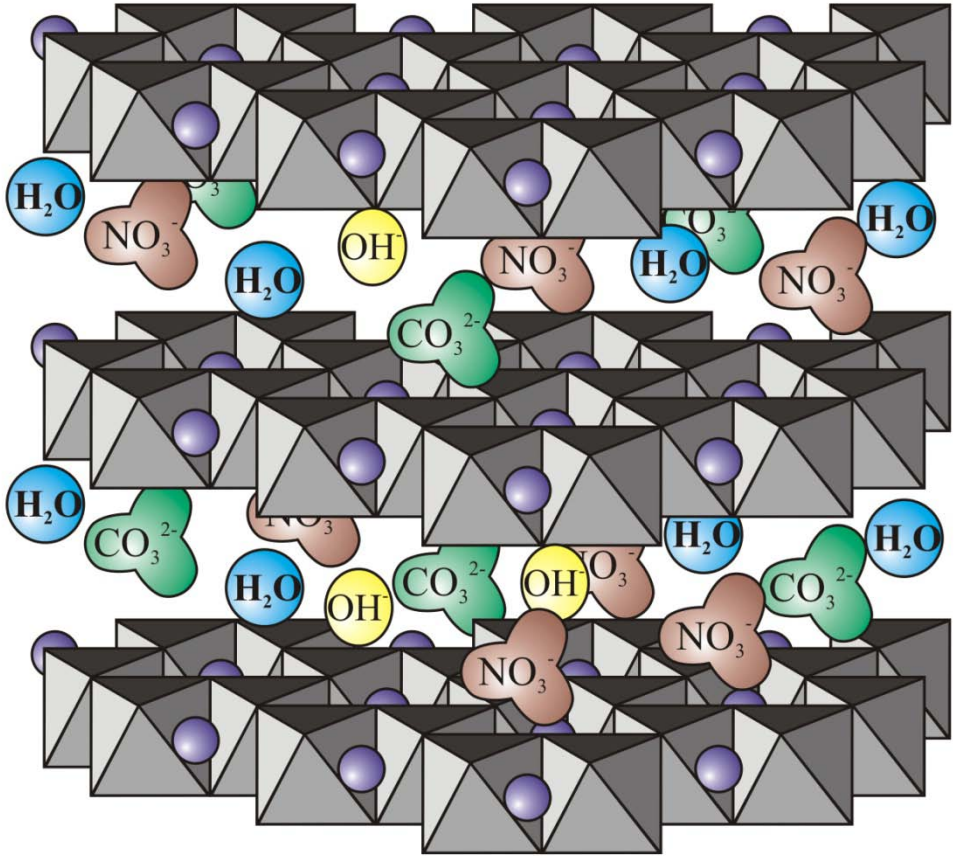
washington river protection solutions

Scouting Experiments for Alumina Reduction in WTP Feed

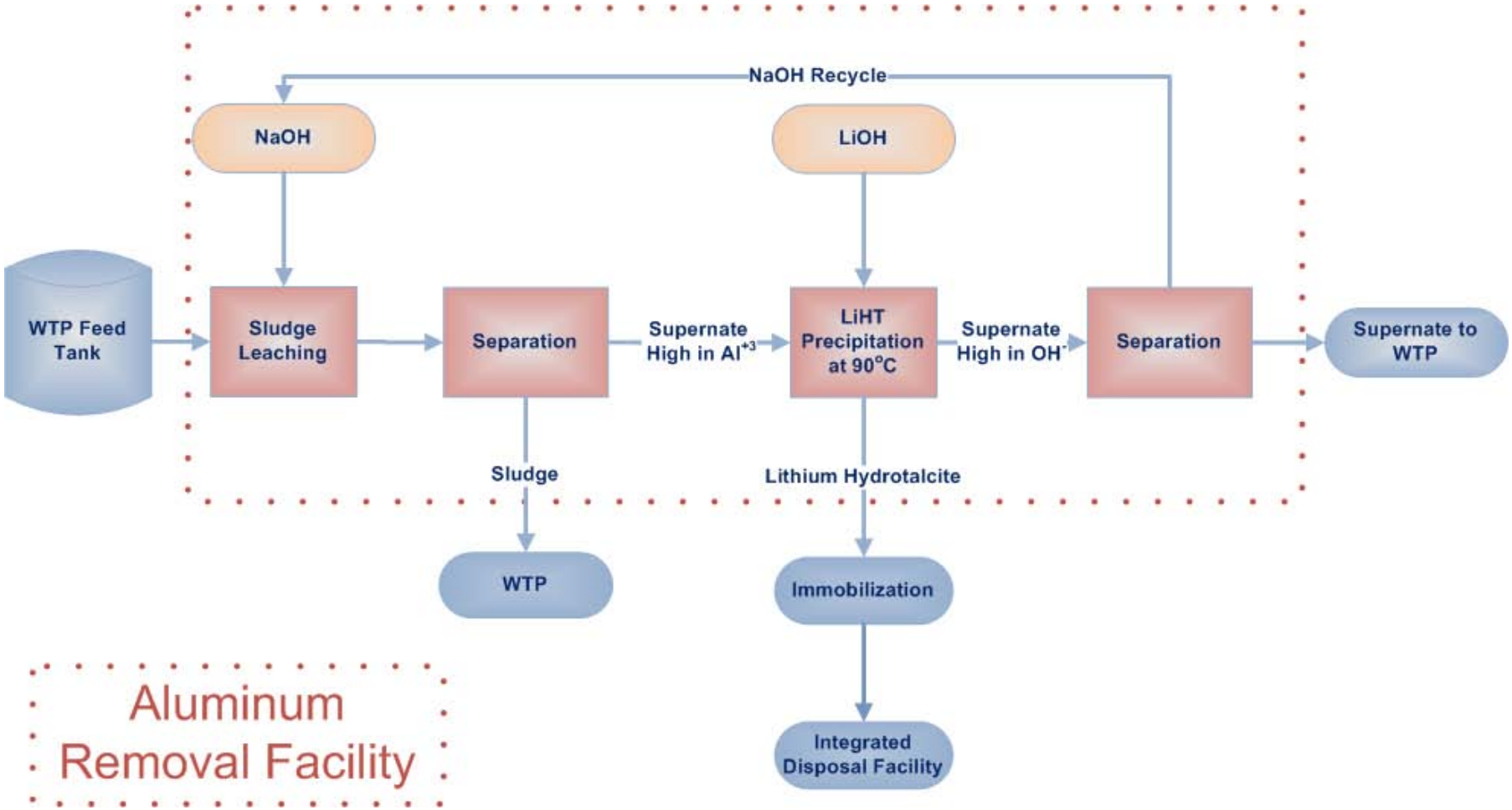


Jim Duncan, PhD
Heinz Huber, PhD

Scouting Experiments for Alumina Reduction in WTP Feed

- The River Project System Plan, Rev 4 (ORP-11242) Establishes a Baseline Case for:
  - Waste Feed Delivery,
  - Single shell tank (SST) Retrieval,
  - **The Alumina Removal Facility (ARF)**,
  - Aluminum solubility in the WTP pretreatment facility,
  - Total sodium requiring vitrification,
