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Industrial Service Centre



Presentation KU Leuven SOLVOMET Group:

Laboratory of Metallurgical Chemistry & Industrial Service Centre

P.T. Jones & K. Binnemans

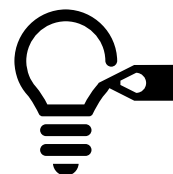
June 10, 2022

SOLVOMET Group

Laboratory of Metallurgical Chemistry



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 and (2) to support its Industrial Service Centre partners in the conceptual and practical development of more sustainable (circular, low-energy input) hydrometallurgical (and solvometallurgical) processes, which are subsequently tested using state-of-the-art lab-scale and mini-pilot-scale experimental facilities.

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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)
- **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 2021-07-06), the “**world’s most cited author**”.





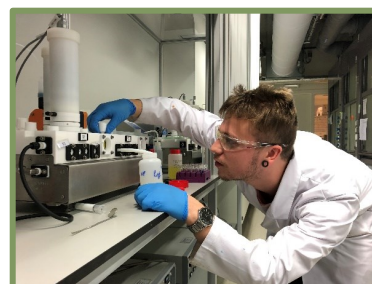
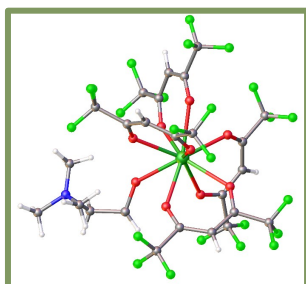
Genuine breakthroughs in hydrometallurgy will not come from the use of neoteric solvents like ionic liquids or deep-eutectic solvents, but rather from a **deep understanding of hydro-processes at a molecular level**. Hydrometallurgy needs to evolve to low-energy-input **circular hydrometallurgy**.

(Prof. Koen Binnemans, August 2021)

SOLVOMET Group Research Topics

Curiosity-driven fundamental and strategic basic research

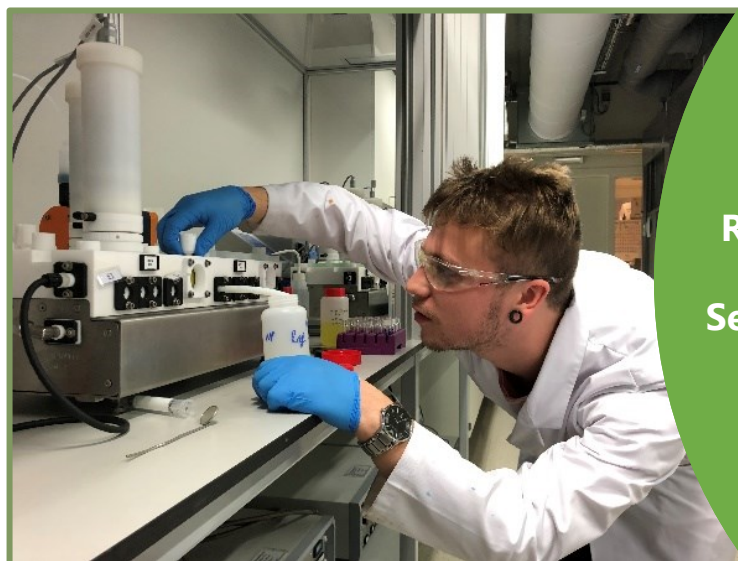
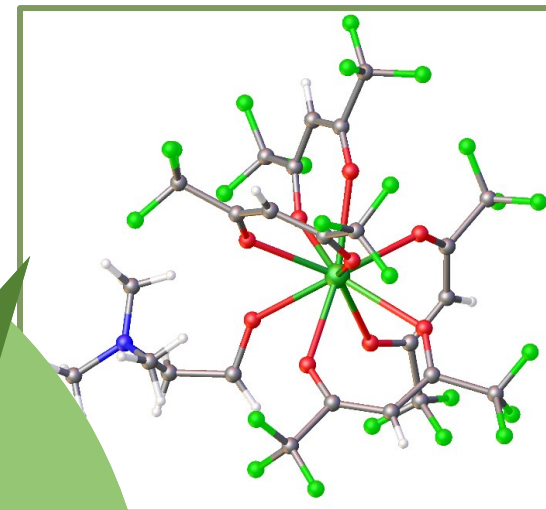
- Solvent-assisted metal recovery (SX, direct reduction or hydrolysis in organic phase after SX, antisolvent precipitation, salt crystallisation, SX-assisted metal carbonation)
- Circular hydrometallurgy
- Molecular thermodynamics (incl. thermodynamic modelling)



