

# Large scale manufacturing: Success and Lessons Learned

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Large scale manufacturing: Success and Lessons Learned  
**Precipitation processes scale-up challenges**

Reaction kinetics

Fluid dynamics and agitation issues

Scale-up of crystallization

Thermodynamics

COGs

Equipment selection

Process interconnection point

Technology transfer

CTQs

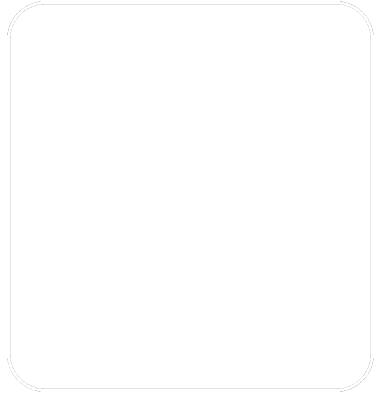
Toller evaluation

**Products**

## Eco-friendly routing

| Pathway                 | Reaction   | Atom utilization(%) |
|-------------------------|--|---------------------|
| Metal Nitrate<br>[18]   | $4 \text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} + 2 \text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} + 2 \text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O} + \text{Na}_2\text{CO}_3 + 16 \text{NaOH}$ $\rightarrow \text{Zn}_4\text{Mg}_2\text{Al}_2(\text{OH})_{16}(\text{CO}_3) \cdot 4\text{H}_2\text{O} + 18 \text{NaNO}_3 + 50 \text{H}_2\text{O}$ | 18.92               |
| Metal Oxide             | $4 \text{ZnO} + 2 \text{MgO} + \text{Al}_2\text{O}_3 + 18 \text{HNO}_3 + \text{Na}_2\text{CO}_3 + 16 \text{NaOH}$ $\rightarrow \text{Zn}_4\text{Mg}_2\text{Al}_2(\text{OH})_{16}(\text{CO}_3) \cdot 4\text{H}_2\text{O} + 18 \text{NaNO}_3 + 5 \text{H}_2\text{O}$   | 34.78               |
| Metal Chloride          | $4 \text{ZnCl}_2 \cdot 4\text{H}_2\text{O} + 2 \text{MgCl}_2 \cdot 6\text{H}_2\text{O} + 2 \text{AlCl}_3 \cdot 6\text{H}_2\text{O} + \text{Na}_2\text{CO}_3 + 16 \text{NaOH}$ $\rightarrow \text{Zn}_4\text{Mg}_2\text{Al}_2(\text{OH})_{16}(\text{CO}_3) \cdot 4\text{H}_2\text{O} + 18 \text{NaCl} + 36 \text{H}_2\text{O}$                                    | 28                  |
| Metal Sulfate           | $4 \text{ZnSO}_4 \cdot 7\text{H}_2\text{O} + 2 \text{MgSO}_4 \cdot 7\text{H}_2\text{O} + \text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O} + \text{Na}_2\text{CO}_3 + 16 \text{NaOH}$ $\rightarrow \text{Zn}_4\text{Mg}_2\text{Al}_2(\text{OH})_{16}(\text{CO}_3) \cdot 4\text{H}_2\text{O} + 9 \text{Na}_2\text{SO}_4 + 39 \text{H}_2\text{O}$              | 19.51               |
| Hydro-thermal synthesis | $4 \text{ZnO} + 2 \text{MgO} + \text{Al}_2\text{O}_3 + 2 \text{NaHCO}_3 + 11 \text{H}_2\text{O}$ $\rightarrow \text{Zn}_4\text{Mg}_2\text{Al}_2(\text{OH})_{16}(\text{CO}_3) \cdot 4\text{H}_2\text{O} + \text{Na}_2\text{CO}_3$   | 90.32               |

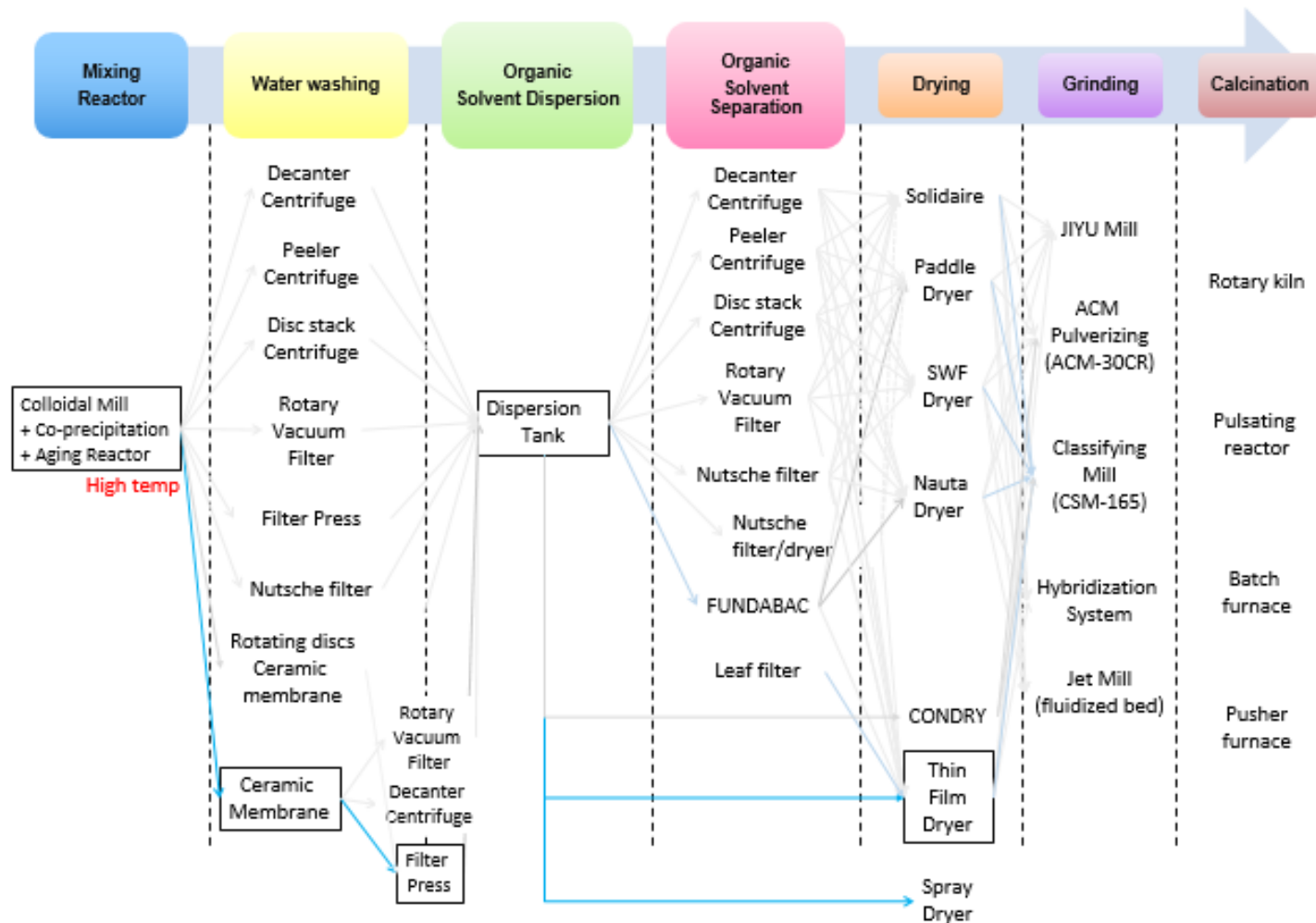
# Crystal scale-up framework



**Conceptual  
Process  
Design**

# Large scale manufacturing: Success and Lessons Learned

## Process synthesis of crystal scale-up



Risk assessment – process flow



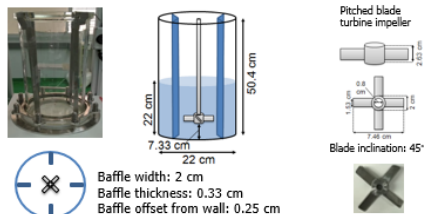
# Crystal Reactor Modeling

- Crystal suspension in a mixing reactor required uniform solid distributions
  - ✓ Kinematic similarity is defined for mixing scale-up
- The CFD model to
  - ✓ Represent a uniform solid distributions of well mixing performance in lab-scale reactor
  - ✓ Utilize the same model to analyze and ensure well mixing of commercial-scale reactor
- Establish the CFD model configurations

|                           |  |  |
|---------------------------|--|--|
| <b>Multi-phase model:</b> | Eulerian granular model                      | → Recommended for modeling solid-liquid system                         |
| <b>Turbulence model:</b>  | Standard <i>k-epsilon</i> mixture            | → Provide sufficiently consistent results<br>→ Save computational time |
| <b>Inter-phase force:</b> | • Drag force<br>• Turbulent dispersion force | → Needed in modeling solid-liquid system                               |
| <b>Drag force model:</b>  | Gidaspow (solid – liquid)                    | → Give consistent results with experiment                              |

- Validate CFD model by comparing with experiment (9L flatted BTM cylindrical vessel with 4 baffles)

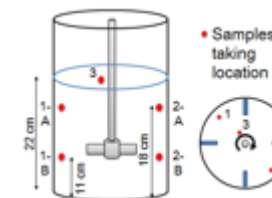
❑ 9L flatted BTM cylindrical vessel with 4 baffles and turbine impeller



❑ Experimental conditions

|                                       |             |
|---------------------------------------|-------------|
| <b>Rotational speed:</b>              | 990 RPM     |
| <b>Solid concentration in slurry:</b> | 14.2 vol. % |
| <b>Suspension time:</b>               | 30 minute   |

❑ Samples taking at different locations to measure solid conc.

