, 4359-4391.
2 T-X. Métro, J. Bonnamour, T. Reidon, J. Sarpoulet, J. Martinez, F. Lamaty, *Chem. Commun.* **2012**, 48, 11781-11783
3 V. Declerck, P. Nun, J. Martinez, F. Lamaty, *Angew. Chem. Int. Ed.* **2009**, 48, 9318-9321.
4 P. M. Murray, F. Bellany, L. Benhamou, D.-K. Bučar, A. B. Tabor, T. D. Sheppard, *Org. Biomol. Chem.* **2016**, 14, 2373-2384
5 M. Lavayssiere, X. Bantreil, F. Lamaty, ChemRxiv. **2025**; doi:10.26434/chemrxiv-2025-zgpnz
6 T. F. Burton, Z. Garisoain, C. Chaix, J. Aassine, E. Virapin, A. Voronova, J. Pinaud, O. Giani, *ACS Omega* **2024**, *9*, 28583-28593.

O-23

Mechanochemical Up-scaling of Co-crystal Syntheses Successful Procedures and Unexpected Problems

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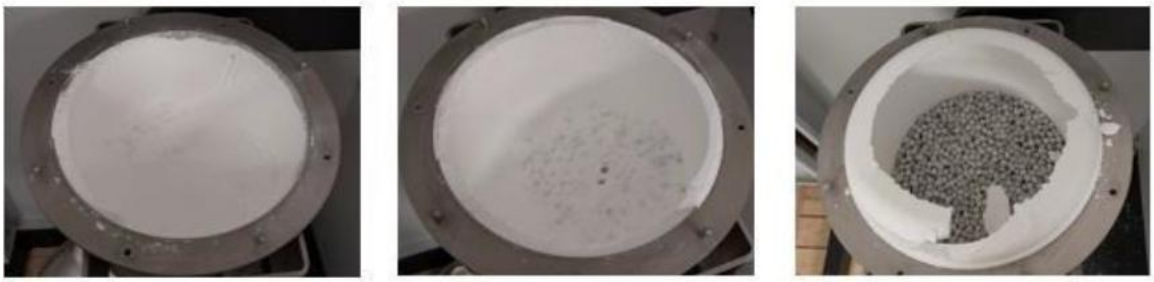
In pharmaceutical research and development, co-crystallisation has emerged as a common strategy for modifying the physicochemical properties of active pharmaceutical ingredients to overcome a variety of challenges in drug formulation. In contrast to conventional solution-based methods, which typically consume significant amounts of solvents and energy, here we discuss a more environmentally friendly and efficient mechanochemical process for the production of co-crystals on a kilogramme scale. Our study pioneers the use of different types of mills for pharmaceutical co- crystal synthesis using the examples of rac-ibuprofen:nicotinamide[1,2] and paracetamol:oxalic acid.[3] Problems that occur are described and possible solutions are shown.



The pictures show the time-dependent changes in the morphology of the reaction mixture of rac-ibuprofen:nicotinamide co-crystals during the mechanochemical synthesis. From left to right: fine powder after 30 min, fine powder and hard solid on the wall after 60 min, hard solid on the wall after 120 min.

The authors (M.F., J.-H.S., D.G.) acknowledge IMPACTIVE (Innovative Mechanochemical Processes to synthesize green ACTIVE pharmaceutical ingredients), the research project funded from the European Union's Horizon Europe research and innovation programme (European Health and Digital Executive Agency) under grant agreement No. 101057286.

1 Jan-Hendrik Schöbel, Frederik Winkelmann, Joel Bicker, Michael Felderhoff, *RSC Mechanochem.* **2025**, *2*, 224-229.

2 Sarah Triller, Frederik Winkelmann, Jan-Hendrik Schöbel, Michael Felderhoff, *RSC Mechanochem.* **2025**, *2*, 538-543.

3 Dhyanesh Gopinath, Master Thesis Ruhr-University Bochum, Germany **2025**.

O-24

Mechanochemical Activation: Revolutionizing the production of Green Cements

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One of the key strategies for reducing the embodied carbon of Portland cement (PC) production is minimizing the use of clinker (the most reactive component of PC), either by increasing the incorporation of cement replacements, referred to as supplementary cementitious materials (SCMs), or by developing clinker-free alternative cementitious binders. A major challenge in both approaches is ensuring that precursor materials are sufficiently reactive, scalable, and consistent in composition and properties. To date, most research on activated resources for use as SCMs or precursors for clinker-free cement production have focused on thermal activation (calcination), which is highly effective when applied to some resources, such as kaolinitic clays. However, this method is less effective for activating minerals other than kaolinite or other industrial wastes, which are highly abundant and currently underutilized.
As an alternative, mechanochemical activation (MCA) is an electrified process that enhances the reactivity of a broader range of materials and minerals through structural disordering and amorphization. MCA has a demonstrated capacity of altering the crystalline structure and chemical reactivity of various clay minerals (e.g., mixed-layer clays, montmorillonite, muscovite, and illite), mine tailings (e.g. gold, silver, iron, etc.) and industrial by-products (e.g. air-cooled slags, ashes, etc.). Moreover, MCA can significantly increase the dissolution rate and reactivity of 2:1 clay mineral, providing a promising pathway for the broader adoption of alternative SCMs in low-carbon cementitious systems production. The MCA process is primarily governed by the grindability of each material, making it a versatile and scalable approach to create SCMs with bespoke reactivities. From a technological point of view, adopting MCA has the potential to provide a wider range of SCMs from different sources and expand the range and adoption of green cements.

This study will deliver an overview on how MCA can be applied to optimize the chemical reactivity of different unreactive materials for use as PC replacements or as precursors for clinker-free cements production. Additionally, it will provide a brief discussion on existing research questions and opportunities for the development in low-carbon cementitious systems. this being discussed by comparing the effectiveness of MCA to the more traditional thermal activation.

O-25

Mechanically Driven N_2O Decomposition over NiO at Near-Room Temperature

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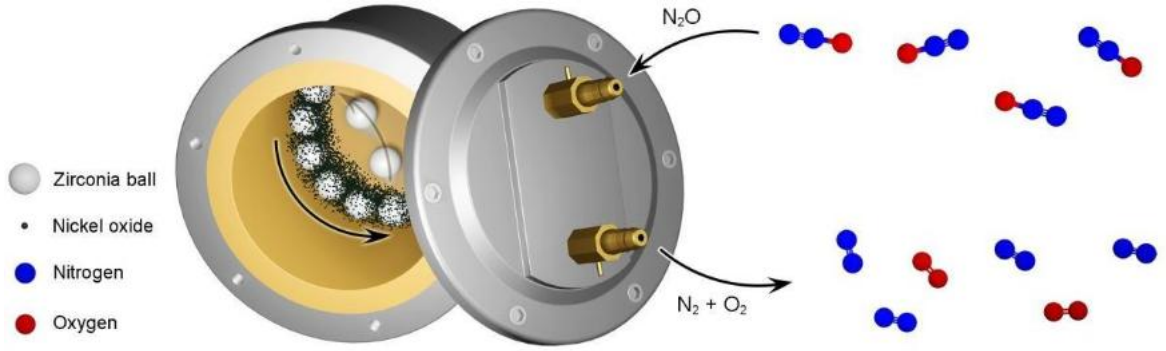
Nitrous oxide (N_2O) is a highly stable greenhouse gas, making its catalytic breakdown particularly difficult under conventional thermal conditions. Traditional methods often require extreme temperatures (~445 °C) and still suffer from low conversion rates. In this work, we introduce a mechanocatalytic route utilizing nickel oxide that enables efficient N_2O decomposition at near- room temperature. Operating at just 42 °C, the process achieves a remarkable conversion efficiency of 99.98% with a reaction rate of 1761.3 mL h^{-1}. This enhanced reactivity under mild conditions is attributed to the mechanically induced non-equilibrium surface states, which facilitate N_2O activation beyond the limitations of thermodynamic equilibrium. Our findings highlight the potential of mechanochemistry as a low-energy alternative for greenhouse gas mitigation.



O-26

Mechanochemical Reduction of Nickel Oxide with Continuous H_2 Flow

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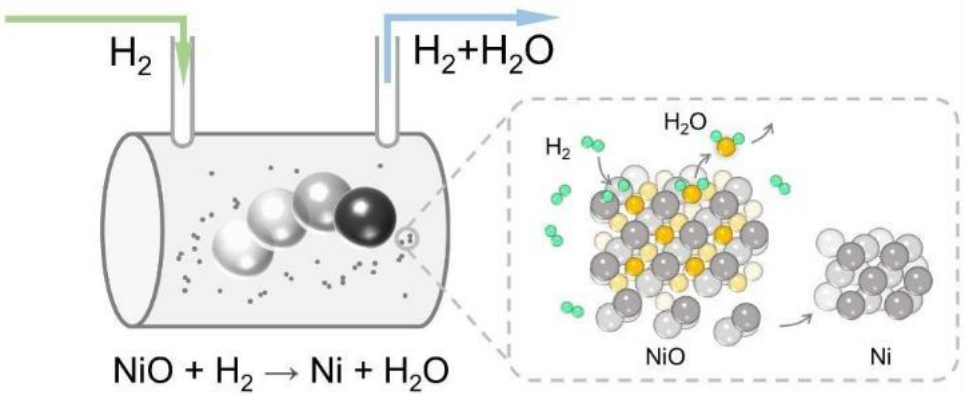
Metal oxide reduction is the key step in the process of both primary and secondary metallurgy to produce raw metals. Direct reduction technology enables carbon-free production of metals with hydrogen as reductant.[1] Due to the high stability of metal oxides, however, metallurgical routes are heavily energy-consuming. Mechanochemistry allows certain reactions to happen under lower temperature and/or lower pressure, with the help of mechanical forces. With less electrical power needed, mechanochemistry leads to wide potentials for more sustainable metal oxide reduction. [2] In this work, the possibility of mechanochemically reducing metal oxides at room temperature with hydrogen is confirmed via ball-milling using NiO as an example. In a commonly used planetary ball-mill system, NiO reacts mechanochemically with hydrogen under formation of water vapor, which leads to a partial reduction of NiO after reaching equilibrium. The generated water is proved to hinder further reduction of NiO. A home-built gas-flow shaker-mill system was then adopted, which enables the continuous removal of the generated water vapor. The reduction of nickel oxide could therefore be realized with a much higher reduction degree. In order for a faster water desorption, mild heating (<100 °C) was adopted, leading to a significantly shortened reduction time. 62 wt% Ni was reduced after 11 h of ball milling with H_2 flow at room temperature, and 88 wt% after 10 h of milling with mild heating (100 °C). Efficient mechanochemical reduction benefits from the generation of abundant oxygen vacancies, increased surface area, continuously renewed particle surface, and constant removal of moisture.



[1] D. Raabe, *Chem. Rev.*, **2023**, 123, 2436-2608.
[2] G. B. Schaffer; P. G. McCormick, *Appl. Phys. Lett.*, **1989**, 55, 45-46.

O-27

Mechanochemical/Thermal Synthesis of Ternary Chromium Selenospinels

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Ternary chromium spinels ACr_2X_4 (A = Cd, Co, Cu, Fe, Hg, Zn; X=S, Se, Te) are characterized by a great versatility of magnetic, electrical and magnetoresistive properties because of their relatively high critical Curie temperatures, T_C above the room temperature and the ability to contain various atoms in the crystal structure[1]. Considering that the presence of a ferromagnetic arrangement can have a strong influence on the thermoelectric properties[2] a series of ternary chromium selenospinels with composition $Cu_{1-x}Zn_xCr_2Se_4$, where x = 0; 0.2; 0.5; 1 have been prepared by simple and relatively fast mechano/thermal synthesis i.e. by high-energy milling for 16 hours and 1 min heating to 473 K from elemental powder precursors. The products were identified by X-ray diffraction. The samples for physical measurements were hot-pressed. The electrical conductivity, Seebeck coefficient, total thermal conductivity, and figure of merit ZT as a function of the temperature of synthesized $Cu_{1-x}Zn_xCr_2Se_4$ compounds were measured and compared. The positive sign of the Seebeck coefficient suggested the p-type conductivity. Lattice thermal conductivity was
≈ 1.6 $W.m^{-1}K^{-1}$ at room temperature and decreased with increasing temperature, so the $Cu_{1-x}Zn_xCr_2Se_4$ spinels seemed from this point of view good candidates for TE applications. Although the ZT achieved a more than 6-fold increase to 0.06 at 575 K for $ZnCr_2Se_4$, Zn-doping has not produced the desired effect. The changes in the magnetic state of prepared selenospinels from ferromagnetic to antiferromagnetic were also recorded.

Acknowledgements: This work was realized within the Slovak Grant Agency (VEGA 02/0036/23).

[1]Marco Esters, Andreas Liebig, Jeffrey J. Ditto, Matthias Falmbigl, Manfred Albrecht, David C. Johnson, *J. Alloys Compd.* **2016**, *671*, 220-225.
[2]Naohito Tsujii, Takao Mori, *Appl. Phys. Express*, **2013**, *6*, 043001.

Formation of Racemic Phases of Amino Acids by Liquid-Assisted Resonant-Acoustic Mixing Monitored by Solid-State NMR Spectroscopy

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Mechanochemistry has become a fundamental method in a variety of fields such as biology and chemistry. This technology can have a huge potential for future sustainable developments due to the absence of organic solvents, new synthesis pathways or potentially shorter reaction times.[1] Despite its current popularity, the mechanisms behind mechanochemical reactions are often not very well understood.

In previous work,[2] we investigated molecular-recognition processes of molecules capable of forming racemic phases in ball mill devices. The combination of mechanochemistry and solid-state nuclear magnetic resonance (NMR) spectroscopy also allowed to better analyze proceedings of molecular-recognition events. We now extended our investigations to mechanochemically-induced racemic-phase formations of amino acids, in our case focusing on serine and alanine, in a resonant acoustic mixing (RAM) device. This technique shares some similarities with ball milling while providing a less harsh reaction environment.[3] This may be beneficial for reactions that cannot handle the forces of the ball mill, but still require efficient mixing. When using RAM, our data reveals the importance of the optimization of reaction parameters, which is in our case the importance of adding small amounts of solvents (here water) to facilitate the underlying solid-state recognition processes. To further investigate the role of water, we performed deuterium magic-angle spinning NMR experiments. This also revealed that RAM enables efficient hydrogen to deuterium exchange in enantiopure serine, paving the way to deuterate organic compounds in the RAM device.

1 J.L. Howard, Q. Cao, D.L. Browne, *Chem. Sci.*, **2018**. 9(12), 3080-3094.
2 C. Quaranta, I.D.A.A. Silva, S. Moos, E. Bartalucci, L. Hendrickx, B.M.D. Fahl, C. Pasqualini, F. Puccetti, M. Zobel, C. Bolm, T. Wiegand, *Angew. Chem. Int. Ed.* **2024**, *63*, e202410801.
3 Resodyn Acoustic mixers, White Paper on "*ResonantAcoustic®Mixing*" (https://resodynmixers.com/2010/02/15/white-paper-ram-mixingtechnology/).

Prediction of Low Melting Eutectic Formation as Intermediate for Mechanochemical Synthesis

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Multicomponent molecular materials, such as cocrystals, are largely used in high valuable arenas such as pharmaceutical, food and agrochemical industries to finely tune physical properties of the active compounds or to induce synergistic effect to its biological properties.
Although cocrystal are often synthesized via mechanochemical protocols, it is not clear yet how the reaction proceeds within the jar. Indeed, once the jar is closed, it is hard to directly probe the reaction and, although many studies appeared in recent literature focusing on the in-situ monitoring of different mechanochemical reactions[1-2], the mechanism of the reaction paths is far from being understood in deep detail.
It has been suggested that proceeding through liquid intermediates may facilitate the activation of the mechanochemical process.[3] The *a priori* knowledge of whether a reaction mixture could give rise to a low melting eutectic mixture would be of great impact on the experimental design of mechanochemical protocols.
Here an accurate time-resolved investigation of cocrystals syntheses via X-Ray Powder Diffraction and Low-frequency Raman Spectroscopy is proposed, with a focus on the estimation of the likelihood of the low melting eutectic formation by means of a friendly predictive tool (PoEM, i.e. Predictor of Eutectic Mixtures) we have recently developed[4] based on the intrinsic thermodynamic parameters of the coformers and the intermolecular interactions involved.

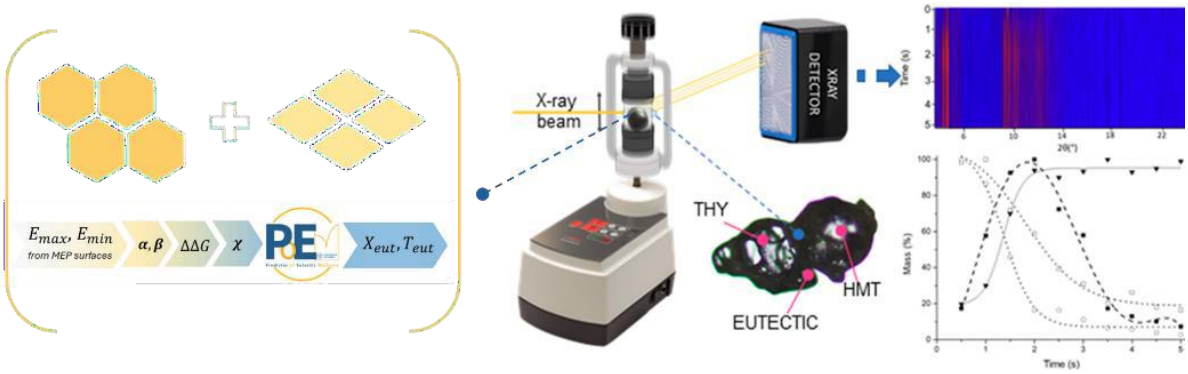

Figure 1. Workflow of cocrystal design and time resolved in-situ monitoring of mechanochemical reaction proceeding though low melting eutectic intermediate

[1] G. I. Lampronti, A. A. L. Michalchuk, P. P. Mazzeo, A. M. Belenguer, J. K. M. Sanders, A. Bacchi, F. Emmerling, *Nature Comm.,* **2021**, *12*, 6134
[2] P. P. Mazzeo, G. I. Lampronti, A. A. L. Michalchuk, A. M. Belenguer, F. Emmerling, *Faraday Discuss.*,**2023**, *241*, 289
[3] P. P. Mazzeo, M. Prencipe, T. Feiler, F. Emmerling, A. Bacchi, *Crystal Growth & Design* **2022**, *22*, 7, 4260- 4267
[4] M. Prencipe, P. P. Mazzeo, A. Bacchi, *RSC Mechanochem.*,**2025**, *2*, 61

POSTERS

Mechanochemical Deracemization: A Solvent-Minimized Approach to Enantiopure Isoindolinones

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In the pharmaceutical sector, the efficacy of many drugs hinges on their chiral nature, with approximately half of those on the market containing a chiral center vital for their effectiveness. This is pivotal as the enantiomer exhibiting the desired pharmacological activity may not always guarantee the same effect in its counterpart; it could be inert or even induce adverse reactions. [1]

As a result, regulatory bodies such as the FDA and EMA strongly advocate for the development of enantiopure drugs. [2] The predominant method to achieve this is the physical separation of enantiomers from a racemic mixture. However, this approach typically yields a maximum of 50%. [3] Alternatively, if the compound is prone to racemization, the unwanted enantiomer can be converted into the desired one, a process known as deracemization, potentially reaching a 100% yield. [4]

Chiral molecules have two potential crystalline forms: conglomerates (where enantiomers form separate structures) or racemic (where both enantiomers coexist in the same structure). Viedma ripening, a process involving grinding the solid in suspension in a solvent, allows for the deracemization of conglomerates. [5]

In 2014, Steendam et al. investigated the deracemization process of isoindolinones (Figure 1). [6] In our study, we take this process to the next level, removing the solvent. We do so, transposing the Viedma-ripening process to a solvent-free medium, in a mechanochemical deracemization process. We highlight the numerous advantages this approach offers over classical Viedma-ripening. [7]

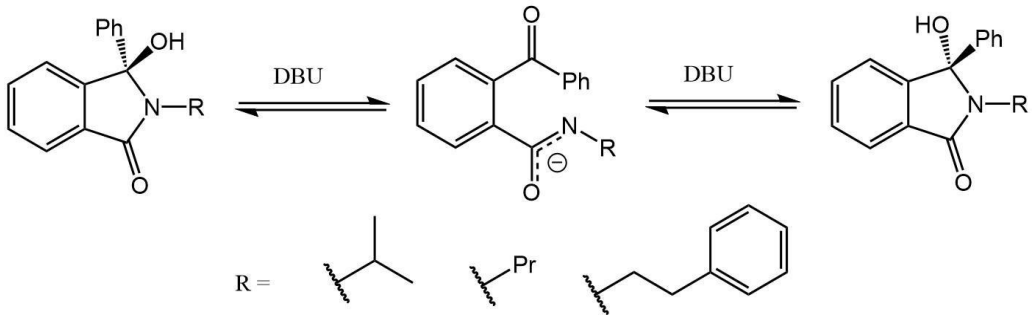

Figure 1. These compounds crystalize as conglomerate and can racemize via an achiral intermediate under the action of a base (DBU).

[1] Sanganyado, E. et al. *Water Res.* **2017**, 124, 527–542.

[2] *Chirality* **1992**, 4, 338–340 ; *European Medicines Agency Guideline* **1994**, 381–391.

[3] Lorenz, H.; Seidel-Morgenstern, A. *Angew. Chemie - Int. Ed.* **2014**, 53, 1218–1250.

[4] Nohira, H.; Sakai, K. *Dordrecht*, **2004**, 165–191.

[5] Sögütoglu, L.C et al. Chem. Soc. Rev. 2015, 44, 6723–6732.

[6] Steendam, R.R.E. et al. *Chem. - A Eur. J.* **2014**, 20, 13527–13530.

[7] Gieling, J. Et al. *Chem. Eur. J.* **2025**, 31, e202404120

P-02

Green Mechanochemical Synthesis of a Novel Drug-Drug Eutectic Mixture of Acetylsalicylic Acid and Pyrazinamide for Enhanced Drug Delivery

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This study presents the successful mechanochemical synthesis of a novel drug-drug eutectic mixture (DDEM) of acetylsalicylic acid (ASA) and pyrazinamide (PZA). This green, solvent-free approach offers an efficient and sustainable method for creating enhanced drug delivery systems. The DDEM was thoroughly characterized using various techniques, including DSC, PXRD, and FT-IR. Binary phase diagrams confirmed its formation at a 2:1 ASA:PZA molar ratio. A key finding was the significant increase in aqueous solubility: ASA's solubility improved by 61.5% and PZA's by 85.8% within the DDEM compared to the pure drugs. DFT calculations also provided insights into the molecular interactions.

These results highlight the substantial potential of mechanochemically synthesized DDEMs to improve drug properties and delivery, offering a viable and environmentally friendly alternative to traditional methods. The synergistic interaction within this eutectic mixture, efficiently formed through mechanochemistry, warrants further investigation into its potential for enhanced therapeutic efficacy [1]

Acknowledgments: This work received financial support from the PT national funds (FCT/MEIC, Fundação para a Ciência e Tecnologia and Ministério da Educação, Ciência e Inoivação), through the project UID/50006 Laboratório Associado para a Química Verde – Tecnologias de Processos Limpos. M. C. Sarraguça thanks FCT for funding through the Individual Call to Scientific Employment Stimulus DOI 10.54499/2022.01388.CEECIND/CP1724/CT0003. C. C. Santos acknowledges the support of FAPEMA (Grant INFRA-02050- 21) and CNPq (Grant N° 310127/2023-8). Ribeiro acknowledges the support of FAPEMA (Grant UNIVERSAL-06736/22). The researchers are grateful to the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001, the Fundação de Amparo à Pesquisa e Desevolvimento Científico do Estado do Maranhão (FAPEMA) and Digital Research Alliance of Canada for providing computational results for the development of the theoretical study, through a collaboration with Canadian researchers.

[1] Queiroz, L. H. S.; Lage, M. R.; dos Santos, C. C.; Sarraguça, M. C.; Ribeiro, P. R. S. Thermodynamic and Structural Characterization of a Mechanochemically Synthesized Pyrazinamide-Acetylsalicylic-Acid Eutectic Mixture. Pharmaceuticals 2025, 18 (2). DOI: ARTN 21110.3390/ph18020211.

Mechanochemical Deracemization of a Naproxen Ester

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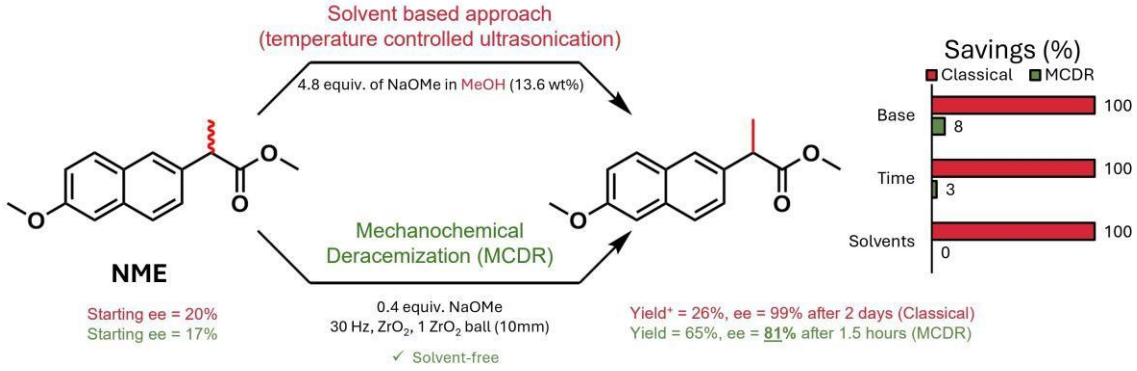
Enantiopurity is of paramount importance in the pharmaceutical industry, where the therapeutic efficacy and toxicity of chiral drugs can depend heavily on their stereochemistry. Naproxen, a widely used non-steroidal anti-inflammatory drug, exemplifies this: while the S-enantiomer is pharmacologically active, the R-enantiomer is hepatotoxic. Although asymmetric synthesis has advanced significantly, industrial production still often relies on racemic intermediates, followed by resolution processes that inherently waste 50% of the material.

To address this inefficiency, deracemization strategies such as Viedma ripening (VR)[1]—attrition-enhanced deracemization—have been developed. However, conventional VR processes are hindered by long processing times and the use of large volumes of toxic solvents. In this study, we demonstrate a novel, solvent-free, mechanochemical approach to deracemization using Naproxen methyl ester as a model compound and compare it to the traditional solvent based approach[2]. By adapting Viedma ripening to a ball milling setup, we achieve complete deracemization within just 90 minutes—significantly faster than traditional methods—while eliminating the need for solvents entirely.

This work,[3] highlights a promising pathway toward greener, safer, and more scalable enantio-separation in pharmaceutical manufacturing, aligning well with the goals of sustainable chemistry and the mechanochemistry community.



[1] C. Viedma, Phys. Rev. Lett. 2005, 94, 065504.

[2] W. L. Noorduin, B. Kaptein, H. Meekes, W. J. P. van Enckevort, R. M. Kellogg, E. Vlieg, Angew. Chem. Int. Ed. 2009, 48, 4581–4583.

[3] J. Gieling, G. Wéry, C. Lopes, J. de Meester, C. Brandel, Y. Cartigny, T. Leyssens, D. M. Baier, Chem. – Eur. J. 2025, 31, e202404120.

Mechanochemical Design of High-Performance Battery Electrodes: A Route Toward Scalable and Solvent-Free Synthesis

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Mechanochemistry offers a promising pathway toward sustainable and scalable synthesis of battery electrode materials by eliminating solvents and reducing thermal processing steps. In this work, we present a solvent-free mechanochemical synthesis of lithium iron phosphate ($LiFePO_4$, LFP) and silicon-carbon (Si/C) composite electrodes as case studies for cathode and anode development, respectively[1]. High-energy ball milling was used to activate precursor materials, followed by moderate annealing under controlled atmosphere to enhance crystallinity[2].

The structural and morphological characterization (XRD, SEM, TEM, Raman) confirmed the formation of phase-pure LFP with uniform nanoscale morphology and intimate carbon coating. Si/C anodes prepared via mechanical alloying demonstrated a homogenous dispersion of silicon nanoparticles within the carbon matrix. Electrochemical testing in coin cell configurations revealed that the LFP cathode achieved an initial discharge capacity of 156 mAh/g at 0.1C and retained over 90% capacity after 200 cycles at 1C. The Si/C anode delivered a reversible capacity of 3100 mAh/g (based on Si) with stable cycling performance and suppressed volume expansion.