  - Supplemental low activity waste (LAW) treatment capacity,
  - Mission duration, and
  - Waste Treatment Plant (WTP) pretreatment throughput.

Scouting Experiments for Alumina Reduction in WTP Feed

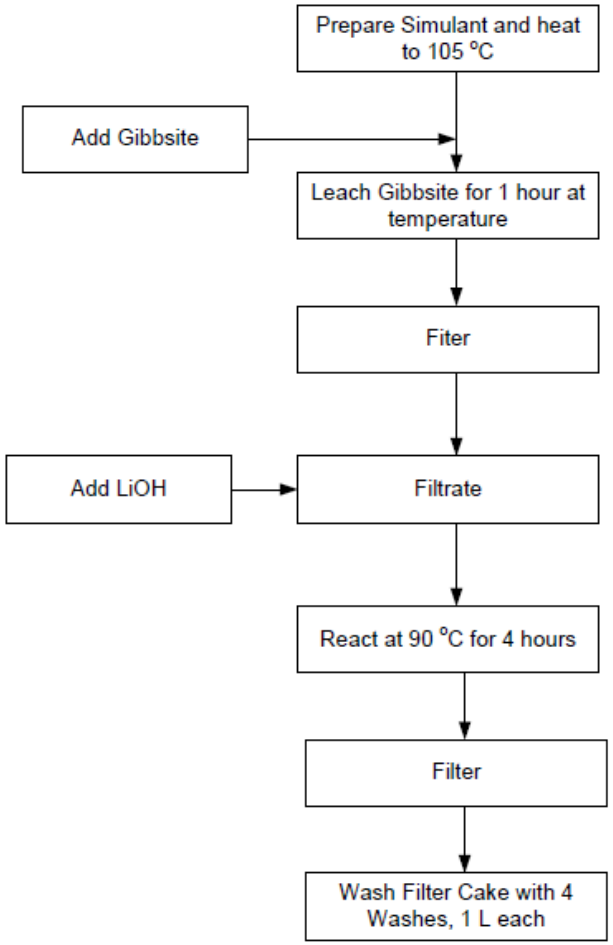


Scouting Experiments for Alumina Reduction in WTP Feed

Simulant

Reagent	DST [mol/L]
$NaOH$	2.59
Na_2CO_3	0.06
$Na_2C_2O_4$	0.003
KNO_3	0.19
$NaNO_3$	0.91
$NaNO_2$	1.15
Na_2SO_4	0.007
$Na_3PO_4 \cdot 12H_2O \cdot 0.25NaOH$	0.007
$NaCl$	0.12
$NaC_2H_3O_2 \cdot 3H_2O$	0.011
NaF	0.001
$Na_2Cr_2O_7 \cdot 2H_2O$	0.04
$CsNO_3$	1.29E-05
Gibbsite	0.4

