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Presentation KU Leuven SOLVOMET Group:

Laboratory of Metallurgical Chemistry & Industrial Service Centre P.T. Jones & K. Binnemans

September 27, 2022

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Laboratory of Metallurgical Chemistry [LAB] / Industrial Service Centre [ISC]





SOLVOMET's vision is that metallurgical chemistry expertise allows to develop more efficient, eco-friendly hydrometallurgical and solvometallurgical processes to provide the critical metals that are needed for the transition to a climate-neutral society.





SOLVOMET's mission is (1) to perform excellent research in metallurgical chemistry and to educate and train young researchers in this domain [LAB] and (2) to support its Industrial Service Centre partners in the conceptual and practical development of more sustainable (circular, lowenergy input) hydrometallurgical (and solvometallurgical) processes, which are subsequently tested using state-of-the-art lab-scale and mini-pilot-scale experimental facilities [ISC].



Background info on Prof. Koen Binnemans & SOLVOMET Group





- Full professor at the Department of Chemistry, specialised in circular hydrometallurgy and solvometallurgy
- Core expertise in critical metals and solvent extraction (SX)
- Author of more than 560 papers, H-index = 81, > 29,000 citations
- Former ERC Advanced Grant holder (SOLCRIMET: Solvometallurgy for critical metals)
- Former ERC Proof of Concept holder (SOLVOLi: Solvometallurgy for battery-grade refining of lithium)
- Co-founder **SOLVOMET Industrial Service Centre** for Hydro/solvometallurgy
- Steercom Member KU Leuven Institute for Sustainable Metals and Minerals (SIM² KU Leuven)
- Former Steercom Member European Rare Earth Competency Network (ERECON)
- Elected member Royal Flemish Academy of Belgium for Science and the Arts (KVAB)
- For research domains "hydrometallurgy" & "solvent extraction", according to Google Scholar (data retrieved 2022-09-07), the "**world's most cited author**".









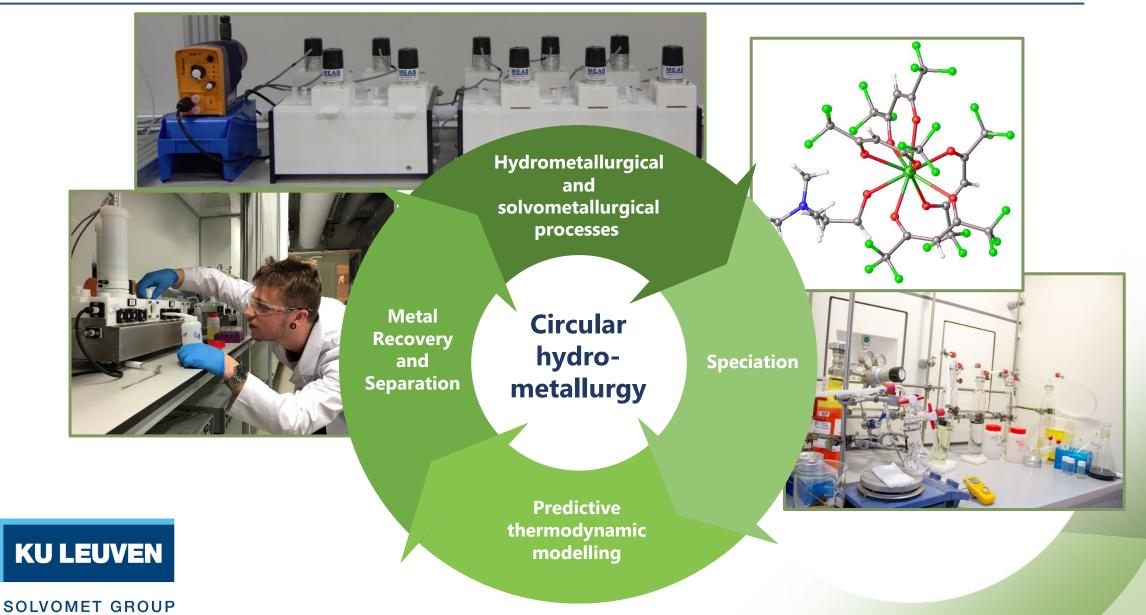
Genuine breakthroughs in hydrometallurgy will not come from the use of neoteric solvents like ionic liquids or deep-eutectic solvents, but rather from a deep understanding of hydro-processes at a molecular level. Hydrometallurgy needs to evolve to low-energy-input circular hydrometallurgy.

