Producing Battery Grade Nickel Sulphate from Varying Feed Sources

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PURITY - Free from impurities

SUSTAINABILITY - Low carbon footprint

VALUE - High ratio of performance / cost

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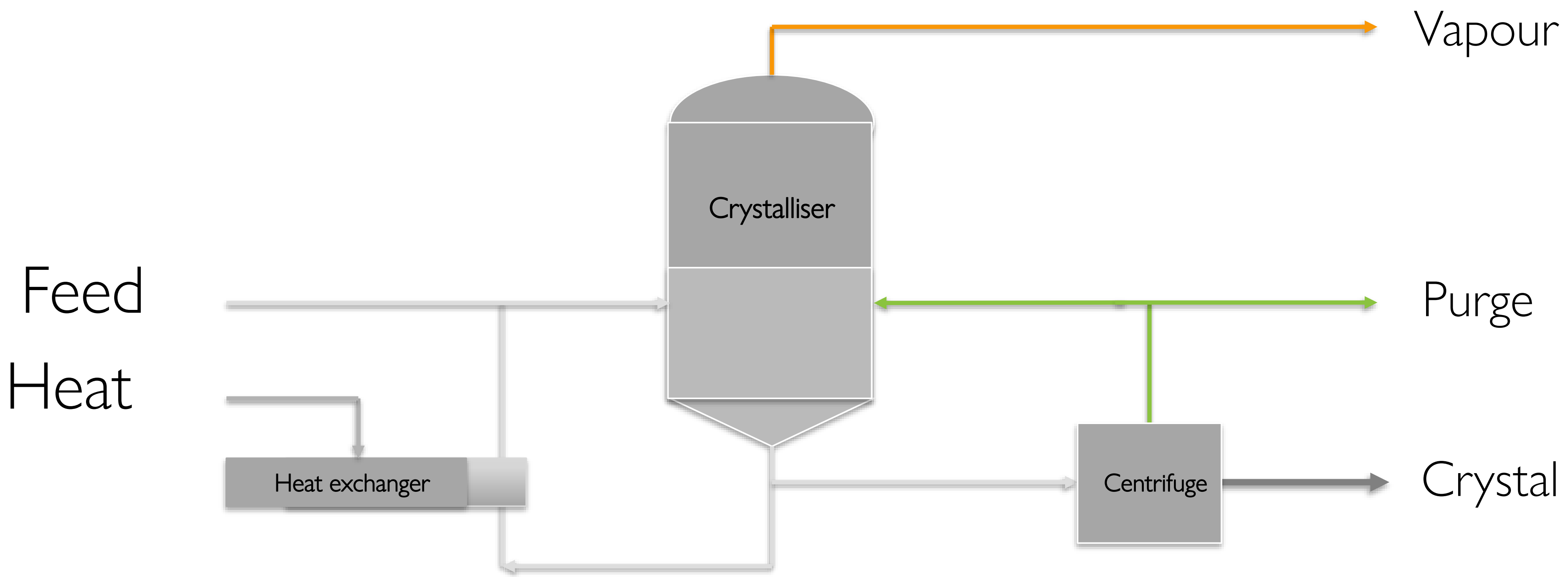
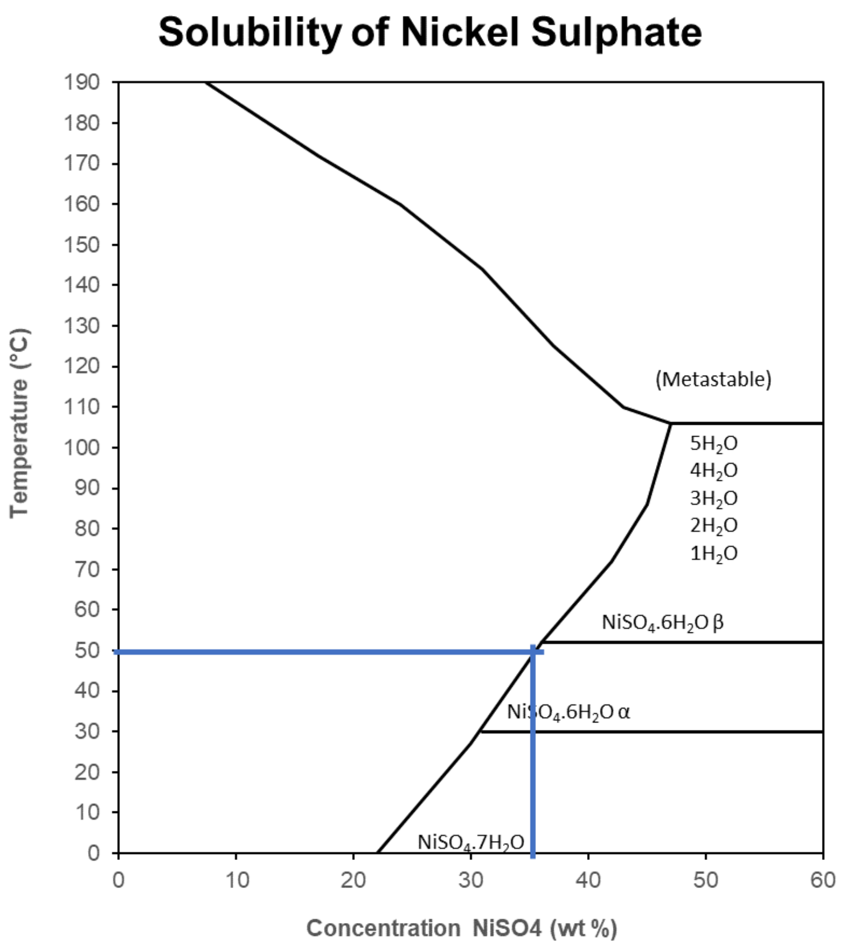
NiSO4 Crystallisation and Product Forms