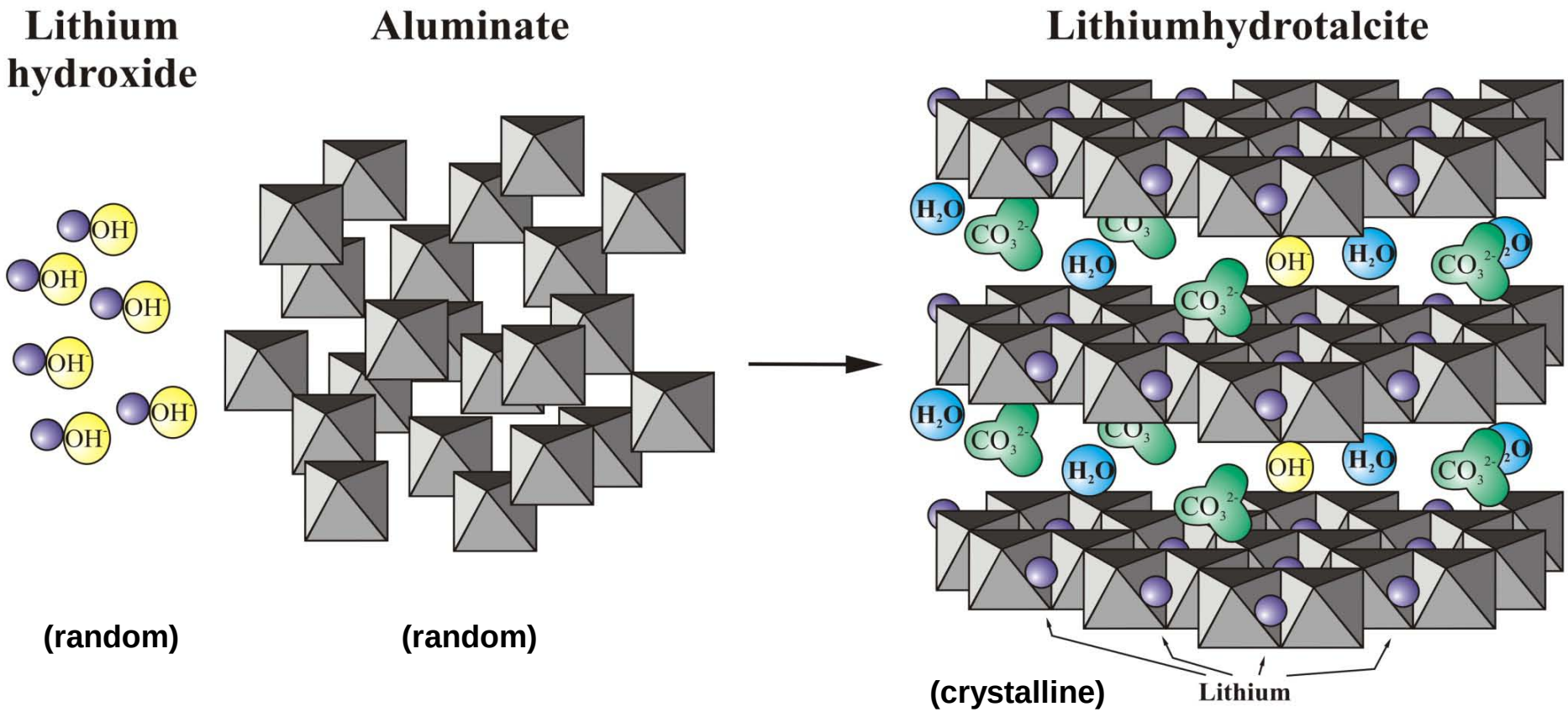
Flow Sheet for the Preparation of Lithium DST Hydrotalcite



Overall Reaction:

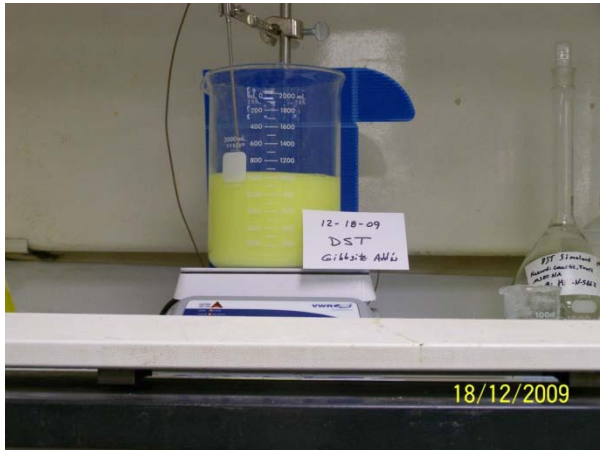
$$2LiOH + 4NaAl(OH)_4 + Na_2CO_3 + nH_2O \rightarrow$$