This study highlights the potential of mechanochemical synthesis for eco-friendly battery production by reducing solvent usage, energy consumption, and synthesis time while maintaining competitive electrochemical performance. Our results provide a green alternative for the scalable fabrication of next-generation battery materials[3].

[1] James, Stuart L., Christopher J. Adams, Carsten Bolm, Dario Braga, Paul Collier, Tomislav Friščić, Fabrizia Grepioni et al. "Mechanochemistry: opportunities for new and cleaner synthesis." *Chemical Society Reviews* 41, no. 1 (2012): 413-447.

[2] Tan, Davin, and Tomislav Friščić. "Mechanochemistry for organic chemists: an update." *European Journal of Organic Chemistry* 2018, no. 1 (2018): 18-33.

[3] Park, Inkyu, Hanbyeol Lee, and Oh B. Chae. "Synthesis Methods of Si/C Composite Materials for Lithium-Ion Batteries." *Batteries* 10, no. 11 (2024).

P-05

Harnessing extrusion forces for the thermo-mechanochemical synthesis of amides through Machine Learning.

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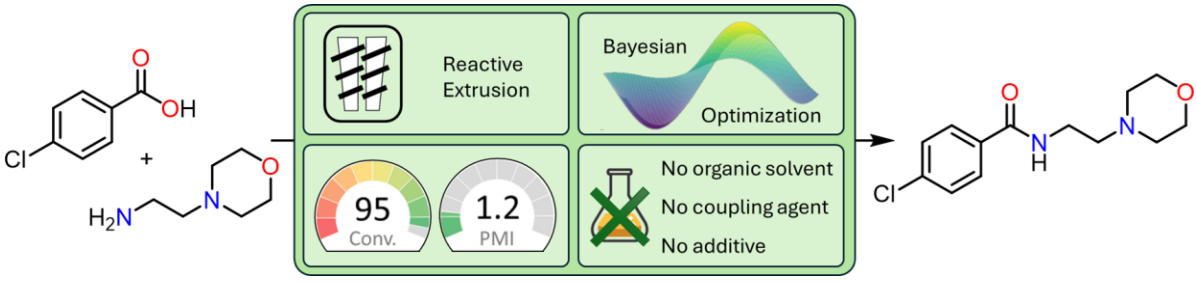
In the field of green chemistry, mechanochemistry has emerged as a powerful and sustainable approach in fine chemical synthesis, harnessing mechanical forces to drive chemical reactions.[1] Using a twin-screw extruder as a chemical reactor we explored the potential of reactive extrusion for the synthesis of amides[2] and peptides[3] in both case reducing by tenfold the amount of waste generated compared to conventional methodologies. To improve further, it was decided to form the amide bond without coupling agents or catalysts, while also leveraging Bayesian Optimization to develop tailored conditions for this thermo-mechanochemical synthesis of amides. A twin-screw extruder equipped with a recirculation valve and heating elements allows for precise control over reaction parameters such as reaction time and temperature. This accuracy was a key point in unlocking new optimization methodologies through Machine Learning, which along with a careful design of the target produces a strong tool to quickly define reaction condition that accommodate both high conversion and excellent sustainability metrics. This recent and innovative approach allowed us to develop efficient processes for the synthesis of amides free from solvents, coupling agents or any additives.[3] This solvent-less flow mechanochemistry approach aligns seamlessly with the industry's growing demand for productive and sustainable manufacturing processes.



1. Bento, O.; Luttringer, F.; Mohy El Dine, T.; Pétry, N.; Bantreil, X.; Lamaty, F. Sustainable Mechanosynthesis of Biologically Active Molecules. *Eur. J. Org. Chem.* **2022**, *2022*, e202101516.
2. Lavayssiere, M.; Lamaty, F. Amidation by Reactive Extrusion for the Synthesis of Active Pharmaceutical Ingredients Teriflunomide and Moclobemide. *Chem. Commun.* **2023**, *59*, 3439–3442.
3. Mohy El-Dine, T.; Lavayssiere, M.; Adihou, H.; Subra, G.; Métro, T.-X.; Ludemann-Hombourger, O.; Lamaty, F. Synthesis of Peptides by Reactive Extrusion. Application to the Continuous and Solventless Preparation of Aspartame. *ChemistryEurope* **2024**, *2*, e202400007.
4. Lavayssiere, M.; Bantreil, X.; Lamaty, F. Optimizing Reactions in Reactive Extrusion by a Bayesian Approach: The Case of Thermal Amidation. *ChemRxiv* **2025**. https://doi.org/10.26434/chemrxiv-2025-zgpnz.

P-06

Mechanochemical Synthesis of Cr_3C_2: Investigating the Role of Pressure and Temperature

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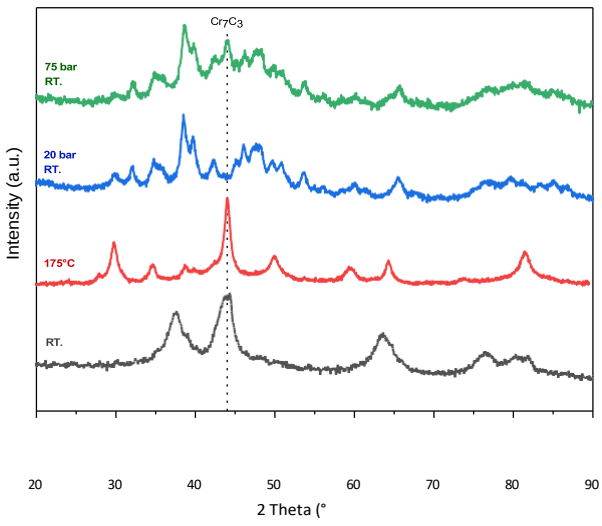
Chromium carbides is a well establish material known for its exceptional hardness, corrosion resistance, and thermal stability, making it highly valuable for applications in protective coatings, cutting tools, and wear-resistant components. [1, 2] Traditional synthesis methods for Cr_3C_2 often require high temperatures, strongly reducing gas atmospheres, and long reaction times, all of which contribute to excessive energy consumption, equipment corrosion, and high production costs. We have recently demonstrated that mechanochemistry offers a promising, energy-efficient alternative for synthesizing transition metal carbides. Through our approach, we not only improved the synthesis procedure but also introduced a new property to the final material: high electrical conductivity. This advancement positions Cr_3C_2 as a potential alternative filler material for fuel cell applications. Despite the potential of mechanochemical approaches, achieving single-phase Cr_3C_2 via ball milling remains challenging due to incomplete reactions and the formation of mixed phases. As a final step, annealing at around 800 °C is required to obtain the pure phase. This study aims to address these limitations by investigating the effects of pressure and applied temperature on the mechanochemical synthesis of Cr3C2.[3,4] The main objective is to identify the key driving force pressure or temperature for pure phase Cr3C2 formation and to establish optimal conditions for its synthesis. The synthesis was carried out using a P6 Fritsch planetary ball mill under an inert atmosphere. For the temperature-controlled experiments, a shaker mill equipped with external heating and continuous argon flow was employed to maintain an inert environment. The products were characterized thoroughly. The results show that applying pressure significantly improves the reactivity of Cr_3C_2, which is attributed to the increased frequency and energy of particle collisions under high-pressure conditions. In contrast, temperature-assisted milling showed only marginal improvements in reaction kinetics and did not substantially affect phase selectivity, indicating that pressure served as a more dominant driving force than temperature. Importantly, the final electrical conductivity values remained within comparable ranges across different synthesis conditions, suggesting that conductivity performance would not be compromised for practical applications. This study provides new insights into pressure-applied mechanochemistry as a viable route for synthesizing transition metal carbides.



[1] S. T. Oyama (Ed.) The Chemistry of Transition Metal Carbides and Nitrides, Springer Netherlands, Dordrecht, 1996.
[2] M. Lei, H. Z. Zhao, H. Yang, B. Song, W. H. Tang, Journal of the European Ceramic Society 2008, 28, 1671.
[3] J. Meyers, Ö. Agbaba, H. Buchholz, F. Schüth, Submitted.
[4] W. Schmidt, P. Losch, H. Petersen, M. Etter, F. Baum, J. Ternieden, C. Weidenthaler, RSC Mechanochem. 2025, 2, 273.

P-07

CZA Catalysts Synthesis by One-Pot Mechanochemical Route Applied to Carbon Dioxide to Methanol Conversion

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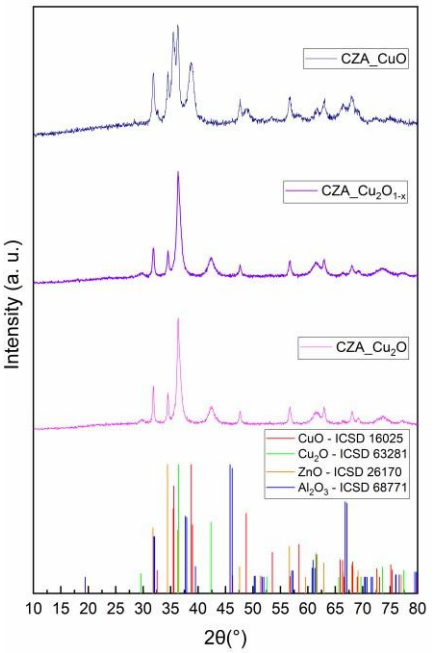
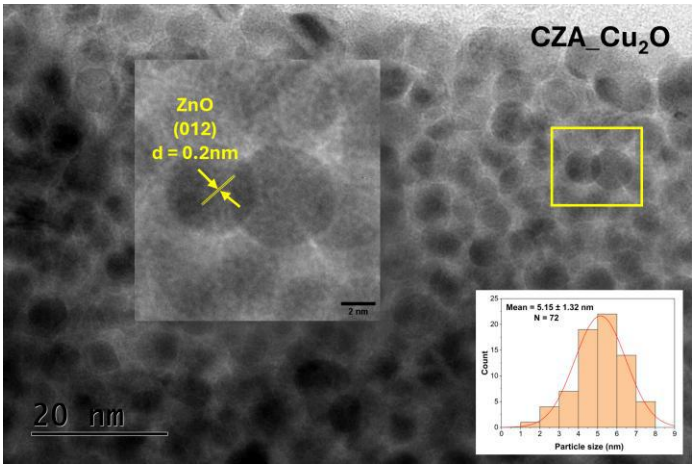
Defined as a simple, eco-friendly, and rapid synthesis approach, the mechanochemistry field





has been growing as an alternative to solution-based methods for obtaining various distinguished materials.[1] Still within the green chemical approach, CZA catalysts —materials based on a mixture of aluminum, copper, and zinc oxide —have been widely employed in the industrial segment for carbon dioxide conversion, yielding satisfactory results.[2] The percentage composition of this catalyst and any inserted metals or other compounds in the sample structure is unknown and is kept secret by the patent. In this work, combining these two aspects, CZA materials were synthesized employing the same pre-established mechanical conditions on a planetary milling equipment and oxides mass composition (6:3.5:5 – Cu:Al:Zn), changing exclusively the nature of the copper precursor (bulking and defective copper oxide(I) and bulking copper oxide (II). TEM and XRD were employed to confirm the maintenance of the initial bulking crystalline structure of oxides separately and to verify the nanoscale material reduction after the milling procedure.

Once these materials were successfully synthesized, their application was based on carbon dioxide conversion, employing a continuous flow reactor with a hydrogen reduction pre-treatment to ensure a clear surface during the catalysis. Promising initial results have been achieved for this class of samples, mainly in the conversion analysis, demonstrating a potential study employing mechanochemistry as a green alternative to obtaining CZA compounds. Moreover, after reactions, these compounds were structurally characterized to observe possible changes with the catalysis.

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[1] X. Liu, Y. Li, L. Zeng, X. Li, N. Chen, S. Bai, H. He, Q. Wang, C. Zhang, *Advanced Materials* **2022**, *34*. [2] S. Kattel, P. J. Ramírez, J. G. Chen, J. A. Rodriguez, P. Liu, *Science (1979)* **2017**, *355*, 1296.

P-08

Supported Palladium NPs Over Oxide Zinc: Structure Analysis of Potential Catalysts Obtained From One-Pot Planetary and Vibratory Milling

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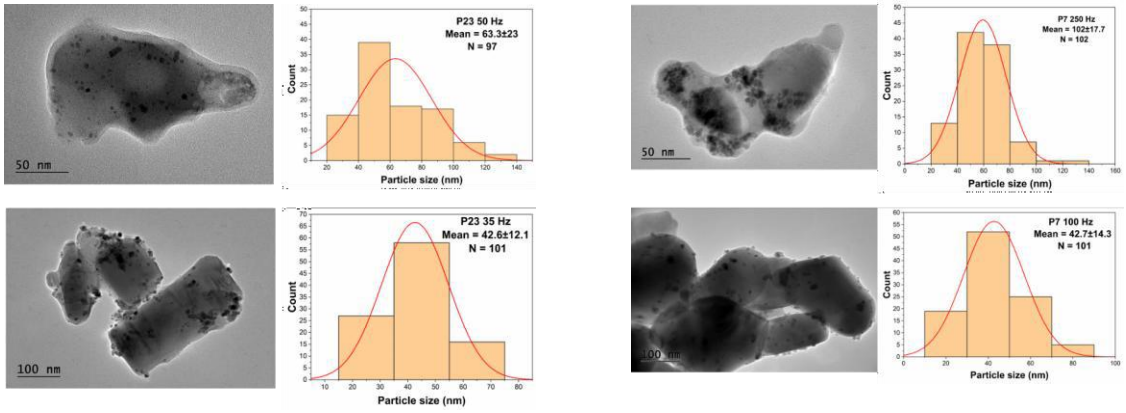
Mechanochemistry has been employed as a green-friendly method to synthesize different potential materials, including metal-organic frameworks (MOFs), metallic nanoparticles (NPs), organic compounds etc.[1,2,3] Characterized as a simple and fast route, the interest in this alternative method has been increasing significantly. However, once the material of interest is chosen, various variables within the mechanical routes, such as frequency, number of balls, jar volume, and milling device, seem uncertain. Could the selected group of variables in the synthesis of a catalyst affect the final product of the reaction and limit its potential application? In this work, it was observed how the structure of palladium nanoparticles supported over oxide zinc (Pd@ZnO) is modified by milling devices and parameters. TEM images demonstrated that, independent of the milling device, the increase in frequency resulted in bigger palladium NPs.



Once these materials were obtained from the milling procedures, they were employed as catalysts in the Suzuki-Miyaura cross-coupling reaction as a model to observe the relationship between particle size and the conversion. The reaction was performed in a solution of H_2O/EtOH (1:1 v/v) using 4-bromobenzaldehyde and phenylboronic acid as reactants in a basic medium. The product of interest, given by 4-biphenyl carboxaldehyde, was quantified by Gas chromatography coupled with a flame ionization detector (GD-FID). Preliminary results demonstrating clear evidence of the increase in conversion occur in dependence on smaller particle size in both milling equipment. This experiment indicates that at high frequencies, these particles tend to agglomerate in a milling medium, also decreasing the surface area of catalytic sites as a consequence. This means that higher milling energy does not necessarily produce smaller particles. Also, it was demonstrated the importance of the integrity of the metal oxide support. Replacing ZnO by Al_2O_3 led to a decrease in the reaction conversion, even for smaller PdNPs, due to a strong reduction in the particle size of the support, resulting in a Pd dispersed within Al_2O_3 with hindered surfaces.

[1] E. Hao, G. C. Schatz and J. T. Hupp, J Fluoresc, 2004, 14, 331–341. [2] Y. Xia, X. Xia and H.-C. Peng, J Am Chem Soc, 2015, 137, 7947–7966. [3] A. R. Tao, S. Habas and P. Yang, Small, 2008, 4, 310–325

P-09

The Mechanochemical Modification of Biomaterials

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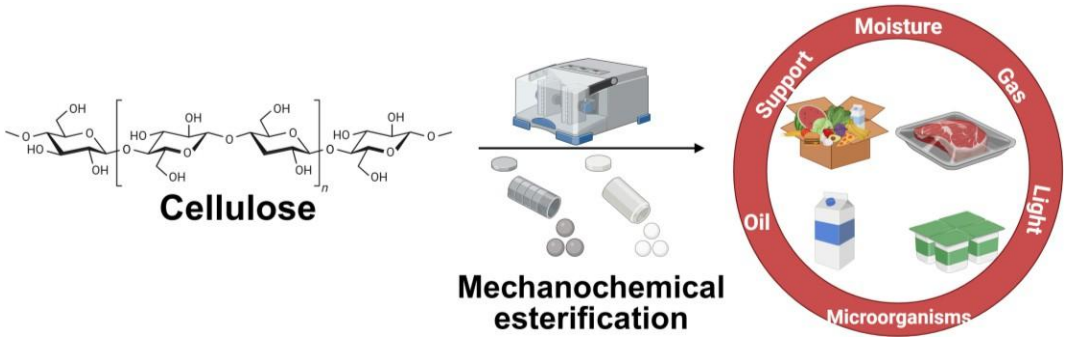
Cellulose, as a high-potential renewable feedstock, holds a significant promise for the development of sustainable thermoplastic, biocomposite, and textile materials. [1,2] Esterification of cellulose represents a crucial pathway for converting it into functional materials with desirable properties. [3, 4] However, conventional solvent-based cellulose modification methods often require large quantities of solvents and high temperatures, posing safety risks and increasing costs. In contrast, mechanochemical modification via ball milling has demonstrated its effectiveness in covalently modifying cellulose with target functional groups while minimizing solvent use and eliminating the need for heating.[5]Building on these advantages, our work focuses on utilizing mechanochemistry to modify cellulose for applications in packaging and as additives in biocomposites.

Figure 1. The roadmap for mechanochemical modification of biomaterial



[1]Saedi, S., Garcia, C. V., Kim, J. T., & Shin, G. H. (2021). Physical and chemical modifications of cellulose fibers for food packaging applications. Cellulose, 28(14), 8877-8897.

[2] Li, N., Zhang, S., Liu, Y., Nica, V., & Coseri, S. (2023). Surficial modification of cellulose with oleic acid via amidation for developing water-resisting property. *Industrial Crops and Products*, *203*.

[3] Langerreiter, D., Attallah, N. L., Schlapp-Hackl, I., Kostiainen, M. A., & Kaabel, S. (2024). Mechanochemical modification of cellulose nanocrystals by tosylation and nucleophilic substitution. Green Chemistry, 26(18), 9823-9832.

[4] Hou, D.-F., Li, M.-L., Li, P.-Y., Zhou, L., Zhang, K., Liu, Z.-Y., Yang, W., & Yang, M.-B. (2023). Efficient Conversion of Cellulose to Thermoplastics by Mechanochemical Esterification. ACS Sustainable Chemistry & Engineering, 11(20), 7655-7663.

[5] Perona, A., Hoyos, P., Farrán, Á., & Hernáiz, M. J. (2020). Current challenges and future perspectives in sustainable mechanochemical transformations of carbohydrates. Green Chemistry, 22(17), 5559-5583.

P-10

An Open Reactions Database for Mechanochemical Reactions

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To address current deficiencies in the sharing of mechanochemical reaction data and to aid in reproducibility, we have recently developed the NSF Center for the Mechanical Control of Chemistry Reaction Database, or CMCCDB. Building off of prior work for solvothermal chemistries, this database provides a centralized location for experimental and theoretical results, allowing, among other things, for the creation of reaction prediction models by comparing yields and other products across different reagents and experimental conditions. By developing a standard set of reported properties for mechanochemistry in collaboration with the broader community, this effort will help improve data sharing independent of direct integration with the CMCCDB.

Contributions to the database use a simplified spreadsheet format with standardized headers, extracted directly from the underlying database schema, allowing for the specification of all types of standard mechanochemical reactions with comprehensive coverage for reactant specifications. Strict validation algorithms ensure data quality. The CMCCDB web interface provides facilities for searching reactions by mechanochemical treatment specifiers and significant effort has been placed on the development of tools to simplify the aggregation of mechanochemical reaction data from published works. Extensions and support for further interchange formats are in development.

Understanding the Propensity of Calcium Carbide Towards Mechanochemical Bis-Alkynylation Over Mono-Alkynylation: Isatin as a Model Electrophile

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In recent literature, calcium carbide (CaC_2) has become an alternative source of acetylenic units.[1] However, the low solubility of CaC_2 in organic solvents often restricts its use, with most studies using dimethyl sulfoxide (DMSO) as the solvent of choice due to its properties in CaC_2 activation.[2] On the other hand, under ball milling mechanochemical conditions, the insolubility of CaC_2 is no longer a limitation.[3] In fact, under these conditions, CaC_2 has been shown to alter its chemical selectivity, favoring *bis*-alkynylation products over the mono-alkynylations typically observed in solution.

Motivated by the reported reaction of isatins with CaC_2 in solution, which selectively yielded mono-alkynylation products (i.e., 3-ethynyl-3-hydroxy-2-oxindoles) (**Figure**).[4] In this work, we studied the reaction under mechanochemical conditions, finding that upon ball milling, the acetylenic anions $[C_2]^{2-}$ found in CaC_2 react with isatin leading preferentially to the *bis*-alkynylation addition product (i.e., 3-ethynyl-*bis*(3-hydroxy-2-oxindole) (**Figure**).

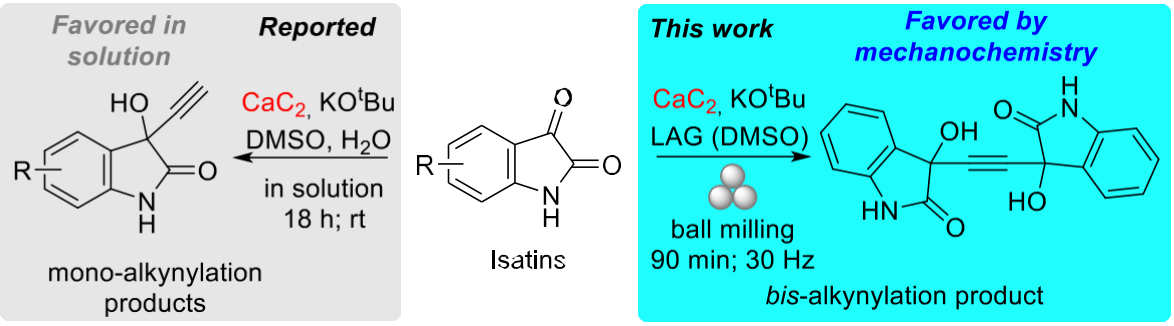

Figure. Selectivity of isatin under solvothermal conditions (left) and mechanochemical conditions (right).

Through experimental work and computational mechanistic studies within the framework of the Density Functional Theory (DFT), we investigated the change in selectivity and identified i) the highly concentrated reaction environment under ball milling and ii) the avoidance of bulk water as conditions to favor the *bis*-alkynylation of two isatin molecules with a single $[C_2]^{2-}$ fragment. In addition, we found that the reaction occurs through several variants of the same overall mechanism in which some cationic metal counterions play a relevant role through non-covalent interactions.[5]

[1] Konstantin S. Rodygin, Maria S. Ledovskaya, Vladimir V. Varonin, Kristina A. Lotsman, Valentine P. Ananikov, *Eur. J. Org. Chem.* **2021**, 43–52.
[2] Mikhail V. Polinski, Mariia D. Sapova, Valentine P. Ananikov, *Chem. Sci.* **2020**, *11*, 13102–13112.
[3] Alejandro Cortés-Lobo, José G. Hernández, *ChemPlusChem* **2024**, *89*, e202400257.
[4] Runze Qi, Zheng Li, *Synth. Commun.* **2022**, *52*, 2241–2248
[5] Alejandro Cortés-Lobo, Karen J. Ardila-Fierro, Cacier Z. Hadad, José G. Hernández. *Under review*.

P-12

Mechanochemically Induced Loss of Stereochemistry in Atropisomers

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Atropisomerism is a form of axial chirality arising due to hindered bond rotation. [1] Unlike compounds with stereocenters, which often racemize via bond breaking and formation processes, atropisomers racemize via bond rotation. Biaryls such as 1,1'-bi-2-napthol (BINOL), and its derivatives have attracted interest in the scientific community since these axially chiral scaffolds can be utilized as chiral catalysts and ligands in asymmetric synthesis.[2,3] Asymmetric synthesis is typically solvent-based, but greener methods like mechanochemistry are gaining momentum due to solvent-related waste and toxicity.[4] With mechanochemistry increasingly used in asymmetric synthesis and catalysis, studying milling's impact is essential.[5] It is yet unknown whether a change in stereochemistry of these biaryl atropisomers can be induced by using solely mechanical energy. Therefore, in this study, we investigate the stereochemical integrity of different biaryl atropisomers under typical mechanochemical conditions for asymmetric synthesis. We explored a variety of biaryl substrates, different organic and inorganic bases, the presence of liquid during milling, and different reaction times. It was found that under various conditions, biaryl atropisomers undergo degradation, which can be attributed to different reaction mechanisms. The most prominent ones are racemization and side product formation via intermolecular ring closure, like C-O and C-C bond formation, resulting in extended, insoluble π-π stacked structures. These findings indicate that use of ball milling leads to changes in stereochemistry, but also loss of it, as well as formation of side products. This leads to a decrease in catalytic activity, which can be crucial for the effectiveness of asymmetric synthesis. Such findings remain critical for the employment of mechanochemistry in asymmetric synthesis and need to be deeply investigated on other substrates.

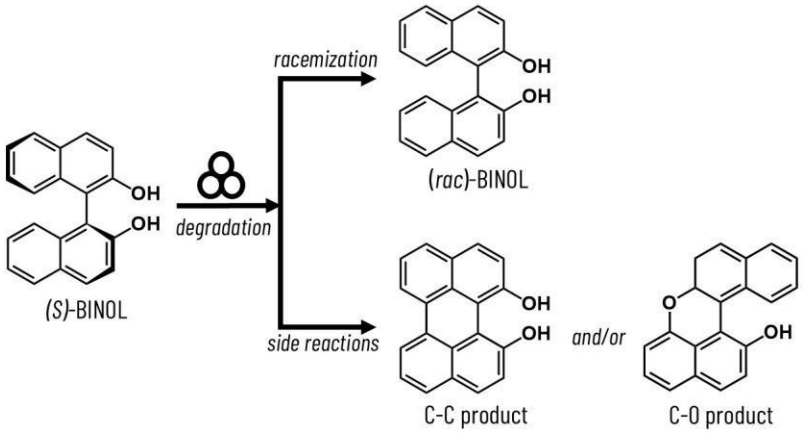

Figure 1. Reaction scheme of milling of (*S*)-BINOL.

[1] George Hallatt Christie, James Kenner, *J. Chem. Soc. Trans.* **1922**, *121 (0)*, 614–620.
[2] Jean Michel Brunel, *Chem. Rev.* **2005**, *105 (3)*, 857–898.
[3] Jie Zhang, Kun Wang, Can Zhu, *JACS Au* **2024**, *4 (2)*, 502–511.
[4] Yong-Bin Wang, Bin Tan, *Acc. Chem. Res.* **2018**, *51 (2)*, 534–547.
[5] Guan-Wu Wang, *Chem. Soc. Rev.* **2013**, *42 (18)*, 7668.

Mechanochemically Synthesized Ni-based Metal-Organic Framework Excels in Electrocatalytic Alcohol Oxidation Reaction

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Mechanochemical synthesis, a solvent-free approach that induces chemical reactions through mechanical energy, has emerged as a sustainable and scalable alternative to conventional wet-chemistry methods for catalyst fabrication. Unlike traditional techniques that often involve hazardous solvents, generate significant waste, and require multi-step processing, mechanochemistry offers a greener and more efficient route for synthesizing a wide range of materials, including nanoparticles, metal oxides, metal organic frameworks (MOFs), and covalent organic frameworks (COFs). Despite its advantages, catalysts synthesized via mechanochemical routes often display distinct structural and morphological characteristics compared to their conventionally synthesized counterparts differences that critically affect catalytic performance but remain poorly understood.

In this study, we report the synthesis of a novel Ni-based MOF using both solvothermal and mechanochemical methods. Although both methods yield the same crystalline framework, the resulting materials exhibit markedly different morphologies. Detailed characterization reveals that the mechanochemically synthesized Ni-MOF possesses a more favorable structure with enhanced surface accessibility and a higher density of catalytically active sites. These features contribute to its significantly improved performance in electrocatalytic alcohol oxidation reactions (AORs), an important process for sustainable hydrogen production and energy conversion. Our findings highlight the potential of mechanochemical synthesis in tuning catalyst properties and underscore the need for further investigation into structure-activity relationships in such materials.