Problem-driven applied and competitive research

- Development of (near-circular, low-energy-input) hydro/solvometallurgical processes (incl. leaching, SX)
- Modelling of hydro/solvometallurgical processes
- Solvent formulation for SX processes
- Validation of leaching and separation processes on mini-pilot scale

SOLVOMET Group Research domains



Metal
Recovery
and
Separation

Hydrometallurgical
and
solvometallurgical
processes

**Circular
hydro-
metallurgy**

Speciation

Predictive
thermodynamic
modelling



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SOLVOMET Industrial Service Centre:

Flagship services offered (general overview)



1. Recovery of metals from ores, solid industrial process residues and urban waste

- ✓ Leaching at lab scale in batch reactors (up to 5 L), mortar grinder or in columns
- ✓ High-pressure leaching in autoclave reactor

2. Removal of metals from liquid (aqueous or organic) process streams

- ✓ After leaching, metal ions are recovered from pregnant leach solutions by (non-)aqueous SX (see next section), (non-) aqueous ion exchange (IX), cementation or precipitation

3. Solvent extraction processes (SX)

- ✓ SX processes studied from chemical point of view: mechanistic studies and kinetic studies + long-term stability studies of extractants and diluents
- ✓ SX tests in lab-scale mixer-settler batteries, batch extractors, pulsed columns or in series of separatory funnels

4. Analytical services

- ✓ QXRD, WDXRF, TXRF, Raman, ICP-OES, ICP-MS, NMR, UV-VIS, SEM-EDS, (EPMA)...

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)