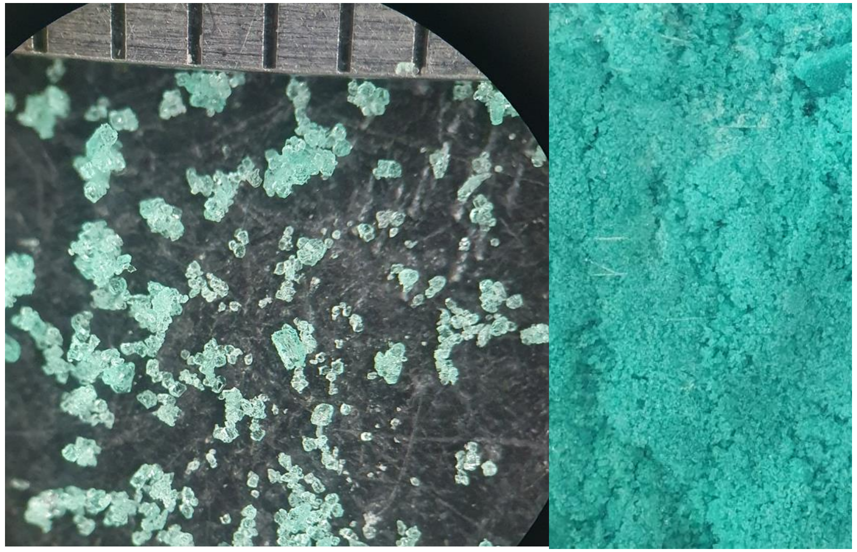
Simplified crystallisation process




Solubility of Nickel Sulphate
Temperature (°C)
Concentration NiSO4 (wt %)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
0
10
20
30
40
50
60
(Metastable)
5H2O
4H2O
3H2O
2H2O
1H2O
NiSO4.6H2O β
NiSO4.6H2O α
NiSO4.7H2O