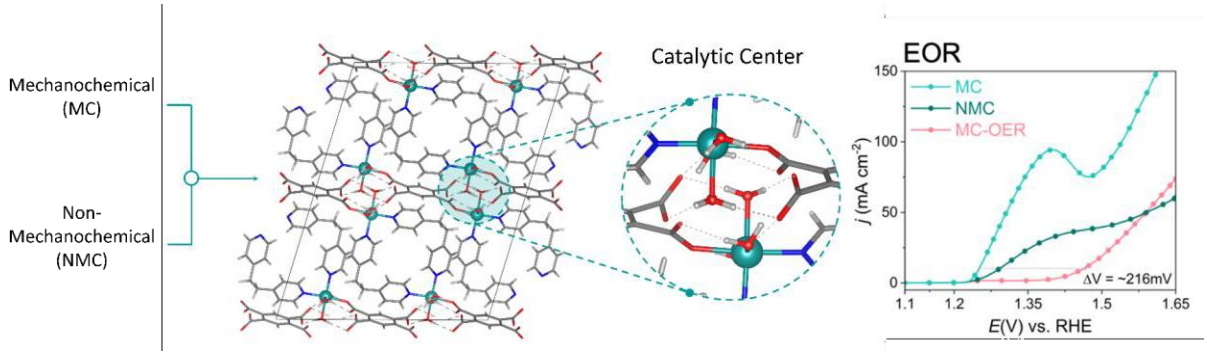

Figure 1 | Schematic representation of Ni-MOF, synthesized by mechanochemical process (MC) and solvothermal process (NMC) and showing better catalytic activity by Ni-MOF prepared by MC process.

Analyzing Molecular-Recognition Processes Exploiting the Power of Solid-State NMR Spectroscopy

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Mechanochemistry has gained increasing attention across various fields of chemistry such as organic synthesis, catalysis, metal-organic frameworks (MOFs), co-crystallization for pharmaceuticals, supramolecular chemistry, and more.[1] Despite this growing interest, the fundamental mechanisms and physicochemical effects occurring during ball milling remain often unclear.[2]
To improve mechanistic insights, various analytical techniques like *in situ* RAMAN Spectroscopy,[2–4] powder X-ray diffraction (pXRD),[5,6] and solid-state Nuclear Magnetic Resonance (NMR) spectroscopy[7,8] have been employed. In our work, we utilized solid-state NMR spectroscopy to investigate different effects taking place during milling, such as mixing, pressure, sublimation, and changes in particle size and morphology. To disentangle these individual effects, we applied different techniques including ball milling, resonant acoustic mixing (RAM), and hydraulic compression. Our model transformation consists of the conversion of racemic conglomerates into true racemic phases by mechanochemical molecular recognition under mechanochemical conditions, which was analyzed by exploiting the distinct ^{13}C chemical shifts of different phases in solid-state NMR magic-angle spinning (MAS) spectra. As model systems, we used α-(trifluoromethyl)lactic acid – which undergoes an extremely rapid phase transformation (30 s upon ball milling) – as well as biologically and prebiotically relevant amino acids such as alanine and serine.[8]
This work underlines the complex and synergistic interplay of mechanochemical effects that presumably contribute to the high efficiency in ball milling reactions. Moreover, it demonstrates the power of solid-state NMR spectroscopy for the analysis of mechanochemical transformations, offering a non-destructive analysis method that preserves the solid-state information typically lost upon dissolution for other techniques.[8]

[1] Stuart L. James, Tomislav Friščić, *Chem. Soc. Rev.* **2013**, *42*, 7494–7496.
[2] Stipe Lukin, Luzia S. Germann, Tomislav Friščić, Ivan Halasz, *Acc. Chem. Res.* **2022**, *55*, 1262–1277.
[3] Davor Gracin, Vjekoslav Štrukil, Tomislav Friščić, Ivan Halasz, Krunoslav Užarević, *Angew. Chem. Int. Ed.* **2014**, *53*, 6193–6197.
[4] Martina Tireli, Marina Juribašić Kulcsár, Nikola Cindro, Davor Gracin, Nikola Biliškov, Mladen Borovina, Manda Ćurić, Ivan Halasz, Krunoslav Užarević, *Chem. Commun*. **2015**, *51*, 8058–8061.
[5] Hannes Kulla, Franziska Fischer, Sigrid Benemann, Klaus Rademann, Franziska Emmerling, *CrystEngComm.* **2017**, *19*, 3902–3907.
[6] Lisa Batzdorf, Franziska Fischer, Manuel Wilke, Klaus-Jürgen Wenzel, Franziska Emmerling, *Angew. Chem. Int. Ed.* **2015**, *54*, 1799–1802.
[7] Ettore Bartalucci, Christian Schumacher, Leeroy Hendrickx, Francesco Puccetti, Igor d'Anciães Almeida Silva, Riza Dervişoğlu, Rakesh Puttreddy, Carsten Bolm, Thomas Wiegand, *Chem. Eur. J.* **2023**, *29*, e202203466.
[8] Calogero Quaranta, Igor d'Anciães Almeida Silva, Sven Moos, Ettore Bartalucci, Leeroy Hendrickx, Benjamin M. D. Fahl, Claudia Pasqualini, Francesco Puccetti, Mirijam Zobel, Carsten Bolm, Thomas Wiegand, *Angew. Chem. Int. Ed.* **2024**, *63*, e202410801.

Maleimide–Anthracene Mechanophores for Reusable Enzyme Supports

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Immobilized enzymes are the workhorse of continuous-flow biocatalysis, commonly tethered to methacrylate or silica-based carriers *via* covalent handles for robustness and reusability under process conditions.[1] In practice, once activity drops the entire packed bed is replaced, which consumes carriers and generates avoidable solid waste and cost. We propose a circular, mechanochemically regenerated support that decouples enzyme replacement from carrier disposal. The design leverages a maleimide–anthracene (M–A) Diels–Alder (DA) mechanophore embedded at the resin–tether interface: enzymes are immobilized to a derivatized anthracene linker *via* standard lysine coupling (e.g., reductive amination routes),[1] while the linker is joined to a maleimide- functionalized silica surface through the M–A DA adduct. Under modest mechanical stimuli available during ultrasonication, the interfacial M–A adduct undergoes force-induced retro-DA scission, releasing the spent biocatalyst and cleanly exposing the surface maleimide without damaging the carrier, an effect established for M–A mechanophores positioned at silica/organic heterointerfaces.[2]

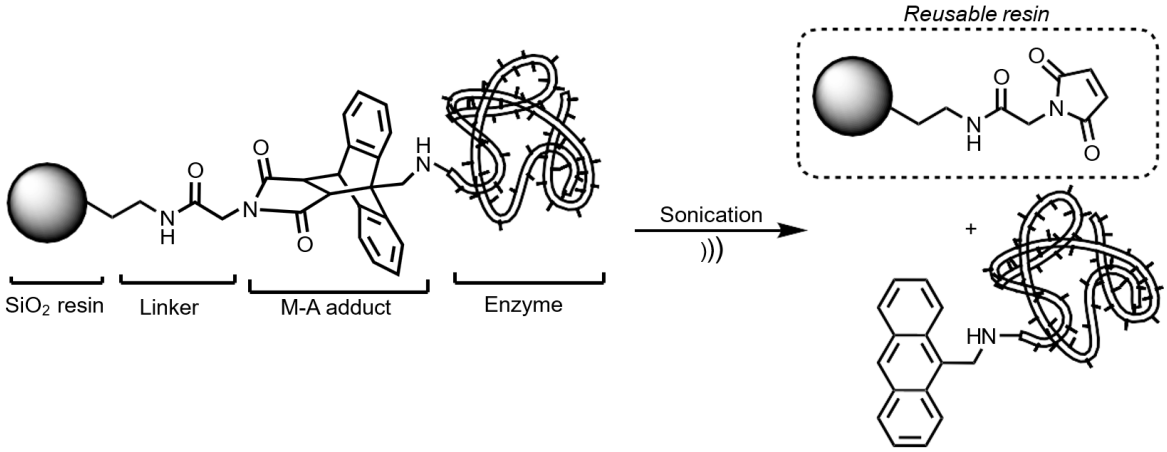

Figure 1: Ultrasound induced, retro Diels-Alder reaction, releasing the spent enzyme and regenerating the silica resin for further use.

After removal of residual protein, the carrier is “re-armed” by thermal DA coupling with a fresh anthracene linker (reported to proceed efficiently under moderate heating in polymer systems),[3] restoring the reactive handle motif for the next enzyme loading. This two-step, “cut-and-click” cycle transforms supports from consumables into reusable assets: (i) enzyme exchange without repacking,
(ii) reduced carrier E-factor, and (iii) compatibility with existing flow hardware and immobilization chemistries. We will present a proof-of-concept using silica microbeads bearing surface maleimides, an anthracene reactive linker, and model enzymes operated in continuous flow. Mechanochemical release will be quantified spectroscopically by measuring the resulting anthracene fluorescence and protein concentration. Recoupling will be demonstrated over multiple cycles with several different enzymes. Beyond enabling greener operations, the approach showcases how mechanophores can be tethered into biocatalyst interfaces to deliver “on-demand” unlinking and rapid carrier turnaround.

[1]C. L. Fernández Regueiro, D. Roura Padrosa and F. Paradisi, *Comptes Rendus. Chimie*, **2025**, 28, 349–359. [2]J. Li, T. Shiraki, B. Hu, R. A. E. Wright, B. Zhao and J. S. Moore, *J Am Chem Soc*, **2014**, 136, 15925–15928. [3]H. Durmaz, B. Colakoglu, U. Tunca and G. Hizal, *J Polym Sci A Polym Chem*, **2006**, 44, 1667–1675.

P-16

Local Structure In Situ Monitoring of Mechanochemical Reactions

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Mechanochemistry has emerged as a greener, more sustainable route to materials synthesis as compared to traditional solution-based approaches. [1] Moreover, mechanochemical synthesis enables the formation of metastable phases that are inaccessible through other solid-state methods.[2] Despite the many advantages of mechanochemical processes, integrating them into the modern landscape of academic and industrial chemistry remains a challenge, largely due to the lack of fundamental understanding of how mechanochemical reactions occur.

A useful route to studying the mechanisms of any physical or chemical transformation is by probing and learning to manipulate their kinetics. Investigations into the kinetics and mechanisms of mechanochemical reactions have revealed that these processes typically proceed through three stages:[3] (1) an induction period, where no macroscopic changes are observed; (2) a reaction period, where the reagents rapidly convert into the products; and (3) a product period, where the final product of the reaction is observed. It is believed that the existence of the induction period relates to some form of local structural distortions, but little is understood about what these distortions could be, especially within the (sub)nanometer domain. To begin to elucidate these distortions, we first need a technique for the *in-situ* investigation of their formation under ball milling conditions.

X-ray absorption spectroscopy (XAS) is a powerful technique that can directly probe the local structure of materials. Though early attempts have been made to use XAS to monitor mechanochemical reactions *in situ*,[4] they have been restricted to near edge (XANES) analysis and slow (14 min) time resolution. These slow XANES spectra have thus been restricted to information on long-lived changes in oxidation state or electronic structure of the target atom. We have now developed a new approach to time-resolved *in situ* (TRIS) XAS, which offers orders-of-magnitude increase in the temporal resolution (30 seconds) and the first capability to measure full extended X- ray absorption fine structure spectra (EXAFS). We report our developments of TRIS-XAS for the milling of α-$NiMoO_4$, in which we demonstrate how the local (first-coordination shell) geometry around Mo ions changes markedly over the course of ball milling, Figure 1, suggesting that milling introduces distortions into the $NiMoO_4$ structure.

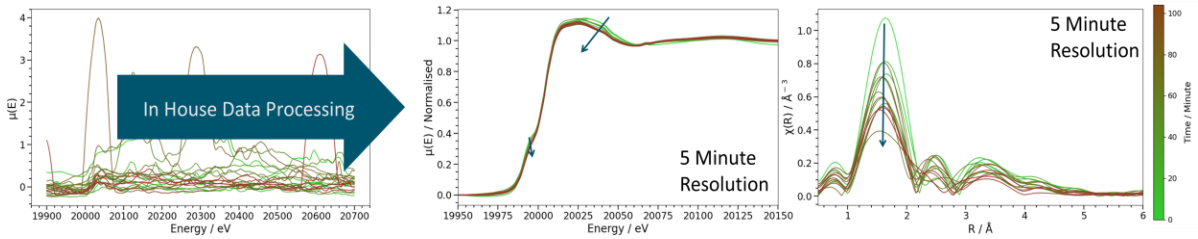

Figure 1. X-ray Absorption spectra with an acquisition time of 0.03 seconds (left) being processed into XANES (middle) and EXAFS (right) with 5-minute resolution times.

[1] O. Galant, O. et al. ACS Sustain. Chem. Eng. 2022, 10 (4), 1430–1439.
[2] L. Driscoll, et al. Energy Environ. Sci. 2023, 16 (11), 5196–5209.
[3] AAL Michalchuk, et al. Angew. Chem. Int. Ed. 2022, 61 (21), e202117270.
[4] AG Buzanich et al; *J. Chem. Phys* 7 December 2022; 157 (21): 214202.

P-17

Mechanochemically synthesized COF for PFAS adsorption

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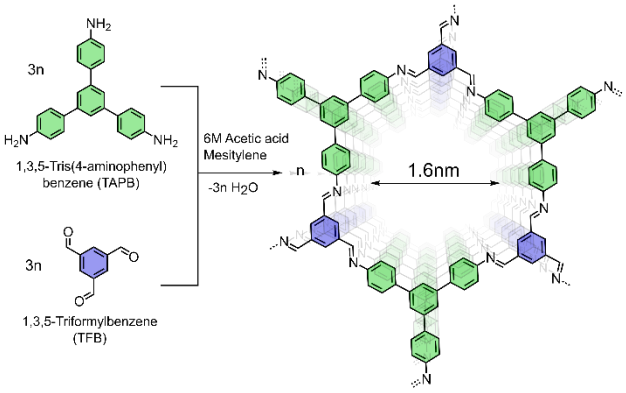
Since the 1950s, per- and polyfluoroalkyl substances (PFAS) have been widely used in a range of consumer products, including clothing, paints, cookware and fast-food packaging. Properties such as high thermal resistance and water and oil repellency contribute to their wide usage. After decades of production, these substances have eventually found their way into our drinking water. Given the adverse effects of PFAS molecules on human health, the removal of these substances from our drinking water is a matter of urgency.[1] This study employs the use of materials known as covalent organic frameworks (COFs) to adsorb PFAS molecules from an aqueous medium. COFs are crystalline, highly porous, two- or three-dimensional polymers with tunable topology and functionalities.[2] For the purposes of this study, a COF was synthesised using 1,3,5-tris(4-aminophenyl)benzene (TAPB) and 1,3,5-triformylbenzene (TFB) via a mechanochemical process. Mechanochemistry represents a green synthesis method that uses mechanical energy to initiate chemical reactions, as opposed to using harmful solvents and heat.[3] Adsorption tests were carried out to test the effectiveness of the material in question, against PFAS. The COF was exposed to a solution of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid PFOS). Following a 13-hour period, it was observed that 90% of PFOA and 99% of PFOS had been adsorbed by the COF. Most of the adsorption appeared to have occurred within the first 10 minutes of exposure. It can therefore be concluded that the TAPB-TFB COF is a promising material for the adsorption of PFAS molecules.



[1]) FitzGerald, L. I.; Olorunyomi, J. F.; Singh, R.; Doherty, C. M. Towards Solving the PFAS Problem: The Potential Role of Metal-Organic Frameworks. ChemSusChem, 15 (19), e202201136 (2022).

[2] Pachfule, P.; Acharjya, A.; Roeser, J.; Langenhahn, T.; Schwarze, M.; Schomäcker, R.; Thomas, A.; Schmidt, J. Diacetylene Functionalized Covalent Organic Framework (COF) for Photocatalytic Hydrogen Generation. J. Am. Chem. Soc., 140 (4), 1423–1427 (2018).

[3] Michalchuk, A. A. L., Boldyreva, E. V., Belenguer, A. M., Emmerling, F. & Boldyrev, V. V. Tribochemistry, Mechanical Alloying, Mechanochemistry: What is in a Name? Front. Chem., 9, 685789 (2021).

Using a Radical Scavenger to Track the Kinetics of Mechanochemical Depolymerization

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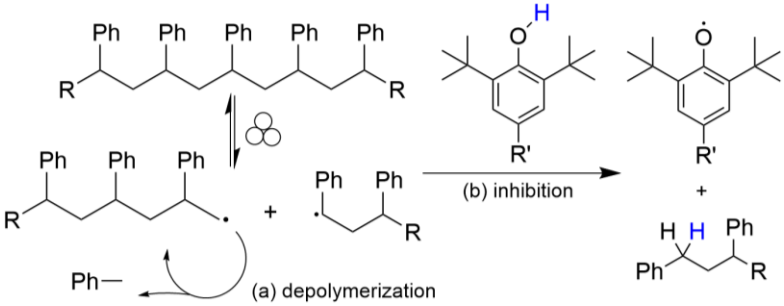
Polyolefins and other polymers can be depolymerized mechano- chemically in a ball mill. This depolymerization is initiated by homolytic cleavage of the backbone, and monomers are then formed from the reactive radical chain ends (see scheme 1). However, recombination and disproportionation of the chain end radicals compete with



Scheme 2. (a) Reaction scheme for the radical depolymerization of polystyrene to styrene, including (b) radical scavenging with tBHT.

depolymerization. To improve the efficiency of the reaction, it is crucial to quickly measure radical kinetics under different milling conditions. Therefore, with the aim of developing in situ radical analysis during ball milling methods in the future, here we investigated the kinetics of the radical reactions by vibrational spectroscopy first ex situ. Electron paramagnetic resonance (EPR), a typical method for radical analysis, cannot be used in situ because the cavity is too small to accommodate a milling vessel. However, vibrational spectroscopy can be applied during ball milling using fiber optics. Therefore, we used the radical scavenger pentaerythritol tetrakis (3,5-di-tert-butyl-4- hydroxyhydrocinnamate) (tBHT, scheme 1), where the hydrogen of the OH group terminates the polymer radical, which leads to a loss of the OH group. The OH group of the tBHT molecule exhibits vibrations in the infrared (IR) and near-infrared (NIR) spectral region (Fig. 1a-b). For future in situ analysis, NIR can have advantages over typical mid-IR measurements, as it penetrates more deeply into the sample and is insensitive to moisture and CO_2 in the environment. These vibrations were used to track the kinetics of the formation of polymer radicals as the inverse of the loss in OH concentration (Fig. 1c). As a result of the hydrogen transfer an oxygen centered radical is formed on the tBHT. This radical has a long lifespan because it delocalizes in the aromatic ring and the tert-butyl groups shields it, and it is easily detectable using EPR spectroscopy (Fig. 1d). Radical scavenging experiments were carried out with milling 1 gram of polystyrene and 15 wt % of tBHT in a 25 mL WC container, with eight 10 mm ZrO spheres at 30 Hz. Experiments were also performed without tBHT, eluting the gaseous products from the milling vessel using a N_2 flow of 60 mL/min and analyzing them with an online GC to obtain monomer formation kinetics (pathway a in Scheme 1). In future studies, we will use NIR diffuse reflectance for in situ radical analysis during ball milling using a fiber optics probe.

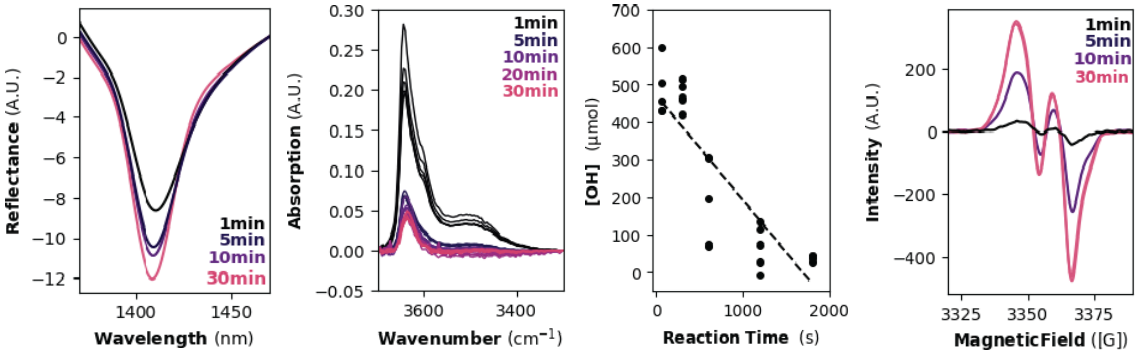


Figure 1. (a) NIR diffuse reflectance spectra of the OH peak (b) ATR-IR spectra of the OH peak (c) calculated OH concentration with linear fit (d) EPR spectra of the tBHT radical

[1] Seitzinger CL, Wigger TL, Hergesell AH, Meisner J, Vollmer I. Kinetic insights into mechanochemical polypropylene depolymerization during ball milling. ChemRxiv. 2025; doi:10.26434/chemrxiv-2025-2ht8r

Mechanically Induced Sequential One-Pot Wittig Olefination–Diels-Alder Reaction: A Solvent-Free Approach to Complex Bicyclic Scaffolds

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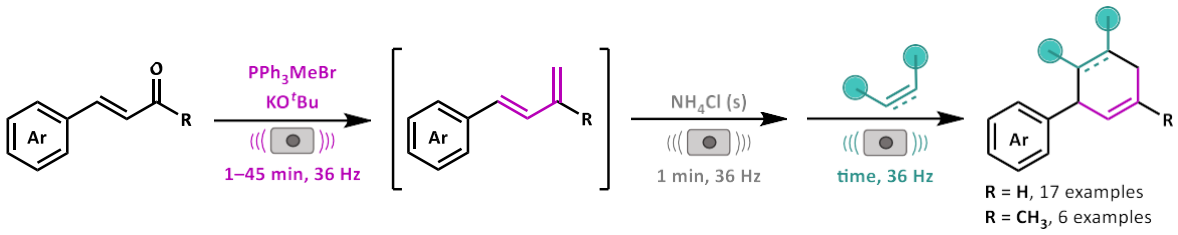
The development of one-pot sequences under mechanochemical conditions is highly desired, as this approach often comes with reduced solvent consumption, shorter process times, and improved overall process efficiency. Herein we present a one-pot reaction protocol that sequentially combines Wittig olefination and Diels-Alder cycloaddition under neat mechanochemical conditions. While both reactions have been independently demonstrated under mechanochemical conditions,[1,2] their integration into a solvent-free one-pot process has not yet been reported. The Wittig reaction, established in our group, proceeds efficiently in the ball mill – typically within 30 seconds.[1] The subsequent Diels-Alder step displayed unexpected behavior: when the reaction does not reach completion during milling, further conversion can occur during the subsequent workup and solvent evaporation process, resulting in a misleadingly high (false) yield. This discrepancy raises important questions about how reaction progress and product formation are evaluated in mechanochemical systems, particularly when unquenchable intermediates or reagents are involved. To distinguish between product formation during milling and product isolation, we conducted competitive reaction experiments by adding a second dienophile or diene in excess after the milling step.

After careful optimization of the milling parameters and reagent addition, we developed an efficient protocol for converting various α,β-unsaturated aldehydes and ketones with electron-deficient dienophiles into the corresponding cycloadducts. The reaction proceeds *via* diene intermediates and affords exclusively *endo* Diels-Alder adducts. The substrate scope includes 12 aldehydes or ketones and 4 dienophiles, yielding 24 structurally diverse products. Furthermore, we extended the sequence by a solvent-free oxidation, achieving a three-step synthesis in a single milling vessel without intermediate workup or purification. This operationally simple protocol shows excellent green metrics compared with solution-based protocols. Beyond demonstrating the synthetic utility of mechanochemistry for multistep reactions, this work highlights the need for more critical assessment of reported yields in mechanochemical studies, particularly when the workup conditions and/or product isolation process necessitate the use of solvents.



[1] J. Templ and M. Schnürch, *Angew. Chem. Int. Ed.*, **2024**, *63*, e202411536.
[2] Z. Zhang, Z.-W. Peng, M.-F. Hao and J.-G. Gao, *Synlett*, **2010**, *19*, 2895-2898.

Scalable Mechanochemical Synthesis of Biotin[6]uril

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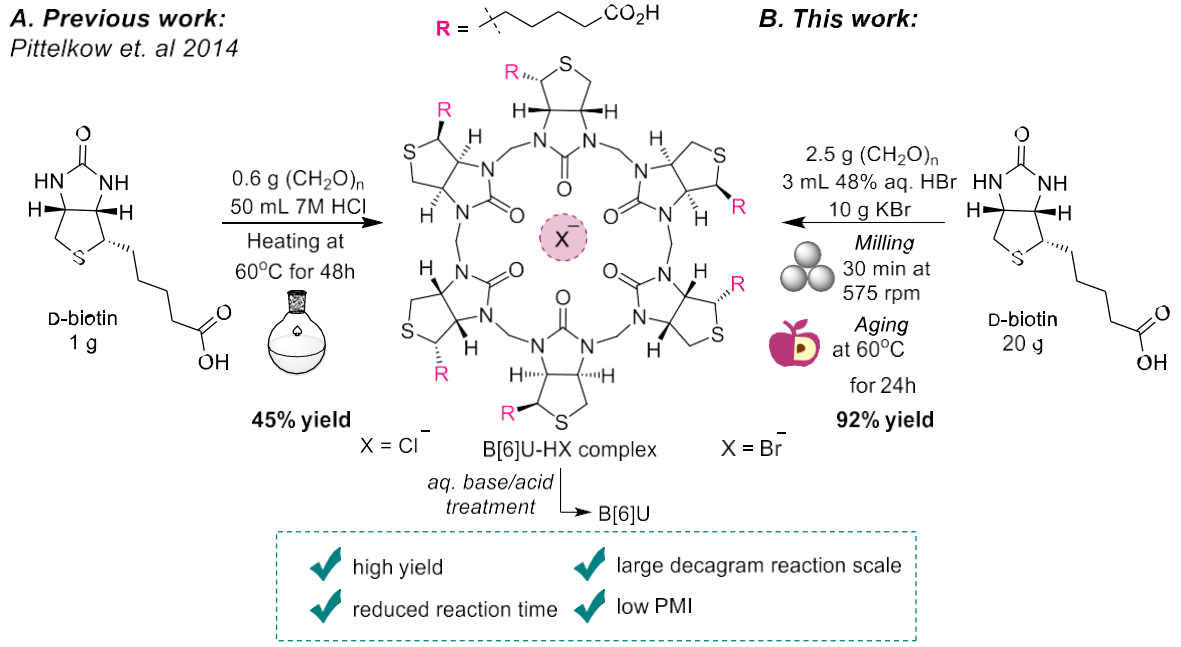
Biotin[6]uril is a chiral, water-soluble macrocycle capable of binding anions. Previously it has been synthesized via the polycondensation reaction of D-biotin with paraformaldehyde in 7M aqueous HCl as solvent, under heating at 60 °C for 2 days in moderate yield.[1]



Herein, we present a practical method for the synthesis of biotin[6]uril under solvent-free conditions using a shaker mill, followed by successful scale-up to decagram quantities in a planetary mill.[2] Optimization studies primarily focused on identifying additives for best templation, aging time and mechanical parameters, as these are key factors influencing the yield of the desired product. The optimized protocol involves mixing solid D-biotin and paraformaldehyde (1 equiv.) in the presence of liquid templating agent (0.3 equiv. of 48% aq. HBr) in a shaker or planetary mill, followed by aging at 60 °C for 24 hr. The reaction was successfully scaled up 82-fold, producing nearly 20 g of product with a high isolated yield of 92% and 91% purity. This approach offers several advantages over conventional solution-based method, including higher yields, shorter reaction times, enhanced scalability, and very low process mass intensity.