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

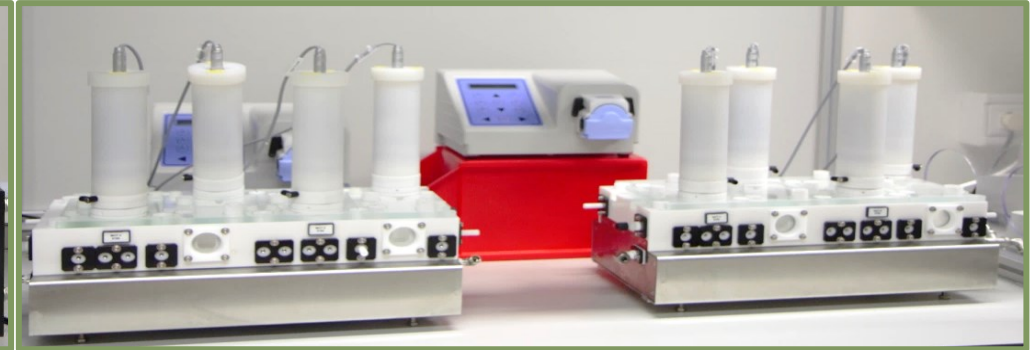


Batch leaching reactors

- 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



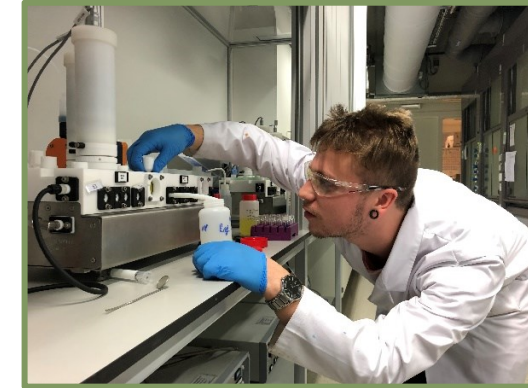
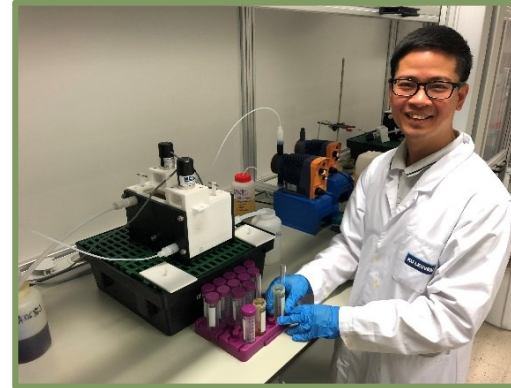
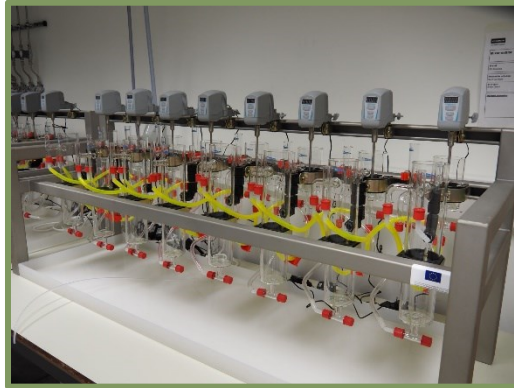
3 SX mixer-settler set-ups



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



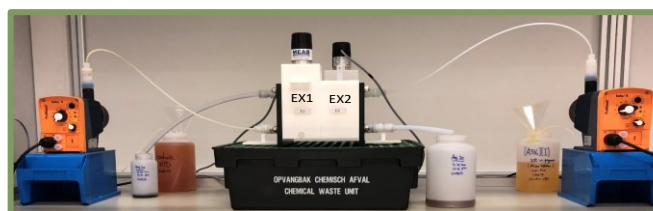
Characteristics	SX Kinetics	MEAB	Rousselet Robatel
Temperature	Only room temperature	Only room temperature	Heatable (water or oil)
Operability	Robust, easy to operate. Easy to assemble.	Robust, easy to operate.	Robust. Easy to assemble. The operation requires more attention. Mainly for research.
Visibility	Transparent	Opaque	Opaque but with a window in the settling chamber
Capacity	Mixer: 0.270 L, Settler: 1.050 L	Mixer: 0.12 L, Settler: 0.48 L	Mixer 0.035 L, Settler 0.143 L
Flows (depends on settling velocity)	Max flow: 10 L/h*	Max flow: 10 L/h	Max flow: 2-4 L/h



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



	Process	Equipment	Collaboration
PLATIRUS project	Pt, Pd and Rh recovery from spent autocatalysts	MEAB MS	JM Johnson Matthey Inspiring science, enhancing life vito
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	

