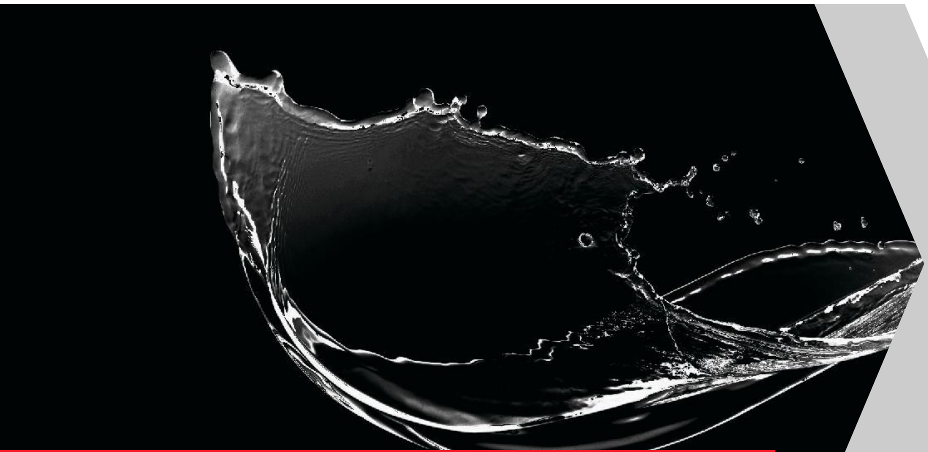

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Energizing Chemistry

Lewatit® ion exchange resins in the sustainable recycling of lithium ion batteries

Dr. Dirk Steinhilber, Technical Marketing Manager, LANXESS Deutschland GmbH

ALTA 2024 – Perth, May 31, 2024

Versatile specialists – comprehensive product portfolio provides advanced solutions

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Products and brands	
Lewatit® Lewatit® Scopeblue	Ion exchange resins, adsorbers, and functional polymers for use in many industries and applications
Bayoxide®	Granular iron oxide adsorbers for water treatment
LewaPlus®	Software for designing and optimizing ion exchange resin plants

Customer industries

Strong growth of battery market growth driven by e-mobility & renewables

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Key drivers

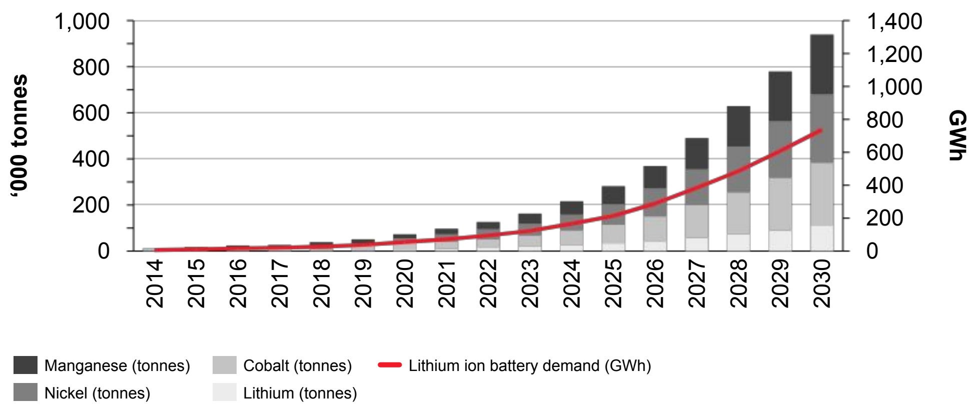
Megatrends driving the battery market

- Global awareness of global warming pushing for adoption of green solutions (CO_2 emissions reduction)
- Widespread introduction of **renewable energies** (stationary storage)
- **Population increase** and **city growth** challenging mobility and energy solutions (e-mobility)

Li-Ion battery is key technology for new concepts of mobility and energy

E-mobility drives demand for battery chemicals

Global lithium-ion and materials demand forecast from EV sales, 2015–2030 (thousand of tonnes, GWh)



Source: Bloomberg New Energy Finance

Monodisperse droplet generation by jetting process

Stable scaffolds for demanding metals processing applications!

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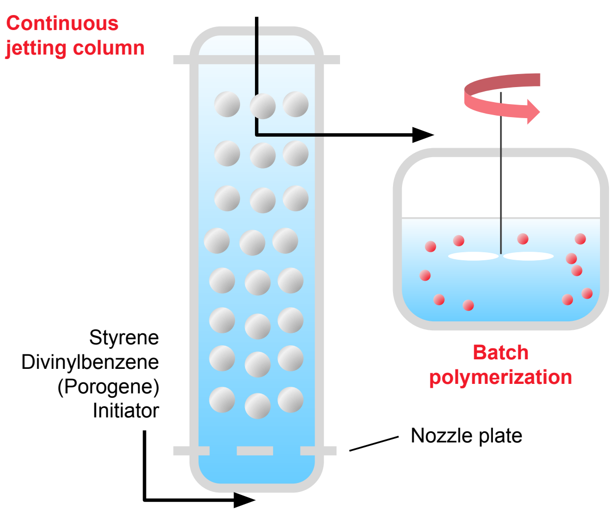
Formation of monodisperse droplets