(Prof. Koen Binnemans, August 2021)



SOLVOMET Group Research domains





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SOLVOMET Group: LAB Fundamental research/curiosity & hypothesis driven

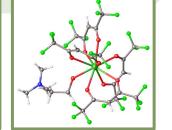
Research topics

- ✓ Speciation studies in concentrated, multicomponent electrolytes
- Development of methods for quantitative analysis of metals in complex matrices
- ✓ Advanced separation processes for hydrometallurgy
- ✓ Thermochemistry of hydrometallurgical reactions
- Thermodynamic and kinetic modelling of multiphase, multicomponent metallurgical systems
- Synthesis of new extractants and new synthesis methods for extractants
- ✓ Electrocoordination chemistry











- ✓ Fundamental research
- Curiosity-driven and hypothesis-driven research
- Low TRL
- ✓ Bottom-up
- ✓ Development of new methods and tools
- ✓ Academia-oriented
- Answering research questions
- Insight and understanding at a molecular level





SOLVOMET Group: Industrial Service Centre (ISC)

Industry-driven & result-oriented research



Research topics (interlinked)

- ✓ Development of (near-circular, low-energy-input)
 hydrometallurgical & solvometallurgical flowsheets
- ✓ Validation of hydrometallurgical processes on mini-pilot scale
- Thermodynamic modelling of hydrometallurgical processes
- \checkmark Advanced leaching processes
- Hydrometallurgical applications of solvent extraction and ion exchange
- ✓ Chemical & mineralogical characterisation ores, concentrates & industrial process residues
- ✓ Forensic hydrometallurgy

Research philosophy

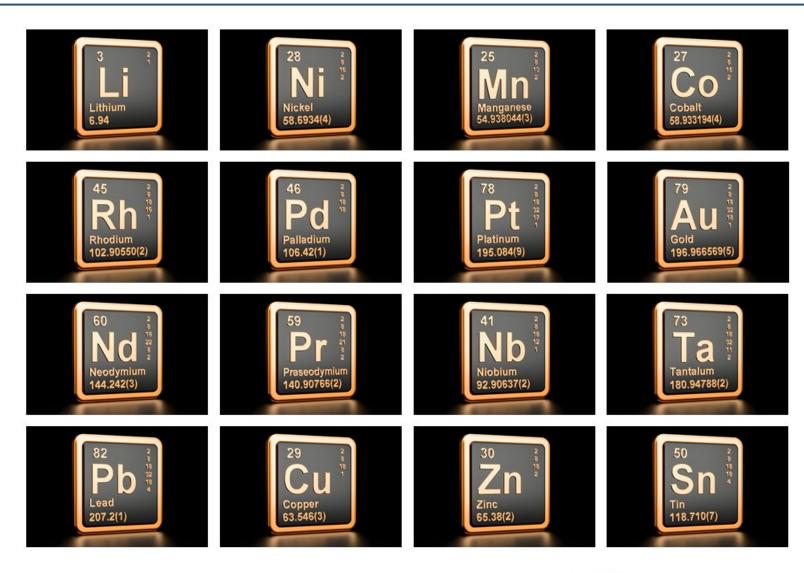
- ✓ Applied research
- ✓ Medium to high TRL
- ✓ Top-down (EU calls and bilateral projects with industry)
- ✓ Development of new processes
- ✓ Industry-driven
- ✓ Solving industrial problems
- Insight and understanding at a molecular level



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SOLVOMET ISC's key metals of interest





SOLVOMET Industrial Service Centre: Mini-pilot plant facilities for leaching

High pressure reactor

- Effective capacity of 800 mL
- Made from stainless steel with PTFE liner
- Max. pressure = 200 bar
- Max. T = 230 °C with PTFE liner (and 300 °C without)





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Multiple reactor system

- 6 reactors (V_{max} = 40 mL) with internal stirring
- Individual T & p control
- Max. p = 200 bar
- Max. T = 300 °C, heating rates up to 15 °C/min





Batch leaching reactors

- Two jacketed reactors (1 & 5 L)
- pH and T control
- Digital overhead stirrer
- Filtration system included