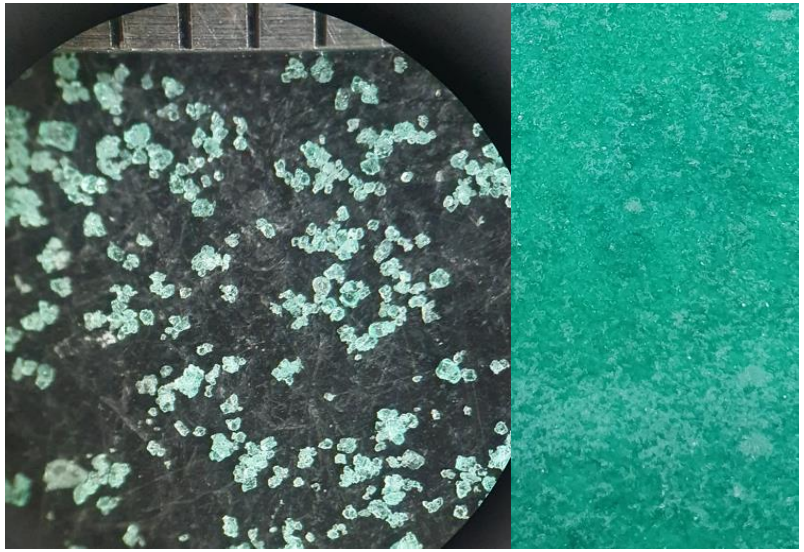
NiSul Crystals – before drying

α - Alpha



LT at 50 °C
Clarity: Slightly opaque
Colour: Turquoise

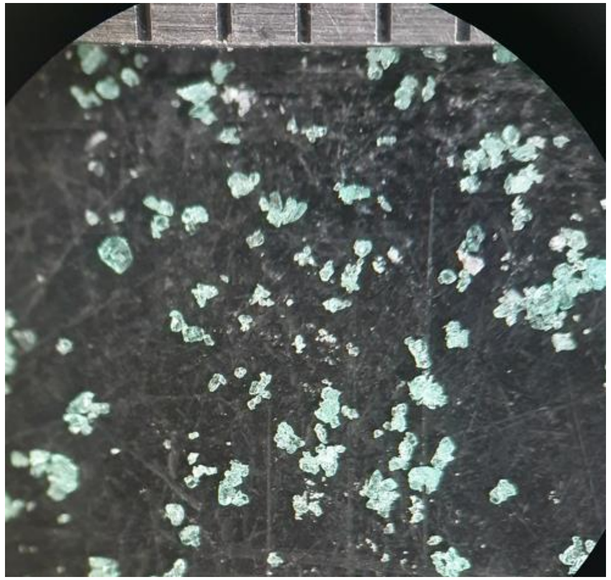
β - Beta



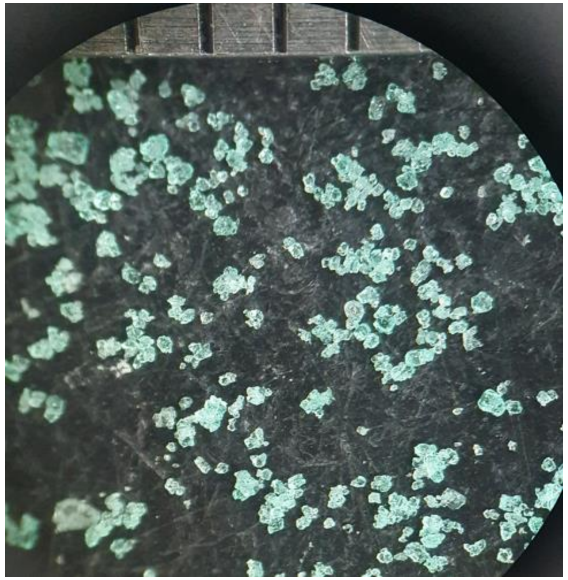
LT at 75 °C
Clarity: Translucent
Colour: Green