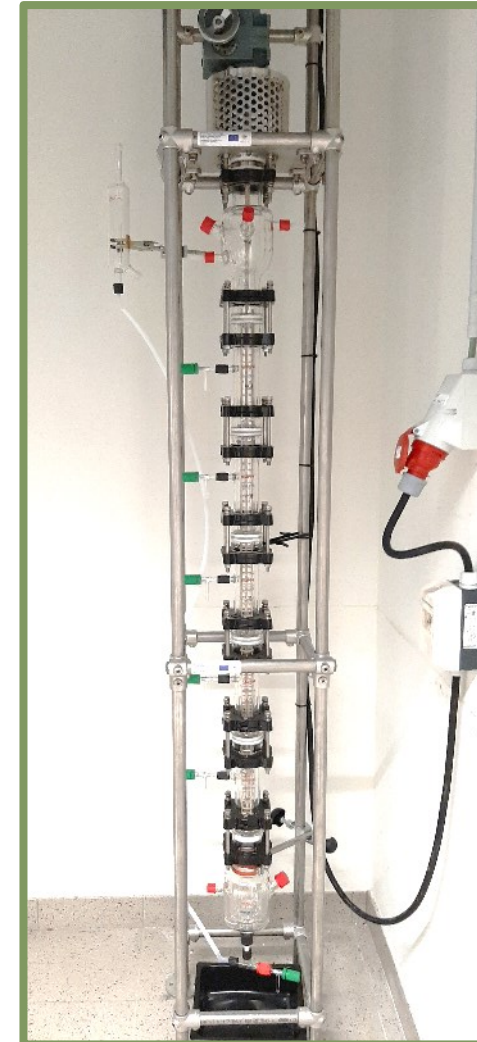


SOLVOMET Industrial Service Centre: Mini-pilot facilities for continuous, countercurrent SX – (Agitated column SX)

Kühni-type agitated column

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

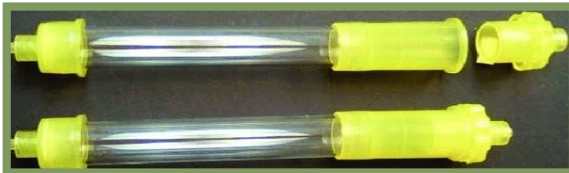
- Jacketed column made from glass with internals made from PEEK
- Max active volume: 0.9 L
- Active height: 1.2 m
- Total Flow: 5-25 L/h (both phases)



SOLVOMET Industrial Service Centre: Lab facilities for Ion exchange work (lab-scale column IX set-up)

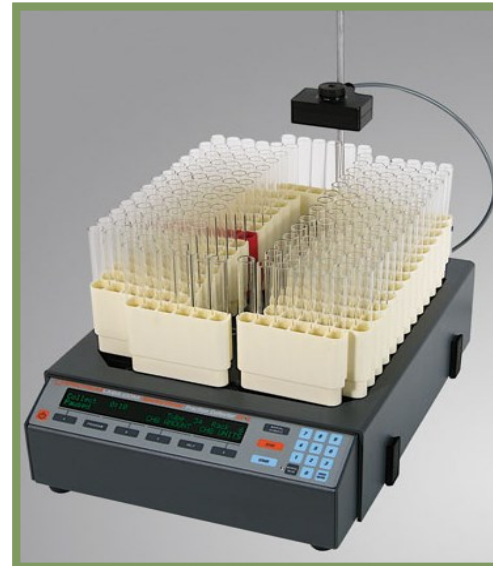
Econo-chromatography columns

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



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

TXRF



WDXRF



XRD



ICP-OES



ICP-MS (Multi Quadrupole)

SOLVOMET Industrial Service Centre: Key analytical facilities & services

UV-VIS-NIR



FT-IR/Raman



NMR

Raman microscopy



Publications deriving from bilateral projects with industry – on (solvo)leaching

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

RESEARCH ARTICLE



Ammoniacal Solvleaching of Copper from High-Grade Chrysocolla

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

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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 solvleaching 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.

Journal of Sustainable Metallurgy
<https://doi.org/10.1007/s40831-020-00305-3>

RESEARCH ARTICLE



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

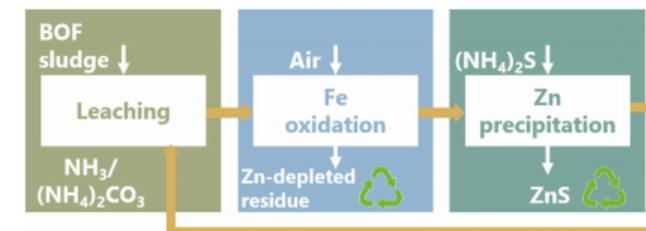
Nerea Rodriguez Rodriguez¹ · Lukas Gijsemans¹ · Jakob Bussé¹ · Joris Roosen¹ · Mehmet Ali Recai Önal¹ · Victoria Masaguer Torres² · Álvaro Manjón Fernández² · Peter Tom Jones² · Koen Binnemans¹

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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:ammonia 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 successfully up-scaled from 10 mL to 1 L scale.