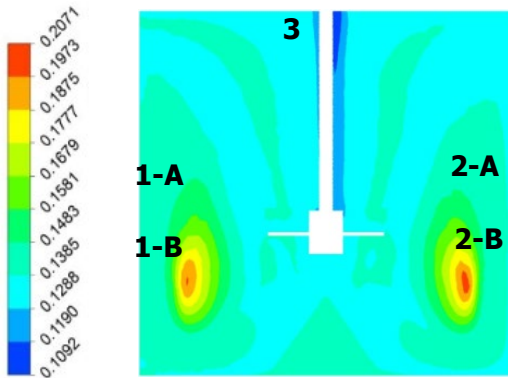
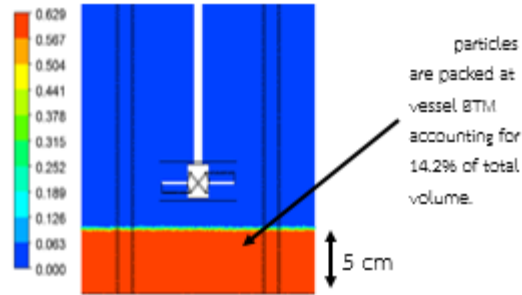


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## Crystal Reactor Modeling

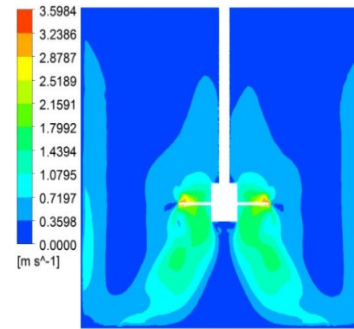
### ➤ CFD simulation VS experimental result

#### ☐ Initial time

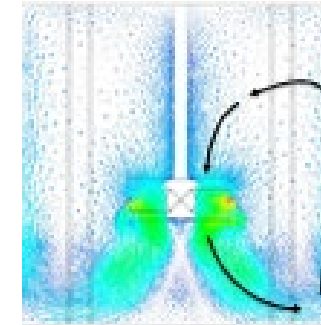


Solid volume fraction at steady stage

#### ☐ Simulation progress until velocity inside reactor constant, i.e. steady stage



Solid Velocity Distribution

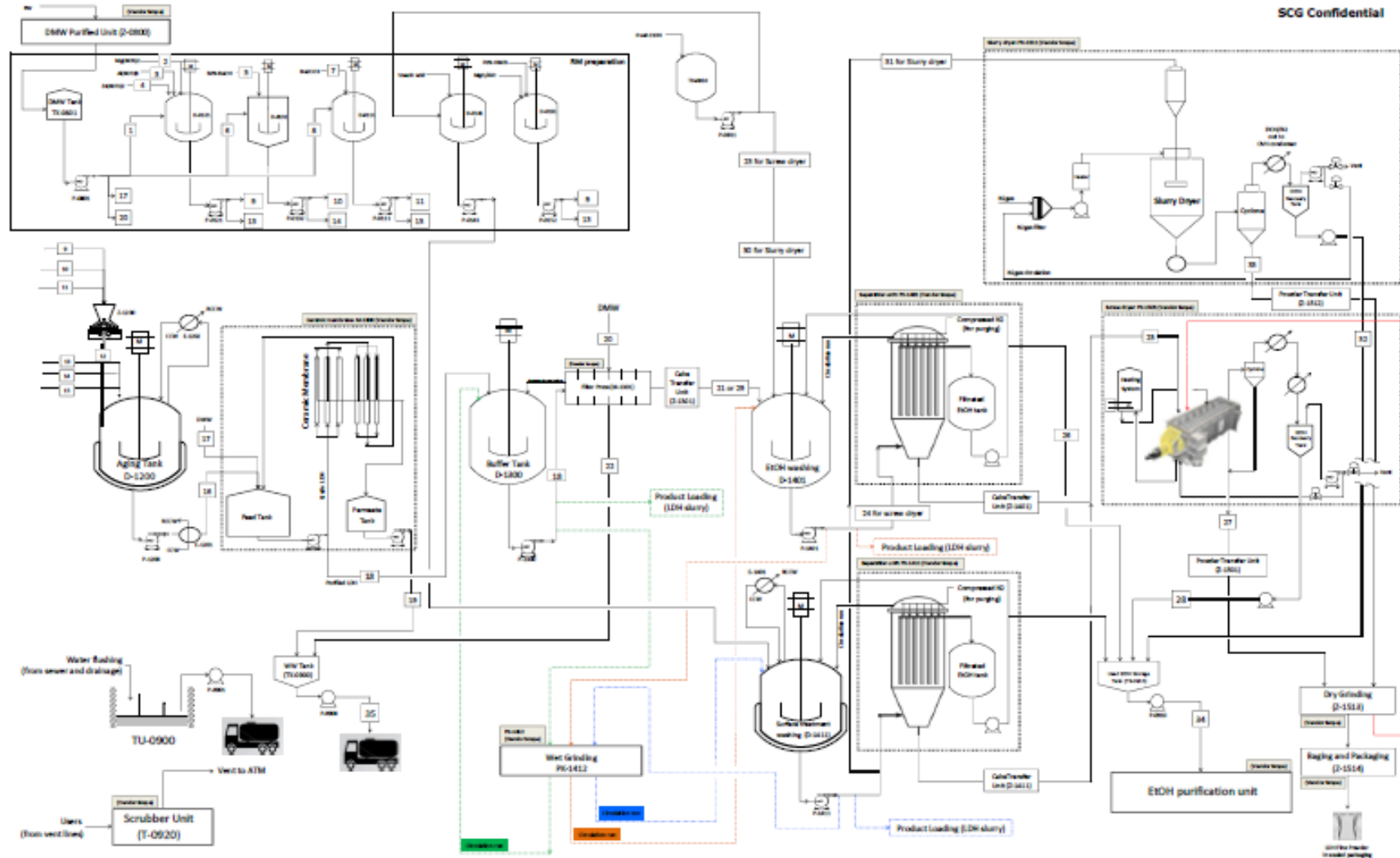


Solid Flow Pattern

|                     | 1-A           | 1-B           | 2-A           | 2-B           | 3             |
|---------------------|---------------|---------------|---------------|---------------|---------------|
| Experimental Result | 13.919%       | 13.983%       | 13.825%       | 13.675%       | 13.814%       |
| Simulation Result   | 13.848%       | 13.922%       | 13.771%       | 13.851%       | 13.565%       |
| Percent Difference  | <b>0.510%</b> | <b>0.436%</b> | <b>0.391%</b> | <b>1.287%</b> | <b>1.803%</b> |

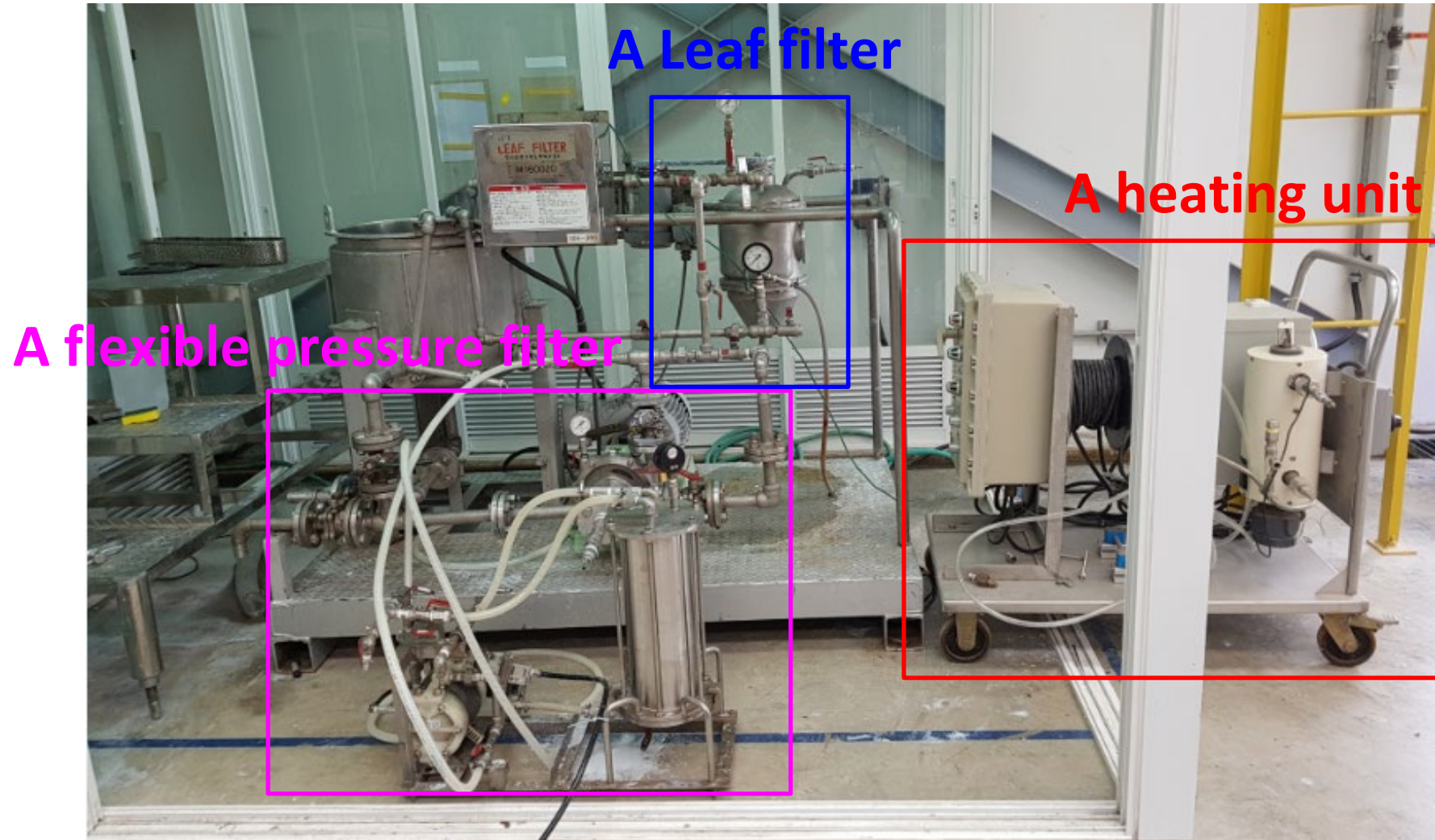
- CFD model configuration is able to represent crystal particles suspension
  - ✓ Prediction of solid volume fraction is very similar to the experiments, less than 2% difference
  - ✓ Confidence of using CFD model for commercial reactor design

# PFD with less concern with process interconnection point





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Process interconnection point



# Simulation of Crystal filtration and Process interconnection

## Flexible pressure filter pilot tests



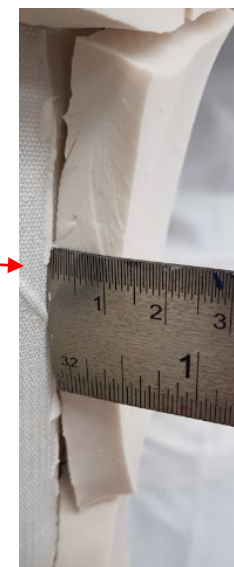
After filtration step



Compressed N2 blow back



Inside the vessel





## Simulation of crystal filtration and Process interconnection

### Leaf filter pilot tests



**After discharge -> sticky**  
(Solid content = 25wt%)



**Heat is applied:**  
jacket 90C, hot N2 90C  
(1 hr during separation and  
bone dry cake)



**Before discharge**  
(Solid content = 35wt%)

- Measure specific filtration resistance
- Check the crystal compressibility
- Model the filtration dynamics and equipment selection through crystal properties and process technologies
- Know amount of filtrate, filtration area, and total cycle time to calculate capacity