NiSul Crystals – after drying

α - Alpha

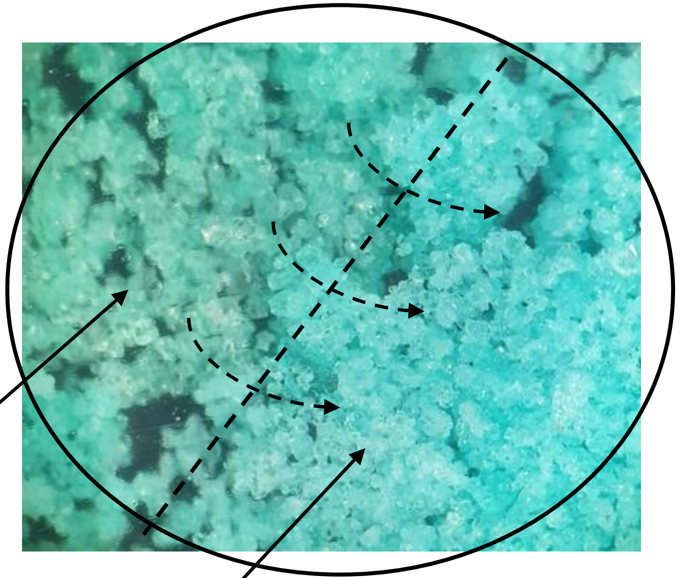


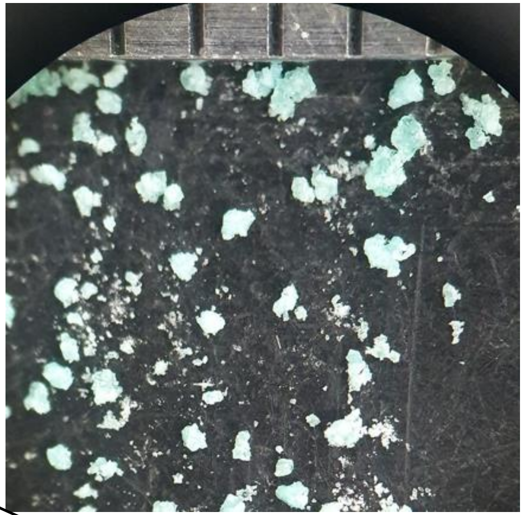
β - Beta



No major changes observed in crystal PSD after drying
(1 hr at 50 °C) for both a and β crystals.

NiSul β Crystals – after 24 hr





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Comparing α and β crystals.

- Distinct differences observed between α and β crystals.
- No changes observed in α and β crystal PSD's after 1hr of drying at 50 °C.
- After 24 hours we observed β reverts to the more stable a form over time (slow kinetics) causing crystals to splinter, and break creating fines.
- Observation on dried crystal clumps (~10 mm): β clumps are substantially harder to break than α clumps.

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Design Considerations

Design Approach

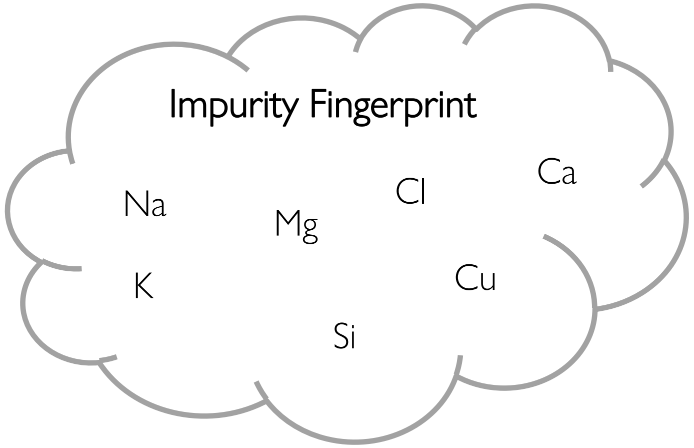
- Maximise purity
- Maximise yield
- Minimise CAPEX and OPEX costs
- Ease of operation with robust design

- Simulation using thermodynamic software
- Mass & energy balances
- Test work
- Flowsheet optimisation
- Equipment design

- Sufficient residence time to allow large crystals to grow
- Sufficient mixing to expose large crystals to the zone with highest level of supersaturation
- Control of slurry density
- Control of temperature, pressure and level