Description

- Continuous process
- Raw materials are fed through a nozzle plate at the bottom of the column
- The resulting monomer jet is chopped into droplets of the same size
- Particle size can be controlled by adjustment of the whole width of the nozzle plate
- The droplets formed at the bottom start to encapsulate as they proceed to the column head
- Polymerization of the monodisperse encapsulated droplets is completed afterwards

The structure of macroporous resins

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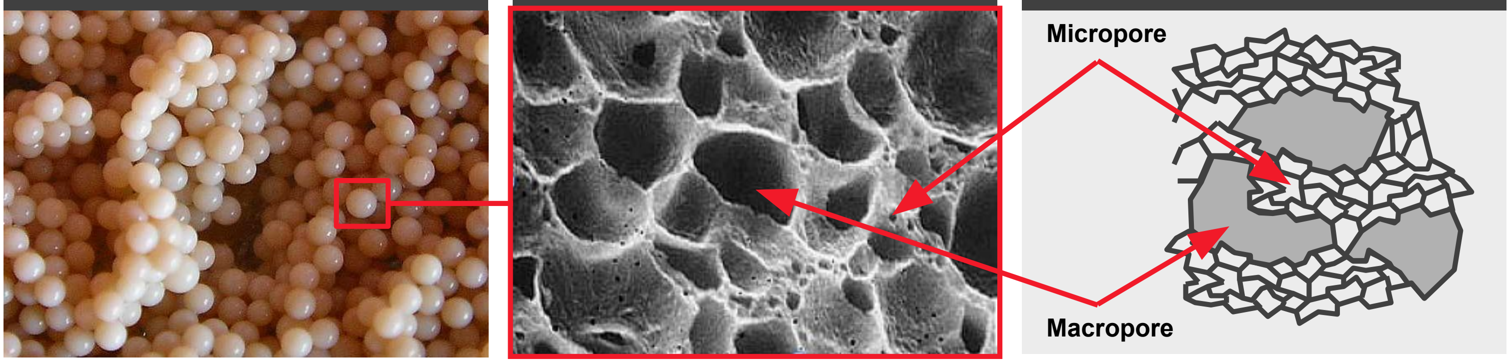
Small opaque beads are actually of a highly permeable sponge-like structure

Microscopic image | **SEM** | **Schematic structure**



Ion exchange groups

A strong portfolio of solutions for critical separation challenges

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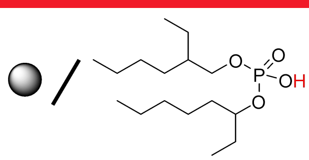
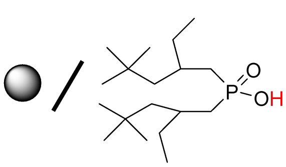
Solvent impregnated resins

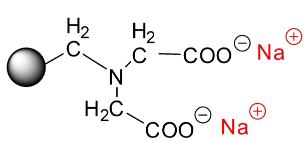


Zn, Cu removal from Ni, Co, Li
Lewatit® VP OC 1026

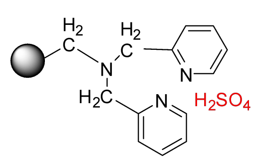


Co from Ni concentrate
Lewatit® TP 272

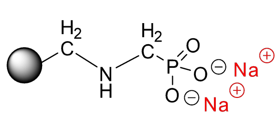
Selective chelating resins



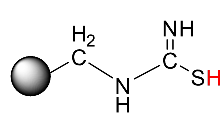
Ca+Mg from Li concentrate
Lewatit® MDS TP 208



Ni from Co and Fe(III)
Lewatit® MDS TP 220

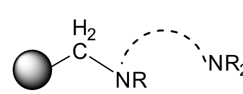


Al from Li, Bi+Sb from Cu
Lewatit® MDS TP 260

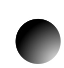


Pt group metals from Ni
Lewatit® MonoPlus TP 214

Anion exchange and adsorber



PFAS from black mass leachate
Lewatit® TP 108, Lewatit® MP 62 WS



Silica from Li and Ni
Bayoxide®, FeO(OH)

Key properties of ion exchange resins

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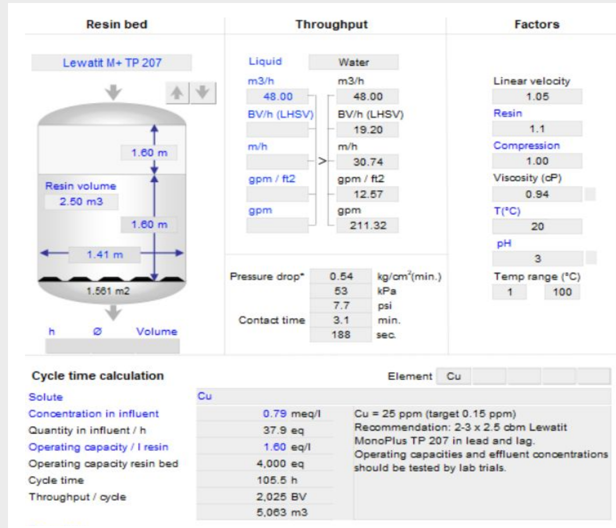
Precise control of resin parameters for critical separation challenges