Graphical Abstract



Publications deriving from bilateral projects with industry – on SX

ACS Sustainable Chemistry & Engineering

pubs.acs.org/journal/ascecg

Research Article

Solvent Extraction of Gold(III) with Diethyl Carbonate

Stijn Raiguel, Lukas Gijsemans, Arne Van den Bossche, Bieke Onghena, and Koen Binnemans*

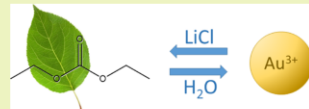
Cite This: *ACS Sustainable Chem. Eng.* 2020, 8, 13713–13723

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ABSTRACT: Diethyl carbonate (DEC) was evaluated as a green, renewable alternative to methyl isobutyl ketone and dibutyl carbitol for the recovery of gold from copper-rich sources, such as anode slimes, by solvent extraction from chloride solutions. DEC is a powerful extractant at high chloride concentrations and can be used to sequester and concentrate gold from large volumes of dilute aqueous solutions containing high concentrations of copper. Stripping can be performed using pure water. Different variables were investigated: DEC concentration, chloride concentration, chloride source, phase volume ratio, and equilibration time. The selectivity and phase disengagement times were studied, and possible mechanisms of extraction are discussed and evaluated. In addition, the stability of DEC against hydrolysis was measured.

KEYWORDS: anode slime, chloride hydrometallurgy, copper, precious metal refining, solvent extraction



Hydrometallurgy 177 (2018) 146–151

Contents lists available at ScienceDirect

Hydrometallurgy

journal homepage: www.elsevier.com/locate/hydromet

Effect of the diluent on the solvent extraction of neodymium(III) by bis(2-ethylhexyl)phosphoric acid (D2EHPA)

Nagaphani Kumar Batchu, Koen Binnemans*

KU Leuven, Department of Chemistry, Celestijnenlaan 200 F, bus 2404, B-3001 Heverlee, Belgium

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ARTICLE INFO

Keywords:
D2EHPA
Diluents
Lanthanides
Rare earths
Solvent extraction

ABSTRACT

The effect of different types of diluents on the extraction of neodymium(III) from a chloride aqueous feed solution by the acidic extractant bis(2-ethylhexyl)phosphoric acid (D2EHPA) was investigated. A total of 11 aliphatic, mixed aliphatic-aromatic and aromatic diluents were considered. D2EHPA was very well miscible with all diluents, even at 90% (v/v) of extractant. Aliphatic diluents gave the highest extraction efficiencies, while aromatic diluents did suppress the formation of emulsions or gels. Although a good separation of Dy(III) over Nd(III) was observed at 0.5 mol/L D2EHPA in all diluents, aromatic diluents were found to yield the highest separation factors. This study shows that the effect of the diluent on the extraction behaviour of Nd(III) is primarily determined by the aliphatic/aromatic content of the diluent. Diluents with the same concentration of aromatics show very similar behaviour. The choice of a diluent can be based on factors other than the extraction behaviour, for instance price and volatility.

CHEMICAL ENGINEERING RESEARCH AND DESIGN 161 (2020) 304–311

Contents lists available at ScienceDirect

Chemical Engineering Research and Design

journal homepage: www.elsevier.com/locate/cherd

Selection criteria of diluents of tri-*n*-butyl phosphate for recovering neodymium(III) from nitrate solutions

Mercedes Regadio*, Nagaphani Kumar Batchu, Koen Binnemans

KU Leuven, Department of Chemistry, Celestijnenlaan 200 F, bus 2404, B-3001 Heverlee, Belgium