FEED QUALITY FROM VARIOUS SOURCES

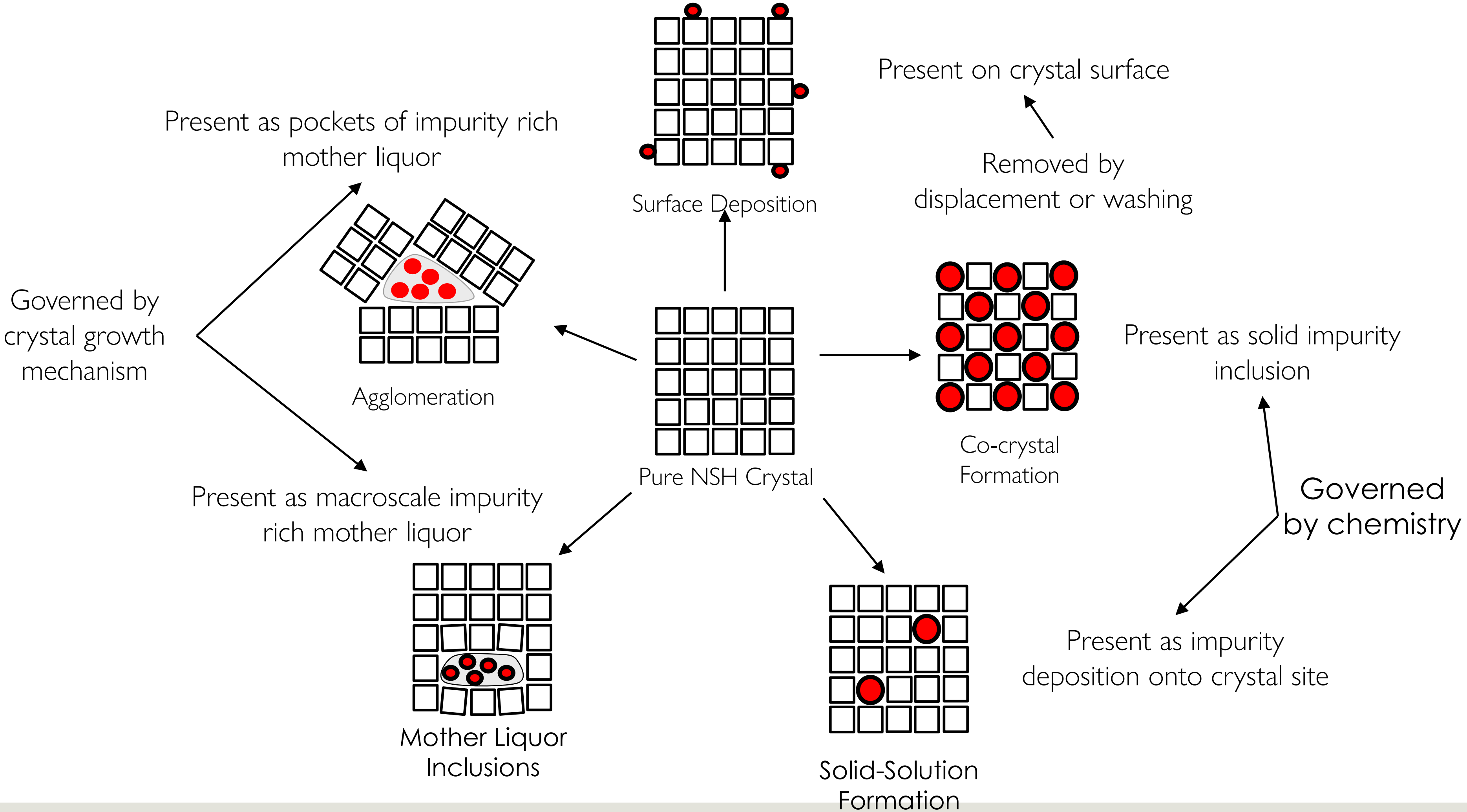
Element	Unit	Excellent Feed	Good Feed	Typical Feed
Ni	g/L	162	162	162
$NiSO_4$	wt%	30	30	30
$NiSO_4$	g/L	426	426	426
Total impurities	mg/L	72	720	1450
Impurities / NSH	mg/kg	100	1000	2000



TYPICAL PRODUCT SPECIFICATIONS

Element	Unit	Typical 1	Typical 2
Ni	% min	22.3%	22%
$NiSO_4.6H_2O$	% min	99.9% (“3 nines”)	98.5%
Co	ppm max	10	10
Na	ppm max	1	5-20
Mg	ppm max	1	5-20
Cl	ppm max	5	10
Cu	ppm max	1	1-5
Si	ppm max	1	5
K	ppm max	1	5