SOLVOMET Industrial Service Centre: Mini-pilot facilities for

continuous, countercurrent Solvent Extraction (SX) - Mixer-settlers





3 SX mixer-settler set-ups





SOLVOMET Industrial Service Centre: Mini-pilot facilities for

continuous, countercurrent Solvent Extraction (SX) - Mixer-settlers

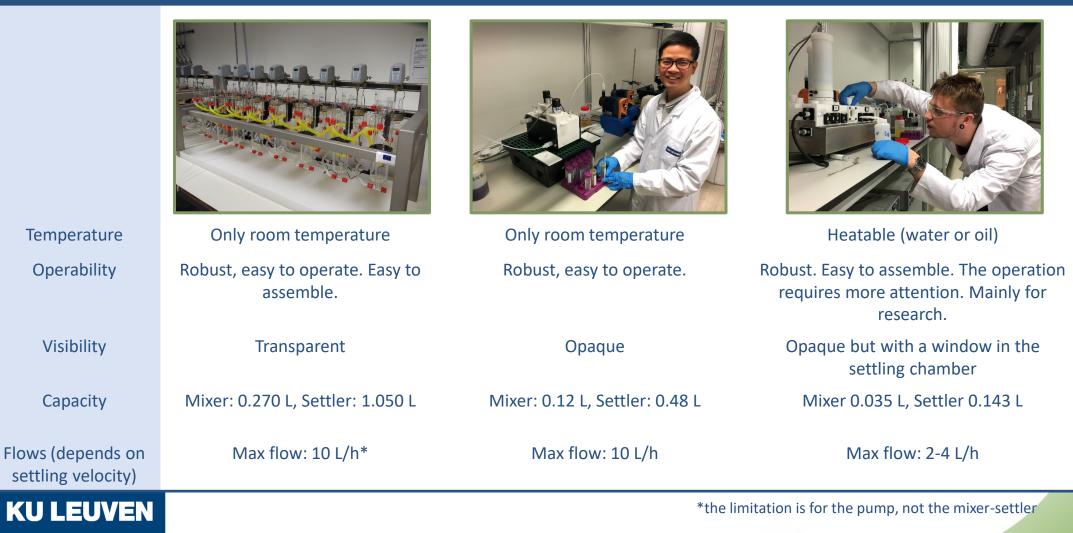


Characteristics

SX Kinetics

MEAB

Rousselet Robatel

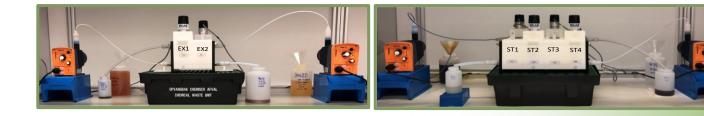


continuous, countercurrent Solvent Extraction (SX) - Mixer-settlers **SOLVOMET Industrial Service Centre: Mini-pilot facilities for**



	Process	Equipment	Collaboration
PLATIRUS project	Pt, Pd and Rh recovery from spent autocatalysts	MEAB MS	JM Johnson Matthey Inspiring science, enhancing life
Bilateral project with industry	Cu recovery from high-grade Chrysocolla	1 L Hitec Zang leaching reactor and Rousselet MS	Shell
Fundamental research	Li and Mg separation using binary extractants	Rousselet MS	
	Nd and Dy separation using ionic liquids	MEAB MS	
	Fe, Pb and Zn separation from DES	Rousselet MS	
	Y and Eu separation using non- aqueous solvent extraction	Rousselet MS	





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SOLVOMET Industrial Service Centre: Mini-pilot facilities for continuous, countercurrent SX – (Agitated column SX)



Kühni-type agitated column

For processes with low mass transfer, average residence time and high number of stages.

- Jacketed column made from glass with internals made from PEEK
- Max active volume: 0.9 L
- Active height: 1.2 m
- Total Flow: 5-25 L/h (both phases)



SOLVOMET Industrial Service Centre: Lab facilities for

Ion exchange work (lab-scale column IX set-up)



Econo-chromatography columns

- Low-pressure (<1 bar) or gravity flow separations
- Used in various dimensions (*e.g.* 0.7 x 30 cm)