- Functional group (type of chelating)
- Polymer Matrix (styrenic or acrylic)
- Morphology (gel or macroporous)
- Crosslinking
- Bead size (mono- vs. heterodisperse)
- Kinetics
- Resin swelling



Lewatit® ion exchange resins in applications for purification and refining of battery metals

LANXESS Energizing Chemistry

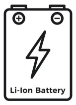
A strong portfolio of solutions for the preparation critical battery materials

	Nickel and Cobalt						Copper				Lithium		$LiPF_6$
	Recycling	Recovery Fixed Bed	Recovery Resin in Pulp	Concentrate Purification	Separation	Waste Water	Recovery Resin in Pulp	Recovery Fixed Bed	Waste Water	Concentrate Purification	Brine Purification	Recycling	Purification
Lewatit® MonoPlus TP 209 XL		■	■			■	■						
Lewatit® MonoPlus TP 207	■	■		■		■		■	■			■	
Lewatit® VP OC 1026	■			■									
Lewatit® TP 272				■									
Lewatit® MDS TP 220				■	■			■	■				
Lewatit® MDS TP 260	■			■		■		■		■	■	■	
Lewatit® MDS TP 208											■		
Lewatit® MonoPlus TP 214				■									
Lewatit® TP 308											■		
Lewatit® MP 62 WS				■									■
Lewatit® TP 108	■												
Bayoxide® E IN 30				■							■		

Lithium ion battery material life cycle

Lewatit® is a crucial part in the recycling flow sheet!

- End of the cycle batteries
- Discharge
- Dismantle
- Crush



- Acid Leaching black mass
- Impurity dissolution

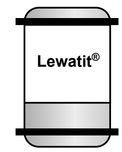
100 g/L Ni, Co, Mn, Li

1-10 ppm Zn, Cu, Al, Fe

NMC 622,
NMC 811

- Lewatit® MDS TP 260 (Zn, Cu, Al, Fe)
- Lewatit® VPOC 1026 (Zn, Cu, Al, Fe)
- Lewatit® MonoPlus TP 207 (Cu)

100 g/L Ni, Co, Mn, Li



Process flowsheet refining of black mass leachate

Lewatit® ion exchange resins are required in the most critical streams within the recycling flow sheet

Black mass

↓ Acid Leach

Ni, Co, Mn, Li, Zn, Al, Cu, Fe

↓ Neutralization

Ni, Co, Mn, Li, Zn, Al, Cu, Fe

↓ Impurity removal

Lewatit® MDS TP 260

↓

Ni, Co, Mn, Li

→ SX →

$NiSO_4$ raw	$CoSO_4$ raw	Li_2SO_4 raw	$MnSO_4$ raw
↓	↓ Trace impurity removal	↓	↓
Lewatit® VPOC 1026 Lewatit® TP 272	Lewatit® VPOC 1026	Lewatit® M+ TP 207	Lewatit® M+ TP 207
↓	↓	↓	↓
$NiSO_4$ battery	$CoSO_4$ battery	Li_2SO_4 battery	$MnSO_4$ battery

Polishing black mass leachate Lewatit MDS TP 260

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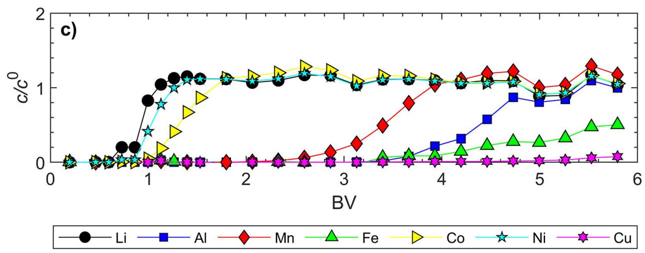
Energizing Chemistry

High selectivity of MDS TP 260 for Al, Fe and Cu in presence of Li, Ni, Co, Mn!

Op. Conditions	
Resin in Na form	
Al	1.3 g/L
Fe	0.6 g/L
Cu	1.7 g/L
Co	13.2 g/L
Ni	1.6 g/L
Li	3.9 g/L
Mn	2.0 g/L
SV	2 BV/h
Temp	60°C
pH	3
Breakthrough	0.1 c/c0
Op. Capacity	
Cu	> 0.4 eq. Cu/L
Al	0.4 eq. Al/L
Fe	0.1 eq. Fe/L



Virolainen et al. Hydrometallurgy, 2021, 20, 105602

Benefits

- High selectivity of MDS TP 260 towards contaminants like Al, Zn, Cu, and Fe allow efficient separation
- Savings on CAPEX. Fast exchange kinetics allows small compact filters
- Savings on OPEX, high loading capacities, less frequent regeneration and lower chemical consumption
- Longer lifetime of the resin due to higher stability and less frequent regenerations

Selective elution of Aluminium and Zinc from Lewatit® MDS TP 260

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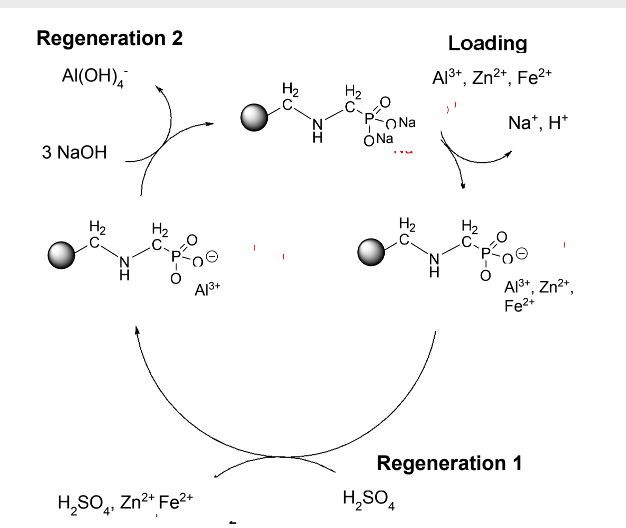
Efficient and easy elution of Al and Zn by use of NaOH!