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ARTICLE INFO

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Keywords:
Diluents
Neutral extractant
Rare earths
TBP
Solvent extraction

ABSTRACT

The selection of a proper diluent should be based on several criteria such as the distribution ratio, phase disengagement time, cost, safety and environmental impact of the process. The effect of different diluents on the solvent extraction of Nd(III) by the neutral extractant tri-*n*-butylphosphate (TBP) from nitrate feed solutions was studied. The nature of the diluent had little effect on the extraction kinetics of Nd(III) by TBP above 2.5 min. In general, phase disengagement times were relatively shorter for aromatic diluents compared to aliphatic diluents. Conversely, extraction efficiencies were the highest for aliphatic diluents, slightly lower for mixed aliphatic-aromatic diluents and much lower for aromatic diluents. The poorer extraction efficiencies of aromatic diluents may be due to the lower concentration of free extractant as a result of the stronger interactions of the diluent with water and/or of the diluent with the extractant. Under the experimental conditions, the differences in extraction between aliphatic and aromatic diluents decreased with increasing the salting-out effect of nitrate ions in the feed. At nitrate concentrations of 4.5 mol L⁻¹ or more, the different diluents had a limited influence on the metal extraction with 1 mol L⁻¹ TBP from feed solutions of 1 g L⁻¹ Nd(III). Thus, under these conditions, the selection of the diluent can be preferably based on its cost, safety and biodegradability rather than on its physico-chemical properties.

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Some key achievements by SOLVOMET

Methanesulfonic acid: a sustainable acidic solvent

Recovery of rare earths from the green lamp phosphor $\text{LaPO}_4:\text{Ce}^{3+}, \text{Tb}^{3+}$ (LAP) by dissolution in concentrated methanesulphonic acid



From the journal:
RSC Advances



Remaghic

Methanesulfonic acid: a sustainable acidic solvent for recovering metals from the jarosite residue of the zinc industry†



From the journal:
Green Chemistry



Zero Waste
CRATES
EU MSCA-ETN

Recovery of Lead and Silver from Zinc Leaching Residue Using Methanesulfonic Acid



From the journal
ACS Sustainable
Chemistry and
Engineering



nyrstar

KU LEUVEN

SOLVOMET GROUP

Broad industrial network

Bilateral projects

Previous collaborations / Active collaborations within H2020 projects



KU LEUVEN

SOLVOMET GROUP

SOLVOMET's academic network

Some key Projects



SAMEX



HE ENiCoN

HE HEPHAESTUS

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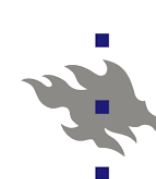
Key Partners