## Large scale manufacturing: Success and Lessons Learned

### Simulation of crystal drying



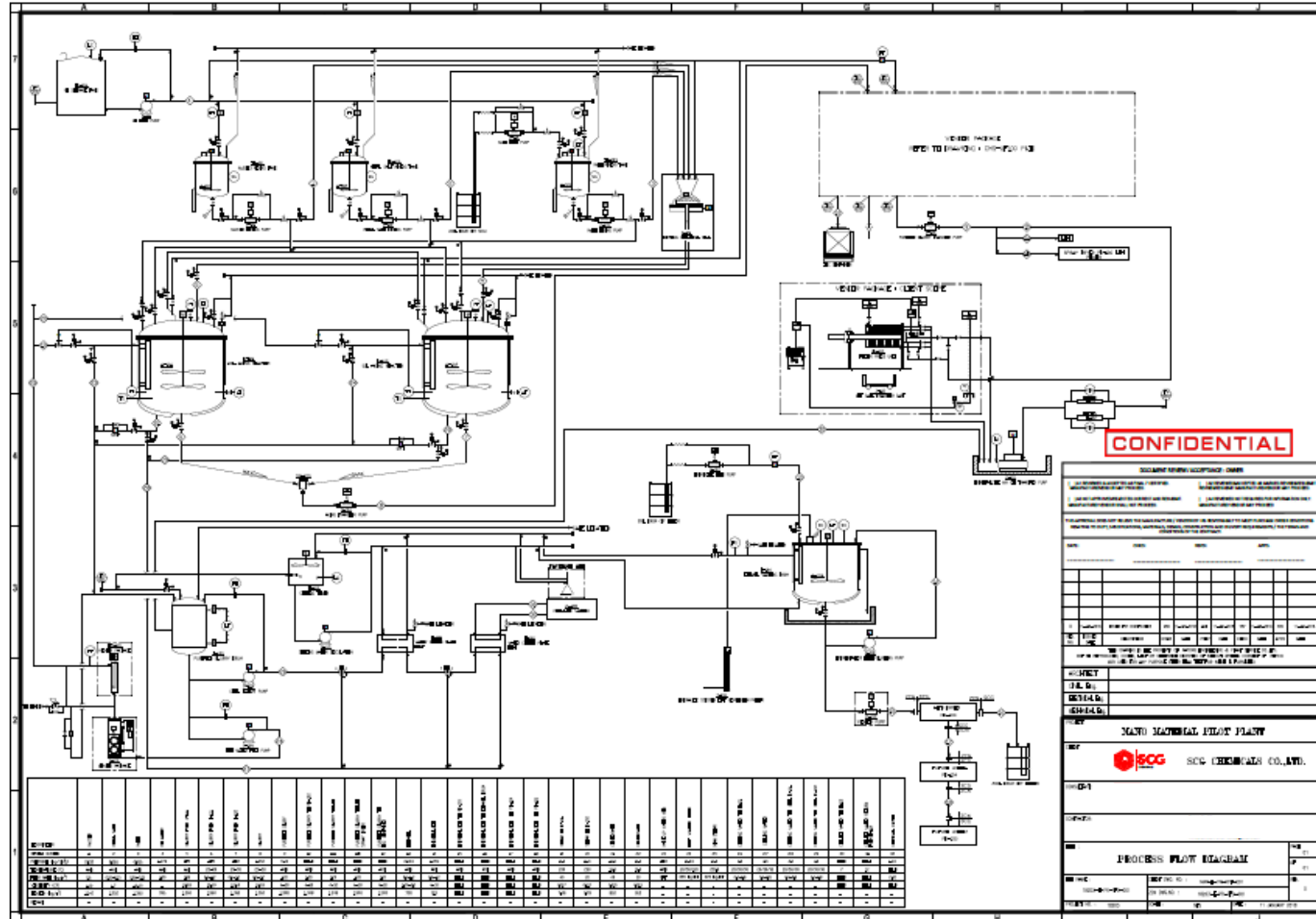
- Leave filtrated cake onto surface of filter media (mimic the condition)
- After drying for 1 hr

Scale-up

- Find the suitable equipment
- Set the optimum drying condition
- Predict the actual drying time



- Predict and model optimal conditions for drying via mock-up test with equipment vendors
- As shown, stable upscaling is achieved through continuous rotation to prevent conglomerate crystallization and by using shear force of agitator at the same time of drying





Large scale manufacturing: Success and Lessons Learned  
Precipitation pilot plant



### Connecting Application-A Needs

| Properties              | Parameters  |
|-------------------------|---|
| Chemical composition    | Mg <sub>x</sub> Zn <sub>(3-x)</sub> Al-CO <sub>3</sub> with/without Zn stearate, or other stearate types.<br>Mg <sub>x</sub> Zn <sub>(3-x)</sub> Al-CO <sub>3</sub> can potentially be:<br>- Mg <sub>3</sub> Al-CO <sub>3</sub> ,<br>- Mg <sub>2</sub> Zn <sub>1</sub> Al-CO <sub>3</sub> ,<br>- Mg <sub>1</sub> Zn <sub>2</sub> Al-CO <sub>3</sub> ,<br>- Mg <sub>1.5</sub> Zn <sub>1.5</sub> Al-CO <sub>3</sub> ,<br>- Mg <sub>2.5</sub> Zn <sub>0.5</sub> Al-CO <sub>3</sub> |
| Primary particle size   | Platelet shape : 60 - 150 nm, preferable ~ 100nm (analyzed by particle size analyzer on wet samples)  |
| Secondary particle size | Platelet shape : 200 nm - 5 μm, preferable ~ 2 μm (D50: Medium value of the particle size distribution)   |
| Density of LDHs         | 1,150 - 1,400 kg/m <sup>3</sup>   |
| Shear Viscosity         | 6.8-E02 - 1.0+E01 Pa.s  |
| Surface area            | 50 - 100 m <sup>2</sup> /g, preferable ~ 90 m <sup>2</sup> /g   |
| Moisture content        | ≤ 2% (dry powder)   |
| Bulk density            | 150 - 400 kg/m <sup>3</sup>   |

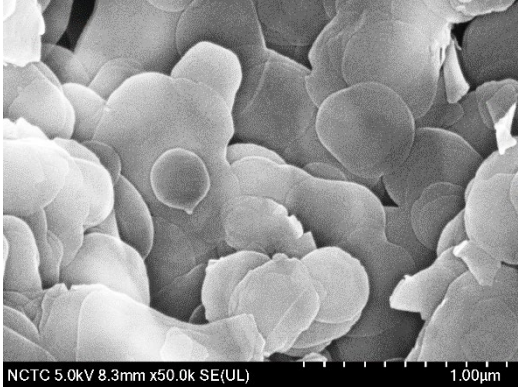
### Connecting Application-B Needs

| Properties   | Parameters   |
|--|--|
| Chemical composition                                 | Mg <sub>2</sub> Al-CO <sub>3</sub>   |
| Primary particle size                                | 500nm - 3 μm with platelet shape (analyzed by particle size analyzer on wet samples) |
| Aspect ratio (via Atomic Force Microscope)           | 20 - 200   |
| LDHs Cake in EtOH or Ethyl acetate LDHs (suspension) | 20 - 30% solid content   |
| LDHs Slurry in EtOH or Ethyl acetate                 | 5 - 10% solid content  |
| Water content  | ≤ 4% in LDHs Slurry  |

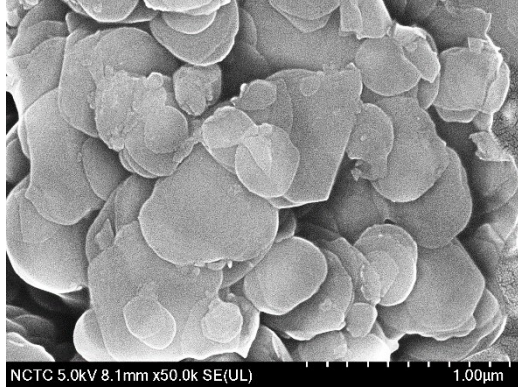


# Key Results: product properties similarity and product characteristics similarity

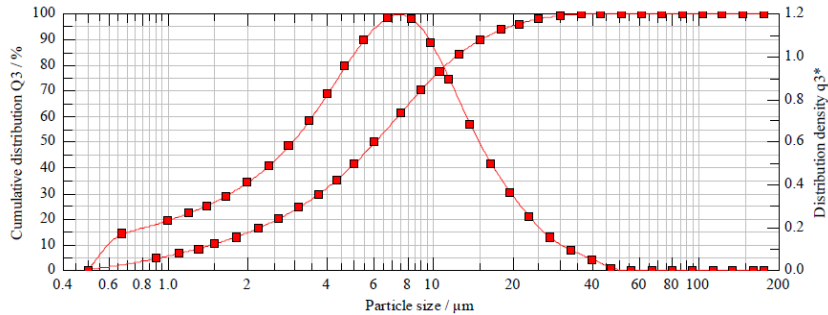
PD-1331 (Lab)



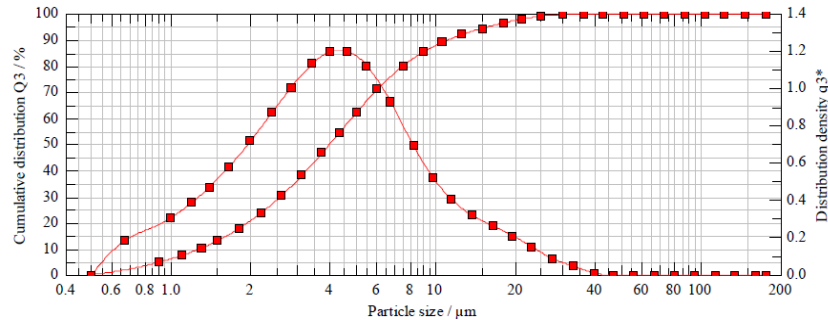
PD-1331 (Pilot Plant)



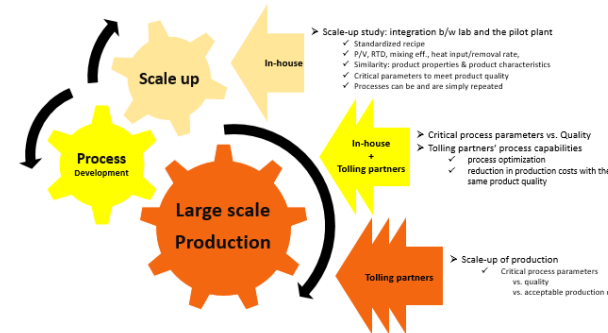
PD-1331 (Lab)



PD-1331 (Pilot Plant)



| Physical Properties               | PD-1331 (Lab) | PD-1331 (Pilot Plant) |              |
|-----------------------------------|---------------|-----------------------|--------------|
| BET (m <sup>2</sup> /g)           | 25.08 ± 0.61  | 27.94 ± 2.53          |              |
| Bulk Density (g/cm <sup>3</sup> ) | 0.18 ± 0.06   | 0.13 ± 0.01           |              |
| Tap Density (g/cm <sup>3</sup> )  | 0.28 ± 0.10   | 0.17 ± 0.02           |              |
| Moisture Content at 110°C (%)     | 0.78 ± 0.35   | 1.10 ± 0.24           |              |
| PSD (Dried Powder Form) µm        | D10           | 1.67 ± 0.54           | 1.27 ± 0.15  |
|                                   | D50           | 7.22 ± 3.25           | 4.12 ± 0.36  |
|                                   | D84           | 15.76 ± 7.11          | 9.19 ± 0.94  |
|                                   | D90           | 19.53 ± 8.31          | 12.10 ± 1.46 |
| Composition Mg : Al               | 5.14 ± 0.03   | 5.09 ± 0.14           |              |



➤ Scale-up study: integration b/w lab and the pilot plant

- ✓ Standardized recipe
- ✓ P/V, RTD, mixing eff., heat input/removal rate,
- ✓ Similarity: product properties & product characteristics
- ✓ Critical parameters to meet product quality
- ✓ Processes can be and are simply repeated

- Applying product properties similarity and product characteristics similarity to scale-up and produce LDH-BR prototypes via pilot plant seem to hit the target which physical & chemical properties are quite close to one producing in the lab scale.