Benefits

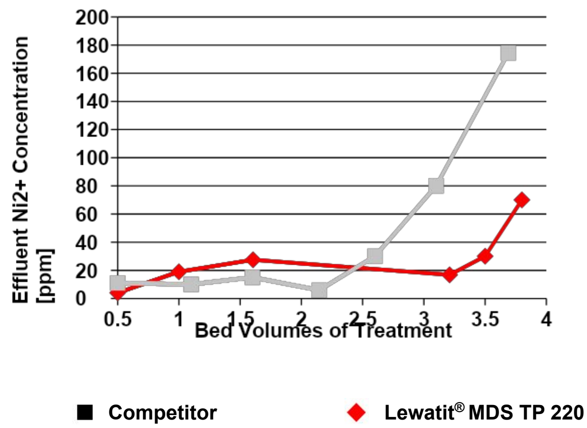
- Al, Zn regeneration efficiency (r.e.) > 99 % by use NaOH
- Conventional acid regeneration yields < 60% r.e.
- Technique by Virolainen et al. yields high r.e. but requires additives (EDTA- and oxalic acid salts)[1]
- NaOH is cheap, readily available, renewable NaOH available
- NaOH elutes Al and Zn selectively. Coloaded Ni, Co, Mn recovered by acid elution and feeding upstream

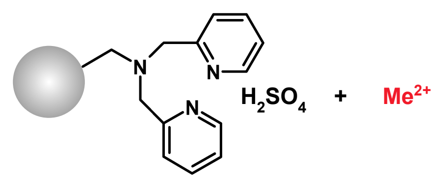
Virolainen et al. Hydrometallurgy, 2021, 20, 105602[1]

Nickel recovery in presence of high concentration of ferric using Lewatit® MDS TP 220

The resin has a high selectivity for nickel over ferric and cobalt!

Nickel	2.6 g/L
Ferric	17 g/L
pH	1.8
Temperature	r.T.
Specific velocity	10 BV/h



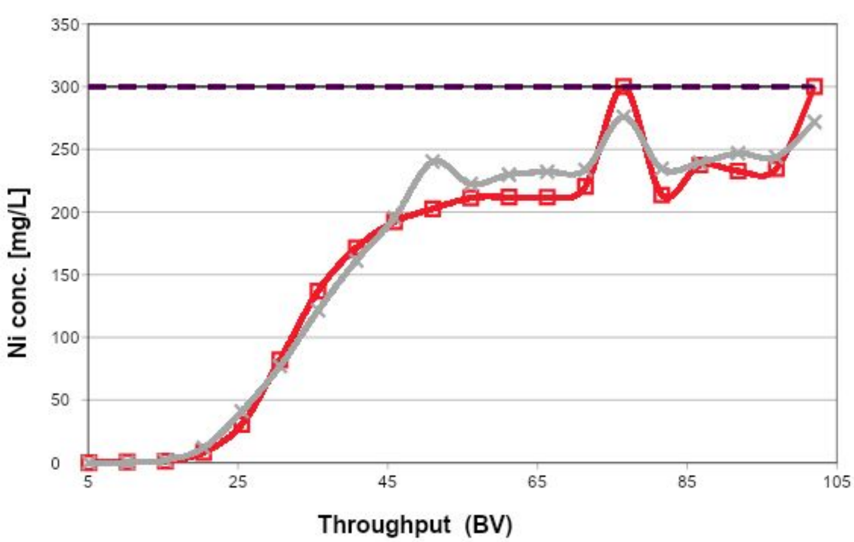


Other application fields

- Purification of cobalt electrolytes (nickel/cobalt separation)
- Copper recovery at low pH (<2)
- Separation of nickel and copper from chromium (III) and ferric solutions
- Selectivity Series: Cu^{2+}> UO_2^{+}> Pb^{2+} > Ni^{2+}> Fe^{3+}> Zn^{2+}>Co^{2+}> Cd^{2+}> Fe^{2+}>>>Cr^{3}

Removal of nickel from cobalt concentrate

Breakthrough curves of benchmark show higher capacity for Lewatit® MDS TP 220



Operating conditions

Ni^{2+}, feed	300 mg/L
Co^{2+}, feed	37 g/L
pH	2.0
SV	5 BV/h
Temp	60°C
breakthrouh	300 mg/L

Operating capacity

MDS TP 220	14.3 g Ni/Liter
Competitor	13.9 g Ni/Liter

Lewatit® TP 272 for Ni/Co separation

A selective resin for cobalt over nickel!