[1] Micke Lisbjerg, Bo M. Jessen, Brian Rasmussen, Bjarne E. Nielsen, Anders Ø. Madsen and Michael Pittelkow, *Chem. Sci.*, **2014**, 5, 2647–2650

[2] Elina Suut-Tuule, Eve Schults, Tatsiana Jarg, Jasper Adamson, Dzmitry Kananovich and Riina Aav, *ChemSusChem*, **2025**, 18, e202402354

P-21

Mechanistic Insights Into Co-Crystal Formation via Mechanochemistry: In-Situ Monitoring and Kinetic Modeling

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Co-crystals are of significant interest to the pharmaceutical industry as a platform to supply APIs (Active Pharmaceutical Ingredients). In fact, by combining an API in the solid state with other chemically distinct entities, it is possible to enhance its physico-chemical properties, including thermal stability, water solubility, dissolution rate, as well bioavailability and processability.
Within the possible methods for the synthesis of co-crystals, those based on mechanochemistry are drawing the attention of the pharmaceutical industry, which is asked to reduce its environmental footprint on behalf of governments and institutions. In fact, the drastically reduced use of solvents and energy, along with reactions that often result in 100% yields of single products, make mechanochemistry a sustainable and eco-friendly method. [1]
However, though mechanochemical methods are very promising, their translation to industry remains hindered by a lack in their mechanistic understanding and selectivity, and this is exacerbated as the kinetic and thermodynamic rules of conventional solution chemistry tend not to apply. To tackle this challenge, methods for time-resolved in-situ (TRIS) monitoring of mechanochemical reactions have been developed, thus paving the way for obtaining (in)accessible information on intermediates or new products.[2,3] Moreover, the collection of TRIS data also provides access to kinetic profiles which, when modelled analytically, offer exciting insights into fundamental behavior of solids under mechanochemical conditions.[4]
These insights are presented in the context of the Horizon project IMPACTIVE, aimed to scale-up the synthesis of target active pharmaceutical ingredients via mechanochemistry. The synergistic contribution of TRIS-PXRD and other experimental and theoretical approaches is shown to represent a key step in the understanding of the mechanistic behaviour.
By gaining a better understanding of how this type of chemistry works, the novel reaction pathways offered by mechanochemistry may no longer be seen as limitations, but rather as valuable assets unlocking numerous opportunities for the pharmaceutical industry.

The authors acknowledge IMPACTIVE (Innovative Mechanochemical Processes to synthesize green ACTIVE pharmaceutical ingredients), the research project funded from the European Union's Horizon Europe research and innovation programme (European Health and Digital Executive Agency) under grant agreement No. 101057286.

[1] Jasna Alić, Moritz-Caspar Schlegel, Franziska Emmerling, Tomislav Stolar, *Angew Chem Int Ed*, **2024**, 63 (50).
[2] Adam A. L. Michalchuk, Franziska Emmerling, *Angew Chem Int Ed,* **2022**, 61 (21).
[3] Tomislav Friščić, Ivan Halasz, Patrick J. Beldon, Ana M. Belenguer, Frank Adams, Simon A.J. Kimber, Veijo Honkimäki, Robert E. Dinnebier, *Nature Chem*, **2013**, 5 (1), 66-73.
[4] Leonarda Vugrin, Maria Carta, Stipe Lukin, Ernest Meštrović, Francesco Delogu and Ivan Halasz, *Faraday Discussions,* **2023**, 241 (0), 217-229.

P-22

High Entropy Oxide support for Ag and Au nanoparticles

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High Entropy Oxides (HEO) have been a hot topic on chemistry and materials science since Rost et al[1] firstly synthesized the $(Co_{0,2}Cu_{0,2}Mg_{0,2}Ni_{0,2}Zn_{0,2})O$ back in 2015. This new class of ceramic materials is defined by a solid solution of oxides in equimolar amounts and having five or more cations in its composition. Stabilization of a single crystalline phase rises from the configurational entropy, S_{config}, in which values above 1,5*R* already imply on a HEO. The properties of the simple oxides are usually enhanced when in a HEO by the mixture due to the cocktail effect, and the same time new properties may appear, whether because of the different ionic radius and a potential lattice distortion, or the placement of the cations on the solution, which tends to be random.[2] Some reports have showed that HEO can be good catalysts on electrochemical reactions[2], act as support for metal nanoparticles (NPs)[3] and even convert CO on its surface[2]. The syntheses of these materials are usually carried out at high temperature (> 900 ˚C) in solid state or hydrothermal sets,[4] in order to obtain a single-phase crystal structure. The mechanochemical approach has been investigated as an alternative route, being able to obtain a HEO only by milling the metal oxides precursors. In this work, we aimed to synthesize the HEO $(Cu_{0,2}Mg_{0,}Mn_{0,2}Ni_{0,2}Zn_{0,2})O$ by ball milling without post-treatment. Different milling devices (planetary and vibratory), milling set material (zirconium oxide, tungsten carbide and stainless steel), as well as different number and size of milling balls were investigated to reach the optimum conditions of mechanical energy input and temperature rising due to the milling process. The best results were obtained on a stainless steel set, using a 45 mL jar and 18 balls of 10 mm, after 20 h milling of the simple oxides – CuO, MgO, MnO, NiO and ZnO. Powder X-ray Diffraction (PXRD) data confirmed the formation of a single cubic phase. The final HEO was then used as support material for active plasmonic nanoparticles (1, 2, 5 and 10 % metal content) of Ag and Au, prepared by the chemical reduction of metal salt precursor under further milling.[5] These hybrid systems (Ag@HEO and Au@HEO) were characterized by XRD and TEM images. The so-called antenna-reactor system can now be used on different applications that require plasmonic effect, as Ag and Au plasmonic property might be enhanced by the HEO support[3].

[1] Rost, C., Sachet, E., Borman, T. *et al.* Entropy-stabilized oxides. *Nat Commun,* **2015,** 6, 8485.

[2] Zou, D., Liu, Y., Gao, Y., Yu, H. High-entropy oxides for catalysis: Status and perspectives. *Applied Catalysis A: General,* **2022**, 631, 118478.

[3] Gao, X., Liu, B., He, C. *et al.*, Quasi-metallic high-entropy spinel oxides for full-spectrum solar energy harvesting. *Matter,* **2024,** 1, 7, 140-157.

[4] Polychronopoulou, K., Haidemenopoulos, G. N., Aljaberi, A., Albedwawi, S. H. High entropy oxides-exploring a paradigm of promising catalysts: A review. *Materials & Design,* **2021,** 202, 109534.

[5] Oliveira, P. F. M., Camargo, P. H. C., Emmerling, F., Torresi, R. M. Challenges and opportunities in the bottom-up mechanochemical synthesis of noble metal nanoparticles. *J. Mater. Chem. A*, **2020**, *8*, 16114-16141.

P-23

Ball Milling for Better Binding: Mechanochemical Molecular Imprinting of Febuxostat

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Building upon our group's pioneering work on solvent-free, mechanochemically assisted synthesis of molecularly imprinted polymers (MIPs) for atenolol, we report the successful application of this method to febuxostat, a xanthine oxidase inhibitor used to treat hyperuricemia. This solvent-free approach, employing liquid-assisted grinding (LAG), offers an environmentally benign and potentially higher-efficiency synthetic strategy.[1]

A standardized planetary ball mill was used to systematically optimize the mechanochemical synthesis of febuxostat-imprinted polymers. Parameters investigated included milling time, vial type, milling media, co-milling agents, LAG solvent volume, monomer-to-template ratio, atmosphere, initiators, and catalytic solvents. Template rebinding capacity, a direct measure of binding affinity, was used to assess the impact of each parameter. The selectivity of mechanochemically synthesized MIPs was compared to those prepared using conventional liquid-phase methods.

Results demonstrated that the mechanochemical approach yielded MIPs with superior febuxostat binding capacity, reduced waste generation, and simplified processing, establishing it as a promising, environmentally friendly, and efficient alternative for high-performance MIP synthesis.

[1] B-C. Iacob, A. E. Bodoki, D. F. Da Costa Carvalho, A. A. Serpa Paulino, L. Barbu-Tudoran, E. Bodoki, *Int. J. Mol. Sci.* **2024**, 25(10), 5504.

P-24

The Mechanochemical Story of the Sydnone Family: From APIs to Coordination Complexes

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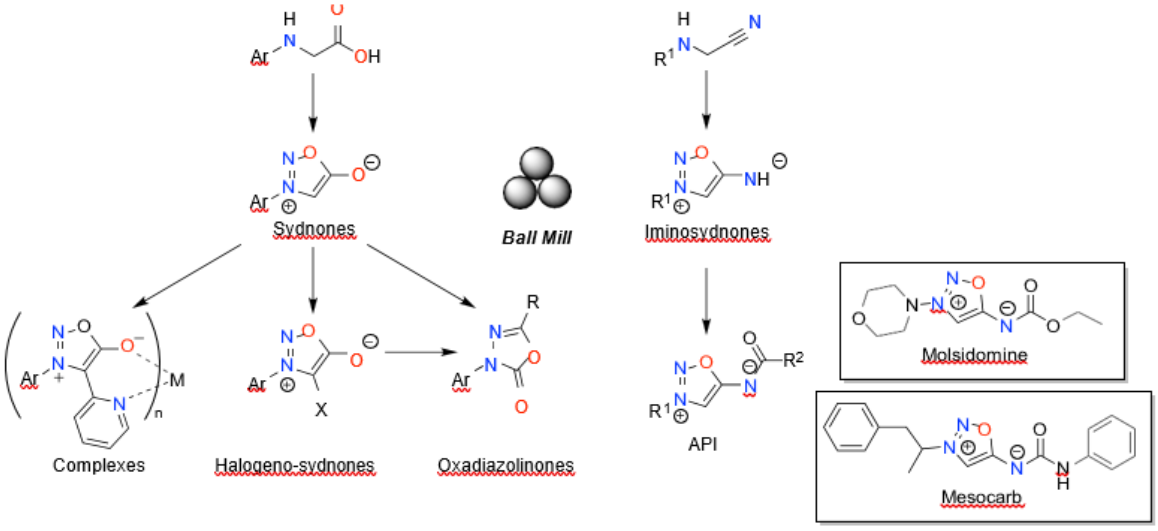
Sydnones and iminosydnones are the most representative members of mesoionic compounds, a family of molecules intensively studied for more than 60 years for their unusual chemical structures and properties.[1] These dipolar heterocycles, besides their remarkable biological properties, recently proved to be valuable molecular platforms for different applications, notably in the field of biorthogonal chemistry. [2] In our will to provide innovative and sustainable methods for organic synthesis, we undertook to develop an eco-friendly and efficient access to mesoionic compounds. By drawing on our expertise in mechanochemistry, we designed first a solventless synthesis of sydnones, iminosydnones and of their precursors (arylglycine and aminoacetonitrile) thanks to ball-milling techniques. [3] Subsequent studies allowed us to derivatize our initial building-blocks into unprecedented coordination complexes [4] and iminosydnone-based Active Pharmaceutical Ingredients (APIs). [5] Moreover, careful tuning of mechanochemical conditions during the halogenation of sydnones allowed us to demonstrate an original molecular rearrangement leading to 1,3,4-oxadiazolin-2-ones.[6]
Thus, in this communication, we will give an overview of our works on the mechanochemistry of mesoionic compounds.



[1] Il'ya Cherepanov, Sergey Moiseev, In *Advances in Heterocyclic Chemistry*; Academic Press: **2020;** Vol. 131, p 49-164.
[2] Karine Porte, Margaux Riomet, Carlotta Figliola, Davide Audisio, Frédéric Taran, *Chem. Rev.* **2021**, *121*, 6718- 6743.
[3] Nicolas Pétry, Florian Luttringer, Xavier Bantreil, Frédéric Lamaty, *Faraday Discuss.* **2023**, *241*, 114-127.
[4] Nicolas Pétry, Thibaut Vanderbeeken, Astrid Malher, Yoan Bringer, Pascal Retailleau, Xavier Bantreil, Frédéric Lamaty, *Chem. Commun.* **2019**, *55*, 9495-9498.
[5] Nicolas Pétry, Xavier Bantreil, Frédéric Lamaty, *Eur. J. Org. Chem.* **2024**, *27*, e202400387.
[6] Florian Luttringer, Nicolas Pétry, Eric Clot, Xavier Bantreil, Frédéric Lamaty, *ChemistryEurope* **2025**, *3*, e202400054.

Adding value to the forest through mechanochemistry

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The effective use of side streams is one of the key steps in strengthening the forest bioeconomy.[1] Lignin, a polyphenolic biopolymer and major byproduct of pulping and biorefinery processes, represents an underutilized renewable feedstock due to its structural heterogeneity and recalcitrance to processing. While chemical modification of lignin is necessary to broaden its application in materials science, conventional solution-state methods often rely on toxic solvents and excessive reagent use—factors that undermine both economic viability and sustainability. More importantly, technical lignins have varying structures depending on their botanical origin and isolation process, consequently impacting their solubility and reactivity.

To address these challenges, our recent work demonstrates the use of reactive extrusion (REx) as a solventless and scalable approach for lignin functionalization.[2] In a twin-screw extruder, lignin was esterified with cyclic anhydrides, achieving over 80% substitution at aliphatic hydroxyl groups within minutes. The use of a liquid esterification reagent allowed the extrusion to be conducted at lower temperatures, minimizing unwanted lignin condensation reactions. The resulting lignin esters exhibited thermoplastic properties and enhanced reactivity, opening pathways for their use in polymer blends and elastomers.

The solventless nature of the REx process substantially reduced the E-factor compared to its solution-state counterpart. Furthermore, the method was applicable to varying lignin feedstocks, demonstrating its potential as drop-in technology that can be integrated in current biorefineries.

We foresee that mechanochemistry will offer immense untapped potential for the forest bioeconomy—not only in simplifying lignin processing but in enabling new material functionalities. To this end, we are seeking to advance fundamental understanding of mechanochemical processes in biobased systems and explore alternative lignin functionalization pathways. By bridging lignin chemistry and mechanochemistry, we aim to unlock the value of forest resources.

[1] M. Österberg, M. Karjalainen, J. Lintunen, T. Tammelin, A. Asikainen, E. Vakkilainen, R. Toivonen, P. Virta, A. Henn, E.-M. Nuutinen, J. Kohl, J. Hassinen, From timber to medicine – Value added for the forest sector through broadening the product portfolio, Report of the Finnish forest bioeconomy science panel (Metsäbiotalouden tiedepaneeli), Helsinki, **2024**. ISBN: 978-952-65456-3-9

[2] Elijah Mark Garcia, Paula Nousiainen, Uula Hyväkkö, Monika Österberg, *ACS Sustain. Chem. Eng.* **2025,** *13* (30), 12198–12209. DOI: 10.1021/acssuschemeng.5c04676

P-26

Pyrazinamide–Pimelic Acid Cocrystals: A Mechanochemical and Thermal Study

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The primary focus of our study was the pyrazinamide (PZA) : pimelic acid (PA) cocrystal. This cocrystal system is of particular interest due to the necessity of mechanochemical approaches for synthesis and the interconversion of polymorphs through ageing, as previously reported.[1] The objective of the present study was to provide a complementary analysis to the preceding research on the PZA:PA cocrystal system, with a particular focus on the coformer PA[2] and temperature effects on milling.
Thermal and mechanochemical aspects of the cocrystal systems were examined. Thermal analysis was conducted using a Thermal Analysis Monitor (TAM) and Differential Scanning Calorimetry (DSC). The transition enthalpy between the cocrystal polymorphs was determined using the TAM method. It was evident from the DSC that a eutectic with a significantly lower melting point was formed between PZA and PA. The generation of a binary phase diagram for PZA and PA was enabled by DSC. Furthermore, temperature-controlled milling experiments were conducted with temperatures reaching up to 70 °C and milling experiments with mechanically pre-treated PA, monitored by time-resolved in situ powder X-ray diffraction (TRIS-PXRD). This measurement highlighted the significance of coformer polymorphism and the associated behavior, as evidenced by the observation that a polymorph transition of PA, induced by elevated temperatures (e.g., 70°C) or mechanical treatments, led to a decrease in the rate of cocrystallization. A subsequent investigation was conducted to determine the effect of milling temperature on polymorph stability, building upon previous findings on the effect of milling energy on polymorph stability.[1] As anticipated, elevating the milling temperature led to the stabilization of the metastable polymorph and a substantial deceleration of the ageing process.
The present study has demonstrated that milling temperature constitutes a pivotal parameter within mechanochemistry. This parameter has been demonstrated to exert a substantial influence on not only the kinetics of the cocrystallization process but also the stability of the resulting cocrystals.

[1] Hannes Kulla, Christian Becker, Adam A.L. Michalchuk, Kevin Linberg, Beate Paulus, Franziska Emmerling, *Cryst. Growth Des.* **2019**, *19*, 7271-7279.
[2] Emile R. Engel, Satoshi Takamizawa, *Cryst. Growth Des.* **2022**, *22*, 1229-1236.

Mechanochemical Synthesis of Pristine and Li-Doped Zinc Orthostannate from Separate and Blended Binary Oxide Precursors

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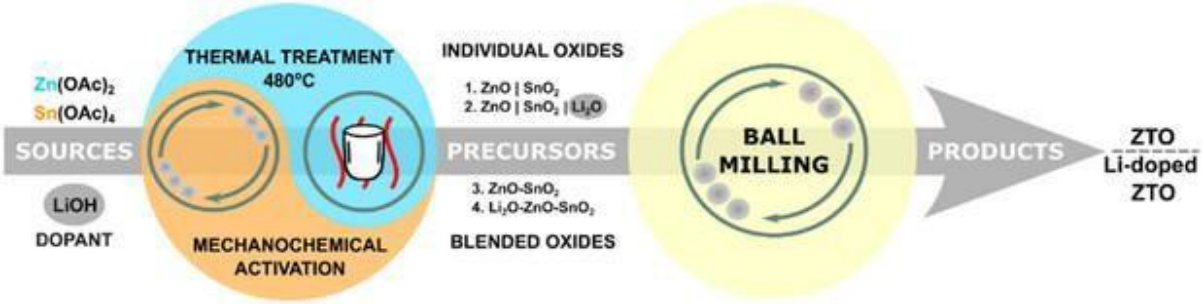
Mechanochemistry can be considered as a top-down and bottom-up synthetic methodology, but for the latter, there are some limitations, especially regarding particle size (nanometric species). It is presented that in specific precursors' preparation and mild mechanosynthetic conditions, these limitations may be overstepped, which results in the formation of cubic zinc orthostannate monocrystals (0.1 – 1.3 µm) embedded into the polycrystalline bodies.



Zinc orthostannate (ZTO) is a promising wide-bandgap (3.6 eV) n-type semiconductor material for various applications, and to develop efficient, facile, and relatively mild synthetic methods, we selected important advantages driving the high-temperature solid-state process and implemented them into mechanochemical reactions, and utilized the low-melting properties of metal acetates and the wetting abilities of Li-based dopants. Therefore, we report on two synthetic methods of undoped and Li-doped ZTO materials, prepared by the direct mechanochemical reactions from ZnO and SnO_2, and ZnO- SnO_2 blend, prepared at 480 °C from mechanochemically activated (500 rpm, 20 min) zinc(II) and tin(IV) acetates, are reported. The reactions' progress is followed by ex situ powder X-ray diffractometry, Raman spectroscopy, and ultraviolet and visible light spectrometry after grinding within 20–200 min in an agate planetary ball mill at 500 rpm. The scanning electron microscopic morphological analysis uncovered the presence of monocrystalline ZTO cubes embedded in polycrystalline ZTO bodies obtained from ball milling of the Li_2O–ZnO–SnO_2 blend. These morphological changes significantly affect the bandgap from the commonly accepted 3.6 eV to about
4.0 eV. Moreover, the X-ray photoelectron spectroscopy studies showed the presence of a Zn-rich surface (Zn:Sn ratio: 5.85:1) on the starting material after thermal blending and decarbonization of precursors.

P-28

Fast, Clean, Pillared: Mechanochemical Routes to Interpenetrated MOFs

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The mechanochemical synthesis of pillared Metal-Organic Frameworks (MOFs) is a well-established and sustainable strategy for constructing porous crystalline architectures. And it is particularly advantageous for assembling frameworks made by poorly soluble ligands, where traditional solvothermal methods face significant limitations. In this study, we investigate the formation of interpenetrated MOFs based on carboxylate-linked 2D layers, pillared by a custom-designed bis-pyridinic bis-amidic ligand featuring a rigid, aromatic backbone. Due to its extremely low solubility—only dissolving in hot DMF—this ligand is ideally suited for mechanochemical synthesis. Among the synthesized materials, PUM198 [1,2] (Parma University Material 198) stands out as a double-interpenetrated double-pillared MOF composed of terephthalate based 2D layers pillared by the bis-amidic ligand. Its mechanochemical analogue, PUM198-m can be synthetized within minutes using only microliter quantities of solvent.

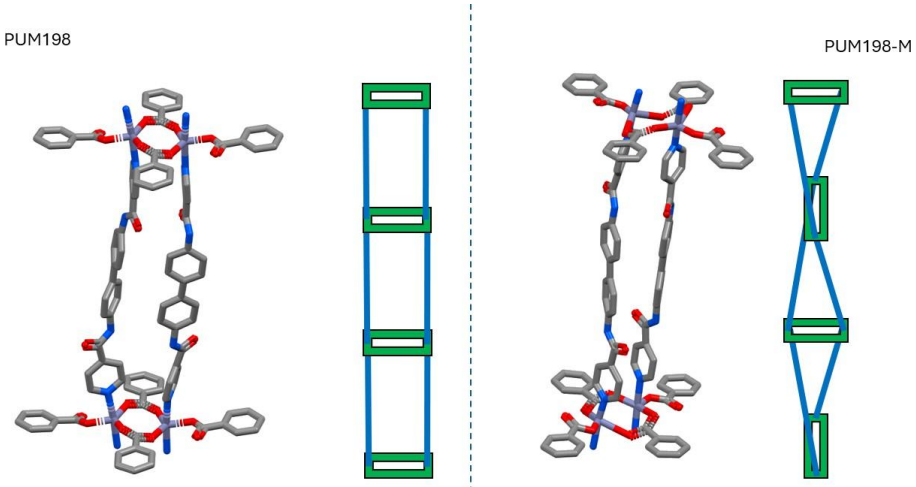

Fig. 1 - PUM198-m presents a twisted framework, representing a distinct polymorph of PUM198.

The structure of PUM198-m was resolved by 3D electron diffraction (3DED) directly from the mechanochemical powder. Due to the high-vacuum conditions during data collection, an evacuated form of the MOF was obtained. Using a combination of Rietveld and periodic DFT calculations, we were able to model and precisely locate DMF guest molecules within the freshly synthesized framework. While solvothermal conditions often lead to competition between homoleptic and heteroleptic framework formation, ball milling allows for a highly selective and rapid assembly of the heteroleptic pillared structure, and can open access to new MOFs with particular structural features.

[1] Balestri, Costa, Bacchi, Carlucci, Pelagatti, *Polyhedron* ***2018****, 153, 278-285*.

[2] Fornari, Bianchi, Riboni, Casoli, Bacchi, Mazzeo, Pelagatti, Careri, *Journal of Chromatography A 1671* ***2022*** *463010*

P-29

Scratching Beneath the Surface: Catalyst Evolution and Reusability in the Direct Mechanocatalytic Sonogashira Reaction

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We report a solvent-free Sonogashira coupling of aryl halides with terminal alkynes using palladium (0) and copper (0) under mechanochemical conditions. The study examines factors that govern carbon–carbon bond formation, including the in-situ generation of an active catalyst from precursors typically used in solution. We compare palladium powder and foil as catalytic sources inside a copper milling vessel and assess how thermal activation and ligand presence influence reactivity. Palladium was found to embed into the copper vial surface, enabling multiple reaction cycles without additional Pd, as confirmed by surface analysis. Moreover, thermal control enables chemoselective activation of one halide over another, this results in broadening the scope of selective functionalization. These findings provide valuable insights into designing catalytic systems from discrete components, presenting a cost-effective and sustainable approach to solvent-free organic transformations. This work underscores the potential of mechanochemical methods in developing reusable catalytic systems with enhanced efficiency and selectivity.

P-30

Shaken, not Stirred! Phase Diagrams of Pharmaceutical Solvates from Mechanochemistry

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Crystalline solvates – including hydrates – hold untapped potential in pharmaceutical development, yet their exploitation remains minimal due to the difficult and laborious task of unequivocally establishing their physical stabilities. Without well-defined solvate stability boundaries, these forms risk unwanted desolvation during crystallisation, processing or storage, with consequences for formulation, manufacturability and drug performance. Here we introduce Controlled Solvent Activity Liquid-Assisted Grinding (CSA–LAG), a novel mechanochemical methodology which enables accurate mapping of solvate regions of stability and thus the determination of critical solvent activities. CSA–LAG offers significant advantages over the traditional 'slurry' screenings including being less laborious, much faster, more reliable and requiring only small amounts of materials. The approach uses carefully designed solvent mixtures of known thermodynamic properties (solvent activities) and leverages the efficiency of mechanochemistry to enable rapid (de)solvation reaction in the mill. Through systematic variations of solvent activity in carefully designed CSA–LAG experiments, we resolve the boundaries between neat forms, hydrates, solvates and competing solvates. To illustrate the generality of our methodology, we use CSA–LAG to accurately establish the hydration and solvation behaviour of four compounds of pharmaceutical importance. Establishing solvation boundaries is essential not only for solid form screening and formulation, but also for guidance on chemical synthesis – where solvates must often be deliberately avoided or intentionally targeted. Our method has the potential to influence the workflows of crystallisation scientists by enabling the exploration of previously "avoided" and "misunderstood" solvated solid forms in just minutes, instead of weeks.

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[1]F. Theodosiou, T.J. Blundell, J.S.O Evans, P. Basford, N. Fellah and A.J. Cruz-Cabeza, " Smashing! Phase Diagrams of Pharmaceutical Solvates from Mechanochemistry" manuscript for submission.
[2]Patricia A. Basford, Christopher A. Cameron, and Aurora J. Cruz-Cabeza Crystal Growth & Design 2020 20 (9), 6044-6056 DOI: 10.1021/acs.cgd.0c00768
[3]Patricia A. Basford, Kevin R. Back, Michael Cram, Robert Docherty, Roger J. Davey, and Aurora J. Cruz-Cabeza Crystal Growth & Design 2019 19 (12), 7193-7205 DOI: 10.1021/acs.cgd.9b01066
[4]Firaha, D., Liu, Y.M., van de Streek, J. et al. Predicting crystal form stability under real-world conditions. Nature 623, 324–328 (2023). https://doi.org/10.1038/s41586-023-06587-3

Toward Circularity with Mechanochemistry: Alternative Pathways for Sustainable Materials and Processes

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The Circular Economy has emerged as a key approach to managing resources and promoting sustainability. Supported by policies worldwide, this paradigm shift encourages innovative solutions across various sectors. Within this framework, mechanochemistry has demonstrated significant potential and relevance.

Mechanochemistry is a rapidly expanding field, continuously developing new applications and offering alternative pathways for chemical transformations. This study highlights its vital role, particularly in acid-free processes for battery recycling and the recovery and reuse of rare earth elements from end-of-life permanent magnets.

Furthermore, mechanochemistry has proven effective in accelerating the degradation of polymers, including crosslinked materials, offering environmentally friendly alternatives to traditional degradation methods. On the other hand, it has contributed to extending the service life of materials through the development of autonomic self-healing polymers. This involves the generation of molecular networks via the polymerization of acrylates, initiated by mechanochemically generated macroradicals.

The advantages of these mechanochemical processes have been compared with conventional methods, showing notable reductions in the use of volatile organic compounds (VOCs), solvents, and other substances harmful to occupational health and the environment.

In the context of addressing the many challenges associated with circular processes, there is growing recognition of the need to develop and design new mechanophores, along with the critical importance of introducing and expanding mechanochemical processes to enhance circularity.

(1) Leveraging mechano-chemistry for sustainable polymer degradation , Polymer Journal (2024) 56:249–268

The Solid-State Aspects of Metal-TEMPO Chemistry

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It all began with simple question – how does TEMPO (2,2,6,6-tetramethylpiperidin-1-yl)oxyl) actually react with alkylzinc compounds? TEMPO is widely used inhibitor of radical processes and radical scavenger. A lot of attention has also been given to its application as a ligand in reactions with metal complexes as diversity of TEMPO red-ox forms (Fig. 1) results in a remarkable breadth of variety of metal complexes and modes of ligation. Strikingly, reports on the reactivity of TEMPO and other stable radicals towards organozinc complexes were vague and misleading, e.g., used as an innocent radical scavengers in mechanistic considerations.

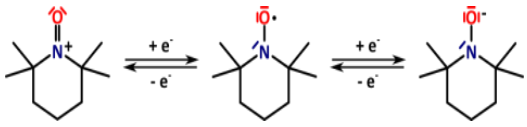

Figure 1. Red-ox reactions of TEMPO.