# Large scale manufacturing: Success and Lessons Learned

## Technology transfer - Raw Materials Specifications (Ex.)

### i. Plant water

| Parameters                                    | Unit  | Value           |
|---|-------|-----------------|
| Appearance                                    | -     | Slightly Turbid |
| pH at 25°C                                    |       | 7-8             |
| Turbidity                                     | NTU   | 1.0             |
| Conductivity at 25°C                          | us/cm | 393             |
| Calcium hardness as CaCO <sub>3</sub>         | mg/L  | 49.5            |
| Chloride as Cl <sup>-</sup>                   | mg/L  | 39.4            |
| Methyl orange alkalinity as CaCO <sub>3</sub> | mg/L  | 72.8            |
| Phosphate as PO <sub>4</sub> <sup>-3</sup>    | mg/L  | 1.3             |
| Iron as Fe                                    | mg/L  | 0.06            |
| Sulfate as SO <sub>4</sub> <sup>-2</sup>      | mg/L  | 38.6            |
| Total dissolved solids dried at 180°C         | mg/L  | 256             |
| Total hardness as CaCO <sub>3</sub>           | mg/L  | 76.8            |

### ii. Reverse Osmosis water

| Parameters                                      | Unit  | Value  |
|---|-------|--------|
| Copper  | mg/L  | 0.0001 |
| Iron  | mg/L  | 0.001  |
| Manganese                                       | mg/L  | 0.0001 |
| Sodium  | mg/L  | 0.1    |
| Calcium hardness as CaCO <sub>3</sub>           | mg/L  | 1      |
| Chloride as Cl <sup>-</sup>                     | mg/L  | 0.2    |
| Conductivity at 25°C                            | us/cm | 0.5    |
| Dissolved oxygen                                | mg/L  | 0.1    |
| Magnesium hardness as CaCO <sub>3</sub>         | mg/L  | 1      |
| Methyl orange alkalinity as CaCO <sub>3</sub>   | mg/L  | 1      |
| pH at 25°C                                      |       | 6-7    |
| Phenolphthalein Alkalinity as CaCO <sub>3</sub> | mg/L  | 1      |
| Phosphate as PO <sub>4</sub> <sup>-3</sup>      | mg/L  | 0.5    |
| Silica as SiO <sub>2</sub>                      | mg/L  | 0.5    |
| Sulfate as SO <sub>4</sub> <sup>-2</sup>        | mg/L  | 0.5    |
| Total dissolved solids dried at 180°C           | mg/L  | 5      |
| Total hardness as CaCO <sub>3</sub>             | mg/L  | 1      |
| Total suspended solid dried at 103-105°C        | mg/L  | 5      |
| Turbidity                                       | NTU   | 0.1    |

### iii. Metal precursors and basic solution

| Specifications                         | Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O | Mg(NO <sub>3</sub> ) <sub>3</sub> ·6H <sub>2</sub> O | Al(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O | NaOH        | Na <sub>2</sub> CO <sub>3</sub> | Sodium Stearate  |
|--|--|--|--|-------------|---------------------------------|------------------|
| Solid/liquid                           | solid (pellet)                                       | solid (pellet)                                       | solid (pellet)                                       | 50%w liquid | solid (pellet)                  | solid (pellet)   |
| Packaging                              |  | 25 kgs PP PE Bag                                     | 25 kgs PP PE Bag                                     |             |                                 | 20 kgs PP PE Bag |
| Assay (min)                            | 96-98%   | 98%  | 98%  | 99%         | 99%                             |                  |
| pH (10% solution)                      | 3.6  |  |  |             |                                 |                  |
| pH (25% solution)                      |  | 3-5  |  |             |                                 |                  |
| Bulk Density (g/L)                     |  |  |  |             |                                 | 200-260          |
| Moisture (%)                           |  |  |  |             |                                 | < 2              |
| Melting point (°C)                     |  |  |  |             |                                 | 210 -220         |
| Sulphate ash (%)                       |  |  |  |             |                                 | 22 - 24.5        |
| Water insoluble (%)                    |  | ≤ 0.01%  |  |             | < 0.002%                        |                  |
| Sieve residue 100 mesh (%)             |  |  |  |             |                                 | < 0.5            |
| Iodine value (g I <sub>2</sub> /100 g) |  |  |  |             |                                 | 1                |
| Free fatty acid (%)                    |  |  |  |             |                                 | < 2              |
| Loss in ignition (%)                   |  |  |  |             | 0.21%                           |                  |
| Impurities (ppm)                       | Ca   | ≤ 500  | < 200  |             |                                 |                  |
|  | Cd   |  |  |             |                                 |                  |
|  | Cu   |  |  |             |                                 |                  |
|  | Cl   | < 700  | ≤ 100  | < 50        |                                 |                  |
|  | Fe   | < 500  | ≤ 5  | < 50        |                                 | 10               |
|  | Hg   |  |  |             |                                 |                  |
|  | K  |  |  | < 200       |                                 |                  |
|  | Mn   |  | ≤ 50   |             |                                 |                  |
|  | N  |  | ≥ 107,000  |             |                                 |                  |
|  | Na   |  |  | < 1,000     |                                 |                  |
|  | Ni   |  |  |             |                                 |                  |
|  | Pb   | < 10   | ≤ 20   | < 50        |                                 |                  |
|  | Na <sub>2</sub> CO <sub>3</sub>                      |  |  |             | < 5,000                         |                  |
|  | NaCl   |  |  |             | < 500                           |                  |
|  | Fe <sub>2</sub> O <sub>3</sub>                       |  |  |             | < 20                            |                  |
|  | NH <sub>4</sub>                                      |  |  | < 300       |                                 |                  |
|  | MgO  |  | ≥ 154,000  |             |                                 |                  |
|  | PO <sub>4</sub>                                      |  | 1,000  |             |                                 |                  |
|  | SO <sub>4</sub>                                      | < 500  | ≤ 200  | < 200       |                                 | 300              |
|  | SiO <sub>2</sub>                                     |  |  |             |                                 |                  |

### Remarks for raw materials selection criteria

- There are some elements needed to be cautious due to the fact that it will affect product characteristics and qualities (such as color) and chemical properties therefore the raw materials must contain the least amount of the following impurities.
- Aluminum precursor is considered as major contributor to Fe impurity in the final product. High Fe levels in the final product have detrimental effect on product performance.

| Elemental analysis | specification | Unit |
|--------------------|---------------|------|
| Na                 | ≤ 300         | ppm  |
| Fe                 | ≤ 70          | ppm  |
| Pb                 | ≤ 1,500       | ppm  |

## I. Mixed base solution

- 1) Weigh amount of RM-1 (solid – pellet) according to synthesis recipe [RM-1 = 531 kg]  **$\pm 1$  kg** accuracy.
- 2) Fill the mixing tank with RO water until reach volume according to synthesis recipe [7,572 L].
- 3) Start an agitator of the mixing tank gently to maintain continuous mixing.
- 4) Gently fill RM-1 (solid – pellet) to the mixing tank.
- 5) Keep Stirring for at least **1 hour** to ensure complete dissolution of RM-1
- 6) Drain RM-1 solution at the bottom or take it from handhold at the cover of the preparation tank to cross-check all solid dissolved, solution should be clear.
- 7) Check RM-1 solution for pH and density vs. temperature using a pH meter, a density meter and a thermocouple, respectively to make sure they are in accepted range according to table-1<sup>B</sup>
- 8) Gradually feed RM-2 (solution 50 wt.%) according to synthesis recipe [2,826 kg]  **$\pm 1$  kg** accuracy to the mixing tank via feeding nozzle<sup>C</sup> and keep stirring for **0.5 hour**.
- 9) Drain mixed base solution at the bottom or take it from handhold at the cover of the preparation tank to cross-check, solution should be clear.
- 10) Check mixed base solution for pH and density vs. temperature using the pH meter, the density meter and the thermocouple, respectively to make sure they are in accepted range according to table-2<sup>D</sup>.

### Remarks

<sup>A</sup> Undissolved  $\text{Na}_2\text{CO}_3$  may cause solution properties off-spec such as a drop in pH. An increase in mixing time and re-sampling of based solution properties are recommended.

<sup>B</sup> Table-1: control value for  $\text{Na}_2\text{CO}_3$  solution

| $\text{Na}_2\text{CO}_3$ solution temperature (°C) | pH | Density (kg/m <sup>3</sup> ) |
|--|----|------------------------------|
| 20   |    |                              |
| 25 (preferred)                                     |    |                              |
| 30   |    |                              |

<sup>C</sup> During mixing, exothermic reaction where heat is liberated.

<sup>D</sup> Table-2: control value for mixed base solution

| Mixed base solution temperature (°C) | pH | Density (kg/m <sup>3</sup> ) |
|--------------------------------------|----|------------------------------|
| 20                                   |    |                              |
| 25 (preferred)                       |    |                              |
| 30                                   |    |                              |



## II. Mixed metal solution

- 1) Weigh amount of each metal salt (solid – pellet) composition according to synthesis recipe [RM-3 = 1,284 kg, RM-4 = 1,879 kg, RM-5 = 2,979 kg] **+1 kg** accuracy each.
- 2) Fill the mixing vessel with RO water until reach volume according to synthesis recipe [6,713 L].
- 3) Start an agitator of the mixing tank gently to maintain continuous mixing.
- 4) Gently pour metals salts to the mixing tank.
- 5) Keep stirring for at least **1 hour** to ensure complete dissolution of metal salts<sup>E</sup>.
- 6) Drain mixed metal solution at the bottom or take it from handhold at the cover of the mixing tank to verify complete dissolution of the solids, solution should be clear.
- 7) Check mixed metal solution for pH and density vs. temperature using the pH meter, the density meter, and the thermocouple to make sure they are in accepted range according to table-3<sup>F</sup>.