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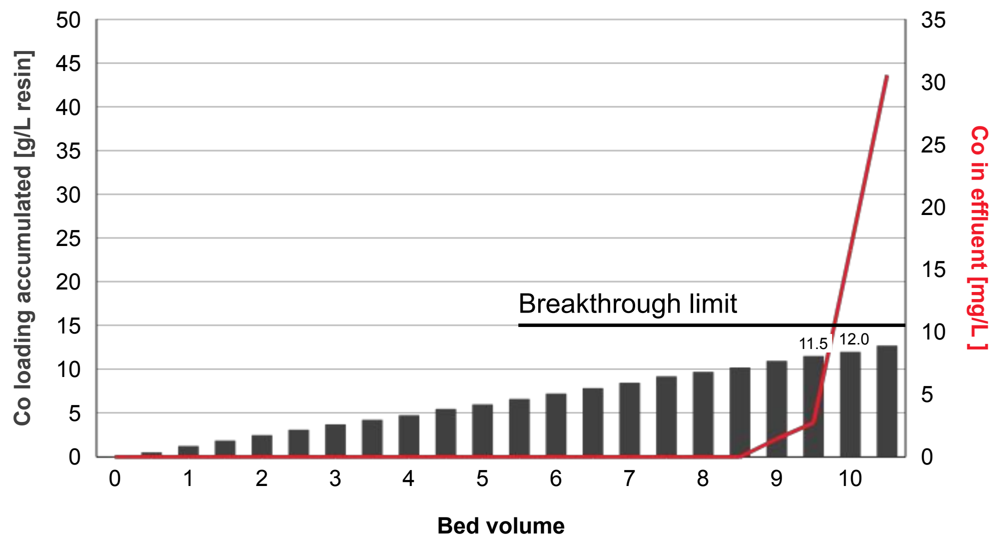
Loading

Nickel	≈ 80 g/L
Cobalt	≈ 990 mg/L
$(NH_4)_2SO_4$	≈ 50 g/L
pH adjustment	ammonia
pH	≈ 5,0
Temperature	≈ 65°C
Specific velocity	3 BV/h



Ion exchange resins for high purity cobalt refining

Zink and copper removal from cobalt electrolyte

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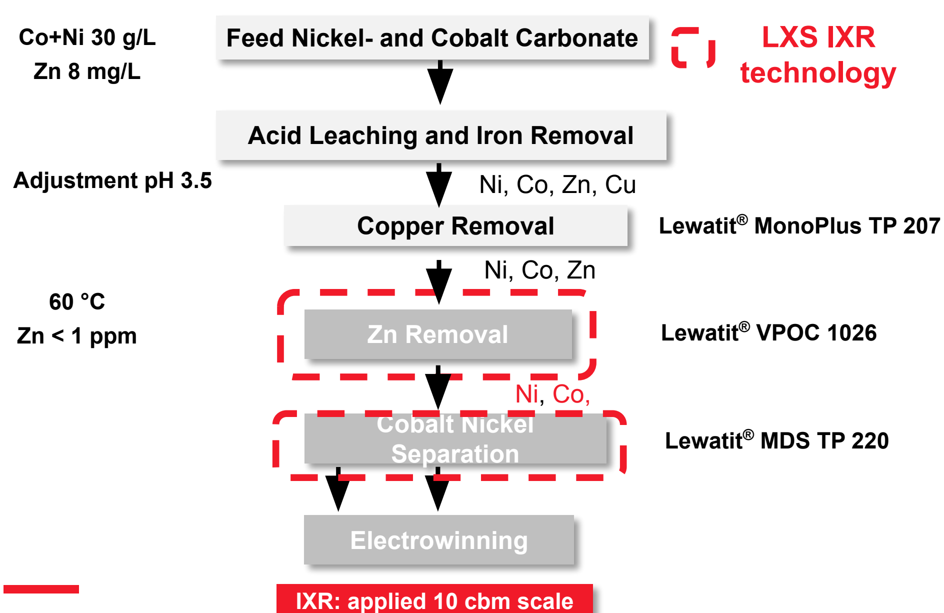
Lewatit® IXR are required in the most critical places within the flow sheet



Lithium concentration from wastewater by loading and elution and scrubbing cycles

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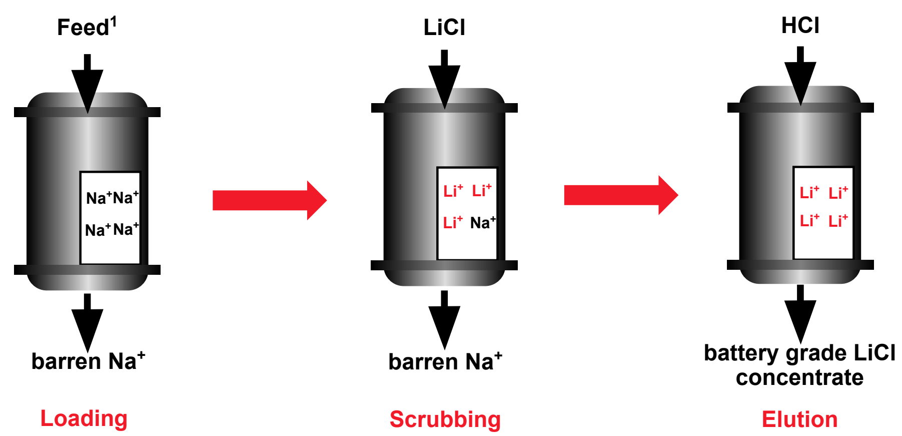
High Li purity in eluate of Lewatit® allows preparation of battery grade LiCl with high yield > 99%!