$$Li_2CO_3 \bullet 4Al(OH)_3 \bullet nH_2O_{(s)} + 6NaOH$$

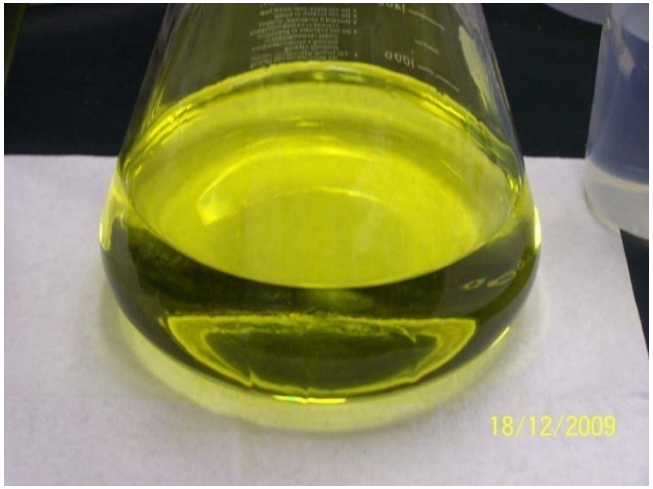


Scouting Experiments for Alumina Reduction in WTP Feed

DST Simulant with Gibbsite added



DST non filterable material after Gibbsite leaching

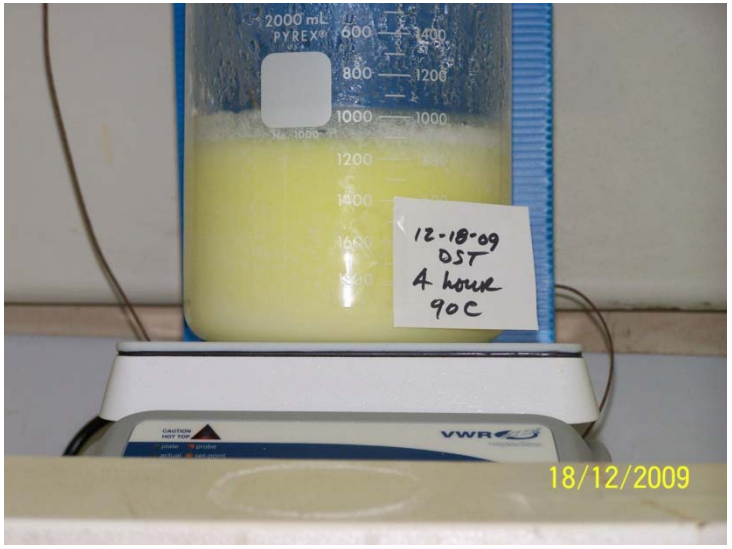


DST Filtrate after Gibbsite leach

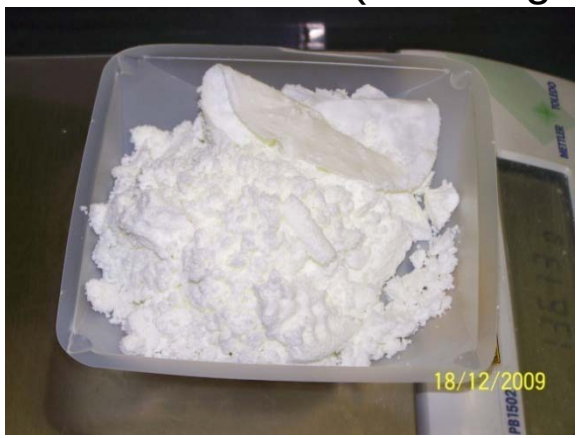
LiOH Addition, DST Simulant

Scouting Experiments for Alumina Reduction in WTP Feed

DST Four hours after
LiOH addition, @ 90 C