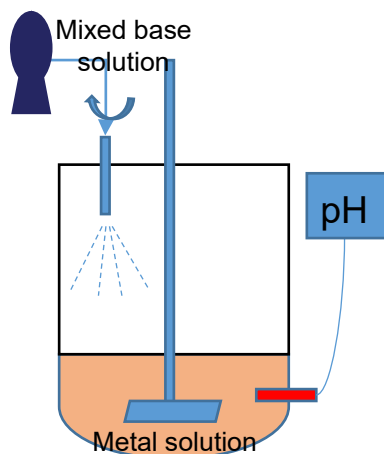
### Remarks

<sup>E</sup> Undissolved metal salts may cause solution properties off-spec such as a drop in pH. An increase in mixing time and re-sampling of mixed metal solution properties are recommended.

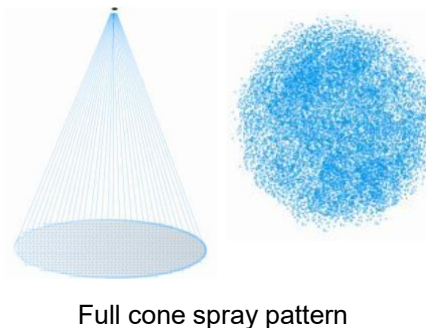
<sup>F</sup> Table-3: control value for mixed metal solution

| Mixed metal solution temperature (°C) | pH | Density (kg/m <sup>3</sup> ) |
|---------------------------------------|----|------------------------------|
| 20                                    |    |                              |
| 25 (preferred)                        |    |                              |
| 30                                    |    |                              |

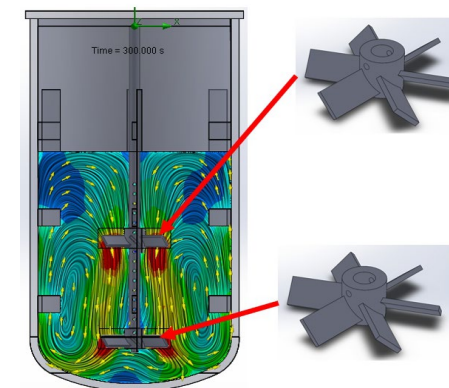
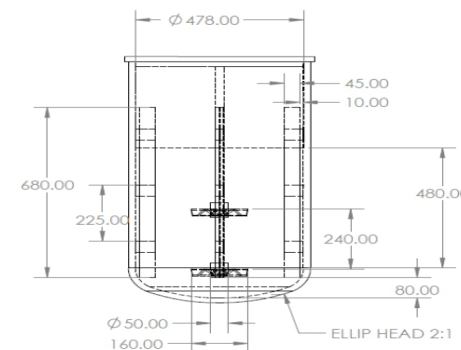
- Chemicals
- Equipment
- Process control



Remark for the spray nozzle



Remarks for the aging reactor



- Slurry pressure while aging at 140°C is around 4.65 kg/cm<sup>2</sup>

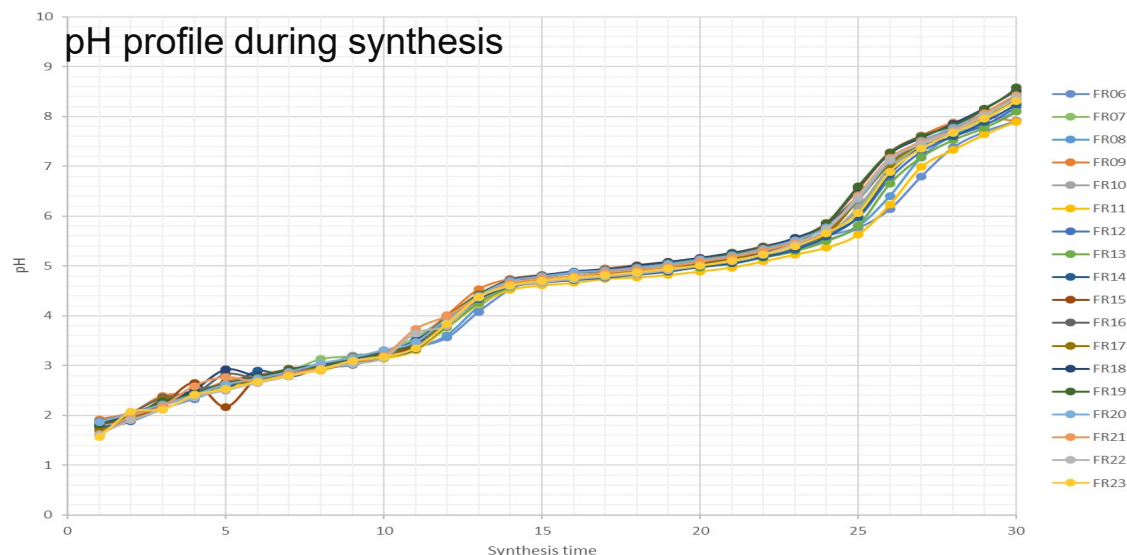
- Geometry of the baffled reactor with 6-blade turbine impeller with two levels (Total Liquid Volume= 101.3L)
- As crystal slurry requires well-mixing during the synthesis and aging step. The multiple-stage turbine impeller for instance 6-blade turbine impeller with two levels with a baffled vessel is suggested to provide well-mixing of slurry in the aging reactor. To confirm well-mixing inside the reactor, computer fluid dynamic such as FLUENT or SOLID WORK flow simulation should be carried out to investigate internal mixing profile and behaviors.
- The co-precipitation is done in the pressure vessel with the two-levels impeller and 4 baffle tank. The 6-pitched blade turbine was selected for preparation of well mixing of slurry in the reactor.

- 1) Feed all mixed metal solution from the mixing tank to an aging reactor to fill up heel volume.
- 2) Start agitator of the aging reactor at  $P/V = 1.0392 \text{ kW/m}^3$  to maintain good mixing efficiency.
- 3) Start monitoring and recording pH value continuously.
- 4) Feed mixed base solution from the mixing tank via the inlet spray nozzle to the aging reactor via the feeding pump within 1-0.5 hours (feed flow rate around 9-18 m<sup>3</sup>/h) while stirring. Record feed rate of the feeding pump in process record. Final pH value should be around  $8.5 \pm 0.2$ .
- 5) Stir for 0.5 hour then record pH value in process record.
- 6) Add slowly base solution 50 wt.% from base solution tank to adjust pH of the slurry to  $10 \pm 0.2$  under continuous stirring.
- 7) Stir for 0.5 hour then record pH value.
- 8) Remove pH probe and seal the reactor.
- 9) Start heating the reactor to final aging temperature  $\pm 5^\circ\text{C}$  accuracy according to synthesis recipe [140°C] with a heating ramp rate of not more than  $1.4^\circ\text{C}/\text{min}$ ; record temperature profile and record slurry temperature while continuously stirring the slurry.
- 10) Aging the slurry as defined according to synthesis recipe [4 hours].
- 11) Cool down slurry below to  $80\text{-}50^\circ\text{C}$  for further processing.

# Large scale manufacturing: Success and Lessons Learned

## Technology transfer - Critical Process Step to make the crystal (Ex.)

pH slowly increase from pH=1 then gradually increase to pH=3 after mixed metal solution is added. The pH of the slurry then showed two significant step ups: The first one is a step during pH reaches 3. The pH will suddenly jump from pH=3 to pH=5 then it remains stable before going through another abrupt elevation at pH=6. The final pH would be pH=9 after everything is mixed together and then would be adjusted to pH = 10 using NaOH solution 50 wt%.



### Remark for the critical item

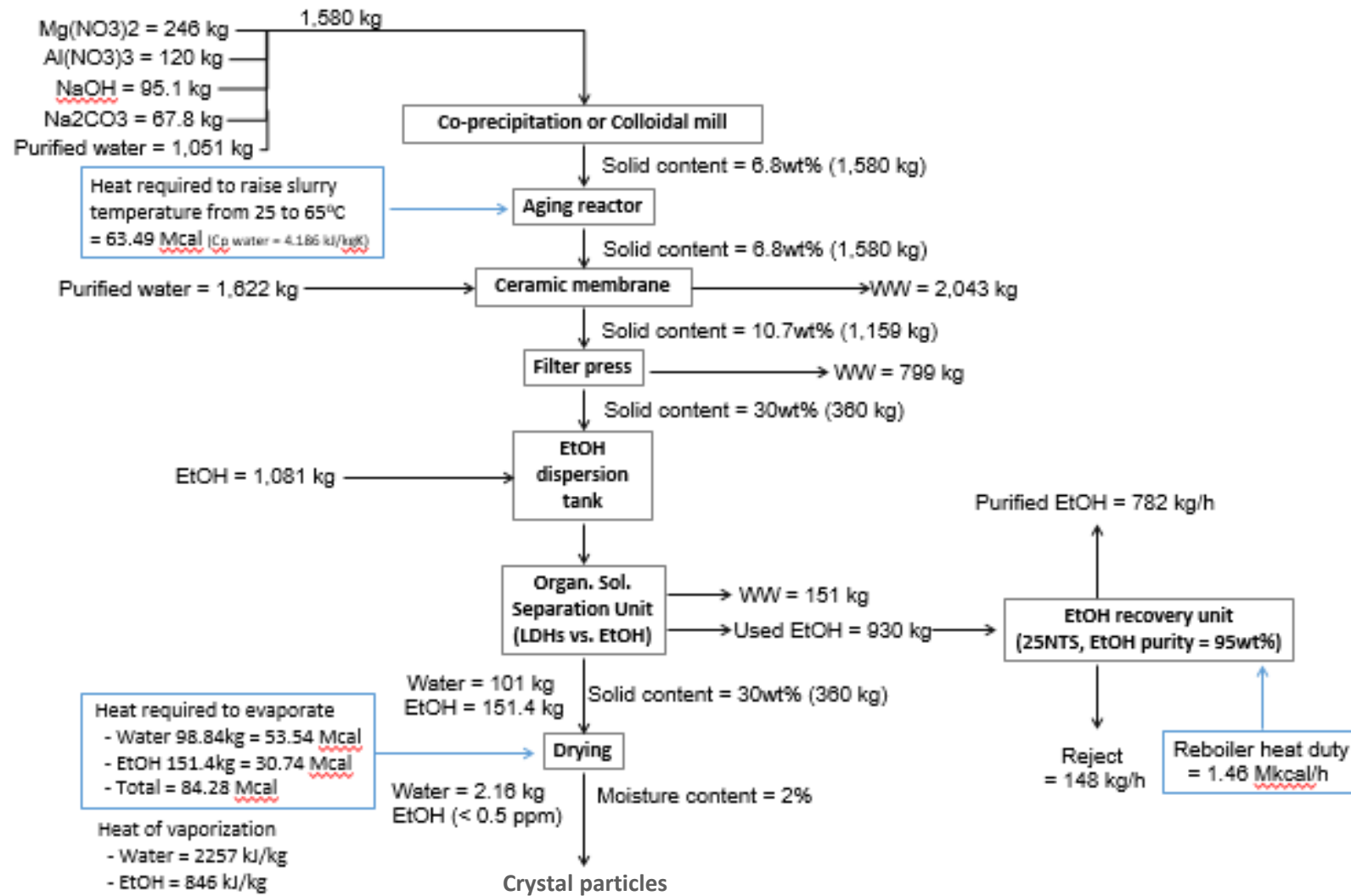
pH profile during synthesis is the crucial step to be successfully produce HFZ2 seed material. The profile of pH can be used as an indicator for guidance of synthesis step. Missing of pH profile during synthesis may be caused by failed quality specifications of raw materials during preparation step, missing of base solution, or base solution feed flow rate. If pH profile is not in accordance with the suggested profile mentioned above, it's recommended to stop synthesis step to re-assure qualities of raw materials including flow rate of calibration curve for the feeding pump.