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CF-2 Fraction collector

- Equipped with drop sensor
- Capacity of 174 tubes
- Coupled with a peristaltic



Ismatec IPC Peristaltic pump

- High-precision 8-channel dispenser
- Flow rates 0.002 44 ml/min



SOLVOMET Industrial Service Centre: Key analytical facilities & services







SOLVOMET Industrial Service Centre: Key analytical facilities & services













Publications deriving from bilateral projects



Check for

with industry – <mark>on (solvo)leaching</mark>

Journal of Sustainable Metallurgy https://doi.org/10.1007/s40831-020-00294-3

Ammoniacal Solvoleaching of Copper from High-Grade Chrysocolla

Lukas Gijsemans¹ · Joris Roosen¹ · Sofía Riaño¹ · Peter Tom Jones² · Koen Binnemans¹

Received: 22 June 2020 / Accepted: 14 September 2020 © The Author(s) 2020

Abstract

The copper silicate ore chrysocolla forms a large potential copper resource, which has not yet been fully exploited, due to difficulties associated with its beneficiation by flotation and metallurgical processing. Direct acid leaching of chrysocolla causes silica gel formation. Therefore, in this work, the feasibility of solvometallurgical methods to leach copper from high-grade chrysocolla while avoiding issues with silica gel formation was assessed. Ammoniacal solvoleaching was performed with a solvent comprising the chelating extractant LIX 984 N or the acidic extractant Versatic acid 10 in an aliphatic diluent (ShellSol D70 or GTL Fluid G70), combined with a small volume of aqueous ammonia. In the three-phase system, aqueous ammonia dissolves copper from milled and sieved chrysocolla, while copper is simultaneously extracted to the organic phase, releasing ammonia that can be reused for further extraction. The best results were obtained with LIX 984 N as extractant: using a 50 vol% LIX 984 N solution, about 75% of copper could be extracted after 60 min of leaching at 25 °C. The stripping of copper from the pregnant leach solution was optimized. Quantitative stripping of copper was achieved with 1.89 M sulfuric acid and the final aqueous solution of copper sulfate had a concentration of 33 g L⁻¹. Experiments in a leaching reactor (1 L) and small battery of mixer-settlers (3 stages, 35 and 143 mL effective volume in the mixer and the settler, respectively, per stage) were successfully conducted and allowed to recover copper with a purity of 99.9%. A conceptual flow sheet has been developed.

Selective Removal of Zinc from BOF Sludge by Leaching with Mixtures of Ammonia and Ammonium Carbonate

Nerea Rodriguez Rodriguez¹¹ · Lukas Gijsemans¹ · Jakob Bussé¹ · Joris Roosen¹ · Mehmet Ali Recai Önal¹ · Victoria Masaguer Torres² · Álvaro Manjón Fernández² · Peter Tom Jones³ · Koen Binnemans¹

Received: 18 July 2020 / Accepted: 13 October 2020 © The Author(s) 2020

Journal of Sustainable Metallurgy

RESEARCH ARTICLE

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https://doi.org/10.1007/s40831-020-00305-3

Abstract

The zinc content of basic oxygen furnace (BOF) sludges is too high for direct recycling into the blast furnace via the sinter plant, as excessive zinc concentrations are detrimental for the refractory lining of the blast furnace. However, by partial and selective removal of zinc from the BOF sludge, the residual sludge can be used as a secondary iron resource in the blast furnace. In this paper, BOF sludge was leached with aqueous ammonia, aqueous solutions of ammonium salts (chloride, carbonate, and sulfate), and aqueous mixtures of ammonia and ammonium salt. The mixtures of ammonia and ammonium salt could leach more zinc with respect to either the aqueous ammonia or the aqueous ammonium salt solution. The ammonia–ammonium carbonate (AAC) mixture was selected as the most suitable lixiviant due to the high zinc leaching efficiency in combination with a high selectivity towards iron; furthermore, this combination does not introduce unwanted chloride or sulfate impurities in the residue. The leaching process was optimized in terms of the liquid-to-solid ratio, total ammonia concentration, ammonium: antonia molar ratio, temperature, and leaching time. The co-dissolved iron was precipitated as a hydroxide after oxidation of ferrous to ferric ions by an air stream, without co-precipitation of zinc, while the dissolved zinc could be easily recovered as zinc sulfide by precipitation with ammonium sulfide. The (almost) closed-loop process was seven the antion of zinc with a the dissolved zinc could be easily necovered as zinc sulfide by recipitation with ammonium sulfide. The (almost) closed-loop process