Benefits

- High Li > 20 g/L concentrations obtained in eluate, recovery as Li_2CO_3
- Pure Li can be obtained by scrubbing off co-loaded Na with Li salt solution
- High Li loading capacities obtained at pH >10 and 60°C
- Empty bed contact times around 12 min. ensure high Li loading
- Process proven on pilot scale and now transferred to industrial scale

[1] Feed: LiCl 1.2 g/L (200 mg Li/L, NaCl 50 g/L, 10 BV/h, 60°C.

Selective fluoride removal from lithium concentrates

Aluminium doped MDS TP 260 (Al) shows high fluoride selectivity in bicarbonate and sulphate concentrates!

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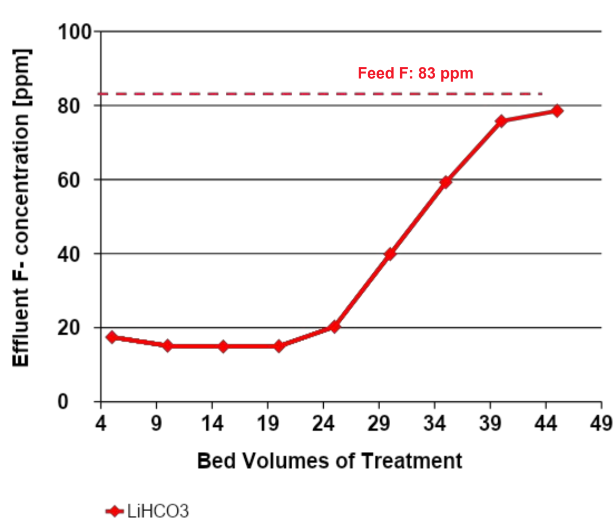
Selective fluoride removal from lithium concentrates: low fluoride leakage, clean lithium!

Op. Conditions	
Resin in Na form	
Fluoride	83 ppm
$LiHCO_3$	75 g/L
pH	8
SV	5 BV/h
Temp	60°C
Breakthrough	30 ppm F
Op. Capacity	
$LiHCO_3$	2.1 g F/L



Benefits

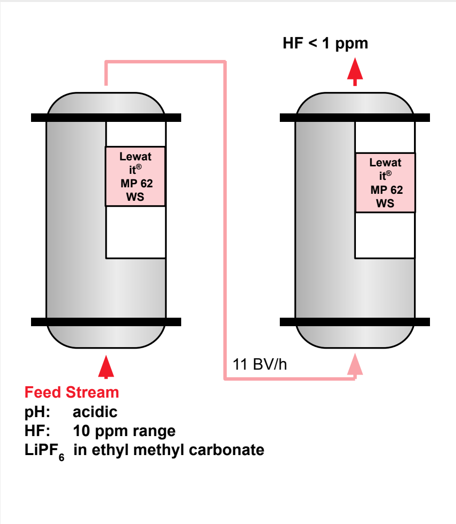
- High selectivity of MDS TP 260 (Al) towards F allow efficient separation
- Savings on CAPEX. High exchange kinetics enable the use of small compact filters
- Savings on OPEX, high loading capacities, less frequent regeneration and lower chemical consumption
- Longer resin lifetime due to higher stability and less frequent regenerations
- F removal from Li_2SO_4 removal efficiency to ppb level

HF removal from $LiPF_6$ electrolyte

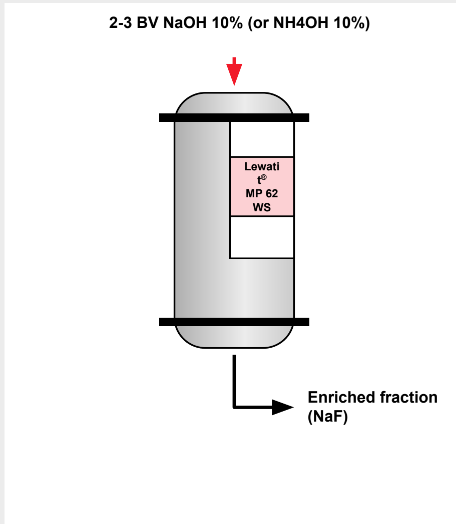
Pure electrolytes free of acid by Lewatit® MP 62 WS

LANXESS Energizing Chemistry

Operation



Regeneration



- HF generated by hydrolysis of $LiPF_6$
- Operating capacities up to 1 eq/L and more possible at high HF feed concentration
- At low feed concentrations operating capacities between 0.5 eq/L and 1 eq/L g/L

Chromatographic effect in ion exchange

Resins with small and monodisperse bead size for a better utilization of resin capacity