First experiments revealed very subtle nature of reactions of TEMPO with Et_2Zn.[1] Further research on the solid-state (mechanochemical and ageing) reactions of R_2Zn (R = *t*Bu, Ph, C_6F_5) with TEMPO has opened a possibility to precisely control different chemical equilibria and the composition and structure of isolable products (Fig. 2).[2]

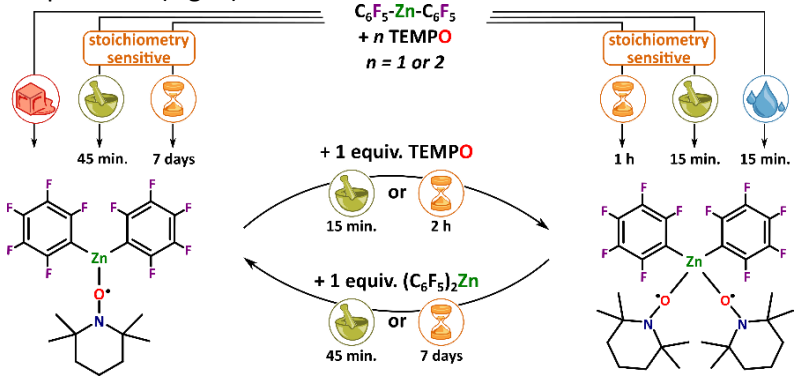

Figure 2. Selected transformations of $(C_6F_5)_2Zn$/TEMPO reaction system.

This presentation will focus on the studies of the reactions between organometallic compounds and TEMPO as model reaction systems utilized to address kinetic and thermodynamic aspects of wet and solid-state processes. The culmination of this research – comparative studies in four different reaction media, i.e. using mechano- and slowchemistry (ageing) synthesis, melted phase and solution protocols – will be discussed in detail. The newest mechanochemical investigations, including EtZnCl/TEMPO [3] and other organometallic reaction systems studied in our parent group will be presented as well.

[1] Krzysztof Budny-Godlewski, Dominik Kubicki, Iwona Justyniak, Janusz Lewiński, *Organometallics* **2014**, *33*, 19, 5093–5096.
[2] Krzysztof Budny-Godlewski, Iwona Justyniak, Michał K. Leszczyński, Janusz Lewiński, *Chem. Sci*. **2019**,*10*, 7149-7155 ; Krzysztof Budny-Godlewski, Michał K. Leszczyński, Adam Tulewicz, Iwona Justyniak, Dawid Pinkowicz, Barbara Sieklucka, Krzysztof Kruczała, Zbigniew Sojka, Janusz Lewiński, *ChemSusChem* **2021**, *14*, 3887.
[3] Krzysztof Budny-Godlewski, Dariusz G. Piekarski, Iwona Justyniak, Michał K. Leszczyński, Jan Nawrocki, Adam Kubas, Janusz Lewiński, *Chem. Eur. J.* **2024**, *30*, e202401968.

P-33

Ligand-Free Reductive Amination via Pd-Coated Mechanocatalysis

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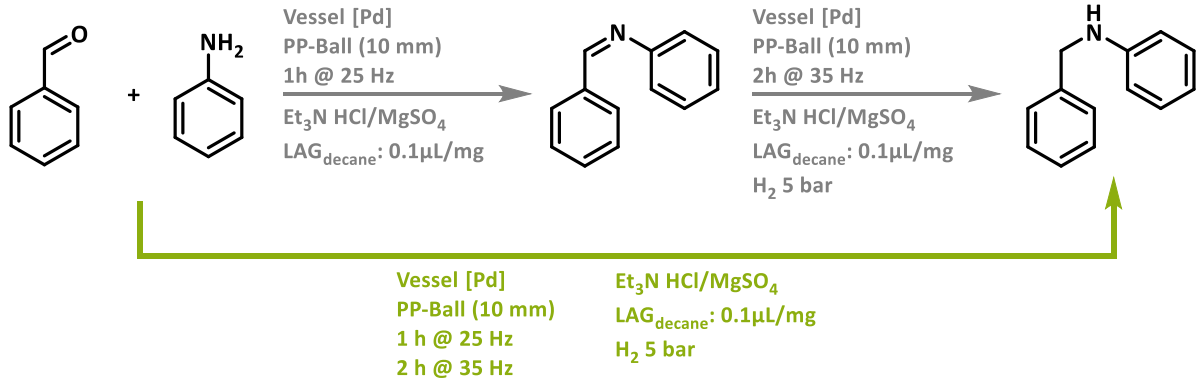
Reductive amination is a widely used C–N bond-forming reaction for producing amines from carbonyl compounds and amines. Conventional approaches often employ solvents, stoichiometric reducing agents, or elevated temperatures, which can require additional separation steps and generate waste. In this study, a solvent-free, ligand-free reductive amination was carried out under direct mechanocatalysis using molecular hydrogen at ambient pressure. Pd-coated milling vessels and polypropylene milling balls were used as both reaction medium and catalyst. A two-step milling procedure with different frequencies was applied to separate the condensation and hydrogenation stages. This reduced the formation of by-products and gave amine yields of up to 88%. Non-polar liquid-assisted grinding additives, such as n-decane, improved the yield, likely by reducing water retention and shifting the equilibrium toward imine formation. Primary amines, including volatile short-chain amines, were used in the form of their hydrochloride salts, allowing solid handling and avoiding direct dosing of volatile reagents. The method was tested with aldehydes and ketones of different structures. Benzylic aldehydes gave the highest yields, aliphatic aldehydes gave moderate yields, and ketones were less reactive. Secondary amines showed lower reactivity than primary amines. The palladium coatings were stable over multiple runs, and no significant leaching was detected (<1 ppm). The results indicate that direct mechanocatalysis with controlled milling parameters can be applied to reductive amination without solvents, ligands, or powdered catalysts, and that the approach is compatible with a range of carbonyl and amine substrates.



[1] M. Wohlgemuth, M. Mayer, M. Rappen, F. Schmidt, R. Saure, S. Grätz, L. Borchardt, *Angew. Chem. Int. Ed.* **2022**, *61*, e202212694.
[2] V. Chantrain, T. Rensch, W. Pickhardt, S. Grätz, L. Borchardt, *Chemistry A European J* **2024**, *30*, e202304060.
[3] M. Wohlgemuth, S. Schmidt, M. Mayer, W. Pickhardt, S. Grätz, L. Borchardt, *Chemistry A European J* **2023**, *29*, e202301714.

P-34

Breaking Down Forever Chemicals With Mechanochemistry

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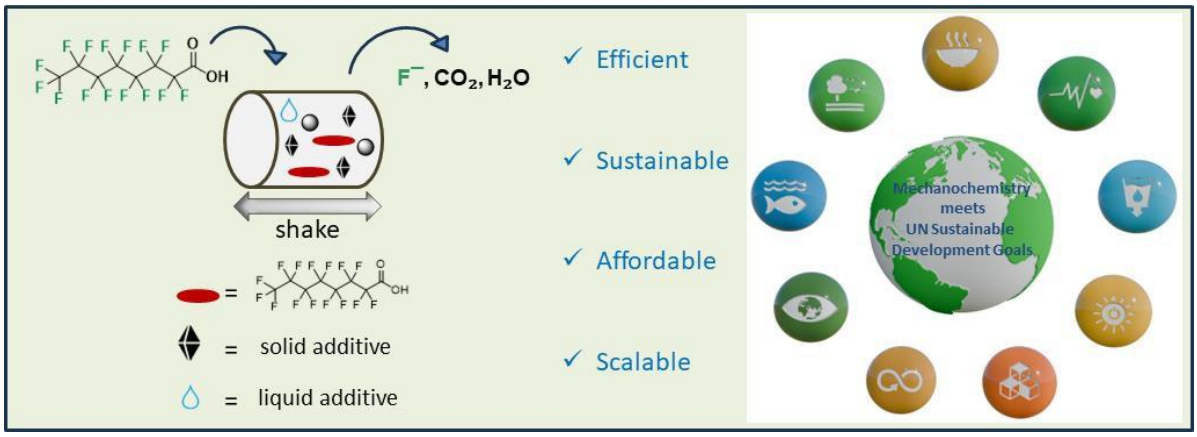
For over 70 years, the uncontrolled production, use, and disposal of per- and polyfluoroalkyl substances (PFAS), commonly known as forever chemicals, have led to widespread global contamination, necessitating the rapid development of innovative and efficient remediation technologies. State-of-the-art strategies rely on energy-intensive incineration, which releases greenhouse gases and smaller, volatile PFAS derivatives.[1] Here, we present a fast, simple, and sustainable method for the complete degradation of PFAS leveraging mechanochemistry[2] to break down the persistent carbon-fluorine bonds. Our findings indicate that liquid-assisted grinding conditions accelerate the degradation of perfluorooctanoic acid compared to neat grinding conditions, resulting in a significant reduction in energy consumption. Moreover, the fluoride released during the process binds to inorganic additives, allowing fluorine recovery as crystalline salts and preventing the formation of secondary toxic waste. The method has strong potential for scaling up and offers a green and viable solution for real-world application in PFAS decontamination.



[1] S. Liu, S. Zhao, Z. Liang, F. Wang, F. Sun, D. Chen, *Sci. Total Environ.* **2021**, *795*, 148468.
[2] J. Alić, M.-C. Schlegel, F. Emmerling, T. Stolar, *Angew. Chem. Int. Ed.* **2024**, *63*, e202414745.

P-35

Mechanochemical Route to OPA-MOF: Structural Control from Milling to Extrusion

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Metal–organic frameworks (MOFs) stand out for their high porosity, chemical versatility, and potential in environmental applications such as gas adsorption and remediation [1]. A promising subclass, bioMOFs [2], is obtained from renewable and low-toxicity ligands, such as biomass-derived organic acids (formic, lactic, citric, and oxalic) combined with metals, enabling more sustainable materials for environmental applications. However, conventional synthesis methods, such as hydrothermal and solvothermal routes, are limited to small-scale production and may require toxic solvents[3]. Mechanochemistry emerges as a greener and scalable alternative, enabling synthesis without liquid solvents and with greater control over reaction conditions[4].

Here, bioMOF OPA-MOF was synthesized at different scales, from ball milling to twin-screw extrusion, resulting in three structurally distinct materials: a novel monoclinic MOF, an orthorrombic MOF (previously reported by Anstoetz et al. 2015) [5] and a new phase obtained exclusively through mechanochemistry. Selective synthesis in the planetary ball mill was achieved by tuning parameters such as ammonia content (implicated as a possible structure-directing agent) and water availability, which proved crucial for directing the formation toward one structure over another.

Morphological and structural analyses revealed clear differences between the obtained materials. Raman spectroscopy indicated variations consistent with changes in phosphate–iron connectivity. Attempts to obtain the monoclinic MOF via ball milling are still ongoing and have reinforced the importance of liquid-assisted grinding (LAG)[6] and η parameter control for achieving phase selectivity. In contrast, twin-screw extrusion enabled the large-scale synthesis of all phases, with controlled water addition and localized heating in the extruder proving decisive for obtaining the monoclinic structure.

Due to the small crystal size, complete structural elucidation remains challenging; therefore, three-dimensional electron diffraction and serial synchrotron crystallography are planned to determine the structures directly from mechanosynthesized crystals.

Acknowledgements for the support of RCGI – Research Centre for Greenhouse Gas Innovation, hosted by the University of São Paulo (USP) and sponsored by FAPESP – São Paulo Research Foundation (2020/15230-5) and Shell Brasil, and the strategic importance of the support given by ANP (Brazil's National Oil, Natural Gas and Biofuels Agency) through the R&D levy regulation.

[1] J. L. C. Rowsell and O. M. Yaghi, *Microporous and Mesoporous Materials*, 2004, **73**, 3–14
[2] I. Imaz, M. Rubio-Martínez, J. An, I. Solé-Font, N. L. Rosi and D. Maspoch, *Chemical Communications*, 2011, 47, 7287.
[3] S. Kumar, S. Jain, M. Nehra, N. Dilbaghi, G. Marrazza and K.-H. Kim, *Coord Chem Rev*, 2020, 420, 213407.
[4] J. M. Marrett, F. Effaty, X. Ottenwaelder and T. Friščić, *Advanced Materials*, DOI:10.1002/adma.202418707.
[5] M. Anstoetz, T. J. Rose, M. W. Clark, L. H. Yee, C. A. Raymond and T. Vancov, *PLoS One*, 2015, 10, e0144169.
[6] T. Friščić, S. L. Childs, S. A. A. Rizvi and W. Jones, *CrystEngComm*, 2009, 11, 418–426.

P-36

Mechanochemical Synthesis of CuAgSe, Study of Its Physicochemical and Thermoelectric Properties

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The aim of this study is the investigation of the physicochemical and thermoelectric properties of $Cu_{2-x}Ag_xSe$ sample series, prepared in a planetary ball mill. Mechanochemical reaction products, synthesized after only 7 min of high-energy milling, were characterized by X-ray diffraction (XRD), particle size distribution (PSD), specific surface area (BET), scanning electron microscopy (SEM) and thermal analysis (DTA). Finally, the thermoelectric properties of the samples were determined.
Thermoelectric materials enable direct energy conversion, from heat to electricity or reversely. That can be beneficial for the environment as a source of green energy. Among various potential thermoelectric materials belong transition metal chalcogenides, such as Cu_2Se, CuAgSe or Ag_2Se.[1,2] Mechanochemical syntheses of $Cu_{2-x}Ag_xSe$ sample series were performed using Pulverisette 6 (Fritsch, Germany) and elemental powders of Cu, Ag and Se. Exact amount of reactants differs in sample series, due to alternation of Cu:Ag ratio. Syntheses were completed after 7 min, which was approved by XRD. In order to consolidate prepared powders and create pellets, spark plasma sintering (SPS) was used.
XRD showed a mixture of at least two phases in $Cu_{2-x}Ag_xSe$ samples, but the same for powders and pellets, denying any significant changes initiated by SPS. As typical products of mechanochemical reaction, the products were composed of agglomerated nanoparticles. It was found, that different chemical composition does not influence specific surface area, neither mean particle size, they were comparable for all samples, meaning dependence only on the synthetic method. Via BSE detector of SEM it was possible to distinguish two different phases at cut of the sample pellet and subsequently verify their identity through EDX. DTA analysis revealed more phase transitions, different for every phase included in the samples, leading to complex behaviour of $Cu_{2-x}Ag_xSe$ samples during heating. Additionally, all phase transitions were found reversible.
Connected to changes in chemical composition, precisely in Cu:Ag ratio, thermoelectric properties can also differ. Thus, by altering composition, properties ideal for the desired application can be found. Since CuAgSe is semiconductor, preparation of both p-type and n-type is possible. The highest ZT reached for p-type $Cu_{1.6}Ag_{0.4}Se$ was 1.47 at 574 K and for n-type $Cu_{0.6}Ag_{1.4}Se$ 0.73 at 570 K, after phase transition.

Acknowledgements
This work was supported by the Slovak Grant Agency VEGA (project 02/0036/23).

[1] Duan, H.Z.; Li, Y.L.; Zhao, K.P.; Qiu, P.F.; Shi, X.; Chen, L.D. Ultra-Fast Synthesis for Ag2Se and CuAgSe Thermoelectric Materials. JOM, **2016**, 68, 2659–2665.
[2] Jiang, S.; Hu, M.; Li, S.; Tian, Y.; Liu, D.; Zhang, H. Enhancing the Thermoelectric Performance of Cu2Se via Ag Doping. Journal of Solid State Chemistry, **2024**, 339, 124951.

Playing with racemic phases in mechanochemistry: Insights from solid-state NMR spectroscopy

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There is a growing interest in mechanochemistry, particularly because it aligns with the principles of green chemistry by eliminating the need for solvents in chemical reactions and enabling new reaction pathways. Furthermore, the straightforward upscaling makes it attractable for industrial applications.[1,2,3] Synthesizing specific molecules selectively and resolving racemic compounds into the enantiopure entities still pose challenges in mechanochemistry. For the latter, many traditional methods such as crystallisation, chiral chromatography, or membrane separation are time- consuming and often require specialized equipment. For ball milling (BM) protocols, metal-based mediators e.g. zinc oxide have been proposed to affect enantiomer separations.[4,5,6]

To better understand how effects such as particle-size reduction and mixing impact the BM process, we have studied several model reactions including the formation of racemic phases by combining their respective enantiopure entities. Such mechanochemically-induced phase formations involve a complex interplay of mixing, pressure, and particle-size alternation, all of which are crucial factors in solid-state reactions. To disentangle these effects further, alternative mechanochemical methods such as Resonant Acoustic Mixing (RAM) have been explored. RAM allows for pure mixing by applying an oscillation force to the sample without inducing significant pressure, heat or altering particle size, focusing purely on enhancing the mixing process itself.[7]

Solid-state Nuclear Magnetic Resonance (NMR) is an ideal tool for analyzing mechanochemical reactions, as it allows for the examination of samples without altering their aggregation state through dissolution, the investigation of dynamic properties and the detection of noncovalent interactions in molecular recognition. Herein, we report on the formation of racemic phases from mixtures of enantiopure compounds using mechanochemical methods, focusing primarily on trifluoromethyl lactic acid (TFLA). By mixing equimolar amounts of enantiopure (*R*)- and (*S*)-TFLA and processing them in a ball mill, we were able to form the racemic phase, which was monitored using solid-state NMR ^{13}C cross-polarization (CP) experiments. Additionally, we investigated racemic-phase formations using RAM, which provided insights into how pure mixing influences molecular-recognition events.

Finally, the study was extended to reactions with amino acids, where attempts were initiated to achieve resolutions of enantiomers following pathways described earlier.[6] In these investigations, ^{13}C and ^{15}N isotope labeled valine was key for gaining further insight into the structure of the developing phase after mechanochemical treatment.

[1] James, S. L.; Adams, C. J.; Bolm, C.; Braga, D.; Collier, P.; Friščić, T.; Grepioni, F.; Harris, K. D. M.; Hyett, G.; Jones, W.; Krebs, A.; Mack, J.; Maini, L.; Orpen, A. G.; Parkin, I. P.; Shearouse, W. C.; Steed, J. W.; Waddell, D. C. Mechanochemistry: Opportunities for New and Cleaner Synthesis. *Chem. Soc. Rev.* **2012**, *41* (1), 413–447. [2] Zhou, J.; Hsu, T.; Wang, J. Mechanochemical Degradation and Recycling of Synthetic Polymers. *Angew. Chem. Int. Ed.* **2023**, *62* (27), e202300768.

[3] G. I. Peterson, W. Ko, Y.-J. Hwang, T.-L. Choi Mechanochemical Degradation of Amorphous Polymers with Ball-Mill Grinding: Influence of the Glass Transition Temperature. *Macromolecules* **2000,** *53* (18), 7795–7802. [4] Lorenz, H.; Seidel-Morgenstern, A. Processes To Separate Enantiomers. *Angew. Chem. Int. Ed.* **2014**, 53 (5), 1218–1250.

[5] Shemchuk, O.; Grepioni, F.; Leyssens, T.; Braga, D. Chiral Resolution via Cocrystallization with Inorganic Salts. *Israel J. Chem.* **2021**, *61* (9–10), 563–572.

[6] Viedma, C.; Lennox, C.; Cuccia, L. A.; Cintas, P.; Ortiz, J. E. Pasteur Made Simple – Mechanochemical Transformation of Racemic Amino Acid Crystals into Racemic Conglomerate Crystals. *Chem. Commun.* **2020**, *56* (33), 4547–4550.

[7] Michalchuk, A. A. L.; Hope, K. S.; Kennedy, S. R.; Blanco, M. V.; Boldyreva, E. V.; Pulham, C. R. Ball-Free Mechanochemistry: *In Situ* Real-Time Monitoring of Pharmaceutical Co-Crystal Formation by Resonant Acoustic Mixing. *Chem. Commun.* **2018**, *54* (32), 4033–4036.

P-38

Kinetic Studies of Ball Mill Reactions: A Diels–Alder Cycloaddition Under Second Order Overall Kinetic Models

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Our research group uses *in situ* Raman spectroscopy, X-ray powder diffraction and occasionally thermography to investigate the kinetics of mechanochemical reactions in ball mills, toward determining their activation barriers, the elucidation of their chemical mechanisms, and the validation of the "stress augmented thermal activation" model. Toward this end, Diels Alder cycloadditions are of interest since their mechanisms are well understood, involving one pericyclic transition state made of two molecules, the diene and the dienophile. This implies that the expected kinetic model is second-order overall, and first order with respect to each reactant, if the chemical reaction is what controls the reaction kinetics. While several combinations of diene and dienophiles were studied, our work focused on 2,3-dimethyl-1,3-butadiene (as the diene) and maleic anhydride (as the dienophile), since reasonably isolated Raman bands, with medium to large intensity, were observed for both reactants and the reaction product, which forms needle-shaped crystallites. Selected bands of acrylic composing the milling vessels were used for scaling and subtracting its signal. Moreover, it was possible to find a liquid additive, Triton X-100©, a non-ionic surfactant, that enables a uniform matter distribution on the inner surfaces of the reaction vessels. These features made possible to collect *in situ* Raman spectroscopy data while ball milling for times between 30 min and 1 hour, leading to kinetic profiles for both reactants and the product. The kinetic data was calculated with MATLAB scripts modified by the authors from those purchased from *InSolido Tech.* Besides the identification of the product and its characterization with X-ray powder diffraction, single crystal X-ray diffraction, optical microscopy and ^{1}H NMR, the presentation will discuss the determination of the rate constants under slightly different amounts of mechanical energy input, occurring by a second order overall kinetic model, as expected if the chemical reaction is what controls the kinetics. Once temperature control while ball milling is implemented, this can lead to the identification of elementary processes (with one transition state) in mechanochemical mechanisms in ball mill reactions, by using Eyring transition state theory modified by the application of mechanical forces at the atomic/molecular levels.

[1] Xiaohe Ma, Wenbing Yuan, Steven E. J. Bell and Stuart L. James, *Chem. Commun*., **2014**, *50*, 1585-1587.

P-39

The Role of Temperature in the Mechanical Impact Sensitivity of Energetic Materials

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The mechanical initiation of energetic materials (explosives, propellants, and pyrotechnics; EMs) represents an important type of mechanochemical reaction. Understanding the elementary mechanisms for EM initiation is crucial for determining their performance, and for designing safer and better-performing materials. Recent successful efforts to understand the mechanochemical reactivity of EMs have focused on how mechanical impact causes the excitation of low-lying phonon modes.[1,2] This excess kinetic energy redistributes quickly within the EM and ultimately causes rupture of a covalent bond: the initiation process.

Aiming to develop further our theoretical models for EM mechanochemistry, we here investigate the effect that temperature has on the mechanical initiation behaviour of EMs. As proof of concept our study focuses on two model EMs, FOX-7 (1,1-diamino-2,2-dinitroethene) and HMX (1,3,5,7-tetranitro-1,3,5,7-tetrazocane), whose sensitivity to mechanical initiation is known to increase with temperature.[3] Through the quasi-harmonic treatment of our EMs, we successfully capture the thermal expansion behaviour of our EMs and the associated phonon-renormalisation, which we benchmark against inelastic neutron scattering. Our models suggest that this renormalisation is key to capturing the thermal-enhancement of mechanochemical reactivity in EMs, thereby underscoring the importance of understanding thermal expansion in mechanochemical reactivity.

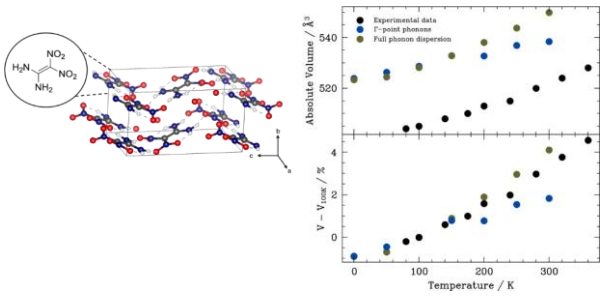

Figure 1: Thermal expansion of FOX-7. The chemical and crystallography structures of FOX-7 (left), alongside the absolute volume vs temperature (top) and the percentage change in volume relative to the volume at 100 K (bottom) for the simulated results using phonons at the **!** (blue) and full integration across the Brillouin Zone (green) compared with experimental data (black).

[1] A. A. L. Michalchuk, S. Rudić, C. R. Pulham and C. A. Morrison, *Chem. Commun.*, 2021, **57**, 11213–11216.
[2] A. A. L. Michalchuk, J. Hemingway and C. A. Morrison, *J. Chem. Phys.*, 2021, **154**, 064105.
[3] M. Yan, L. Wei, Y. Xiao, W. Xian, Z. Wang, S. Li, J. Xu, Y. Liu, F. Nie and S. Huang, *J. Phys. Chem. C*, 2023, **127**, 20838–20848.

P-40

Tracking the Evolution of the Periodic and Local Structure Noble Metal Nanoparticles Prepared by Milling

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The structural properties of metallic nanoparticles are closely linked to their atomic-scale organization. Silver and gold nanoparticles (AgNPs and AuNPs) are widely applied in catalysis, but controlling their atomic structure remains a challenge. Mechanochemical synthesis has emerged as an efficient, solvent-free alternative for nanoparticles synthesis, yet the impact of mechanical stress on atomic structure is still not fully understood.[1] Thus, the aim of this study is to move beyond conventional crystallographic approaches and explore how AgNPs, AuNPs, and AgAuNPs behave at the local structural level under increasing milling time conditions, i.e., under increased stress.

High-resolution X-ray diffraction (HXRD) analysis of AuNPs indicates increased structural ordering with longer milling times, characterized by an increase in crystallite size and a reduction in the isotropic atomic displacement parameter (U_{iso}). In contrast, AgNPs exhibit the opposite trend: a decrease in crystallite size and an increase in U_{iso}, suggesting a greater degree of structural disorder at prolonged milling times. However, the crystallographic information obtained from Rietveld refinement alone is insufficient to fully understand how mechanical stress is accommodated within the silver and gold structures. To gain deeper insight at the atomic level, X-ray total scattering (TS) measurements combined with Pair Distribution Function (PDF) analysis were employed. PDF confirmed the general trends observed in the Rietveld refinement for both materials. However, a more detailed interpretation emerged from the PDF data. For AuNPs, the first interatomic distance peak exhibited a significant broadening compared to its peak shift, indicating that stress accommodation occurs primarily through an increase in non-homogeneous strain, that is, enhanced thermal mobility of the first atomic pair.

For AgNPs, on the other hand, a slight splitting of the second and fourth interatomic distance pair peaks was observed. This suggests that stress accommodation in silver nanoparticles goes beyond increased atomic mobility and likely involves a lattice distortion, possibly indicating a symmetry breaking from the cubic Fm-3m structure to a tetragonal I4/mmm structure.

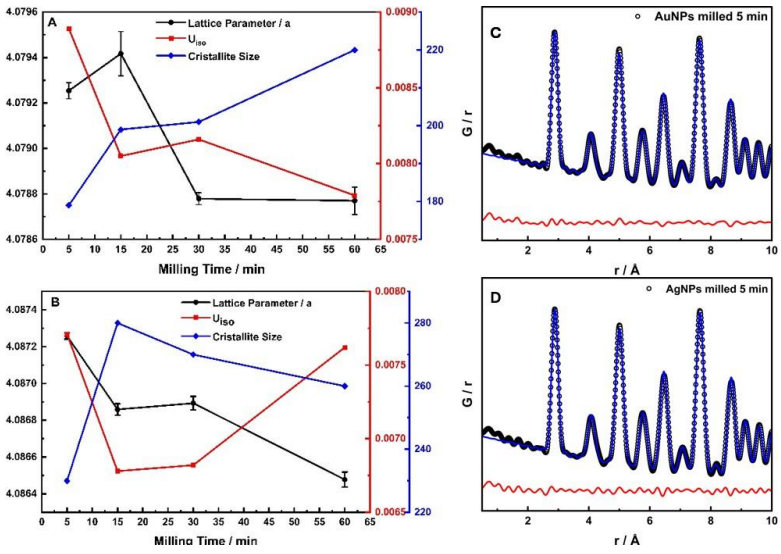

Figure 1. Rietveld refinement results for AuNPs (A), AgNPs (B), and PDF plots of AuNPs (C) and AgNPs (D) milled for 5 minutes.

1 Paulo Filho Marques De Oliveira, Roberto Manuel Torresi, Franziska Emmerling and Pedro Henrique Cury Camargo, J Mater Chem A Mater, **2020**, 8, 16114–16141.