Hydrotalcite in Buchner Funnel
Yield (wet weight) = 130.56 g

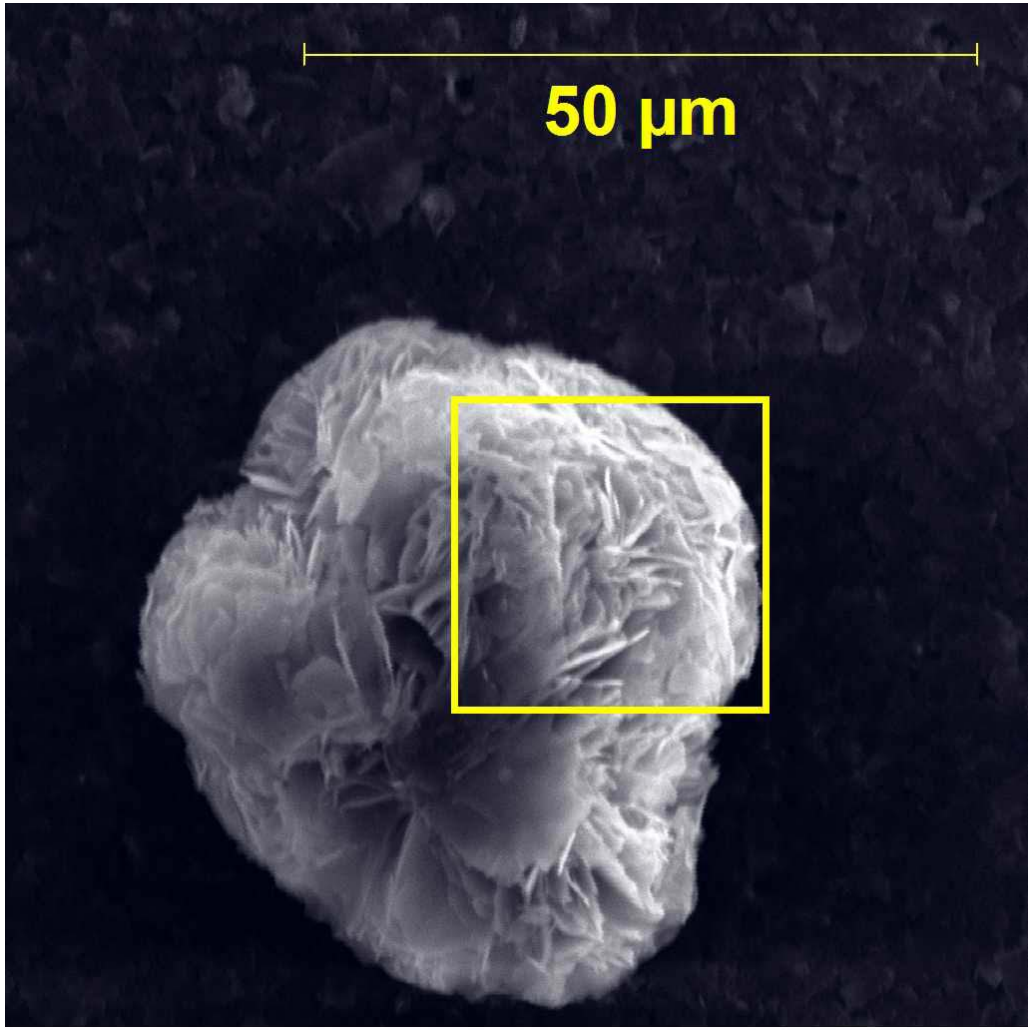


Scouting Experiments for Alumina Reduction in WTP Feed

Filtrates from Initial Filtering through Four Water Washes

Scouting Experiments for Alumina Reduction in WTP Feed

DST Reacted Simulant, Hydrotalcite Particle



Analytical Results

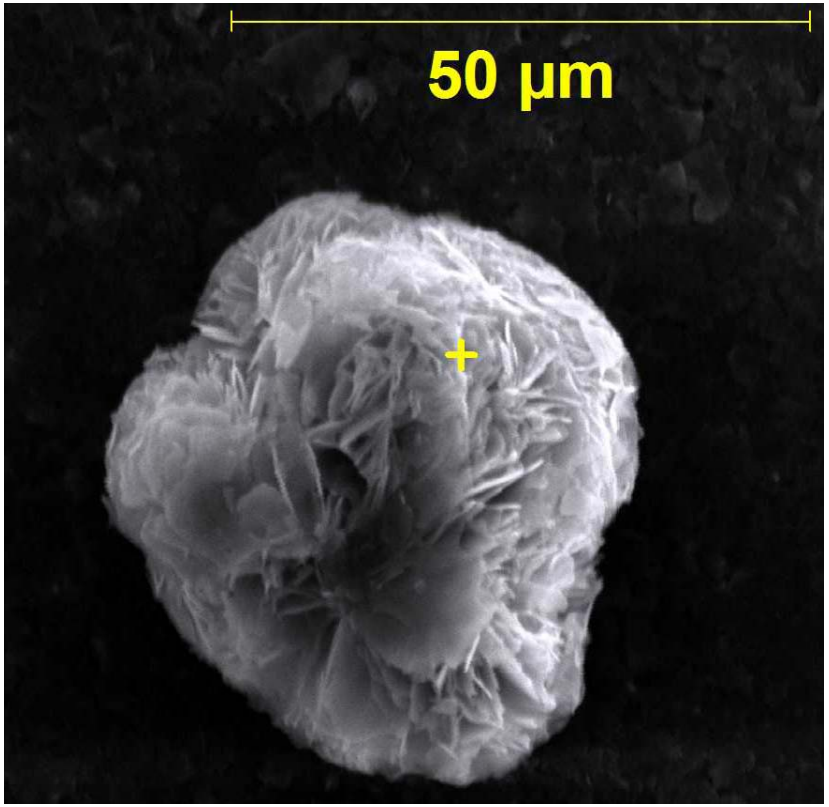
SEM-EDS, XRD,
TGA, Chemical Analysis

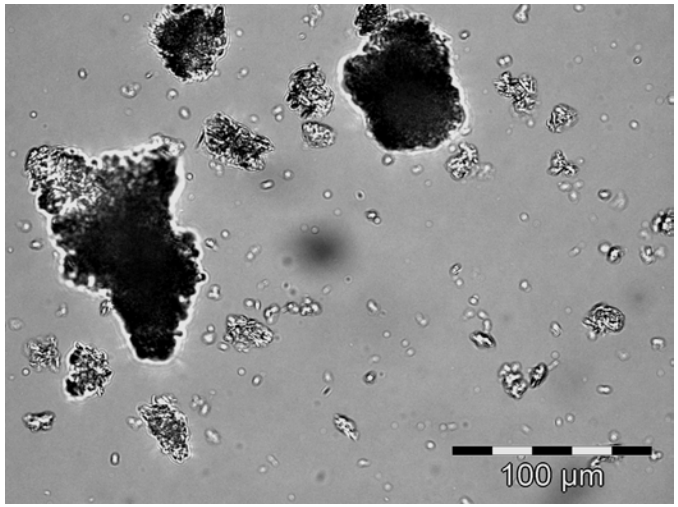
Scouting Experiments for Alumina Reduction in WTP Feed