Graphical Abstract



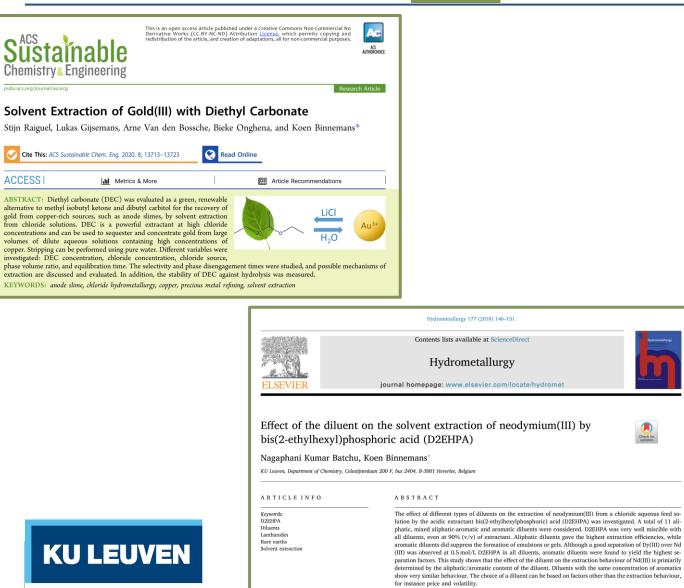


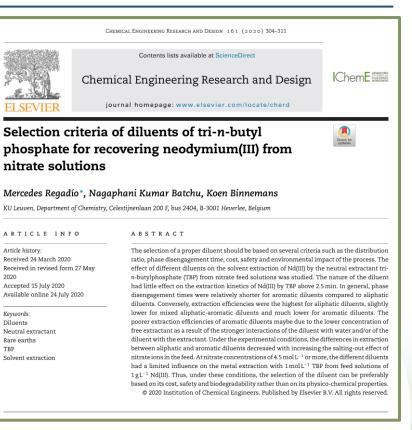
Publications deriving from bilateral projects with industry - on SX

ACCESS

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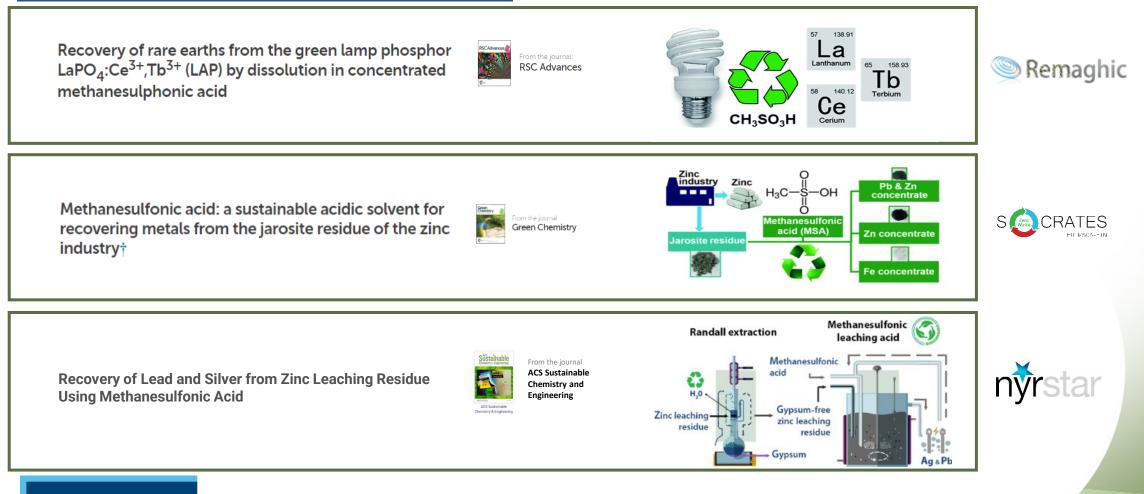
2020