| SCG LDH Production Data Record Sheet |                          | FR Process Description and Control Process           |                  |                                     | Parameters Data Record Sheet   |   | Additional Request : |              |                    |        |  |
|--------------------------------------|--------------------------|--|------------------|-------------------------------------|--|---|----------------------|--------------|--------------------|--------|--|
| Batch number                         |                          | Date   | Record Time      |                                     |  |   |                      |              |                    |        |  |
| no                                   | Process                  | Agitator speed (RPM)                                 | Temperature (°C) | Time (hr)                           | Control Process Parameters   | Record Data   |                      | Sample Label | Sample volume (ml) | Remark |  |
|                                      |                          | Process parameters                                   | Date             | Time                                |  |   |                      |              |                    |        |  |
| 1                                    | Raw material preparation | -  | RT               | -                                   |  |   |                      |              |                    |        |  |
| 2                                    | Feeding + Homogeneity    | 450  | RT               | 30 min                              |  |   |                      |              |                    |        |  |
|                                      |                          | Zn(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O | metal solution   |                                     |  |   |                      |              |                    |        |  |
|                                      |                          | Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O | metal solution   |                                     |  |   |                      |              |                    |        |  |
|                                      |                          | Al(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O | metal solution   |                                     |  |   |                      |              |                    |        |  |
|                                      |                          | NaOH   | NaOH solution    |                                     |  |   |                      |              |                    |        |  |
|                                      |                          | Na <sub>2</sub> CO <sub>3</sub>                      | NaOH solution    |                                     |  |   |                      |              |                    |        |  |
|                                      |                          | NaOH (for pH adjustment)                             | NaOH solution    |                                     |  |   |                      |              |                    |        |  |
| 3                                    | pH Adjust + Homogeneity  | 450  | RT               | 10 min                              | pH <sub>final</sub> = 9<br>pH <sub>inter adjust</sub> = 10   | pH <sub>final</sub> =<br>pH <sub>inter adjust</sub> =   |                      |              |                    |        |  |
| 4                                    | Hydrothermal (aging)     | 450  | 140              | 4 hr<br>(+150 min for raising temp) | Heating time = 100 min   | Heating time =<br>Level in reactor =  |                      |              |                    |        |  |
| 5                                    | Cooling down reactor     | 450  | <50              | 1 hr                                |  | pH <sub>inter aging</sub> =   |                      |              |                    |        |  |
| 6                                    | CRM washing (DI washing) | -  | RT               | 3 hr                                | conductivity <sub>maximum</sub> < 600 uS/cm<br>Volume <sub>DI</sub> = 40-50 L<br>Volume <sub>CR</sub> = 20-40 L<br>Total volume <sub>slurry</sub> < 65 L<br>Temp <sub>in-Slurry</sub> = 10°C | conductivity <sub>permeate</sub> =<br>%solid content <sub>slurry</sub> =<br>weight <sub>slurry</sub> (kg) = |                      |              |                    |        |  |
| 7                                    | Filter press             | -  | RT               | 3 hr                                |  | %solid content =<br>Total wet cake (kg) =<br>Pressure =   |                      |              |                    |        |  |
| 8                                    | Wet surface treatment    | 350  | 80               | 1 hr<br>(+100 min for raising temp) | Slurry volume = 95 L<br>Temp <sub>slurry</sub> = 80  | Amount of water (kg) =<br>Total slurry volume (L) =<br>Temp <sub>slurry</sub> =                             |                      |              |                    |        |  |
|                                      | Prepare NaST 5% Liquid   | -  | 80               | -                                   | Completely dissolved   |   |                      |              |                    |        |  |
| 9                                    | Cooling down reactor     | -  | RT               | 1 hr                                |  | Total wet cake (kg) =<br>Pressure =   |                      |              |                    |        |  |
| 10                                   | Filter press             | -  | RT               | 3 hr                                | %solid content =   |   |                      |              |                    |        |  |
| 11                                   | Washing                  | -  | RT               | -                                   | conductivity <sub>maximum</sub> < 80 uS/cm   | conductivity <sub>permeate</sub> =  |                      |              |                    |        |  |
| 12                                   | Oven dry                 | -  | 110              | -                                   |  |   |                      |              |                    |        |  |
| 13                                   | Centrifugal mill         | -  | RT               | -                                   | Sieve = 80 um<br>RPM = 10,000  |   |                      |              |                    |        |  |
| 12                                   | Oven dry                 | -  | 150              | -                                   | %moisture content < 3%   | %moisture content =   |                      |              |                    |        |  |
| Comment :                            |                          |  |                  |                                     |  |   |                      |              |                    |        |  |

Table-4: process record datasheet

Table-4 above is process record datasheet where process condition and parameters are collected to control final product such as RPM, temperature and time. Also, the criteria which needed to be considered to make decision whether slurry could be processed to the next steps.

# Global H&M balance



# Global H&M balance and cost structure

|   |         |        |           |
|---|---------|--------|-----------|
| - Net Mfg COGS cost benefit                                       | 1386.24 | 1y2q3  |           |
| - P of sales of COGS  | 373     | 4.33   | U% (1°C)  |
| <b>Value realising</b>  |         |        |           |
| - Cost to sell for the year 2e3 realising revenue prod sales 10e3 | 2.8     |        |           |
| - Cost to sell for the year 2e3 realising revenue prod sales 2e3  | 4       |        |           |
| <b>Export</b>   |         |        |           |
| - (Transportation) (COGS)   | 383     |        | C         |
| - (Transportation) (COGS)   | 473     |        | C         |
| - (Transportation) (COGS)   | 373     |        | C         |
| - Total cost of sales of COGS                                     | 3386.84 |        | U%        |
| - P of sales realising revenue IT (COGS)                          | 373     | 4.33   | U% (1°C)  |
| - P of sales realising revenue IT sales (COGS)                    | 383     | 4.48   | U% (1°C)  |
| - P of sales realising revenue IT sales (COGS)                    | 473     | 4.48   | U% (1°C)  |
| - P of COGS realising revenue IT sales (COGS)                     | 383     | 383.37 | U% (1°C)  |
| - P of COGS realising revenue IT sales (COGS)                     | 473     | 388.71 | U% (1°C)  |
| <b>Manufacturing - sell to</b>                                    |         |        |           |
|   | 1)      |        |           |
|   | 1)A)    |        |           |
| <b>Manufacturing - import</b>                                     |         |        |           |
| - P of COGS realising revenue IT sales (COGS)                     | 383     | 383.37 | U% (CC°C) |
| - P of COGS realising revenue IT sales (COGS)                     | 483     | 348.93 | U% (CC°C) |
| <b>Buy material overseas</b>                                      |         |        |           |
| - P of sales of COGS  | 383     | 4.38   | U% (CC°C) |

|   |                 |                 |                 |                   |
|---|-----------------|-----------------|-----------------|-------------------|
| <b>Revenue</b>                          |                 |                 |                 |                   |
| Export COGS, 01 (U)                     | 28,628.68       | 178,283.24      | 713,412.88      | 7,124,128.81      |
| Export COGS, 02 (U)                     | 48,266.49       | 303,833.44      | 818,439.22      | 8,183,343.22      |
| Table M segment account 00e segm :      | 78,432.7        | 00000000        | 00000000        | 00000000          |
| Table M segment account 00e segm :      | 38              | 0000            | 0000            | 4,342.7           |
| Table M segment account 00e segm :      | 78,440          | 00000000        | 00000000        | 00000000          |
| Cost (000)                              | 3.18            | 18.28           | 42.44           | 424.86            |
| <b>Revenue</b>                          |                 |                 |                 |                   |
| Export of prod sales to the 0e COGS (p) | 8.28            | 3.28            | 18.88           | 188.88            |
| Cost (000)                              | 8.81            | 8.86            | 8.34            | 3.37              |
| <b>Revenue</b>                          |                 |                 |                 |                   |
| Net sales COGS (p)                      | 13.38           | 84.68           | 337.44          | 3883.18           |
| Cost of COGS (p)                        | 8.68            | 41.28           | 166.84          | 1783.17           |
| Cost sales (p)                          | 3.21            | 17.91           | 71.48           | 288.93            |
| Profit - sales (p)                      | 4.68            | 41.28           | 166.84          | 1783.17           |
| Profit - sales (p)                      | 8.37            | 1.24            | 8.18            | 84.14             |
| Profit - sales (p)                      | 3.44            | 86.61           | 86.24           | 866.24            |
| <b>Manufacturing - sell to (0)</b>      | 3               | 3               | 3               | 3                 |
| Export to sell to (U)                   | 825,458         | 3,436,314       | 18,425,488      | 188,413,498       |
| Export to export sales (U)              | 1,891           | 8,284           | 21,888          | 328,488           |
| Cost to (U)                             | 8,282           | 48,484          | 161,181         | 1,614,181         |
| Export to export sales (U)              | 116             | 863             | 3,244           | 32,844            |
| Table M segment account 00e Segm :      | 00000000        | 00000000        | 00000000        | 00000000          |
| Table M segment account 00e Segm :      | 848             | 288             | 2,888           | 28,428            |
| Table M segment account 00e Segm :      | 00000000        | 00000000        | 00000000        | 00000000          |
| Cost (000)                              | 14.43           | 23.24           | 388.48          | 3833.83           |
| <b>Revenue</b>                          |                 |                 |                 |                   |
| Table M segment account of seg 01       | CC0 00 (20 (p)) | CC0 00 (20 (p)) | CC0 01 (26 (p)) | CC0 100 (102) (p) |
| Table M segment account of seg 01       | 82,44           | 84,88           | 388,88          | 3,784,48          |
| Table M segment account of seg 01       | 48,848          | 00000000        | 000,268         | 00000000          |
| Cost (000)                              |                 |                 |                 |                   |
| <b>Revenue</b>                          |                 |                 |                 |                   |
| Table M segment account of seg 01       | 4,48            | 21,64           | 86,38           | 488,68            |
| Table M segment account of seg 01       | 88,383          | 28,836          | 00000000        | 00000000          |
| Cost (000)                              | 88.31           | 436.66          | 1281.31         | 12843.66          |
| <b>Revenue</b>                          |                 |                 |                 |                   |
| Export of COGS, 01 (U)                  | 344,816.86      | 00000000        | 00000000        | 00000000          |
| Export COGS (U)                         | 00000000        | 00000000        | 00000000        | 00000000          |
| Table M segment account 00e Segm :      | 00000000        | 00000000        | 00000000        | 00000000          |
| Table M segment account 00e Segm :      | 428             | 2,288           | 4,888           | 46,242            |
| Table M segment account 00e Segm :      | 00000000        | 00000000        | 00000000        | 00000000          |
| Cost (000)                              | 48.88           | 323.38          | 436.34          | 4343.31           |
| <b>Revenue</b>                          |                 |                 |                 |                   |
| Export COGS, 01 (U)                     | 38,326.84       | 126,684.14      | 88,236.28       | 8,867,267.28      |
| Export COGS (U)                         | 58,366.47       | 181,343.31      | 488,343.43      | 4,883,343.33      |
| Table M segment account 00e segm :      | 48,686          | 00000000        | 488,236         | 00000000          |
| Table M segment account 00e segm :      | 82              | 82              | 382             | 3,824             |
| Table M segment account 00e segm :      | 48,326          | 288,823         | 00000000        | 00000000          |
| Cost (000)                              | 1.28            | 6.41            | 28.61           | 286.48            |