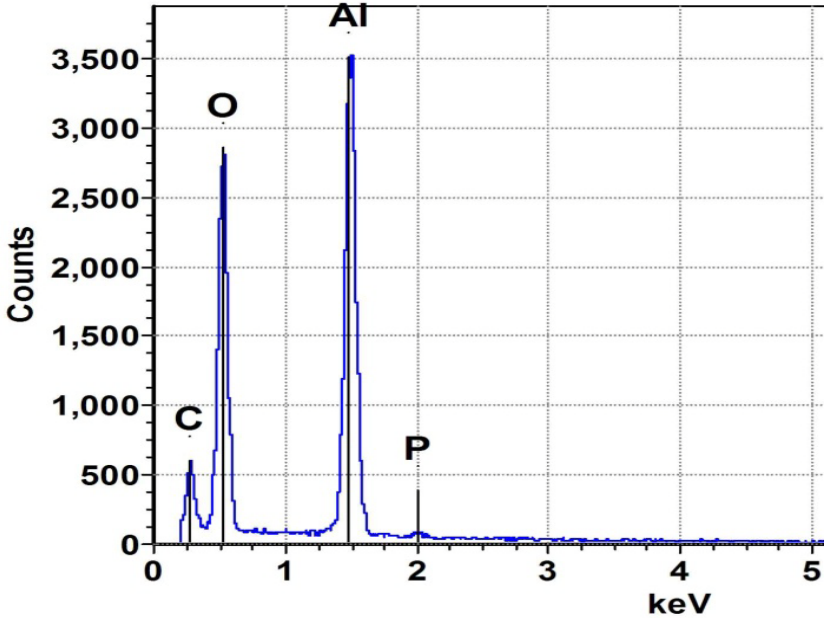
DST Product

Hydrotalcite

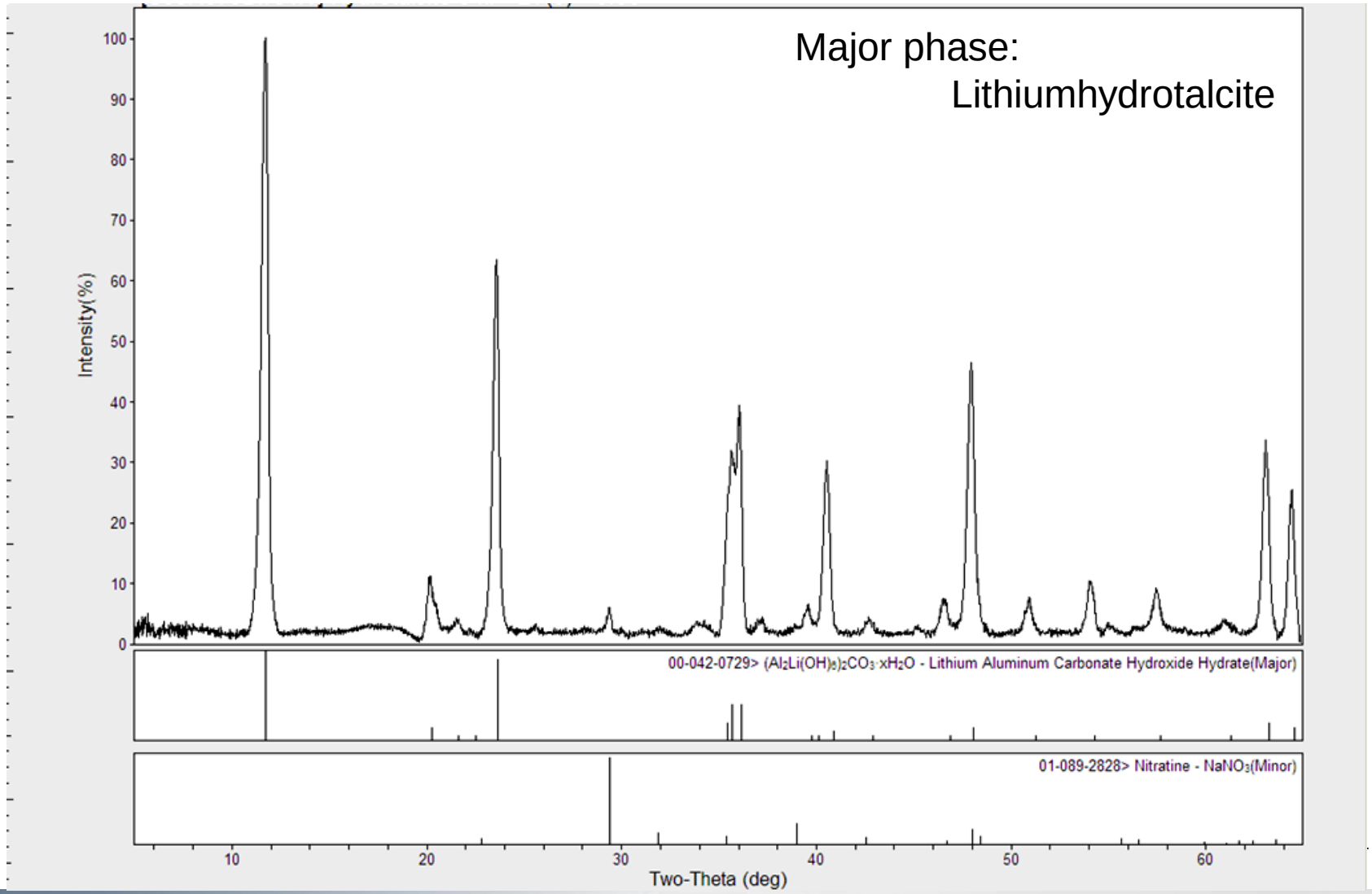
BSE image (bottom), PLM image (right),
and EDS spectrum (bottom, right) of LiHT particle