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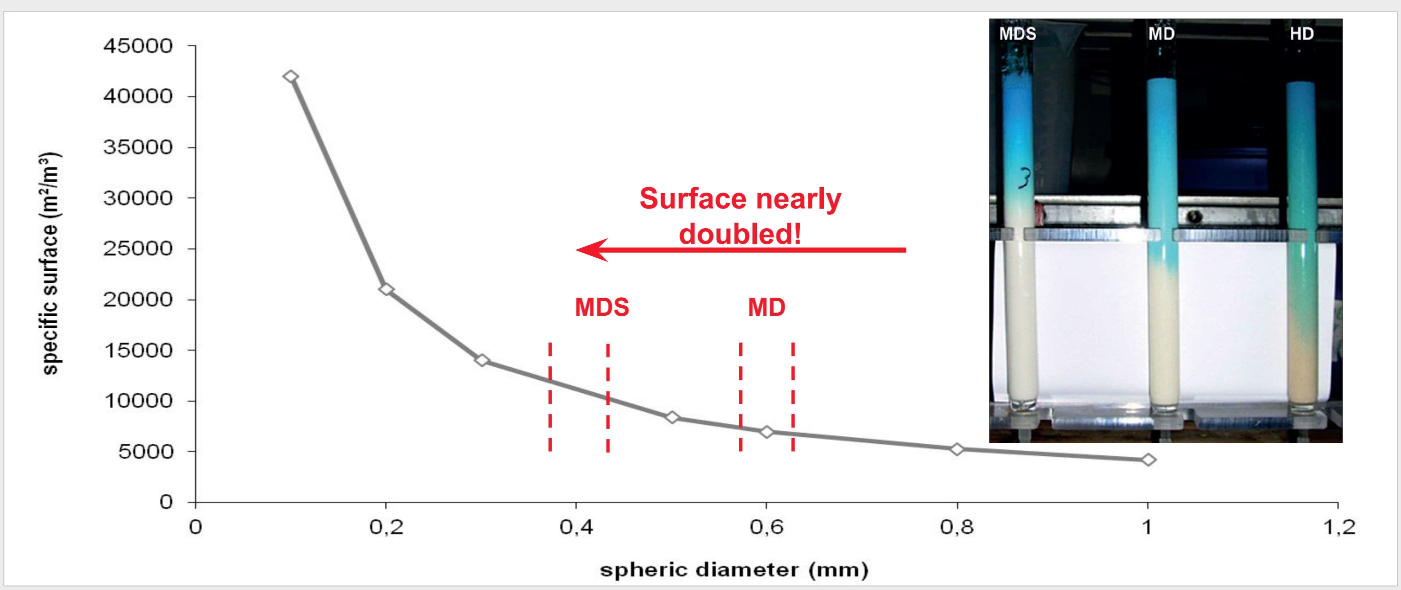
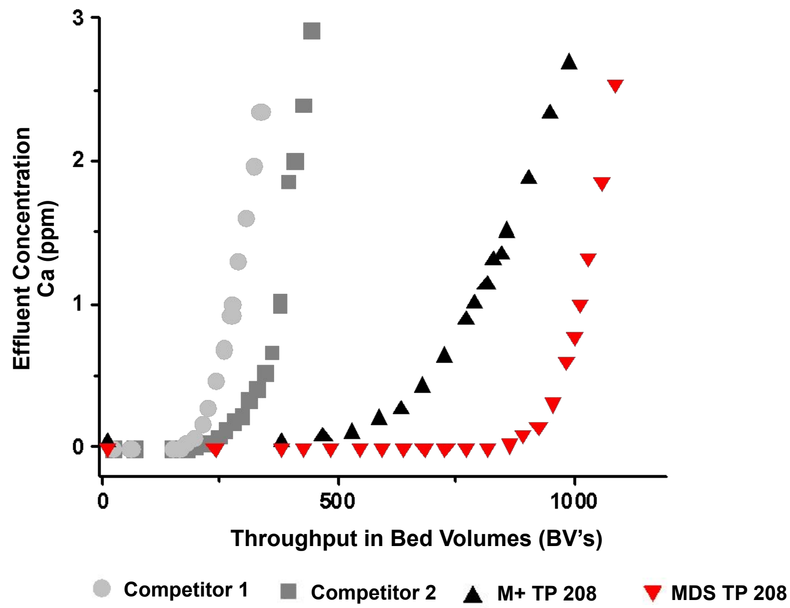


Calcium capacity from LiCl brines

Lewatit® MDS TP 208 has the highest operating capacity and the lowest leakage!

Softening lithium chloride brine to achieve less than 1 ppm of Ca

Op. Conditions	
Resin in Na form	
$CaCl_2$	10 ppm as Ca
LiCl	61.1 g/L (10 g Li/L)
NaCl	60 g/L as NaCl
pH	9
SV	10 BV/h
Temp	60°C
Breakthrough	1 ppm Ca
Op. Capacity	
Competitor 1	2.7 g Ca/L
Competitor 2	3.7 g. Ca/L
Lewatit® M+TP 208	8.3 g Ca/L
Lewatit® MDS TP 208	10.6 g Ca/L