ETH zürich



HZDR



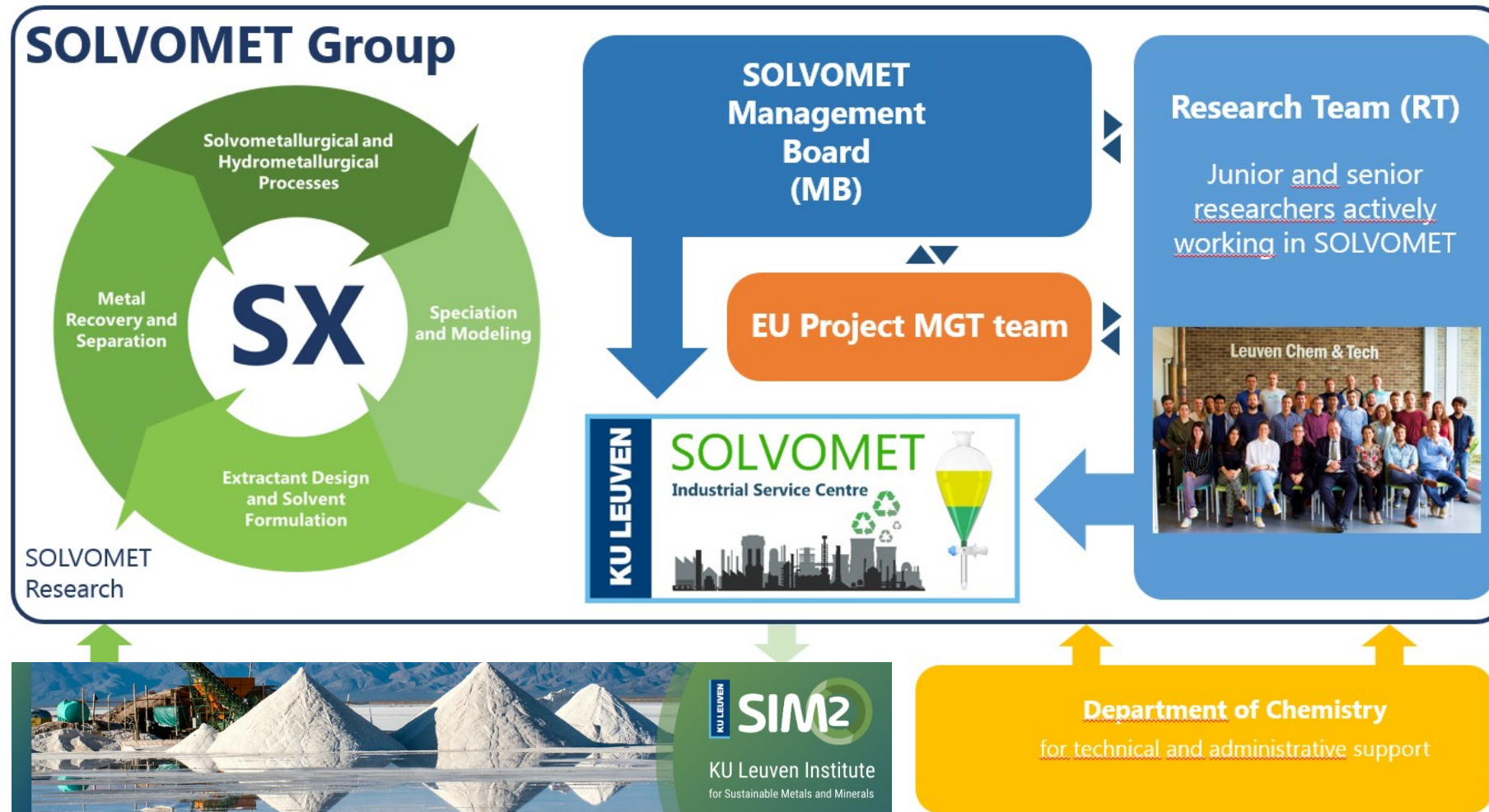
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Die Ressourcenuniversität. Seit 1765.



National Technical
University of Athens



SOLVOMET Group Governance



KU LEUVEN

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SOLVOMET Industrial Service Centre:

Project types



Services Agreement

- **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, hands-on researchers**

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**

Industrial postdoc project

- **Project description:** 4-page document research programme, budget
- **IP:** foreground IP transferred to company
- **Duration:** 1 to 2 years
- **Cost:** ~120,000 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 advanced, first-class training of postdoc researcher that can go and work for the company later**

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



Research Line 1

Geological exploration and advanced resource characterisation



Research Line 2

Remanufacturing and demanufacturing



Research Line 3

Sustainable metallurgical processes



Research Line 4

Upcycling processes for primary and secondary resources



Research Line 5

Sustainability assessment and policy research



Research Line 6

Process intensification and digitalisation

SIM² KU Leuven's mission is to develop, organise and implement problem-driven, science-deep research and future-oriented education, contributing to the environmentally friendly production and recycling of metals, minerals and engineered materials, supporting the transition to a climate-friendly, circular-economy.

<https://chem.kuleuven.be/solvomet>

<https://solvomet.eu>

<https://kuleuven.sim2.be/>

Contact: Peter Tom Jones ([LinkedIn profile](#))