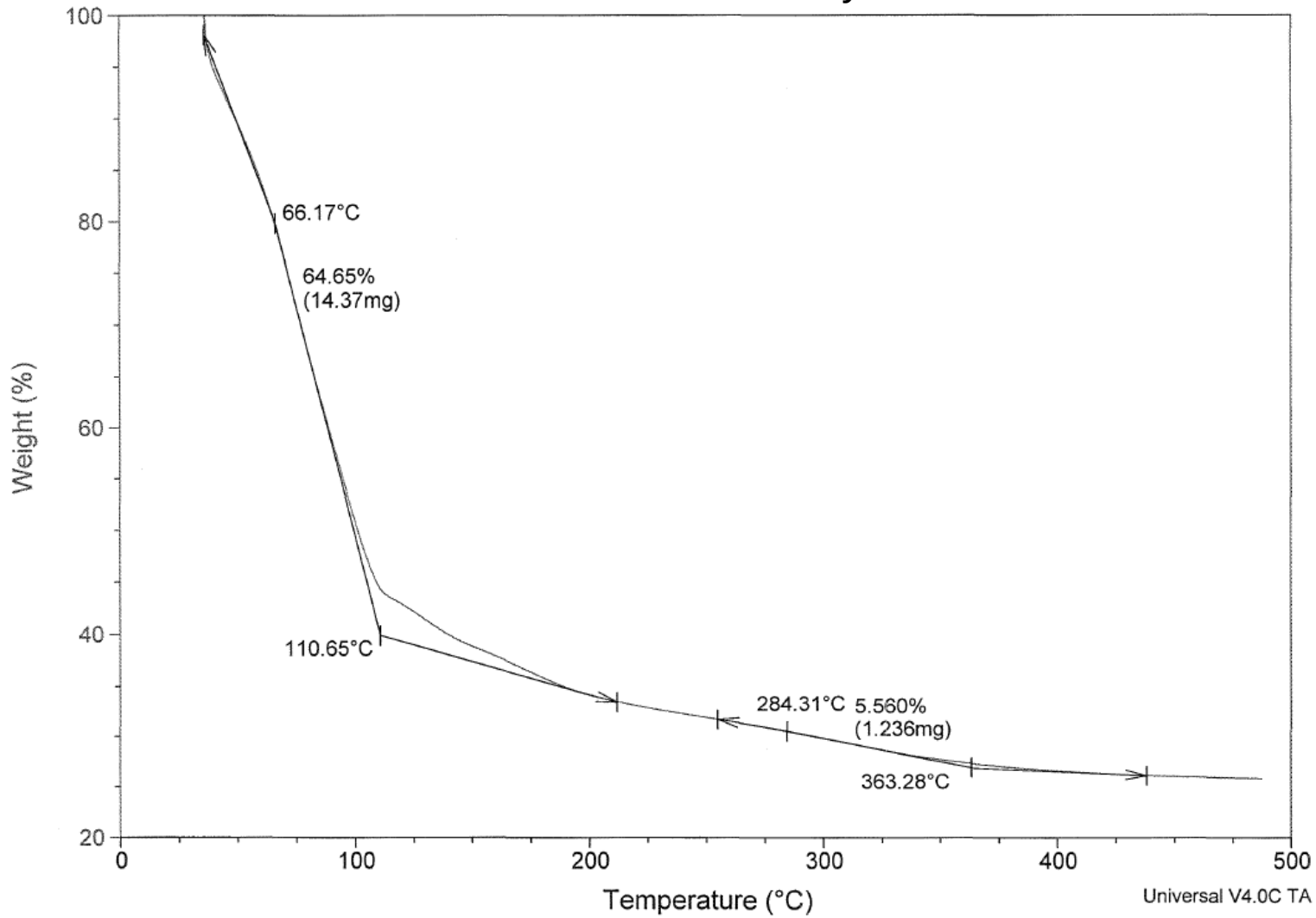


DST Product – XRD spectrum



Scouting Experiments for Alumina Reduction in WTP Feed

TGA for DST Lithium Hydrotalcite



Scouting Experiments for Alumina Reduction in WTP Feed

Amount precipitated in [wt.%]

Analyte	DST Simulant
Aluminum	65
Lithium	65
Hydroxide	< detection limit
Nitrate	0.4
Phosphate	87
Carbonate (TIC)	63

Calculated as the amount of each analyte in the filter cake divided by the sum of filtrate plus all water washes.

Molar ratios of Cations and Anions to Aluminum in Precipitate DST Simulant Filter Cake Analysis

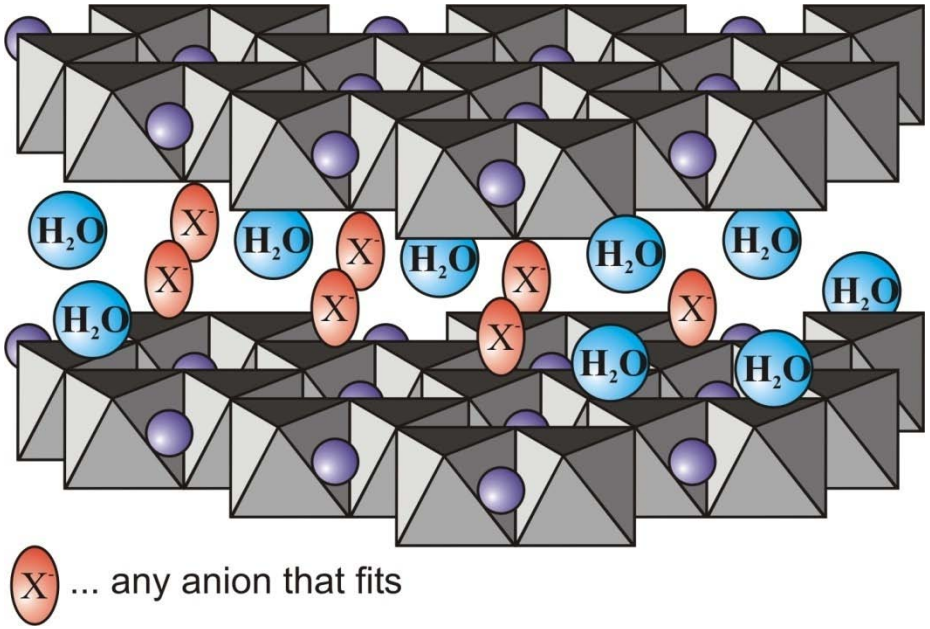
Molar ratios	Li/Al	Carbonate/Al	Nitrate/Al	Phosphate/Al
Filter cake	0.53	0.03	0.02	0.00
If it is Hydrotalcite	0.50	0.25*	0.50*	0.17*

* numbers are based on the molar ratios for the pure individual species of Li-hydrotalcite.

Scouting Experiments for Alumina Reduction in WTP Feed

Future Endeavors (not to be construed as an all-inclusive list):

- Optimize reaction and crystal ripening time.
- Optimize the heating and cooling cycles of the filtrate.
- Select reactors with appropriate materials of construction.
- Monitoring of anions is necessary to identify limiting factors for precipitation.
- In-situ instrumentation will be needed for process monitoring, product formation, particle size, and other process parameters that will be evident for optimization as the effort goes forward.
- Investigate filter cake washing, up-flow bed expansion versus down-flow.

Scouting Experiments for Alumina Reduction in WTP Feed

Thank you for your attention!