TRP

Some key achievements by SOLVOMET



Methanesulfonic acid: a sustainable acidic solvent





Broad industrial network



Bilateral projects

Previous collaborations / Active collaborations within H2020/HE projects



SOLVOMET's academic network

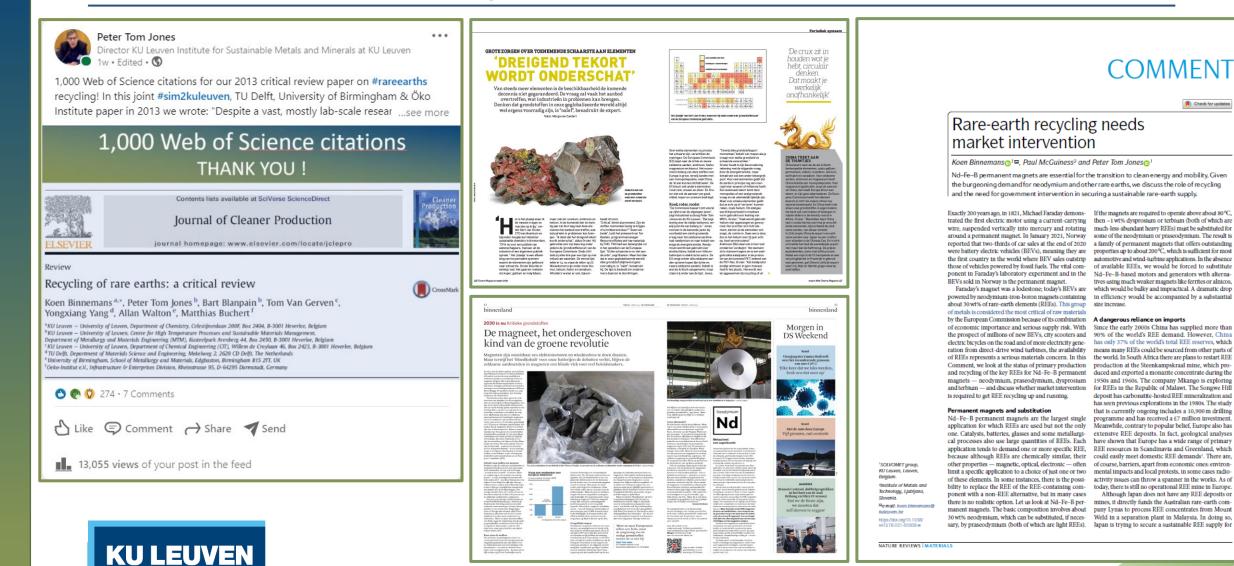






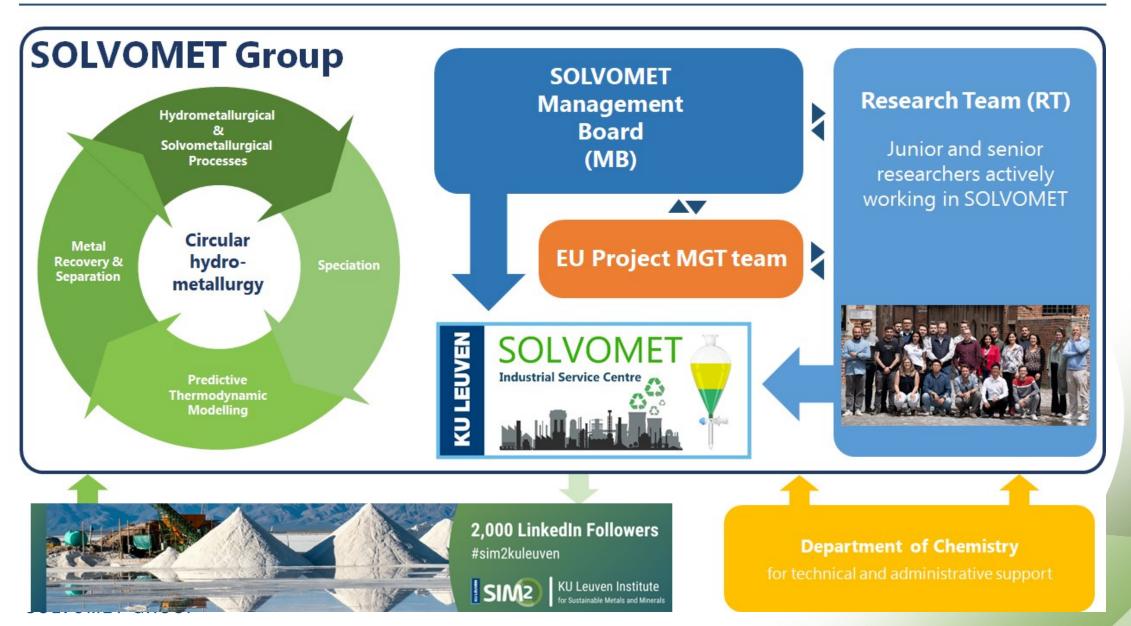
Scientific & societal impact





SOLVOMET Group Governance







Services Agreement (short-term)