# Global H&M balance and cost structure

| VARIABLE COSTS                       |                  |                           |                           |                               |                                      |               |            |             |
|--------------------------------------|------------------|---------------------------|---------------------------|-------------------------------|--------------------------------------|---------------|------------|-------------|
| Raw materials                        | Quantity (kg)    | Cost per Unit (high side) | Cost per Unit (low side)  |                               | Cost of RMs per kg [USD/kg]          | RMs: Zn/Mg/Al | RMs: Zn/Al | % Reduction |
| Zn(NO3)2.6H2O                        | 2893.12          | 2.46                      | 1.22                      |                               | Low side, sourcing by SCGC [USD/kg]  | 2.12          | 1.90       | 10.50       |
| Mg(NO3)2.6H2O                        | 1246.90          | 1.25                      | 0.22                      |                               | High side, sourcing by SCGC [USD/kg] | 5.00          | 3.74       | 25.10       |
| Al(NO3)3.9H2O                        | 1824.24          | 1.28                      | 0.68                      |                               |                                      |               |            |             |
| NaOH 50%w/w solution (ASTEC-2 price) | 3254.46          | 0.54                      | 0.54                      |                               |                                      |               |            |             |
| Na2CO3 (only one source)             | 515.5470632      | 0.40                      | 0.40                      |                               |                                      |               |            |             |
| RO water - synthesis (ASTEC-2 price) | 13875.55         | 0.02                      | 0.02                      |                               |                                      |               |            |             |
| Water washing                        | 150.00           | 0.02                      | 0.02                      |                               |                                      |               |            |             |
| Product (kg)                         | 1752.17          |                           |                           |                               |                                      |               |            |             |
| Process steps                        | Q required (kWh) | Q required (Btu)          | Electricity cost in China | Electricity cost in USA, Geor |                                      |               |            |             |
| Aging                                | 4,247            | 14,489,691                | \$ 407.66                 | \$ 520.62                     |                                      |               |            |             |
| Drying                               | 29,970           | 102,262,791               | \$ 2,877.15               | \$ 3,674.35                   |                                      |               |            |             |
| Grinding - jet mill                  | 2,735            | 9,332,027                 | \$ 262.55                 | \$ 335.30                     |                                      |               |            |             |
| Grinding - impact mill               | 912              | 3,110,676                 | \$ 87.52                  | \$ 111.77                     |                                      |               |            |             |
| Dry surface treatment                | 96,242           | 328,390,290               | \$ 9,239.20               | \$ 11,799.23                  |                                      |               |            |             |
| Wet surface treatment                | 2,534            | 8,645,288                 | \$ 243.23                 | \$ 310.63                     |                                      |               |            |             |

| VARIABLE COSTS             | China  |           | USA   |           | China   |         | USA   |  |
|----------------------------|--|-----------|---|-----------|---|---------|---|--|
|                            | A) Aging + Drying + Dry surface treatment + Grinding - jet m. A) |           | A) Aging + Drying + Dry surface treatment + Grinding - jet mill |           | B) Aging + 'Wet surface treatment + Drying + Grinding - impact mill |         | B) Aging + 'Wet surface treatment + Drying + Grinding - impact mill |  |
| 1) Raw materials high side | \$ 7.55  |           | \$ 7.30   | \$ 3.32   |   | \$ 2.06 | \$ 2.64   |  |
| 2) Raw materials low side  | \$ 4.13  |           |   |           |   |         |   |  |
| VARIABLE COSTS             | China  |           | USA   |           |   |         |   |  |
|                            | Process A  | Process B | Process A   | Process B |   |         |   |  |
| High                       | \$ 14.84   | \$ 9.61   | \$ 10.18  |           |   |         |   |  |
| Low                        | \$ 11.43   | \$ 6.20   | \$ 6.77   |           |   |         |   |  |

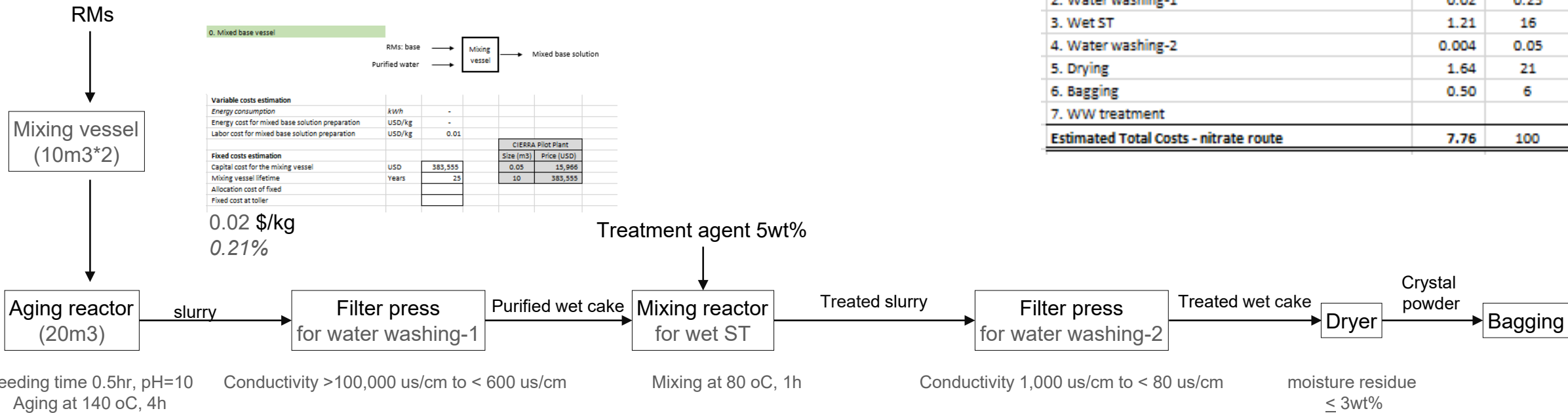
# Large scale manufacturing: Success and Lessons Learned