Calcium Capacity from LiCl Brines

MDS TP 208 can achieve less than 20 ppb hardness in the treated brine

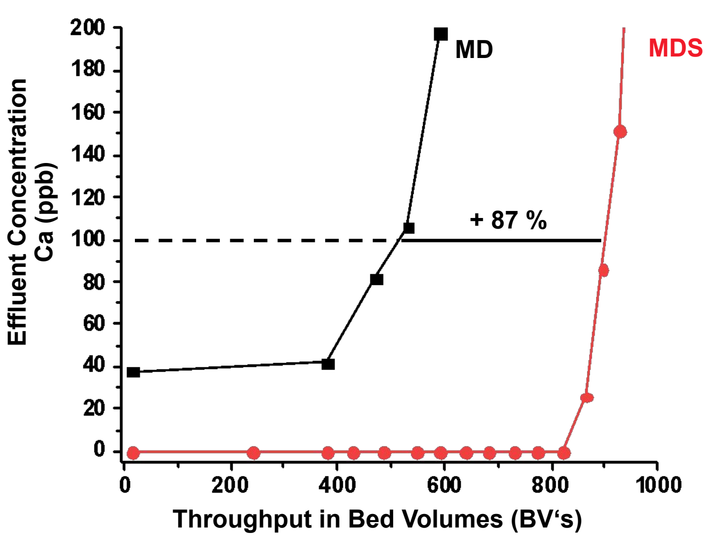
Lewatit® MonoPlus TP 208 vs. MDS TP 208

Op. Conditions

Resin in Na form
$CaCl_2$, feed 10 ppm as Ca
LiCl, feed 61.1 g/L (10 g/L as Li)
NaCl, feed 60 g/L as NaCl
pH 9
SV 10 BV/h
Temp 60°C
breakthrough 100 ppb Ca

Op. Capacity

M+ TP208 5,2 g Ca/L
MDS TP 208 9,0 g Ca/L

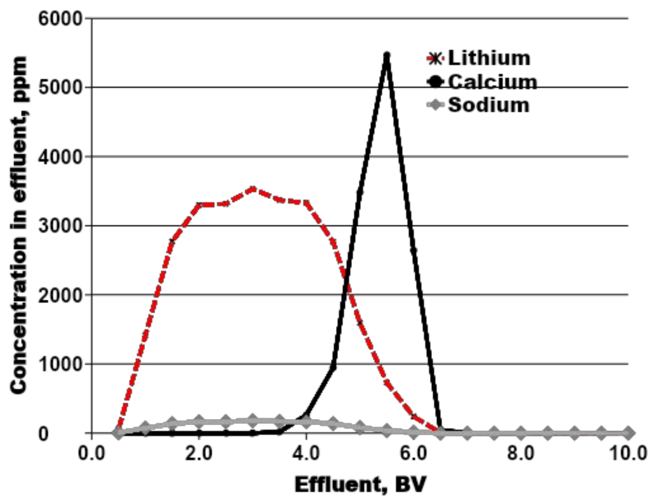


Lithium recovery by selective regeneration of trial product

Efficient separation between lithium and calcium by split elution, high lithium yield!

More than 70% of co-loaded lithium can be recovered by split elution

Loading conditions	
Resin in Na form	
$CaCl_2$, feed	10 ppm as Ca
LiCl, feed	61.1 g/L (10 g Li/L)
NaCl, feed	60 g/L as NaCl
pH	9
SV	10 BV/h
Temp	60 °C
Regeneration conditions	
HCl	1.5%
SV	1.5 BV/h



Silica removal from $NiSO_4$, $CoSO_4$, LiCl, and LiOH concentrates by use of Bayoxide® E IN 30

Bayoxide® has a low silica leakage in various battery metal concentrates

Nickel sulfate

	Co	Fe	Ni	Si
Feed silicate	970	0	81550	78.9
Effluent silicate	445	0	65250	2.1
Removal %	54%	n/a	20%	97%

Lithium chloride

	Fe	Li	Si
Feed silicate	0	14070	68.8
Effluent silicate	0	13535	0.01
Removal %	n/a	4%	100%

Lithium hydroxide

	Fe	Li	Si
Feed silicate	0	7715	24.8
Effluent silicate	0	5145	0.01
Removal %	n/a	33%	100%

10 g of Bayoxide were added to 40 mL of concentrate and shaken for 20 h. After decantation and filtration through 0.4 µm filter, ICP analysis performed. Regeneration procedure is currently optimized.

Smart solutions for more efficient use of scarce battery metals – Energized by LANXESS

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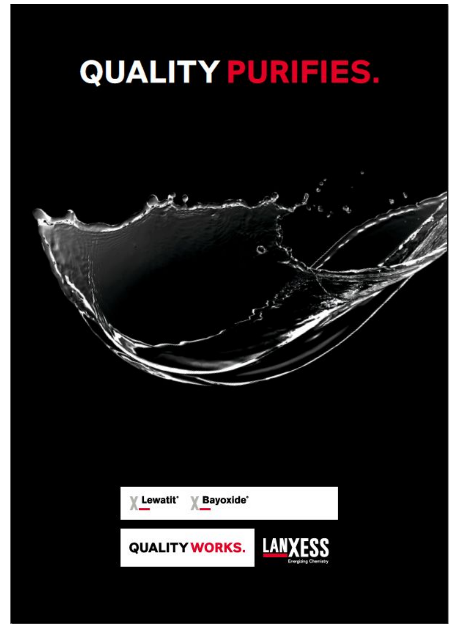
Energizing Chemistry

- One of the leading global solution provider with excellent technical application know-how
- Competence recycling and refining of battery materials
- German standards and certified by international organizations
- Global highly competent sales & technical service team



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