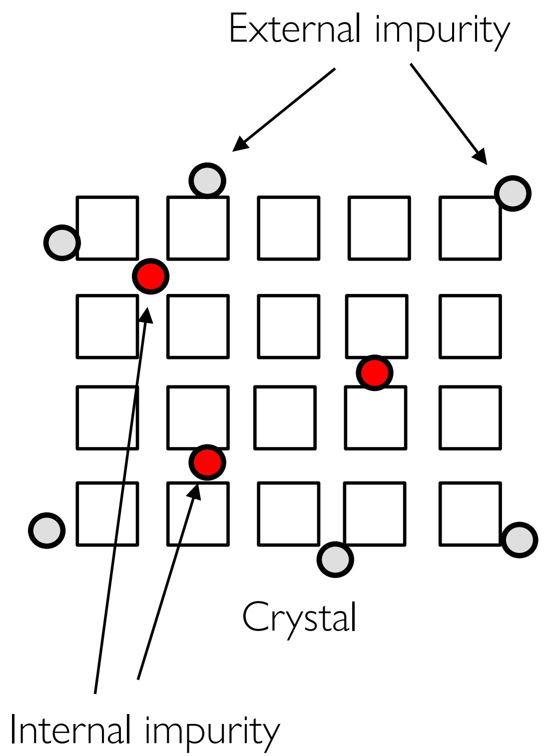
Crystalliser design



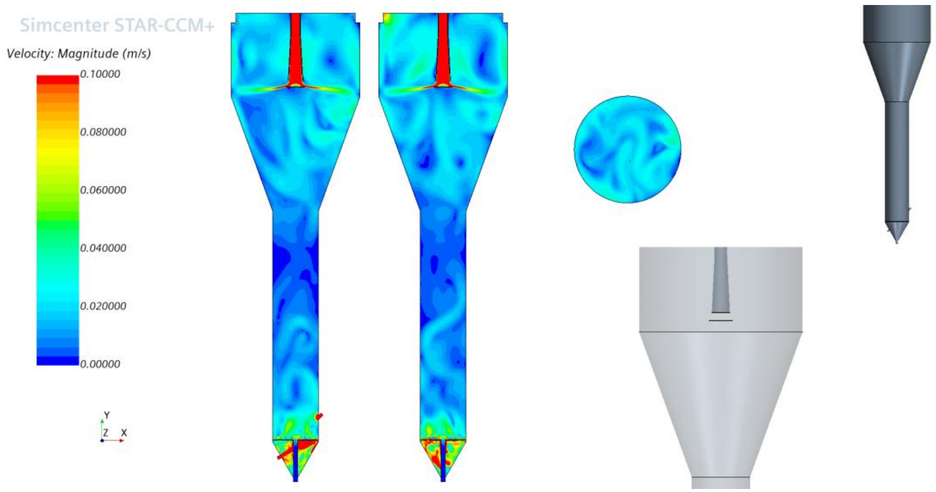
Levers for Purification

Ref: Urwin, S.J., et al. (2020)