Time-Resolved Tandem In Situ Raman and X-Ray Diffraction Monitoring of Mechanically Induced Self-Propagating Reactions in the Nickel-Copper-Sulfur System

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Nickel sulfides and copper sulfides have been widely prepared and utilized in diverse applications, including in solar cells, [1] batteries,[1] water splitting, [2] and sensors, [3] owing to their excellent physicochemical properties. Although mechanochemical synthesis of these compounds has been reported, the detailed mechanistic pathways governing the transformation from precursors to the products remain largely unexplored. Moreover, it was recently revealed that the mechanochemical reactions in the binary systems can proceed within seconds via a mechanically induced self-propagating reaction (MSR),[4,5] a combustive-like process capable of an immediate transformation of reagents into product via an exothermic event[6]. In this work, we report the *in situ* monitoring of the formation of binary systems, starting from Ni:S molar ratios 1:1 and 3:2, Cu:S 1:1, and also a ternary one Ni:Cu:S 1:1:2 via MSR, using a vertical ball mill P23. A tandem time-resolved *in situ* Raman spectroscopy and powder X-Ray diffraction (PXRD) monitoring was conducted at BESSY II synchrotron Facility. The rapid transformation was clearly observed utilizing the selected techniques, as after a short time of activation period, the peaks corresponding to reagents are rapidly transformed into those of the products. Through *in situ* monitoring, the rapid transformation can be seen obviously. This is exemplified for the time-resolved PXRD patterns for ternary Ni-Cu-S mixture in Fig. 1, where diffraction peaks corresponding to elemental sulfur, copper and nickel at 2θ of 43.0°, 43.6° and …°, respectively, disappeared, while new ones at 2θ of 45.3°, 47.8°, and 53.7° corresponding to the product emerged. The changes indicate the formation of a mixture of Cu_2S, NiS_2 and $Cu_{1.8}S$ in the case of the ternary system. During the lecture, also the plots for binary Cu-S and Ni-S systems will be presented as well.

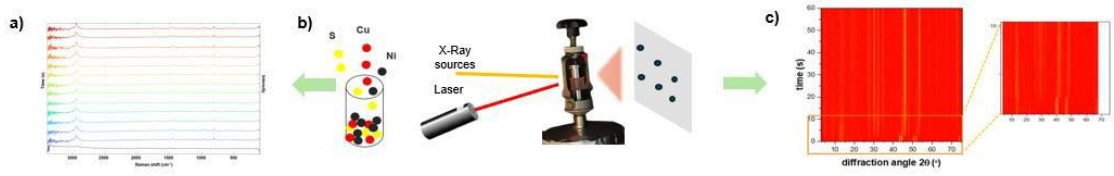

Fig. 1 Tandem time-resolved *in situ* monitoring of mechanochemical treatment of Ni:Cu:S 1:1:2 system: a) Raman spectra, b) scheme of experimental setup, c) XRD patterns heat map.

[1] Paulomi Roy, Suneel Kumar Srivastava, *CrystEngComm*. **2015**, 17, 7801-7815.
[2] Hitha Haridas, Bhargavi Somapur, Harish Sharma Akkera, Nagarjuna Neella, Nagaiah Kambhala, *ChemistrySelect*. **2024**, 9, e202402550.
[3] Mani Arivazhagan, Govindhan Maduraiveeran, *J. Electroanal. Chem*. **2020**, 874, 114465.
[4] Matej Baláž, Anna Zorkovská, Farit Urakaev, Peter Baláž, Jaroslav Briančin, Zdenka Bujňáková, Marcela Achimovičová, Eberhard Gock, *RSC Adv.* **2016**, 6, 87836-87842
[5] Matej Baláž, Róbert Džunda Radovan Bureš, Tibor Sopčák, Tamás Csanádi, *RSC Mechanochem*. **2024**, 1, 94-105.

P-42

Mechanochemical synthesis of essential oil cocrystals

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The Industrial scale use of most conventional pesticides, insecticides and bactericides are unsustainable for ecosystems, users and consumers. Essential oils (EOs) have been demonstrated to possess antibacterial, antiviral, antifungal and insecticide effects [1] . However, their ability to evaporate or sublime quickly makes them more prone to be used in the perfume industry than anywhere else for now. By including them in co-crystal structures or host-guest arrangements in MOFs or HOFs, synthesized in bulk by mechanochemistry, the stabilization of EOs increases the timeframe of action by several orders of magnitudes from several days to weeks of effective action [2] .

We developed cocrystal structures for example between thymol and isonicotinamide or niacin or isonicotonitrile, and carvacrol and isonicotinamide or 8-aminochinoline. We subsequently demonstrated that our carvacrol cocrystals with isonicotinamide released carvacrol constantly over a timeframes greater than 2 weeks at room temperature. This highlights the successful modulation of the release profile of EOs into the environment, thus opening the possibility to exploit their properties in agricultural or food related uses.

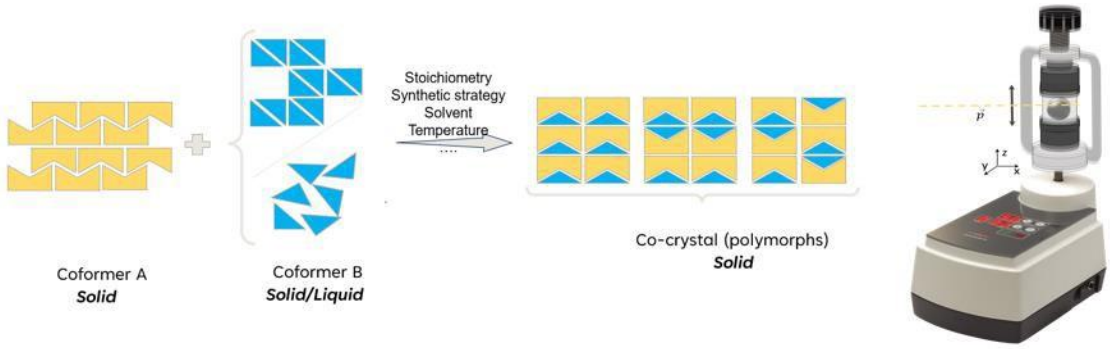

Figure 1. Process of mechanochemical synthesis of cocrystals.

[1] 1. Perricone, M.; Arace, E.; Corbo, M. R.; Sinigaglia, M.; Bevilacqua, A. Bioactivity of Essential Oils: A Review on Their Interaction with Food Components. Front. Microbiol. 2015, 6, No. 76.
2. Hammer, K. A.; Carson, C. F.; Riley, T. V. Antimicrobial Activity of Essential Oils and Other Plant Extracts. J. Appl. Microbiol. 1999, 86, 985–990.
3. Bakkali, F.; Averbeck, S.; Averbeck, D.; Idaomar, M. Biological Effects of Essential Oils- A Review. Food Chem. Toxicol. 2008, 46,446− 475.
[2] Dispensing Essential Oil Components through Cocrystallization: Sustainable and Smart Materials for Food Preservation and Agricultural Applications. Fabio Montisci, Paolo P. Mazzeo, Claudia Carraro, Michele Prencipe, Paolo Pelagatti, Fabio Fornari, Federica Bianchi, Maria Careri, and Alessia Bacchi. ACS Sustainable Chemistry & Engineering 2022 10 (26), 8388-8399 DOI: 10.1021/acssuschemeng.2c01257

P-43

Mechanochemical Synthesis of Perspective Composites for Li-Ion Batteries

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In the past few years, global energy crises and climate change have become major challenges. In this context, lithium-ion batteries represent a promising alternative for energy storage. This study presents the mechanochemical synthesis and subsequent heat treatment of spinels containing several metal elements.

We successfully prepared high-entropy compounds, i.e., the oxide $(Zn_{0.25}Mg_{0.25}Co_{0.25}Cu_{0.25})Fe_2O_4$ (HEOFe), lithiated oxyfluoride $Li_{0.5}(Zn_{0.25}Mg_{0.25}Co_{0.25}Cu_{0.25})_{0.5}Fe_2O_{3.5}F_{0.5}$ (LiHEOFeF), and lithiated oxychloride $Li_{0.5}(Zn_{0.25}Mg_{0.25}Co_{0.25}Cu_{0.25})_{0.5}Fe_2O_{3.5}Cl_{0.5}$ (LiHEOFeCl) with a spinel-based cubic structure by ball milling and subsequent heat treatment. The as-prepared samples show particles with sizes from 50 to 200 nm with a homogeneous atomic distribution. The average elemental composition of the samples is close to the nominal value. ^{57}Fe Mössbauer spectroscopy revealed that incorporating Li and F or Cl and forming oxygen defects do not influence the redistribution of Fe^{3+} cations over the spinel lattice sites and result in their preferred octahedral coordination.

Electrochemical measurements performed using 2032-coin cells with a Li-metal anode have demonstrated voltammetric charge capacities of 450, 694, and 593 mA h g^{-1} for HEOFe, LiHEOFeCl, and LiHEOFeF, respectively. The best electrochemical performance of LiHEOFeCl was ascribed to its smallest particle size. Galvanostatic chronopotentiometry at 1C rate confirmed high initial charge capacities for all the samples, but galvanostatic curves exhibited capacity decay over 100 charging/discharging cycles. Raman spectroelectrochemical analysis conducted on the LiHEOFeF sample proved the reversibility of the electrochemical process for initial charging/discharging cycles. Electrochemical impedance spectroscopy revealed the lowest initial charge transfer resistance for LiHEOFeCl and its gradual decrease for both LiHEOFeCl and LiHEOFeF during galvanostatic cycling, whereas the charge transfer resistance of HEOFe slightly increases over 100 galvanostatic cycles due to the different mechanism of the electrochemical reduction.

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M. F. also thank the Slovak Grant Agency VEGA (2/0058/23). O. P. acknowledges Štefan Schwarz Postdoc Fellowship No. 2024/OV1/003.

Solid-State Synthesis of Cationic Cellulose Fibers from Low-Processed Cotton for Efficient Virus Capture

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Cellulose is a valuable natural resource for the development of innovative biomaterials with a wide range of applications.[1] Mechanochemical functionalization of cellulose is currently gaining growing interest as an efficient, sustainable, and environmentally benign strategy.[2] Here, we present a solid-state approach, combining mechanochemistry and accelerated aging, for the cationization of low processed cotton fibers (CF, Whatman 1 filter paper) via etherification reaction (Figure 1). The developed protocol demonstrated high cationization reaction efficiency, while reducing waste generation. Moreover, the prepared material showed a strong ability for binding viruses (Figure 1),[3] thus offering promising applications in biotechnology and purification processes.

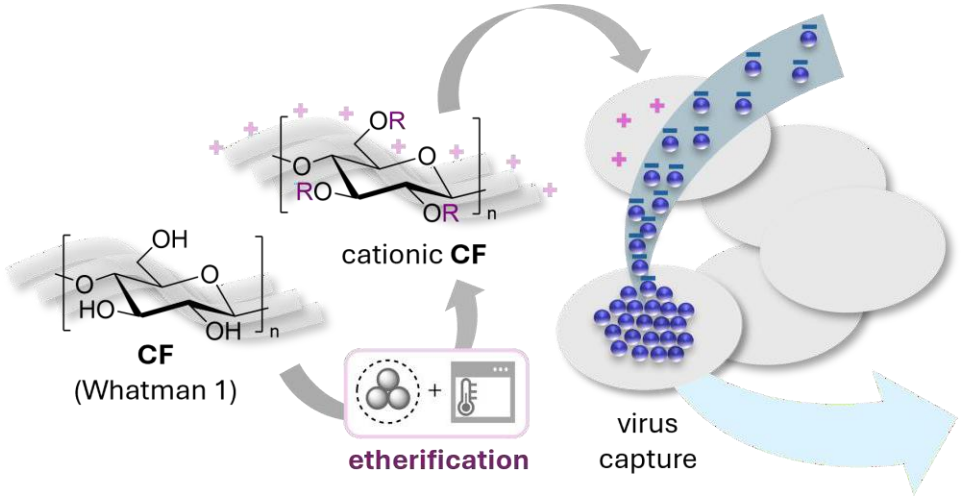

Figure 1. Solid-state synthesis of cationic cellulose and its application for virus capture.

[1] Youssef Habibi, *Chem. Soc. Rev.*, **2014**, *43*, 1519-1542.

[2] Faezeh Hajiali, Tony Jin, Galen Yang, Madison Santos, Edmond Lam, Audrey Moores, *ChemSusChem*, **2022**, *15*, e202102535.

[3] Henna Rosilo, Jason R. McKee, Eero Kontturi, Tiia Koho, Vesa P. Hytönen, Olli Ikkalaa and Mauri A. Kostiainen, *Nanoscale*, **2014**, *6*, 11871-11881.

Butyllithium-Free Access to Lithium Amide Bases via Mechanochemistry

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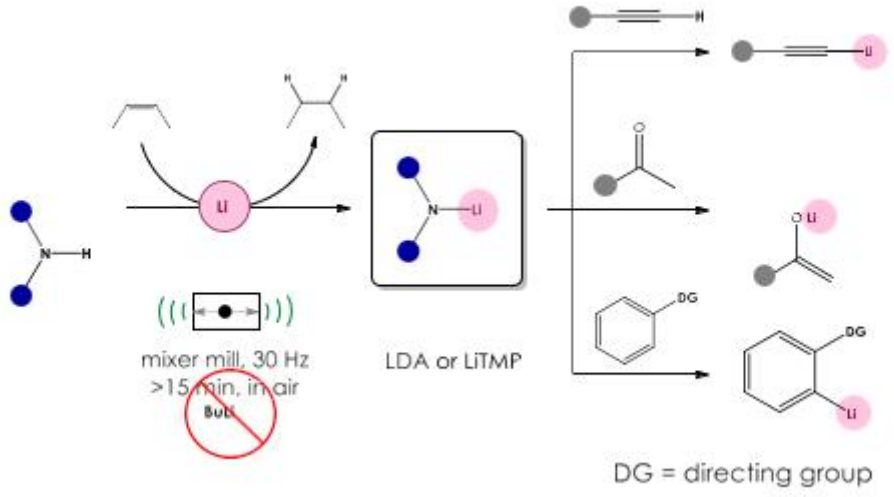
In recent years, mechanochemistry has emerged as a valuable tool for activating zero-valent metals in organic synthesis,[1] enabling the preparation and use of organometallic reagents under ambient conditions and without the need for bulk organic solvents or inert atmospheres. Organolithium compounds are powerful carbon nucleophiles used in the formation of carbon–carbon and carbon–heteroatom bonds. The fundamental method for generating organolithium species involves the reaction of organic halides with lithium metal, a process that has recently been adapted to mechanochemistry.[2] Once organolithium reagents such as butyllithium (BuLi) are prepared, a variety of other organolithium compounds can be accessed via metalation using BuLi itself or strong lithium amide bases derived from it.[3] These methods typically require cryogenic temperatures and an inert atmosphere when conducted in organic solvents.

Herein, we report the mechanochemical generation of strong lithium amide bases, such as lithium diisopropylamide (LDA) and lithium tetramethylpiperidide (LiTMP), directly from lithium metal and the corresponding amine using a mixer mill. This Birch-type transformation employs unsaturated hydrocarbons as sacrificial oxidants, thereby avoiding the use of BuLi. The reaction proceeds efficiently without solvent additives, although the use of THF or hexane as liquid-assisted grinding (LAG) agents accelerates the reaction, achieving full conversion within 15 minutes. We also investigated the stabilization of these lithium amide bases against hydrolysis by atmospheric moisture through the addition of various organic and inorganic additives, thereby extending their shelf life in air. The solid-state generated LDA was successfully applied to a range of transformations, including the enolization of ketones, conversion of terminal alkynes into lithium acetylides, and directed ortho-metalation (DoM)[3] of aromatic substrates under ambient conditions.



[1] Andrew C. Jones, Jamie A. Leitch, Sarah E. Raby-Buck and Duncan L. Browne, *Nat. Synth.* **2022**, *1*, 763–775.
[2] Keisuke Kondo, Koji Kubota and Hajime Ito, *Nat. Synth.* **2025**, *4*, 744–753.
[3] Victor Snieckus, *Chem. Rev.* **1990**, *90*, 879–933.

Oxidation Reactions Catalyzed by Me_3TACN-Mn Catalysts Under Mechanochemical Conditions

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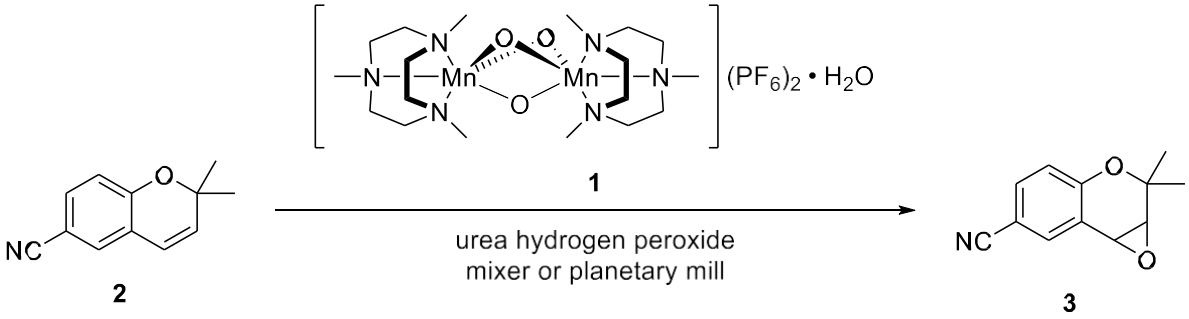

The Me_3TACN-manganese complex **1** is part of a family of peroxide-activating Mn-complexes, firstly described by Wieghardt in the 1980s. [1] Catalyst **1** found application in a detergent by Unilever, however, it showed too high activity for the public market. It was also intensively investigated as catalyst for the oxidation of diverse organic compounds, most prominently for the epoxidation of olefins. [2]

Here, we describe the first employment of complex **1** under mechanochemical conditions. The epoxidation of chromene derivative **2** with urea hydrogen peroxide (UHP) was chosen as the initial reaction system. Various parameters, including reaction time, oxidizing agent, and additives were tested in mixer and planetary mills. A focus was put on grinding additives, with silica gel leading to the highest yields of up to 33%. Also, the oxidation of diphenylmethanol and cinnamyl alcohol to the respective ketone or aldehyde was possible, albeit in low yields. Additionally, magic-angle spinning (MAS) NMR spectroscopy was used to study the catalyst composition.

The undesired catalase activity of **1**, also known from the reactions in solution, was identified as the most likely limiting factor for the product formation. [3] Various strategies to reduce the disproportionation of the peroxide, like addition of carboxylic acids, have already been tested, and will be further explored in the future.

[1] Karl Wieghardt, Ursula Bossek, Bernhard Nuber, Johannes Weiss, J. Bonvoisin, M. Corbella, S. E. Vitols, and J. J. Girerd, *J. Am. Chem. Soc.* **1988**, *110*, 7398–7411.
[2] Kevin F. Sibbons, Kirtida Shastri, and Michael Watkinson, *Dalton Trans.* **2006**, 645–661.
[3] Johann B. Kasper, Jorn D. Steen, Ronald Hage, and Wesley R. Browne, *Adv. Inorg. Chem.* **2021**, *78*, 143–182.

P-47

Synthesis and Characterization of $SrMoO_4$ and $SrMoO_4$@C Powders as Lithium Storage Anode Materials

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In this work, pure $SrMoO_4$ and carbon coated $SrMoO_4$@C were synthesized via a simple mechanochemical synthesis and with glucose ($C_6H_{12}O_6$) as carbon source, respectively. A stoichiometric mixture of $SrCO_3$ and MoO_3 is subjected to intense mechanical treatment in the planetary ball mill (Fritsch–Premium line–Pulversette No 7). The tetragonal $SrMoO_4$ was direct obtained after 5h milling time applied the milling speed of 500 rpm. As prepared sample was dispersed into $C_6H_{12}O_6$ following by heat-treatment in order to prepared $SrMoO_4$@C. The obtained materials were characterized by X-ray diffraction (XRD), infrared (IR) and photoelectron (XPS) spectroscopies. Additionally, cyclic voltammetry (CV) measurements were performed on both samples.

The result of above mention analysis shows that the corbon source and calcination temperature effects on the crystallite size, phase composition and electrochemical properties. The average crystallite size was 22 nm and 61 nm for $SrMoO_4$ and $SrMoO_4$@C, respectively. IR and XPS spectra confirm the combination of $SrMoO_4$ with carbon source. The $SrMoO_4$@C shows the better electrochemical performance than pure $SrMoO_4$. This study demonstrated that $SrMoO_4$@C sample can be a promising candidate as anode material for lithium battery.

[1]

[1] Acknowledgements:
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P-48

In Situ Production of High Entropy Rare-Earth Hexaboride and Tetraboride by Mechanochemical Synthesis

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High entropy rare-earth borides have become very popular in recent decades with high mechanical strength, melting point, good corrosion, wear, and magnetic behavior. However, the production of these borides is very challenging and unique. The production of high entropy rare-earth boride (HEREB) nano-powders via mechanochemical synthesis (MCS) was reported in this study first time in the literature. Prepared as-blended powders and hardened steel balls (6 mm ø) were placed to hardened steel vials (50 ml). The ball-to-powder weight ratio (BPR) was kept constant at 10 : 1. The vials that include powders and balls were sealed under Argon (Ar) atmosphere (Linde, 99.999 % pure) in a MBraun glove box. Then, mechanochemical synthesis (MCS) was conducted on the as-blended powders in a high energy NanoMultiMix mill at 920 rpm for different durations up to 8 h. The high-energy impacts that caused mechanical deformation in the powder particles provided the necessary energy to start the reaction. After MCS, while the rare-earth borides formation was provided, MgO occurred by-product. The selective acid leaching with 4 M HCl solution (HCl, Merck, 37 %) was applied to the MCS'ed powders to obtain rare-earth borides under the ultrasonic stirring by using a Bandelin Sonorex RK-100H ultrasonic bath. The leaching treatment was conducted at 80 °C for 15 min with a solid-to-liquid ratio of 1 g / 10 cm_3. The residue was then separated from the leaching solution by repeated centrifugation (Hettich Rotofix 32 A, 4000 rpm, 30 min), decantation, and rinsing steps. Repeated centrifugation was arranged to the proper pH value of the solution. When this value reached to ~5, the centrifugation was stopped, and the residue was dried in an FN 500 stove at 120 °C for 24 h in air. Supernatant liquids of the leached product were collected in a volumetric flask and subsequently subjected to chemical analysis to control the contaminants arose from debris of the milling media. The nano-structured HEREB powders were analyzed using X-ray diffraction (XRD). Particle size analysis were conducted on powders.

Direct Mechanoorganocatalysis via Surface Functionalization of Milling Reactors

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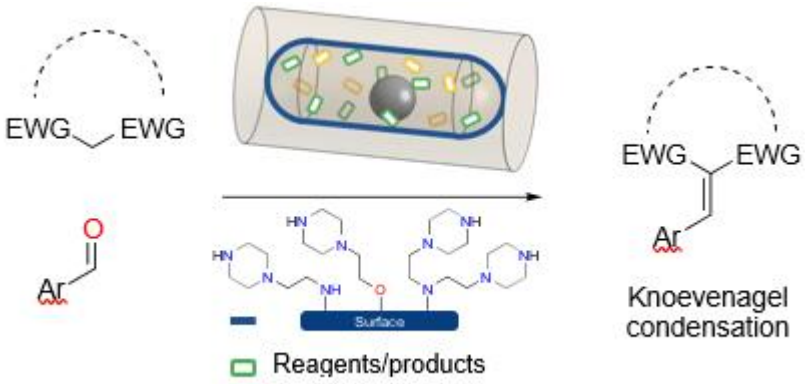
Mechanochemistry, particularly ball milling, has emerged as a green and efficient alternative to traditional solution-based synthesis. This solvent-free approach has found broad applications in organic, medicinal, and materials chemistry.[1] To further expand the scope of these reactions, catalysts can be directly added to the reaction medium, enabling catalyzed transformations. However, this method often requires multiple purification steps to remove residual catalysts, leading to increased waste, longer reaction times, and a reduction in the overall sustainability of the process. To address these limitations, metal-based direct mechanocatalysis has been developed. This approach employs metal-coated vessels, balls, or even metal-based balls[2] to catalyze reactions without introducing catalysts into the bulk reaction mixture.

In contrast, direct mechanoorganocatalysis under ball-milling conditions has been unexplored yet. Therefore, in this work, we present a novel approach using epoxy resin milling jars with pendant groups to anchor organocatalysts onto the jar surface. This design prevents catalyst leaching into the reaction medium, enabling a reusable, heterogeneous catalytic system. These newly functionalized jars demonstrated high efficiency in both the Knoevenagel condensation[3] and a multicomponent reaction,[4] offering a sustainable and practical alternative for mechanochemical synthesis.



[1] J. L. Howard, Q. Cao, D. L. Browne, *Chem. Sci.*, **2018**, *9*, 3080-3094.
[2] W. Pickhardt, S. Gratz, L. Borchardt, *Chemistry,* **2020**, *26*, 12903-12911.
[3] R. Trotzki, M. M. Hoffmann, B. Ondruschka, *Green Chemistry,* **2008**, *10*, 767-772.
[4] M. Amirnejad, M. R. Naimi-Jamal, H. Tourani, H. Ghafuri, *Monatsh. Chem.,* **2013**, *144*, 1219-1225.

P-50

Advancing Mechanochemistry's Impact: Education, Innovation, Professional Development, and Outreach Programs in the NSF Center for the Mechanical Control of Chemistry (CMCC)

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The NSF Center for the Mechanical Control of Chemistry (CMCC) is a Phase II center in the U.S. National Science Foundation's Centers for Chemical Innovation (CCI) program. The CMCC brings together chemists, physicists, and engineers who all work together to achieve the goal of establishing a fundamental understanding of mechanochemistry. By applying new instrumental, chemical, and theoretical approaches, the CMCC aims to advance the ability to design, predict, and scale up mechanically driven chemical reactions. This work is critical to building more sustainable and energy-efficient approaches to chemical synthesis, and the Center's broader efforts reflect that vision. Together with the research activities in the center, the CMCC leads a range of initiatives that support and promote education, innovation, professional development, and public engagement. These include a Research Experience for Undergraduates (REU) program in mechanochemistry, and a new International Research Experience in Science (IRES) program, a Smash Chemistry summer camp for high school students, and a web-based textbook, currently in development, that introduces foundational mechanochemistry concepts, methods, and applications. To support new research, and foster connections with industry, the Mechanochemistry Innovation Hub (MIH) provides open access to tools for mechanochemical synthesis, and a mechanochemical reactions database. The CMCC also collaborates with the Science History Institute to create podcasts, digital exhibits, and other public-facing resources that explore both the science and the history of mechanochemistry. This poster presents an overview of the Center's current activities across research, innovation, education, and outreach. Together, these efforts reflect the CMCC's broader mission: to drive scientific discovery while building public engagement, training future scientists, and making sustainable chemistry more accessible and understandable.

P-51

Building Fire Safety in the Solid State: Scaling Mechanochemistry for Coating Applications

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Mechanochemistry is gaining relevance as a scalable and sustainable route for the synthesis of advanced functional materials. Within the REFUGI project, we report the successful scale-up of solid phosphorus-based flame retardants via solvent-free twin-screw extrusion (TSE), reaching continuous production rates of up to several kg/h. The synthesized additives belong to a family of organophosphorus compounds tailored for high thermal stability and strong flame inhibition.

These materials have been specifically developed for incorporation into solvent-based varnish coatings, targeting applications on construction, furniture and mobility industries. When incorporated into commercial coating formulations, the mechanochemically synthesized flame retardants delivered up to 20 % lower peak heat release rates, 25 % slower flame growth, 30 % reduced total oxygen consumption, and an over 80 % decrease in smoke production compared to conventional additives—dramatically suppressing flame spread and improving overall fire performance.

This work demonstrates the viability of using reactive extrusion as an industrial mechanochemical platform for additive production, enabling the design of high-performance, solvent-free flame-retardant systems compatible with existing coating technologies. REFUGI establishes a solid foundation for the industrial uptake of mechanochemistry in the fire safety sector.

Asymmetric [5,5] Sigmatropic Rearrangement: Excellent Chirality Transfer Enabled by Mechanochemistry

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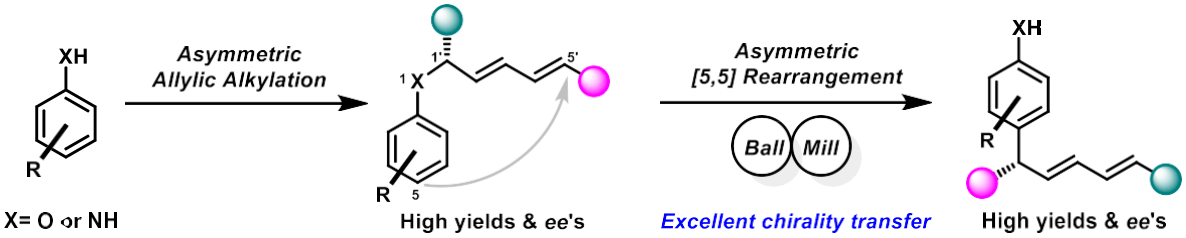
While the well-established [3,3] sigmatropic rearrangements have been thoroughly explored and widely applied, its [5,5] counterpart remains largely overlooked and underdeveloped. Only in recent years, the utility of the [5,5] sigmatropic rearrangement has gained increasing recognition.[1-3] The corresponding asymmetric variant has, to date, remained completely unknown.