## Global H&M balance and cost structure

|   |                                       |   |                   |             |                   |             |                   |             |                   |             |                 |
|---|---------------------------------------|---|-------------------|-------------|-------------------|-------------|-------------------|-------------|-------------------|-------------|-----------------|
| Electric cost in thailand is 3.17 Bath/kWh      |                                       |   |                   |             |                   |             |                   |             |                   |             |                 |
| Rexchange rate on Feb, 4 2020 is 31.3156 bath/฿ |                                       | <b>0.10</b>                                     | <b>฿/kWh</b>      |             |                   |             |                   |             |                   |             |                 |
|   |                                       |   | 8.647571502       |             |                   |             |                   |             |                   |             |                 |
| RM cost (High side - 20MT)                      | Components                            | Price per kg (USD/kg) High side SCG CH sourcing | Reactor size (m3) | 0.1         | Reactor size (m3) | 0.5         | Reactor size (m3) | 2           | Reactor size (m3) | 20          |                 |
|   |                                       |   | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) |                 |
|   | Zn(NO3)2.6H2O                         | 2.46  | 14.28             | 35.18       | 68.98             | 169.98      | 275.07            | 677.78      | 2893.12           | 7128.64     |                 |
|   | Mg(NO3)2.6H2O                         | 1.25  | 6.15              | 7.72        | 29.73             | 37.28       | 118.55            | 148.67      | 1246.90           | 1563.61     |                 |
|   | Al(NO3)3.9H2O                         | 1.28  | 9.00              | 11.52       | 43.50             | 55.63       | 173.45            | 221.84      | 1824.24           | 2333.21     |                 |
|   | NaOH 50%/w/w solution (ASTEC-2 price) | 0.54  | 16.06             | 8.72        | 77.60             | 42.13       | 309.43            | 168.01      | 3254.46           | 1767.03     |                 |
|   | Na2CO3 (only one source)              | 0.40  | 2.54              | 1.02        | 12.29             | 4.92        | 49.02             | 19.61       | 515.5470632       | 206.22      |                 |
|   | RO water - synthesis (ASTEC-2 price)  | 0.02  | 68.48             | 1.08        | 330.85            | 5.23        | 1319.28           | 20.86       | 13875.55          | 219.37      |                 |
|   | <b>RM total cost</b>                  |   |                   |             | <b>65.24</b>      |             | <b>315.17</b>     |             | <b>1256.76</b>    |             | <b>13218.08</b> |
|   | <b>RM total cost per kg product</b>   |   | 8.65              |             | <b>7.54</b>       | 41.78       | <b>7.54</b>       | 166.59      | <b>7.54</b>       | 1752.17     | <b>7.54</b>     |
| RM cost (Low side - 20MT)                       | Components                            | Price per kg (USD/kg) Low side SCG CH sourcing  | Reactor size (m3) | 0.1         | Reactor size (m3) | 0.5         | Reactor size (m3) | 2           | Reactor size (m3) | 20          |                 |
|   |                                       |   | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) |                 |
|   | Zn(NO3)2.6H2O                         | 1.22  | 14.28             | 17.42       | 68.98             | 84.16       | 275.07            | 335.59      | 2893.12           | 3529.60     |                 |
|   | Mg(NO3)2.6H2O                         | 0.22  | 6.15              | 1.37        | 29.73             | 6.63        | 118.55            | 26.44       | 1246.90           | 278.06      |                 |
|   | Al(NO3)3.9H2O                         | 0.68  | 9.00              | 6.12        | 43.50             | 29.58       | 173.45            | 117.94      | 1824.24           | 1240.49     |                 |
|   | NaOH 50%/w/w solution (ASTEC-2 price) | 0.54  | 16.06             | 8.72        | 77.60             | 42.13       | 309.43            | 168.01      | 3254.46           | 1767.03     |                 |
|   | Na2CO3 (only one source)              | 0.40  | 2.54              | 1.02        | 12.29             | 4.92        | 49.02             | 19.61       | 515.5470632       | 206.22      |                 |
|   | RO water - synthesis (ASTEC-2 price)  | 0.02  | 68.48             | 1.08        | 330.85            | 5.23        | 1319.28           | 20.86       | 13875.55          | 219.37      |                 |
|   | <b>RM total cost</b>                  |   |                   |             | <b>35.74</b>      |             | <b>172.65</b>     |             | <b>688.44</b>     |             | <b>7240.77</b>  |
|   | <b>RM total cost per kg product</b>   |   | 8.65              |             | <b>4.13</b>       | 41.78       | <b>4.13</b>       | 166.59      | <b>4.13</b>       | 1752.17     | <b>4.13</b>     |
| RM cost (ASTEC-2)                               | Components                            | Price per kg (USD/kg) ASTEC-2                   | Reactor size (m3) | 0.1         | Reactor size (m3) | 0.5         | Reactor size (m3) | 2           | Reactor size (m3) | 20          |                 |
|   |                                       |   | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) | Weight (Kg)       | Price (USD) |                 |
|   | Zn(NO3)2.6H2O                         | 3.00  | 14.28             | 42.87       | 68.98             | 207.11      | 275.07            | 825.84      | 2893.12           | 8685.82     |                 |
|   | Mg(NO3)2.6H2O                         | 1.49  | 6.15              | 9.20        | 29.73             | 44.44       | 118.55            | 177.20      | 1246.90           | 1863.77     |                 |
|   | Al(NO3)3.9H2O                         | 11.50   | 9.00              | 103.52      | 43.50             | 500.13      | 173.45            | 1994.28     | 1824.24           | 20375.01    |                 |
|   | NaOH 50%/w/w solution                 | 0.54  | 16.06             | 8.72        | 77.60             | 42.13       | 309.43            | 168.01      | 3254.46           | 1767.03     |                 |
|   | Na2CO3                                | 0.83  | 2.54              | 2.11        | 12.29             | 10.21       | 49.02             | 40.70       | 515.5470632       | 428.11      |                 |
|   | RO water - synthesis                  | 0.02  | 68.48             | 1.08        | 330.85            | 5.23        | 1319.28           | 20.86       | 13875.55          | 219.37      |                 |
|   | <b>RM total cost</b>                  |   |                   |             | <b>167.50</b>     |             | <b>809.25</b>     |             | <b>3226.89</b>    |             | <b>33939.12</b> |
|   | <b>RM total cost per kg product</b>   |   | 8.65              |             | <b>19.37</b>      | 41.78       | <b>19.37</b>      | 166.59      | <b>19.37</b>      | 1752.17     | <b>19.37</b>    |

# Large scale manufacturing: Success and Lessons Learned

## Cost structure



| Estimated Total Costs                        | USD/kg      | 7.76       |
|--|-------------|------------|
|  | USD/kg      | %          |
| 0. Mixed base/salts                          | 0.02        | 0.21       |
| 1. Aging                                     | 4.38        | 56         |
| 2. Water washing-1                           | 0.02        | 0.23       |
| 3. Wet ST                                    | 1.21        | 16         |
| 4. Water washing-2                           | 0.004       | 0.05       |
| 5. Drying                                    | 1.64        | 21         |
| 6. Bagging                                   | 0.50        | 6          |
| <b>Estimated Total Costs - nitrate route</b> | <b>7.76</b> | <b>100</b> |

0. Mixed base vessel

RMs: base  
Purified water

Mixing vessel

Mixed base solution

| Variable costs estimation                       |        |      |  |  |
|---|--------|------|--|--|
| Energy consumption                              | kWh    | -    |  |  |
| Energy cost for mixed base solution preparation | USD/kg | -    |  |  |
| Labor cost for mixed base solution preparation  | USD/kg | 0.01 |  |  |

| Fixed costs estimation             |       |         |  |  |
|------------------------------------|-------|---------|--|--|
| Capital cost for the mixing vessel | USD   | 383,555 |  |  |
| Mixing vessel lifetime             | Years | 25      |  |  |
| Allocation cost of fixed           |       |         |  |  |
| Fixed cost at toiler               |       |         |  |  |

| CIERRA Pilot Plant |             |
|--------------------|-------------|
| Size (m3)          | Price (USD) |
| 0.05               | 15,966      |
| 10                 | 383,555     |

0.02 \$/kg  
0.21%

Treatment agent 5wt%

Feeding time 0.5hr, pH=10  
Aging at 140 oC, 4h

Conductivity >100,000 us/cm to < 600 us/cm

Mixing at 80 oC, 1h

Conductivity 1,000 us/cm to < 80 us/cm

moisture residue  
≤ 3wt%

Nitrate-route

1. Aging

RMs → Pressure reactor (20m3) → LDH slurry

Aging at 140 oC, 4h

| Variable costs estimation |        |       |  |  |
|---------------------------|--------|-------|--|--|
| RMs                       | USD/kg | 4.13  |  |  |
| Energy consumption        | kWh    | 4,247 |  |  |
| Energy cost               | USD/kg | 0.23  |  |  |
| Labor cost for aging      | USD/kg | 0.01  |  |  |

| Fixed costs estimation                |       |           |  |  |
|---------------------------------------|-------|-----------|--|--|
| Capital cost for the pressure reactor | USD   | 1,150,664 |  |  |
| Pressure reactor lifetime             | Years | 23        |  |  |
| Allocation cost of fixed              |       |           |  |  |
| Fixed cost at toiler                  |       |           |  |  |

| CIERRA Pilot Plant |             |
|--------------------|-------------|
| Size (m3)          | Price (USD) |
| 0.1                | 47,899      |
| 20                 | 1,150,664   |

4.38 \$/kg  
56%

2. Water washing-1

LDH slurry → Filter press → Purified LDH slurry (wet cake)

Conductivity >100,000 us/cm to < 600 us/cm

| Variable costs estimation      |        |       |  |  |
|--------------------------------|--------|-------|--|--|
| Plant water                    | USD/kg | 0.001 |  |  |
| Energy consumption             | kWh    |       |  |  |
| Energy cost                    | USD/kg |       |  |  |
| Labor cost for water washing-1 | USD/kg | 0.02  |  |  |

| Fixed costs estimation            |       |         |  |  |
|-----------------------------------|-------|---------|--|--|
| Capital cost for the filter press | USD   | 435,477 |  |  |
| Filter press lifetime             | Years | 25      |  |  |
| Allocation cost of fixed          |       |         |  |  |
| Fixed cost at toiler              |       |         |  |  |

| ROSE project |             |
|--------------|-------------|
| Size (m3)    | Price (USD) |
| 1            | 72,169      |
| 20           | 435,477     |

0.02 \$/kg  
0.23%

3. Wet surface treatment

Surface treatment agent  
Purified LDH slurry (wet cake)

Mixing vessel  
NAST Swtch

Treated LDH slurry

| Variable costs estimation |        |       |  |  |
|---------------------------|--------|-------|--|--|
| RMs                       | USD/kg | 0.23  |  |  |
| Energy consumption        | kWh    | 1,669 |  |  |
| Energy cost for wet ST    | USD/kg | 0.96  |  |  |
| Labor cost for wet ST     | USD/kg | 0.01  |  |  |

| Fixed costs estimation             |       |         |  |  |
|------------------------------------|-------|---------|--|--|
| Capital cost for the mixing vessel | USD   | 767,109 |  |  |
| Mixing vessel lifetime             | Years | 25      |  |  |
| Allocation cost of fixed           |       |         |  |  |
| Fixed cost at toiler               |       |         |  |  |

| CIERRA Pilot Plant |             |
|--------------------|-------------|
| Size (m3)          | Price (USD) |
| 0.1                | 31,933      |
| 20                 | 767,109     |

1.21 \$/kg  
16%

4. Water washing-2

Treated LDH slurry → Filter press → Purified/treated LDH slurry (wet cake)

Conductivity <1,000 us/cm to < 80 us/cm

| Variable costs estimation      |        |       |  |  |
|--------------------------------|--------|-------|--|--|
| Plant water                    | USD/kg | 0.001 |  |  |
| Energy consumption             | kWh    |       |  |  |
| Energy cost                    | USD/kg |       |  |  |
| Labor cost for water washing-2 | USD/kg | 0.003 |  |  |

| Fixed costs estimation            |       |         |  |  |
|-----------------------------------|-------|---------|--|--|
| Capital cost for the filter press | USD   | 435,477 |  |  |
| Filter press lifetime (Y)         | Years | 25      |  |  |
| Allocation cost of fixed          |       |         |  |  |
| Fixed cost at toiler              |       |         |  |  |

| ROSE project |             |
|--------------|-------------|
| Size (m3)    | Price (USD) |
| 1            | 72,169      |
| 20           | 435,477     |

0.004 \$/kg  
0.05%

5. Drying

Purified/treated LDH slurry (wet cake) → Dryer → LDH dried powder

moisture residue ≤ 3wt%

| Variable costs estimation |        |        |  |  |
|---------------------------|--------|--------|--|--|
| Plant water               | kWh    | 26,970 |  |  |
| Energy consumption        | USD/kg | 1.64   |  |  |
| Energy cost               | USD/kg |        |  |  |
| Labor cost for drying     | USD/kg |        |  |  |

| Fixed costs estimation                              |       |           |  |  |
|---|-------|-----------|--|--|
| Capital cost for the drying equipment (spray dryer) | USD   | 2,043,750 |  |  |
| Spray dryer lifetime                                | Years | 20        |  |  |
| Allocation cost of fixed                            |       |           |  |  |
| Fixed cost at toiler                                |       |           |  |  |

| Spray dryer @ TRC |             |
|-------------------|-------------|
| Size              | Price (USD) |
| 2,977/yr          | 2,043,750   |

1.64 \$/kg  
21%

0.5 \$/kg

0.5 \$/kg  
6%