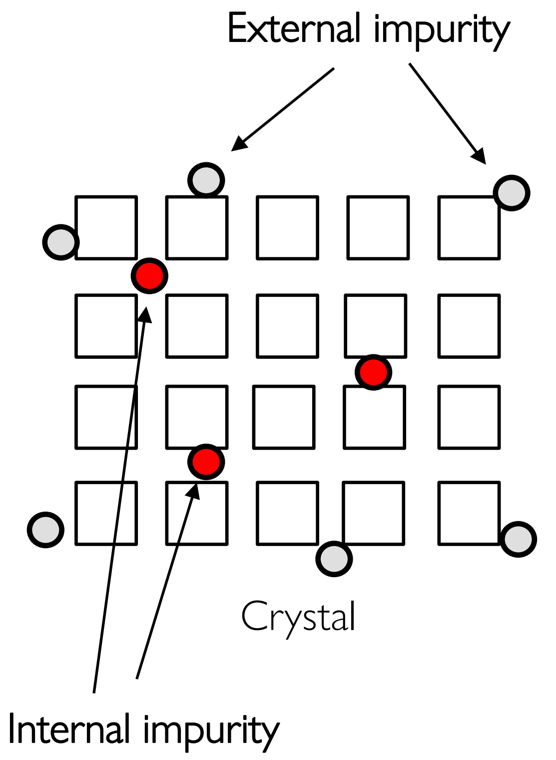
Levers for Purification



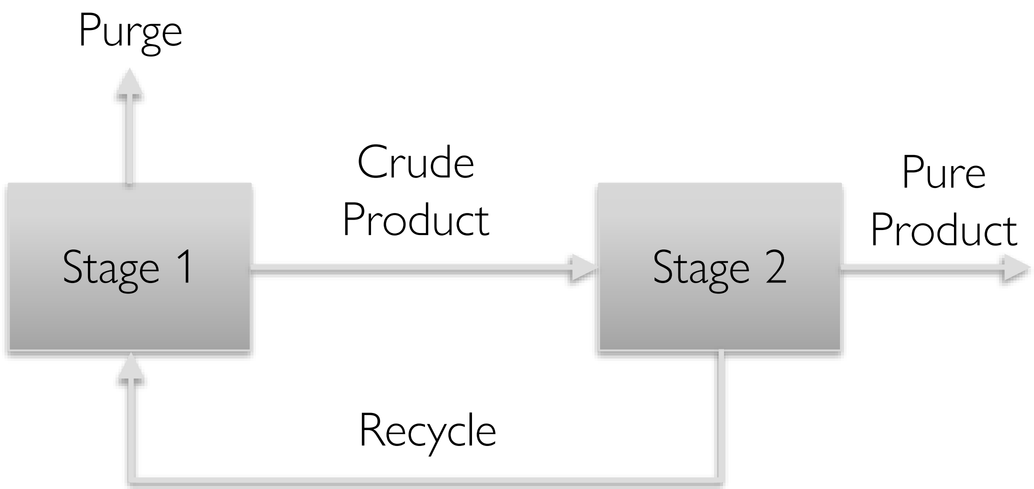
- Centrifugation displaces surface depositions by washing and ML removal.
- Wash legs reduce surface depositions by partly displacing high impurity ML with feed.



Levers for Purification



- Staged crystallisation reduces ML impurity fingerprint profile.
- Purge/Recycle rates reduce ML impurity fingerprint profile.



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Crystalliser Performance

$$\text{Purity Enhancement Factor (PEF)} = \frac{(\text{Impurity} \;/\; \text{desired species})_{\text{feed}}}{(\text{Impurity} \;/\; \text{desired species})_{\text{product}}}$$

REQUIRED PERFORMANCE TO MAKE BATTERY GRADE

		4 nines product		5 nines product	
	mg impurity / kg NSH	mg impurity / kg NSH	Required PEF	mg impurity / kg NSH	Required PEF
TYPICAL	2,000	100	20	10	200
GOOD	1,000	100	10	10	100
EXCELLENT	100	100	n/a	10	10

CRYSTALLISER PERFORMANCE

	purge fraction	entrainment	wash efficiency	Fraction of impurity		PEF
				purged	on crystals	
conventional single stage	5%	5%	80%	91.3	8.7	10.8
optimised single stage	10%	3%	85%	98.02	1.98	44.6
two stage	5%	5%	80%	99.24	0.76	113

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Conclusions

CONCLUSIONS

- Crystallisation is a key step in the processing of battery materials such as nickel sulphate to achieve the desired product purity.

- Alpha and beta form of Nickel sulphate hexahydrate products were evaluated. Although the costs associated with producing a beta product is less, it introduces challenges associated with product quality and product handling.

- A carefully designed crystalliser can produce crystals large enough for effective dewatering and washing.

CONCLUSIONS

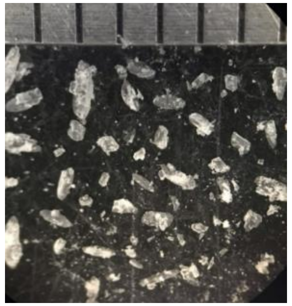
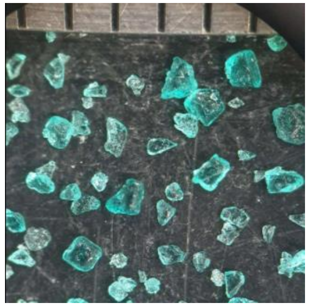
- A carefully designed crystalliser can produce crystals large enough for effective dewatering and washing.

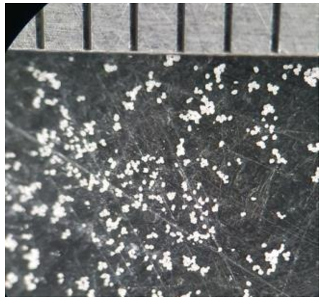
- We need to control liquor chemistry & kinetics to prevent lattice substitution; formation of double salts; inclusions and agglomerates.

- As the demand for the product purity increases, the requirement for optimisation and further enhances in the technology become critical.

Expertise in Battery Chemicals

$LiOH.H_2O$ | $NiSO_4.6H_2O$ | $CoSO_4.7H_2O$ | $MnSO_4.H_2O$



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