Figure 1. Novel aryl-dienyl rearrangement.



In this study, we introduce the first asymmetric [5,5] sigmatropic rearrangement of enantioenriched aryl-dienyl substrates. The reaction proceeds through a well-defined 10-membered transition state, allowing for highly predictable regio- and stereoselectivity. Enantioenriched chiral ethers or amines are synthesized via an optimized Asymmetric Allylic Alkylation (AAA) protocol, affording high yields with exceptional enantiopurity from readily available starting materials. Mechanochemical grinding in combination with mild Lewis-acid catalysis enables an efficient and solvent-free transformation with excellent chirality transfer. This strategy grants access to a diverse range of para-alkylated phenols and anilines with high enantioselectivity. Furthermore, the chiral diene chain offers a versatile synthetic handle for downstream functionalization, for example via Diels–Alder cycloaddition. By expanding the scope of sigmatropic rearrangements into the asymmetric [5,5] domain, this work opens new avenues for selective and sustainable synthesis.

[1] C. Hu, K. Farshadfar, M. C. Dietl, A. Cervantes-Reyes, T. Wang, T. Adak, M. Rudolph, F. Rominger, J. Li, A. Ariafard, A. S. K. Hashmi, ACS Catal. **2021**, 11, 6510–6518.
[2] Y. Liang, B. Peng, Acc. Chem. Res. **2022**, 55, 2103–2122.
[3] M. Kaiser, M. Steinacher, F. Lukas, P. Gaertner, RSC Adv. **2023**, 13, 32077–32082.

Routes to Direct Mechano-*Organo*catalysis (DMOC)

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Recent advancements in mechanochemistry have expanded its scope beyond simple comminution to encompass precise, selective synthesis, enabling complex organic, inorganic, and materials transformations without the need for bulk solvents. Progress has been achieved in areas such as reaction monitoring, mechanistic elucidation, and process intensification. Notably, significant developments in direct mechanocatalysis—where the reaction vessel or milling media themselves serve as a source of catalytically active transition metals—have enabled a range of transformations, including Sonogashira[1] and Suzuki couplings[2], as well as CuAAC reactions.[3] Furthermore, efforts to translate direct mechanocatalysis into twin-screw extrusion (TSE) processes have also been reported, most notably through the coating of screw segments with catalytically active transition metals such as palladium, as demonstrated by Borchardt[4] and James,[5] to catalyse cross-coupling reactions.

To date, direct mechanocatalysis has not been extended to organocatalysis, as common coating technologies such as electroplating and physical vapour deposition are not directly transferrable. Here, we introduce three strategies for immobilising organocatalysts onto reactor vessels and grinding media to realise direct mechano-organocatalysis (DMOC), progressing from ball milling to twin-screw extrusion (TSE). Notably, we demonstrate that gold–thiol self-assembled monolayer (SAM) formation can effectively anchor secondary amines, enabling the solvent-free mechanocatalytic Knoevenagel condensation.

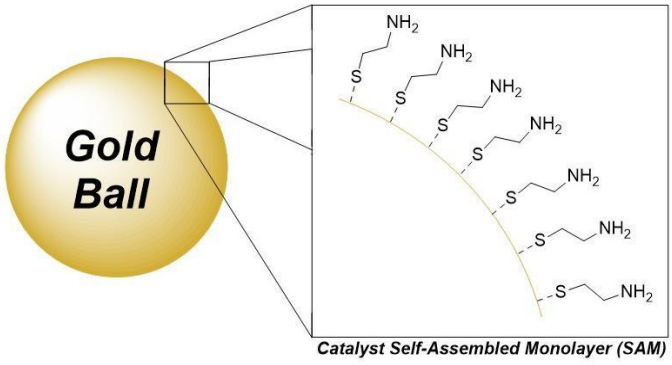

Figure 1: Self-assembled monolayer formation on a gold coated ball bearing.

[1] Dennis A. Fulmer, William C. Shearouse, Shareika T. Medonza and James Mack, Green Chem., 2009,**11**, 1821-1825

[2] Wilm Pickhardt, Claudio Beaković, Maike Mayer, Maximilian Wohlgemuth, Fabien Joel Leon Kraus, Martin Etter, Sven Grätz, and Lars Borchardt, Angew. Chem. Int. Ed. **2022**, 61, e20220500

[3] Teresa L. Cook, James A. WalkerJr. and James Mack, Green Chem., 2013, **15**, 617

[4] Viviene Chantrain, Tilo Rensch, Wilm Pickhardt, Sven Grätz, and Lars Borchardt, Chem. Eur. J., 2024, **17**, e202304060.0

[5] Unpublished results, Aaron S. McCalmont (QUB), Hugh Hamilton (JM), Stefania M. Scalzullo (JM), Deborah E. Crawford (UoB) and Stuart L. James (QUB).

P-54

Ball Milling Enabled High-throughput Mechanochemistry For Solvent to Solventless Organic Reaction Translation

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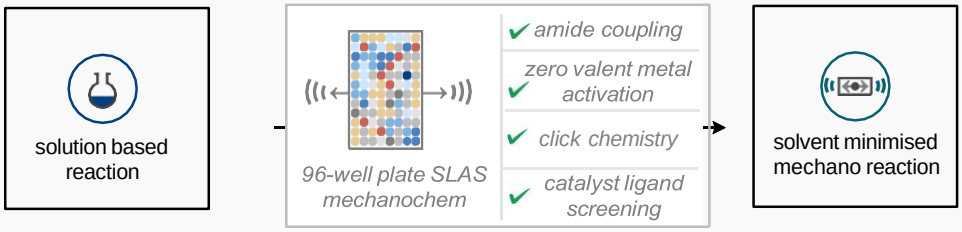

A general 96-well plate design for the translation of solvent based reactions into solvent-minimisedmechanochemical process

High-throughput mechanochemistry offers a powerful approach to sustainable synthesis by enabling solvent-minimized reactions under ambient conditions.[1][2] In this work, we developed a scalable and automation-compatible platform for parallel mechanochemical screening using a modified shaker mill and SLAS-format vial arrays. The system supports both manual and automated workflows and allows efficient exploration of variables such as solvents, additives, catalysts, and ligands.

We demonstrated the platform's utility across diverse reaction classes, including amide coupling, redox chemistry, selective N-alkylation, and metal-catalyzed cross-couplings. The approach enabled both reaction optimization and the discovery of novel reactivity.This platform provides a practical and generalizable method for accelerating reaction development in a greener and more efficient manner, bridging the gap between academic research and scalable synthetic processes.[3]

[1] K. Martina, L. Rotolo, A. Porcheddu, F. Delogu, S. R. Bysouth, G. Cravotto, E. Colacino, *Chem. Commun.* **2018**, *54*, 551-554.
[2] Q. Lemesre, T. Wiesner, R. Wiechert, E. Rodrigo, S. Triebel, H. Geneste, *Green Chemistry* **2022**, *24*, 5502- 5507.
[3] Unpublished work, C. Yu, P. Kollmus, C. Waudby, M. Santagostino, F. Roschangar, D. L. Browne.

P-55

Photoextrusion: Twin-Screw Extrusion for Continuous Solid-State Photochemistry

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Photochemical synthesis provides access to unique reaction pathways and can offer high selectivity under mild conditions; [1] however, challenges with scalability and solvent requirements have limited its wider adoption. To address some of these issues, recent photomechanochemical methods, such as photo–ball milling, integrate mechanical energy and light to enable solvent-free solid-state transformations. [2, 3] Nevertheless, their inherently batch-based operation continues to pose challenges for large-scale industrial implementation.

We address this challenge through photoextrusion—the integration of light irradiation into twin-screw extrusion (TSE), a continuous, solvent-free, and industry-ready processing platform. This approach enables simultaneous mechanical processing and photochemical activation within a continuous system. Using this method, we demonstrate the feasibility of continuous, solvent-free photochemistry via [2+2] cycloadditions and photocatalysed transformations. These results establish photoextrusion as a scalable and practical route to industrially relevant photochemical synthesis.

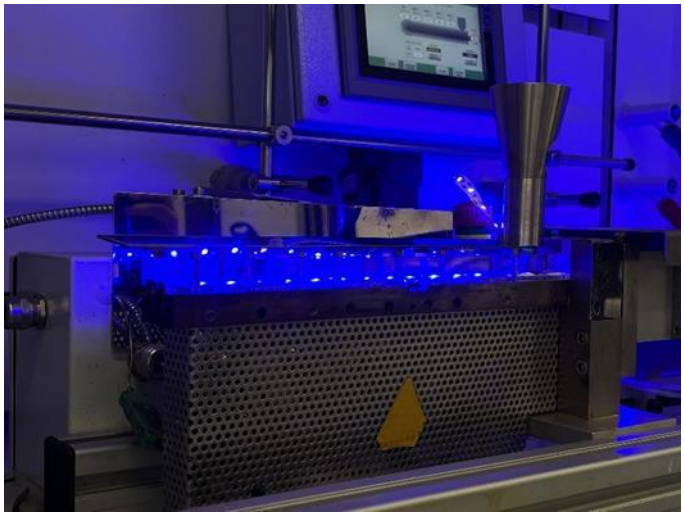

Figure 1: Photoextruder for continuous, solid-state photochemical reactions

[1] N. Hoffmann, N. *Chem. Rev.* **108**, 1052–1103 (2008).
[2] Hernández, J. G. *Beilstein J. Org. Chem.* **13**, 1463–1469 (2017).
[3] Štrukil, V. & Sajko, I. *Chem. Commun.* **53**, 9101–9104 (2017).

P-56

Upscaling Mechanochemical Lignin Modification: Small Ball vs. Planetary Milling

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Lignin is the second most abundant biopolymer on our planet after cellulose and is one of the least utilized renewable raw materials. It is mainly produced as a by-product in the pulp and paper industry, but its heterogeneous structure and broad molecular weight distribution make it difficult to use in a wider range of applications. Homogenization of the molecular weight distribution and targeted functionalization are therefore crucial steps in unlocking the potential of lignin for high-value applications. Mechanochemistry offers a solvent-free and energy-efficient approach to this end.

In this work, technical lignin was functionalized using sodium percarbonate and sodium hydroxide. Two reagents that are already established in industrial processes. The aim was to narrow the molecular weight distribution and introduce additional oxygen-containing functional groups, in particular carboxyl groups. Initial experiments were carried out in a laboratory ball mill[1]. For upscaling, a planetary mill was then used to evaluate the transferability of the process to larger scales. Differences in reaction yield, degree of functionalization, and molecular weight distribution between the two grinding processes are presented, as are practical challenges in scale-up.

The results provide insights into mechanistic and process-related influencing factors in lignin modification at different scales and underscore the potential of mechanochemistry as a sustainable and industrially relevant approach to lignin valorization.

[1] Friedrich Fink, Tomasz M. Stawski, Jörg M. Stockmann, Franziska Emmerling, Jana Falkenhagen, *Biomacromolecules*, **2023**., *24(9)*, 4274-4284.

P-57

Efficient and Eco-Friendly Recovery of PGMs from Spent Automotive Catalysts by Mechanochemical Approach

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The recovery of platinum group metals (PGMs) such as Pt, Pd, and Rh from spent automotive catalysts is of growing strategic importance due to their limited supply, high economic value, and essential role in automotive emission control and various high-tech applications. Traditional recovery methods rely on high-temperature pyrometallurgy or aggressive hydrometallurgical leaching, which are energy-intensive and involve release of hazardous emissions both toxic gases, polluted water, solid and liquid wastes. Mechanochemistry, which uses mechanical energy to drive solid-state reactions, offers an environmentally friendly and cost-effective alternative that aligns with green chemistry principles.

In this study, mechanochemical activation was applied to enhance the accessibility and extractability of PGMs from real spent automotive catalyst (SAC) samples containing various metal combinations supported on cordierite. The performed high-energy ball milling promoted particle size reduction, increased surface area, and generated fresh reactive surfaces, facilitating more efficient PGM leaching in subsequent mild chemical processing. The treated materials were characterized by X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), X-ray fluorescence (XRF), scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS), and Fourier-transform infrared spectroscopy (FTIR) to monitor structural changes, surface composition, and PGM dispersion.

The results demonstrated that the established treatment protocol led to redistribution and exposure of PGMs on the catalyst surface, increasing extraction yields while significantly reducing reagent consumption and processing time. This approach decreases the need for using bulk aggressive leaching agents and the environmental impact, providing a scalable route for sustainable PGM recycling. Mechanochemistry thus represents a promising pathway toward optimized industrially viable, low-impact recovery of critical metals from end-of-life automotive catalysts, contributing to circular economy goals.

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P-58

Green Synthesis and Solubility Profile of New Lenalidomide:Quercetin Cocrystal

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Considered by IUPAC one of "top ten emerging technologies in chemistry",[1] mechanochemistry is a sustainable method, that uses mechanical force to drive chemical reactions, minimizing the amount of waste and unnecessary by-products.[2] In this work, we report two different mechanochemical approaches to obtain a novel cocrystal of lenalidomide, an immunomodulatory agent used in the treatment of multiple myeloma,[3] with quercetin. Cocrystal structure is stabilized by $O\text{-}H_{QUER}\cdots H_{LENA}$ / $O\text{–}H_{QUER}\cdots N_{LENA}$ / $N\text{–}H_{LENA}\cdots O_{QUER}$ hydrogen bonds and face-to-face π-π interactions along the *c* axis. Accelerated stability studies showed that this cocrystal remains stable, presenting good properties for handling and storage in the solid state. In addition, solubility studies showed that regardless of synthetic method, LENA:QUER cocrystal has a lower solubility compared with pure LENA, suggesting that the cocrystal may function for an extended release of LENA. This study provides valuable insights into the design and development of cocrystals using a cleaner route to modify the physicochemical properties of drugs.

Acknowledgements
The authors acknowledge IMPACTIVE (Innovative Mechanochemical Processes to synthesize green ACTIVE pharmaceutical ingredients),[4,5] the research project funded from the European Union's Horizon Europe research and innovation programme (European Health and Digital Executive Agency) under grant agreement No. 101057286. A.C.S.C, V.A., M.T.D acknowledge: Centro de Química Estrutural is a Research Unit funded by Fundação para a Ciência e a Tecnologia through projects UIDB/00100/2020 (https://doi.org/10.54499/UIDB/00100/2020) and UIDP/00100/2020 (https://doi.org/10.54499/UIDP/00100/2020). Institute of Molecular Sciences is an Associate Laboratory funded by Fundação para a Ciência e a Tecnologia through project LA/P/0056/2020 (https://doi.org/10.54499/LA/P/0056/2020). M.S. acknowledge: This work received financial support from the PT national funds (FCT/MCTES, Fundação para a Ciência e Tecnologia, and Ministério da Ciência, Tecnologia e Ensino Superior) through the project UID/50006 - Laboratório Associado para a Química Verde - Tecnologias e Processos Limpos. M. Sarraguça thanks the Individual Call to Scientific Employment Stimulus 2022 (DOI 10.54499/2022.01388.CEECIND/CP1724/CT0003).

[1] Isaiah R. Speight, Karen J. Ardila-Fierro, José G. Hernández, Franziska Emmerling, Adam A. L. Michalchuk, Felipe García, Evelina Colacino, James Mac, Nat. Rev. Methods Prim. **2025**, 5:29, 1-18.
[2] Or Galant, Giacomo CerfedaAaron S. McCalmont, Stuart L. James, Andrea Porcheddu, Francesco Delogu, Deborah E. Crawford, Evelina Colacino, Sabrina Spatari, *ACS Sustain. Chem. Eng.* **2022**, 10, 1430–1439.
[3] Martin A. Screen, Gary Tomkinson, James F. McCabe, Sean Askin, Clare S. Mahon, Mark R. Wilson, Jonathan W. Steed, New J. Chem. **2025**, 49, 6535-6543.
[4] IMPACTIVE: Innovative Mechanochemical Processes to synthesize green ACTIVE pharmaceutical ingredients. https://mechanochemistry.eu/ (accessed 17 June, **2025**).
[5] Juan José Sáenz de la Torre, Leyre Flamarique, Fernando Gomollon-Bel, Evelina COLACINO, *Open Res. Europe*, **2025**, 5, 73.

P-59

Mechanochemical Synthesis of Framework Materials for the Electrocatalytic Reduction of Nitrate to Ammonia

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The electrocatalytic reduction of nitrate to ammonia offers a sustainable pathway to address nitrate pollution while providing an alternative route for ammonia production under mild conditions. This reaction has gained significant attention as an energy-efficient and environmentally friendly alternative to the Haber–Bosch process. Framework materials, such as metal–organic frameworks (MOFs), present highly tunable structures, large surface areas, and accessible metal centers, making them attractive candidates for catalytic applications. However, their limited long-term electrochemical stability often necessitates post-synthetic modifications. Mechanochemical synthesis of framework materials has not only emerged as a solvent-minimized, sustainable, and rapid synthetic approach, it has also shown the potential to generate unique active catalytic phases not readily accessible via conventional solvothermal synthesis. It allows precise compositional control, defect generation and facile incorporation of multiple metal species.

In this work, we explore a series of MOF-based systems for nitrate electroreduction. To overcome intrinsic conductivity and stability limitations, the MOFs are subjected to controlled calcination, yielding a conductive graphitic support in which the nature of the resulting metal species can be tuned by varying calcination parameters. We investigate different mechanochemical synthesis strategies, including alloying and multi-metal incorporation, to explore synergistic effects between active sites. Initial findings suggest that these multi-metallic materials exhibit enhanced catalytic activity compared to single-metal counterparts. Furthermore, mechanochemically derived catalysts show promising performance trends that surpass those prepared by traditional solvothermal methods. Overall, our results indicate that the combined mechanochemical–calcination strategy is a versatile and effective route toward robust, high-performance electrocatalysts for nitrate-to-ammonia conversion.

P-60

Mechanochemical Activation of Spent Lithium Cobalt Oxide Cathodes: A Sustainable Approach for Metals Recovery

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Lithium-ion batteries (LIBs) containing lithium cobalt oxide ($LiCoO_2$) cathodes are widely used in electric vehicles, leading to a growing volume of battery waste[1, 2]. This waste poses a significant environmental hazard due to the presence of heavy metals, while simultaneously representing a valuable secondary source of critical raw materials. One of the promising methods is hydrometallurgical treatment for the efficient recovery of metals. The concept of this work involves mechanochemical strategy for the reduction of refractory $LiCoO_2$ to individual lithium and cobalt oxides using aluminum and carbon as internal reducing agents to enhance material's reactivity. The experiments were carried out using a planetary ball mill under different conditions designed via Taguchi method (500–800 rpm; 15–60 min; BPR 35–80; ball size 4–10 mm) to optimize processing parameters.

The mechanochemical activation significantly altered the phase composition and morphology of the cathode material. Aluminum addition facilitated the formation of CoO, $CoAl_2O_4$, and AlCo phases via redox and alloying reactions, while carbon induced partial reduction and amorphization without forming new crystalline compounds. These transformations enhance the material's reactivity, making it more vulnerable to subsequent hydrometallurgical leaching. Morphological studies showed a dramatic particle size reduction and surface roughening as a result of milling, indicating enhanced reactivity.

Ammoniacal leaching in 3.0 M $NH_3 \cdot H_2O$ + 1.0 M $(NH_4)_2CO_3$ (L/S = 25 mL g^{-1}; 60 °C; 6 h) yields up to 94.6% Li and 83.7% Co for the Al/C co-activated material. Performance is lower for the untreated cathode sample (34.7% Li, 18.8% Co) and for the sample using only Al (91.8% Li, 80.1% Co) or only the one milled under the optimum conditions determined by Taguchi method one without reductants (79.7% Li, 67.3% Co). Raising the temperature to 80 °C further improves recoveries to ~97.0% Li and 87.9% Co within 6 h. Coupling mechanochemical activation with in-situ Al/C reduction thus enables phase engineering that dramatically enhances ammoniacal leachability under mild conditions while eliminating prior Al/C separation—offering a cleaner, scalable path for LCO cathode recycling.

[1] Biswal, B. K., Zhang, B., Tran, P. T. M., Zhang, J., & Balasubramanian, R. Recycling of spent lithium-ion batteries for a sustainable future: recent advancements. *Chemical Society Reviews,* **2024,** 53(11), 5552-5592.
[2] Tian, G., Yuan, G., Aleksandrov, A., Zhang, T., Li, Z., Fathollahi-Fard, A. M., & Ivanov, M. Recycling of spent Lithium-ion Batteries: A comprehensive review for identification of main challenges and future research trends. *Sustainable Energy Technologies and Assessments,* **2022,** 53, 102447.

Soft Magnetic Composites Prepared by Resonant Acoustic Mixing and Self-Milling Methods

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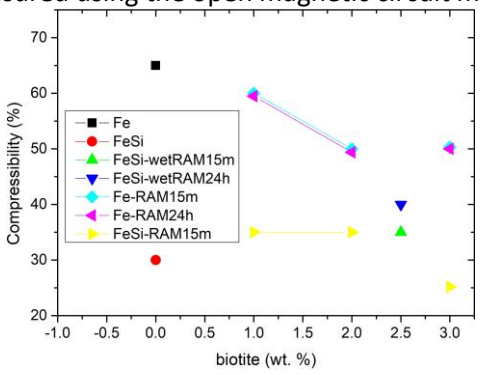
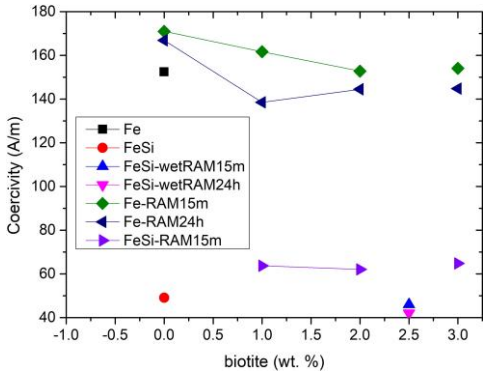
Soft magnetic composites (SMC) are materials made of ferromagnetic powder particles coated with electrical insulation, usually produced using press and sinter methods. The best applied ferromagnetics, excluding advanced amorphous/ nanocrystalline alloys, involve pure iron and iron-silicon alloys. Insulation materials consist of thin layers of polymeric resins, dielectric ceramics, or their combinations. Organic coatings enhance compressibility and act as a binder between ferromagnetic particles, whereas ceramic dielectrics are characterised by their high electrical and thermal resistivity, but they reduce compressibility and restrict manufacturing options. In relation to the necessary high-frequency SMCs for efficient power electronics, there is an urgent need to apply new materials and technologies for designing thin dielectric layers [1 2].

Fe/biotite and FeSi/biotite were prepared using the Resonant Acoustic Mixing (RAM) technique. Three different methods were utilised for coating biotite powder ($d_{0.5}$ = 6 μm) onto the surface of Fe ($d_{0.5}$ = 105 μm) and Fe6.5Si ($d_{0.5}$ = 170 μm and 240 μm) particles: a) dry mixing for 15 minutes, b) wet mixing in etanol for 15 minutes, and c) dry coating using the self-milling process [3] for 24 hours. Prepared composite powders with 1, 2, 3 and wet RAM.2.5 wt.% of biotite were examined to analyse the connection between technology, microstructure, and properties. Compressibility was assessed using in-die continuous compressibility curves. Microstructure focused on the surface morphology of composite powder particles, which was observed with SEM-EDS. Coercive field strength was measured using the open magnetic circuit method.





The compressibility of hard-to-compress FeSi can be enhanced through wet RAM process with 2.5% biotite or a dry RAM (15 m). In Fe, increasing biotite content reduces compressibility regardless of the process duration. The coercivity of all FeSi/biotite samples is slightly higher compared to pure FeSi, but it remains almost independent of biotite content in dry RAM. In wet RAM, however, with 2.5 wt.% biotite, it can maintain the same value as pure FeSi. Investigation of surface morphology of composite powders highlighted the importance of biotite distribution depending on RAM process parameters and their effect on the development of compressibility and coercivity.

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[1] A. Talaat, M.V. Suraj, K. Byerly, A. Wang, Y. Wang, J.K. Lee, P.R. Ohodnicki Jr, *J. Alloys Compd.* **2021**, 870, 159500 -1-19.

[2] R. Bureš, M. Fáberová, V. Milyutin, Z. Birčáková, P. Kollár, J. Füzer. *Sci Data*, **2025**, 12/8, 1-14.

[3] Radovan Bureš, Mária Fáberová. Method of production of composite magnetic powders by autonomous milling: Patent file No. SK 289198 B6. Banská Bystrica: Office of Industrial Property SR, **2024**. 7 p.

Green Hydrometallurgical Approach for Lithium and Cobalt Recovery from Spent $LiCoO_2$ Batteries Using Ammonia-Based Processes

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The intensive production and use of lithium-ion batteries, particularly in the context of the development of electric vehicles and renewable energy storage systems, has made the efficient recycling of these batteries at the end of their service life a pressing issue [1]. Lithium cobalt oxide ($LiCoO_2$), which is frequently used as the cathode material in lithium-ion batteries, contains strategically essential metals such as lithium and cobalt, whose natural reserves are limited and market prices are high [2]. Although existing acid-based hydrometallurgical methods provide high recovery yields, the formation of waste solutions rich in sulfate or chloride ions renders these processes environmentally and economically disadvantageous [3].

In this study, the ammonia leaching method and an energy-efficient ammonia distillation process were comprehensively optimized for the recovery of lithium and cobalt from spent $LiCoO_2$ cathodes. Preliminary thermal treatment (500 °C, 2 hours, in an inert atmosphere) enabled the complete removal of organic binders and residual electrolyte. During ammonia leaching, using solutions of 6 M NH_3 and 1.5 M $(NH_4)_2CO_3$ at a liquid-to-solid ratio of 10:1, a temperature of 70 °C, and a stirring duration of 5 hours, recovery efficiencies of 82.5 % for lithium and 96.1 % for cobalt were achieved. Kinetic analysis indicated that the process followed a first-order reaction, with activation energies determined to be 76.54 kJ/mol for lithium and 97.22 kJ/mol for cobalt, confirming a chemically controlled reaction mechanism.

For selective metal separation, lithium was first precipitated as Li_2CO_3 (yield: 98.5 %) by adding sodium hydroxide to adjust the pH to 10.7–10.8. The subsequent ammonia distillation process was carried out at 95–98 °C, reducing the stability of $Co(NH_3)_6^{2+}$ complexes and enabling the precipitation of cobalt as $CoCO_3$. At 98 °C, the energy consumption was approximately 282 kJ/mol, which was found to be about seven times lower compared to low-temperature processes.

The results of the study demonstrated that the integrated technology combining ammonia leaching and energy-efficient distillation can serve as an alternative to acid-based hydrometallurgy, enabling the high-yield recovery of metals on an industrial scale with the possibility of reagent reuse and reduced energy consumption.

[1] Jin, S., Mu, D., Lu, Z., Li, R., Liu, Z., Wang, Y., Tian, S., & Dai, C. A comprehensive review on the recycling of spent lithium-ion batteries: Urgent status and technology advances. *Journal of Cleaner Production*, **2022**, 340, 130535. https://doi.org/10.1016/j.jclepro.2022.130535

[2] Natarajan, S., & Aravindan, V. An urgent call to spent LIB recycling: Whys and wherefores for graphiterecovery. *Advanced EnergyMaterials*, **2020,** 10(28), 2002238. https://doi.org/10.1002/aenm.202002238

[3] Qi, Y., Meng, F., Yi, X., Shu, J., Chen, M., Sun, Z., Sun, S., & Xiu, F.R. A novel and efficient ammonia leaching method for recycling waste lithium-ion batteries. *Journal of Cleaner Production*, **2020**, 251, 119665. https://doi.org/10.1016/j.jclepro.2019.119665

One-Pot Mechanochemical Synthesis of Spin Crossover Materials

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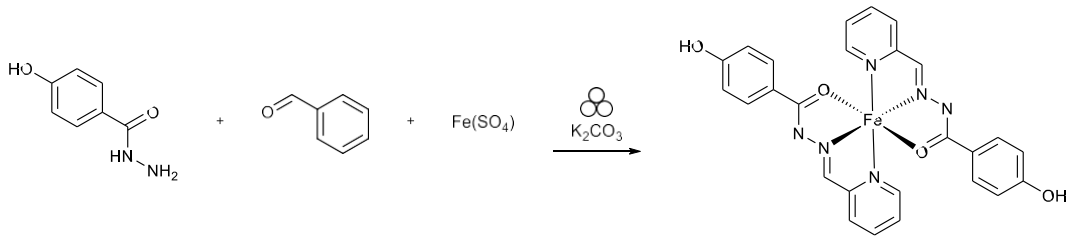
Spin Crossover (SCO) materials are switchable transition metal complexes and coordination polymers with promising applications in sensing, actuation[1] and more recently in solid state cooling.[2] If SCO materials are to achieve their potential in these fields, then it is vital that environmentally sound and industrially scalable methodologies are developed for their synthesis. We have developed mechanochemical[3] and accelerated ageing[4] routes for the synthesis of these materials as well as for their post-synthetic modification.[5] We are also interested in understanding the effect of grinding on their crystallinity and cooperative switching properties.[6]

The Fe^{2+} complex $[Fe(HPMB)_2]$ (Scheme 1, HPMB = 4-hydroxy-N0-((pyridin-2-yl)-methylene) benzohydrazide) has been previously reported.[7] Its SCO properties are ideal for application in barocaloric cooling, with an abrupt transition close to room temperature with narrow hysteresis. In recent work we have developed 'one-pot' processes to synthesise the HMPB ligand and associated metal complex in a single step.[8] Liquid-assisted grinding significantly increases the yield, and the use of a grinding auxiliary allows for in-situ ligand deprotonation, affording the final neutral SCO complex. An annealing step ensures the mechanochemically synthesised complex retains its cooperative SCO properties. This poster will present recent progress and current challenges in this work.