- **Project description**: 4-page document describing tasks, deliverables, milestones, budget, timing etc.
- IP: foreground IP transferred to company
- **Duration**: typically 6 to 12 months
- **Cost**: salaries of the involved researchers + 50% extra for working budget (chemicals, lab use, travel...)).
- **Publications:** not planned unless explicitly desired by company in terms of dissemination goals
- Ideal for fast delivery of (confidential) results by experienced (permanent staff) research experts/managers, experienced postdocs, research associates & lab technicians

Long-term framework agreement

- **Project description**: 4-page document research programme, budget
- IP: foreground IP transferred to company
- Duration: 2 to 5 years
- **Cost**: salaries of the involved researchers + 50% extra for working budget (chemicals, lab use, travel...)).
- **Publications:** not planned unless explicitly desired by company in terms of dissemination goals
- Ideal for long-term, in-depth, research support provided by experienced (permanent staff) research experts/managers, experienced postdocs, research associates & lab technicians

Industrial PhD project

- **Project description**: 4-page document with research programme, budget
- IP: foreground IP transferred to company
- Duration: 4 years
- **Cost**: ~95,000 euro/year [salary + 50% (overhead + working budget for chemicals, lab use, travel...)]
- **Publication clause:** Allowing to publish more generic parts of research while keeping the rest confidential
- Equitable remuneration principle: e.g. preferred partnership for follow-up projects
- Ideal for first-class training of PhD researcher that can go and work for the company later



Our permanent staff Research Managers (SOLVOMET ISC projects)



Dr. Ir. Peter Tom Jones	Dr. Clio Deferm	Dr. Lieven Machiels	Dr. Thomas Abo Atia
Industrial Research Fund (IOF) Valorisation Manager	OZK-Research Manager	OZK-Research Manager	Senior Research Manager (pending completionf of procedure)
Sustainable Metallurgy	Metallurgical Chemistry	Battery raw materials/recycling & valorisation of residues	Circular Hydrometallurgy
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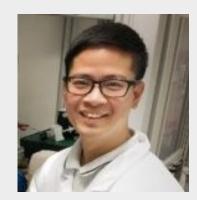


Our permanent staff Research Experts (SOLVOMET ISC projects)



Dr. Ir. Sofía Riaño	Dr. Viet Tu Nguyen	Dr. Rayco Lommelen	
OZK-Research Expert	OZK-Research Expert	Senior Research Expert (pending completion of procedure)	
Upscaling of Solvent Extraction	Aqueous and non- aqueous solvent extraction process development	Hydrometallurgical thermodynamic modelling	
LinkedIn Profile	LinkedIn Profile	LinkedIn Profile	









Our permanent staff Lab Technicians and Research Associates (SOLVOMET ISC projects)



Jakob Bussé	Alexandra Alexandri
Research Associate (ATP) (pending completion of procedure)	Lab Technician (ATP)
Metallurgical Chemist	ICP-OES/MS Lab Technician







Our permanent staff Project Managers & Software developers (SOLVOMET ISC EU projects)



Giorgian Dinu	Lucian Onisei	Rabab Nasser	Elisabeth De Decker
ATP	ATP	ATP	ATP
Software developer & project manager	Software developer & project manager	Project manager & event organiser	Financial-administrative manager SOLVOMET Group
[LinkedIn profile]	[LinkedIn profile]	[LinkedIn profile]	[LinkedIn profile]











Meet our other SOLVOMET colleagues



Postdoctoral fellows: Dr. Jong-Won Choi Dr. Brecht Dewulf Dr. Pieter Geysens Dr. Sharron (Xiaohua) Li Dr. Stijn Raiguel Dr. Panagiotis Xanthopoulos

Researchers:

Carlos Arias Quintero

PhD students: Robin Aerts Meryem Ozge Arman Vincent Cool Nor Kamariah **Filip Kolesar Dominic Maertens Nand Peeters** Willem Vereycken Hongshan Zhu



SOLVOMET is embedded in the KU Leuven Institute for Sustainable Metals and Minerals (SIM² KU Leuven)









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INDIAN



https://chem.kuleuven.be/solvomet https://solvomet.eu https://kuleuven.sim2.be/

Contact: Peter Tom Jones (LinkedIn profile)