Scheme 1. One-pot synthesis of $[Fe(HPMB)_2]$

‡UK participants in Horizon Europe Project First Regenerative Solid-State Barocaloric Refrigerator (FROSTBIT) are supported by UKRI grant number 10134597.

[1] H. J. Shepherd, I. A. Gural'skiy, C. M. Quintero, S. Tricard, L. Salmon, G. Molnár and A. Bousseksou, *Nat. Commun.*, 2013, **4**, 2607.
[2] S. P. Vallone, A. N. Tantillo, A. M. dos Santos, J. J. Molaison, R. Kulmaczewski, A. Chapoy, P. Ahmadi, M. A. Halcrow and K. G. Sandeman, *Adv. Mater.*, 2019, **1807334**, 1–7
[3] J. H. Askew and H. J. Shepherd, *Chem. Commun.*, 2018, **54**, 180–183
[4] R. F. Lonergan, G. A. Conway, P. W. Doheny and H. J. Shepherd, *Chem. - A Eur. J.*, 2022, **28**, e202201823
[5] J. H. Askew and H. J. Shepherd, *Dalt. Trans.*, 2020, **49**, 2966–2971
[6] J. H. Askew, D. M. Pickup, G. O. Lloyd, A. V. Chadwick and H. J. Shepherd, *Magnetochemistry*, 2020, **6**, 1–12
[7] L. Zhang, G.-C. Xu, H.-B. Xu, T. Zhang, Z.-M. Wang, M. Yuan and S. Gao, *Chem. Commun.*, 2010, **46**, 2554–2556
[8] C. Dunsford, Lewis Jackson, Y. Sabater Algarra, H. J. Shepherd, Unpublished work

P-64

Tailoring Accessible Surface Area of Activated Carbons via Mechanochemical Processing

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Controlling the external accessibility of porous carbons without compromising their internal pore network is a challenge with important implications for storage, separation, and containment technologies. In this work, activated carbons were subjected to ball milling in the presence of aromatic compounds to tune their externally measurable specific surface area (SSA). The pristine hard carbon exhibited an SSA of 970 m^2g^{-1}, which was reduced to just 95 m^2g^{-1} under the most intensive milling conditions. This dramatic decrease is attributed to the combined effects of particle fragmentation and partial blockage of pore entrances during milling. While nitrogen adsorption measurements reveal a substantially smaller accessible surface area, complementary analysis suggests that the internal microporosity remains largely intact. The resulting materials thus retain significant internal storage capacity while presenting a far less accessible external surface.

Such selective modification enables the decoupling of internal volume from external reactivity, offering a pathway to design carbons for specialised applications. Potential uses include container and barrier materials for sensitive adsorbates, where preventing premature interaction with the environment is crucial, and composite systems requiring a protective carbon interface. Additionally, these materials could serve as controlled access supports in catalytic or electrochemical systems, where minimising unwanted surface reactions is advantageous. This study demonstrates that mechanochemical treatments can provide a straightforward and effective means to engineer carbon materials with tailored accessibility, unlocking new functionality in established porous systems.

Mapping the Breathing in Mechanochemically Synthesized MOFs: Structure–Property Relationships for Gas Capture

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This work presents an innovative approach to the synthesis of Metal-Organic Frameworks (MOFs) using mechanochemistry as a sustainable, solvent-free method. The study aims to develop MOF materials from biodegradable ligands, such as citrate and glutamate, in line with the principles of green chemistry. The primary focus is on the application of these novel materials for the adsorption and separation of gases of energetic and environmental interest, specifically H_2 and CO_2. A complementary strategic aspect of the work involves using the mechanochemical tool to obtain flexible, three-dimensionally porous materials, with advanced crystallographic methods applied to elucidate the crystal structure of the MOFs.[1]

Mechanochemical synthesis enables the production of MOFs with controlled structures in much more optimized time than conventional methods, for instance solvothermal. The study of the synthetic parameters can enhance the potential for specific interactions with H_2 and CO_2 molecules, improving characteristics of the structure such as surface area, tunable pore sizes, and high selectivity and adsorption capacity.[2] The mechanochemical synthesis of MOFs enables scalability and precise control of pore size, phase transitions, and chemical composition, which is critical for gas sorption mechanisms[3]. The metallic centers provide unique coordination environments and potential active sites for interaction with H_2 and CO_2 molecules. The use of flexible organic ligands like citrate and glutamate provides additional functionality to the MOF structure, contributing to structural stability and increasing the potential for selective adsorption. In this way, mapping the characteristics of both metal and organic ligands can help to optimize the material production.

In this context, the main objective of this work is to study the structure-property correlations that will contribute significantly to the development of more effective and environmentally friendly technologies for gas valorization, advancing the science of porous materials for solutions in energy storage (H_2) and climate change mitigation (CO_2). Mechanosynthesis as a tool for the green route to obtain these porous materials enables industrial-scale application and contributes to the paradigm of reversible and circular purification, with a direct impact on sectors such as gas capture and storage.

[1] Adarsh Singh, Soorya K K, Amit Bhatnagar, Ashok Kumar Gupta. *Separation and Purification Technology*, **2025**, 353, Part C, 128588.

[2] Federica Raganati, Mariangela Bellusci, Francesco Leardi, Francesca Varsano, Paola Ammendola. *Chemical Engineering Journal*, **2025**, 506, 159966.

[3] Wesam A. Ali, Selena E. Richards, Reem H. Alzard, Journal of Industrial and Engineering Chemistry, **2025**, 149, 63-93.Conference 14.-18.09.20

P-66

Native Mechanochemical Ligation

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Native chemical ligation is the predominant strategy for the chemoselective assembly of large peptides or even proteins, starting from an activated C-terminal fragment and a fragment displaying a N-terminal cysteine. A major challenge in native chemical ligation, and peptide chemistry more broadly, is the limited solubility of peptides, particularly when using hydrophobic fragments. With the emergence of mechanochemistry, an alternative to the conventional solvent based approach has been established. Although mechanochemistry is applicable to a broad range of chemical reactions, mechanochemical native chemical ligation has not yet been reported.

We report the development of native mechanochemical ligation, a peptide ligation methodology using mechanochemistry, that offers a robust approach in which peptide solubility is no longer a limiting factor. Our results show successful chemoselective ligations without any detectable epimerization. In addition, an epimerization-free C-terminal peptide activation[1] was applied using mechanochemistry and embedded into our ligation protocol, further overcoming concerns for epimerization and solubility.

In summary, the application of mechanochemistry for C-terminal activation as well as chemical ligation offers a unique opportunity for overcoming peptide solubility concerns, while expanding the toolbox for modern peptide chemistry.

[1] Popovic, S.; Bieräugel, H.; Detz, R. J.; Kluwer, A. M.; Koole, J. A. A.; Streefkerk, D. E.; Hiemstra, H.; van Maarseveen, J. H., *Chem. Eur. J.* **2013**, *19* (50), 16934-16937.

Sustainable Mechanochemical Synthesis of Gallate-Based BioMOFs for Aqueous Chromium Removal

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Metal–organic frameworks (MOFs) are a class of porous materials with adjustable pore sizes and symmetrical ordering, formed through the self-assembly of metal ions or clusters with organic ligands. The wide range of available metals and organic ligands enables the development of customized particles tailored to specific requirements, in which the use of biomolecules as ligands gives display a subclass namely bioMOFs. [1] Typically, solvothermal methods are used, in which parameters such as temperature, reaction time, and reagent concentration play a crucial role in governing material formation. However, the method presents certain limitations, particularly regarding synthesis duration, which can range from hours to several days depending on the MOF, potentially restricting large-scale or time-sensitive applications.[2]

In this work, it is proposed the synthesis of two bioMOFs, namely Fe-Gallate and Mg-Gallate MOF, via mechanochemistry using a horizontal ball mill reactor. Both MOFs contain gallic acid as the organic ligand, a molecule that requires a Lewis base for deprotonation before coordinating with a metal centre. Previous works have reported the synthesis of these materials through solvothermal methods at 120°C, with reaction times of 24 to 48h.[3,4] Through mechanochemistry, the amount of reactant and the synthesis duration were investigated to optimize the materials. Once stablished the parameters, it was attested that the Fe-Gallate MOF can be obtained within 15 minutes, whereas the Mg-Gallate, requires a minimum of 30 minutes. In both cases, morphology only stabilizes within 60 minutes of synthesis, evolving from polydisperse to homogeneous spherical particles. Structure characterization by X-ray diffraction revealed that both materials are isostructural but have different sizes of unit cell, which is consistent with surface area and pore volume data.

Preliminary studies demonstrate that these materials can be applied for Cr(VI) removal via chemical adsorption, in agreement with previously reported findings.[5] However, different removal efficiencies were observed for each MOF, correlating with their respective surface areas, a distinctive feature of the mechanochemical method employed.

Acknowledgments for the support of RCGI – Research Centre for Greenhouse Gas Innovation, hosted by the University of São Paulo (USP) and sponsored by FAPESP – São Paulo Research Foundation (2020/15230-5) and Shell Brasil, and the strategic importance of the support given by ANP (Brazil's National Oil, Natural Gas and Biofuels Agency) through the R&D levy regulation.

[1] Sanjida Afrin, Muhammad Waqas Khan, Enamul Haque, Baiyu Ren, and Jian Zhen Ou. Journal of Colloid and Interface Science. 2022, 623, 378–404.

[2] Sylwia Głowniak, Barbara Szczęśniak, Jerzy Choma, and Mietek Jaroniec. Materials Today. 2021, 46, 109–124.

[3] Aldo Ponce, Lynn B. Brostoff, Sarah K. Gibbons, Peter Zavalij, Carol Viragh, Joseph Hooper, Sufian Alnemrat, Karen J. Gaskell, and Bryan Eichhorn. Analytical Chemistry. 2016, 88, 5152–5158

[4] Lucy Cooper, Tania Hidalgo, Martin Gorman, Tamara Lozano-Fernández, Rosana Simón-Vázquez, Camille Olivier, Nathalie Guillou, et al. 2015. Chemical Communications. 2015, 51, 5848–5851, 2015.

[5] Hongyun Niu, Yang Zheng, Saihua Wang, Sijing He, and Yaqi Cai. Journal of Materials Chemistry. 2017, 32, 16600–16604.

P-68

Mechanochemical Approach for Perovskite-Based Catalysts: A Rapid, One-Pot Synthesis Towards Greener Materials

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Perovskites have emerged as promising multifunctional materials for broad applications, such as in photovoltaics, sensors, electrochemical devices, and photo/thermocatalysis. The conversion of undesired carbon-based molecules, where CO_2 is the most typical example, into high-value-added chemicals can be successfully obtained by employing adequate catalysts, which play a pivotal role in the chemical reaction selectivity.[1] The development of perovskite crystals composed of earth-abundant metals, such as copper and iron, leads to a more attractive, eco-friendly, and versatile material. Iron titanates are types of perovskites with enhanced structural and physicochemical characteristics, possessing improved conductivity, fast surface reaction kinetics, and high stability in a wide pH range.[2] Although promising, the application of these materials in the catalysis field is more significant on photocatalysis and dry reform of methane, with few works employing them in CO_2 hydrogenation. In addition, their obtention via mechanochemistry is scarcer in the literature when compared with other synthetic methods.

In this work, we present a one-step synthesis and characterization of copper- and iron-based titanates in a greener context by using a promising mechanochemical methodology aiming their application towards catalytic CO_2 hydrogenation.[3]

The materials were synthesized by adding hematite Fe_2O_3, anatase TiO_2, and Cu_2O into a stainless-steel jar containing 18 stainless-steel and milling at 400 rpm using a Pulverisette 7 Fritsch orbital-mill. Preliminary syntheses, carried out for 4 h under the conditions above, led to the formation of very low amounts of **$FeTiO_3$**, with the presence of starting materials, especially hematite. The thermocatalytic characterization pointed methanol and CO as main products, with low conversion (<2%) for CO_2 hydrogenation.

The improvement of the synthesis, provided by the increase of 8 h milling, led to a distribution of products composed majorly of **$FeTiO_3$** (62.3%), followed by **Cu_3TiO_4** and **$CuFe_2O_4$**, according to XRD Rietveld refinement.

Further ongoing characterization using TEM, XPS, and Raman as well as the catalytic activity evaluation may well elucidate the role and correlation between the titanates' phase distribution and the product selectivity in CO_2 hydrogenation.

[1] Thalita S. Galhardo, Adriano H. Braga, Bruno H. Arpini, János Szanyi, Renato V. Gonçalves, Bruno F. Zornio, Caetano R. Miranda, Liane M. Rossi, Journal of the American Chemical Society **2021**, *143*, 4268-4280.

[2] Mauricio A. Melo, Higor A. Centurion, Thalles T. A. Lucas, Dereck N. F. Muche, Flavio L. Souza, Renato V. Gonçalves, ACS App. Nano Materials **2020**, *3*, 9303-9317.

[3] Jean-Louis Do, Tomislav Friščić, ACS Cent. Sci. **2017**, *3*, 13-19.

Mechanochemical Aerobic Activation of Metallic Copper for the Synthesis of 1,4-Allenynes

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Mechanochemistry is emerging as a transformative and sustainable approach in the synthetic community.[1] It utilizes mechanical forces to drive chemical reactions, thus complementing thermal, electrical, and photochemical activation modes, and offers advantages such as reduced waste, shorter reaction times, and alternative selectivity.[2] In this scenario the mechanical activation of zero-valent metals is redefining sustainable synthesis, offering access to diverse chemical space under more practical conditions than conventional solution-phase methods.[3] However, unlike its neighboring elements in the periodic table, copper has remained largely unexplored in this domain, particularly for carbon - carbon bond formation.[4] Herein, we introduce a mechanochemical strategy for the copper-catalyzed homocoupling of propargylic esters, enabling the synthesis of over 25 previously unreported conjugated allenynes. This approach harnesses ball milling to activate cost-effective zero-valent copper, sourced from both commercial powders and recycled waste, under solvent-minimized and aerobic conditions. Through this process, Cu(0) is transformed into catalytically active Cu(I) species without the need for air- and moisture-sensitive Cu(I) salts.

The synthetic potential of the allenynes obtained was exemplified through two unprecedented catalytic transformations: a rhodium-catalyzed cyclocarbonylation and a gold-catalyzed cyclization reaction. By demonstrating that ball milling is essential for the in-situ generation of active Cu(I) species, we establish the first mechanochemical approach to allenynes synthesis, leveraging the reactivity of copper allenylidene under sustainable conditions. This work not only expands the mechanochemical toolbox but also underscores the versatility of copper in catalysis. [5]

[1] J. L. Howard, Q. Cao, D. L. Browne, *Chem. Sci.* **2018**, *9*, 3080.
[2] V. Martinez, T. Stolar, B. Karadeniz, I. Brekalo, K. Užarević, *Nat. Rev. Chem.* **2022**, 7, 51.
[3] A. C. Jones, J. A. Leitch, S. E. Raby-Buck, D. L. Browne, *Nat. Synth*. **2022**, 1, 763.
[4] C. G. Vogt, M. Oltermann, W. Pickhardt, S. Grätz, L. Borchardt, *Adv. Energy Sustainability Res.* **2021**, 2, 2100011.
[5] A.M. Constantin, F. Mele, M. Lanzi, G. Maestri, R. Maggi, N. Della Ca', L. Capaldo, L., *ChemSusChem,* **2025**, 18, e202500211

Mechanoenzymatic Oligomerization and Depolymerization Transformations

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Recent investigations have demonstrated the capacity of enzymes to facilitate chemical transformations under mechanochemical conditions.[1,2,3,4] In this work, we report recent findings on the ability of papain to catalyze the oligomerization of L-phenyl alanine methyl ester under ball milling conditions. The oligomeric phenylalanine material was key to the mechanosynthesis of Pro-$(Phe)_n$-CO_2Me peptides, which proved active as organocatalytic materials in asymmetric aldol reactions (Figure).[5]

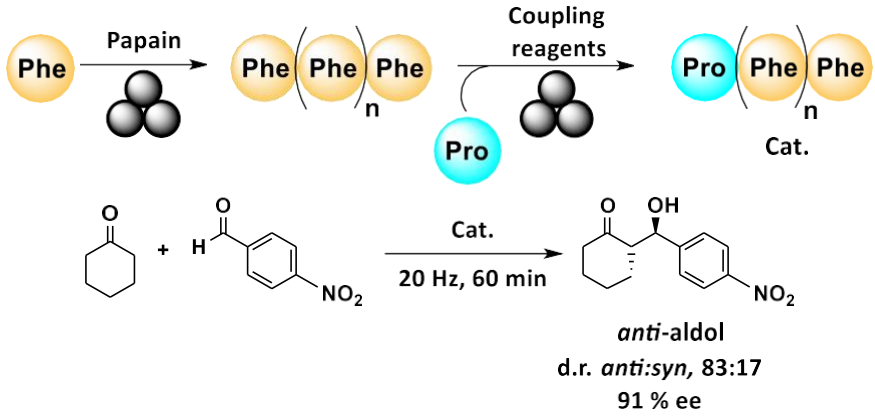

Figure 1. (Top) Mechanoenzymatic and mechanochemical synthesis of organocatalytic peptides. (Bottom) Asymmetric aldol reaction.

Moreover, in this presentation, we will introduce an ongoing project focused on the use of oxidases to facilitate the mechanoenzymatic depolymerization of biomacromolecules.

[1] Carsten Bolm, José G. Hernández, *ChemSusChem* **2018**, *11*, 1410-1420.
[2] Sandra Kaabel, Tomislav Friščić, Karine Auclair, *ChemBioChem* **2020**, *21*, 742-758.
[3] Mario Pérez-Venegas, Eusebio Juaristi, *ChemSusChem* **2021**, *14*, 2682-2688.
[4] Rebecca Hollenbach, -Ing. Katrin Ochsenreither, *ChemCatChem* **2023**, *15*, e202300656.
[5] Alexandra Rios-Echeverri, Carlos E. Puerto Galvis, Karen J. Ardila-Fierro, José G. Hernández, *ChemSusChem*

Ball Mill Reaction of ZnO and Imidazole: Mechanistic Aspects Inferred from Its Kinetics

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Our research group uses *in situ* Raman spectroscopy, X-ray powder diffraction, and occasionally thermography to investigate the kinetics of mechanochemical reactions in ball mills, toward determining their activation barriers, the elucidation of their chemical mechanisms, and the validation of the "stress augmented thermal activation" model. The ball mill synthesis of a zinc imidazolate, ZIF-6 (Cambridge Structural Database refcode EQOCOC) is of interest, since a second-order overall kinetic model based on the kinetic profiles of imidazole at four milling frequencies (10, 15, 20 and 25 Hz), has been published.[11] Our approach used *in situ* Raman spectroscopy (detecting imidazole and the products, without polymorphic distinction), laboratory X-ray powder diffraction (leading to the crystalline phases, their unit cell parameters and total amorphous contents), scanning electron microscopy (verifying the formation of nanomaterials and their morphologies), energy dispersive spectroscopy (for elemental composition), and microcrystal electron diffraction (leading to the unit cell parameters of a new polymorph, and those identifying a known phase). Moreover, the effects of the milling frequency and the investigation of the effects of liquid additives of increasing polarity and varied imidazole solubility, the kinetic isotope effect (replacing imidazole by its deuterated analog), and the effects of deuterated ethanol as a liquid additive were studied. The additives additionally included acidic and basic aqueous solutions of HCl and NaOH, respectively. While the rheology of the mixture ball milled did not always allow the measurement of reliable kinetic data, some additives and experimental conditions led to homogeneous-type kinetic models, as published[1]. However, most additives led to kinetic profiles controlled by the nucleation of the products. The kinetic data was calculated with MATLAB scripts modified by the authors from those purchased from *InSolido Tech.* This presentation will discuss the observations and mechanistic inferences made toward the elucidation of the possible chemical and mass transfer mechanisms, different under the several experimental conditions used. Based on these measurements, a currently proposed chemical mechanism has been outlined.

[1] *Xiaohe Ma, Wenbing Yuan, Steven E. J. Bell and Stuart L. James. Chem. Commun*., **2014**, *50*, 1585-1587.

From Prediction to Milling: Periodic DFT-Guided Mechanochemistry of Halogen-Bonded Materials

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Mechanochemistry has emerged as a green, efficient, and scalable synthetic route, recognized by IUPAC in 2021 as one of the "Top Ten emerging technologies in chemistry."[1] In cocrystallization, conventional solution-based methods are often limited by solubility constraints and the formation of solvated crystal structures. Ball milling, employing either dry grinding or liquid-assisted grinding (LAG), overcomes these challenges by enabling stoichiometric cocrystal formation under solvent-minimized conditions.

In this work, we focus on halogen-bonded materials, particularly those involving iodine (I), which pose computational challenges due to strong dispersion, polarization, electrostatics, and charge-transfer effects, as well as the diffuse nature of the halogen atom.[2] To better model the materials under mechanochemistry, especially using periodic density functional theory (DFT) implemented in the CASTEP,[3] we aim to identify computational methods that can reliably predict solid-state reactivity under milling conditions.

As a first step, we have synthesized the (**tftib**)(**pyr**)$_{1/2}$ [tftib = 1,3,5-trifluoro-2,4,6-triiodobenzene; pyr = pyrazine] and performed a Cambridge Structural Database (CSD) search to identify potential donor/acceptor partners. We calculated interconversion energies using periodic DFT and compared predictions to experimental outcomes from ball milling.[4] We further explore the formation of ternary halogen-bonded cocrystals by overcoming the competition between I···S (Sulfur) and I···N (Nitrogen) via mechanochemistry, supported by in situ PXRD to correlate conversion times with interaction strengths.[5]

This study demonstrates the feasibility of integrating periodic DFT with mechanochemistry to rationally predict interconversion pathways, optimize reaction conditions, and efficiently synthesize stoichiometric and ternary cocrystals under green chemistry principles.

Fig: schematic representation of the work.

[1] *Gomollón-Bel, F. Chemistry International* ***2021****, 43 (4), 13–20.*
[2] *Zhu, Z.; Xu, Z.; Zhu, W. J Chem Inf Model* ***2020****, 60 (6), 2683–2696.*
[3] *Clark, S. J.; Segall, M. D.; Pickard, C. J.; Hasnip, P. J.; Probert, M. I. J.; Refson, K.; Payne, M. C. Kristallogr Cryst Mater* ***2005****, 220 (5–6), 567–570.*
[4] *Kumar, L.; Leko, K.; Nemec, V.; Trzybiński, D.; Bregović, N.; Cinčić, D.; Arhangelskis, M. Chem Sci* ***2023****, 14 (12), 3140–3146.*
[5] *Kumar, L.; Dash, S. G.; Leko, K.; Trzybiński, D.; Bregovic, N.; Cinčić,*

Mechanochemical Synthesis of Metal intercalated Boron Nanostructures

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Borophene, a two-dimensional allotrope of boron, possesses exceptional mechanical strength, electrical conductivity, and electrochemical activity, making it a compelling candidate for next-generation energy storage and advanced fusion applications. However, scalable synthesis remains a major bottleneck[1]. In this study, we demonstrate a simple and scalable ball milling approach to produce borophene nanosheets and copper-intercalated borophene composites in a single step. The high-energy milling facilitates both the exfoliation of bulk boron into thin sheets and the in situ incorporation of copper, resulting in significant structural and electronic modifications. Comprehensive characterization—including X-ray diffraction (XRD), high-resolution transmission electron microscopy (HR-TEM), X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and Fourier-transform infrared (FTIR) spectroscopy—confirms successful exfoliation and copper intercalation. HR-TEM imaging reveals the formation of a novel structure with β^{12} and χ^{3} phases in a 2D planar structure, where β^{12} ring offers potential sites for hydrogen confinement. These copper- intercalated borophene materials show promise as target materials in proton–boron (p–^{11}B) fusion reactions, potentially supporting avalanche fusion mechanisms through their unique structural and electronic features[2].

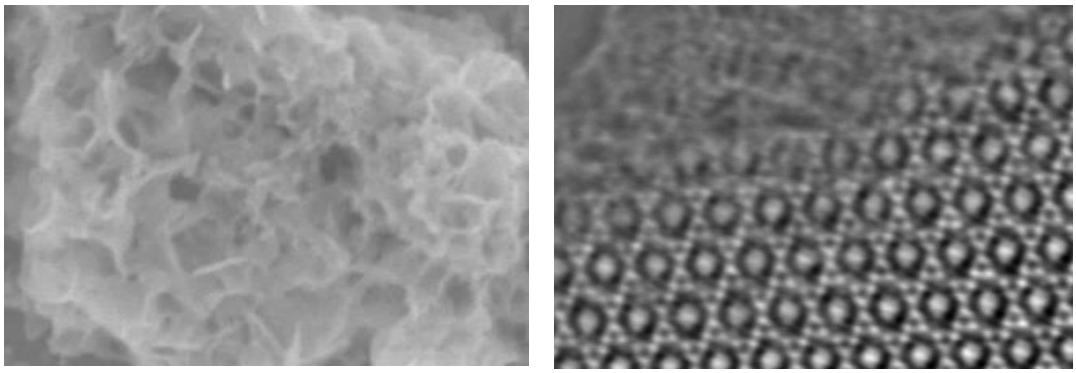

Figure 1. As-synthesized Cu intercalated Borophene sheets a) imaged using SEM and b) atomic resolution HRTEM image displaying the β^{12} nanoring and χ^{3} traingles.

[1] Kumar, P.; Singh, G.; Bahadur, R.; Li, Z.; Zhang, X.; Sathish, C.; Benzigar, M. R.; Tran, T. K. A.; Padmanabhan, N. T.; Radhakrishnan, S., Progress in Materials Science 2024, 101331.
[2] Hora, H.; Korn, G.; Giuffrida, L.; Margarone, D.; Picciotto, A.; Krasa, J.; Jungwirth, K.; Ullschmied, J.; Lalousis, P.; Eliezer, S., Laser and particle Beams 2015, 33 (4), 607-619.

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Borchardt, L.	Ruhr-Universität Bochum	Germany
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Braunschweig, A.	City University of New York	US
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Fisher, L.	Royal Society of Chemistry	UK
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Friscic, T.	University of Birmingham	UK
Gallego, A.	University of Montpellier	France
Gancheva, M.	Bulgarian Academy of Sciences	Bulgaria
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Garcia, E.	Aalto University	Finland
Garrido Nuñez, S.	TU Delft	Netherlands
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Gomes, C.	Universidade de Lisboa	Portugal
Grätz, S.	Ruhr-Universität Bochum	Germany
Gugin, N.	Federal Institute for Materials Research and Testing	Germany
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Karimi, A.	Semnan University	Iran
Kessler, R.	Pentapharm AG	Switzerland
Kickelbick, G.	Saarland University	Germany
Kim, S.	Ulsan National Institute of Science and Technology	Korea
Korfmacher, M.	RWTH Aachen University	Germany
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Kumar, L.	University of Warsaw	Poland
Lamaty, F.	University of Montpellier	France
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Lee, J.	Ulsan National Institute of Science and Technology	Korea
Leyssens, T.	UCLouvain	Belgium
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Luo, H.	University of Surrey	UK
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Morais Mendes, M.	University of São Paulo	Brazil
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