Scaling Up Your Thermal Processes Successfully

Webinar 2 in Harper's Series on Maximizing Production Economics

October 23rd, 2012
11:00am Eastern Time

Welcome!

Scaling Up Your Thermal Processes Successfully

Meet your Presenters:

- Tom Burkholder, Applications Engineering Mgr

  B.S. Chemical Engineering, State University of NY at Buffalo

  Leads engineering team in support of customer's new process development

- Brian Fuller, Sales Engineer

  B.S. Chemical Engineering, University of Colorado at Boulder

  Responsible for projects in Powdered Metals, Technical Ceramics and Energy Materials

About This Webinar Series

Maximizing the Production Economics of Your Thermal Processing System

- Your inside access to Harper's deep technical expertise
- Recordings of the events will be available on demand afterwards
- Other Events in this Series
  - Designing for Energy Efficiency in Thermal Processing (July 2012 – get it online now!)
  - Thermal Processing Research: Designing Flexibility & Performance (2013)
  - Maintenance Optimization: Planning Downtime Efficiently (2013)

Introduction to Harper

- Headquartered outside of Buffalo, NY
- Decades of thermal processing experience
- Dedicated Technology Centers for customer process development & testing
- Multi-disciplined engineering talent
  - Chemical
  - Ceramic
  - Mechanical
  - Electrical
  - Industrial
  - Process & Integration

Introduction to Harper

We work with developers & producers of advanced materials to provide innovative technologies:

- 200°C – 3000°C
- Batch to continuous processing
- Precise atmospheric controls
- High purity requirements
- High temperature Gas-Solid reactions

- Process Refinement
- Scale Up
- Optimization

Introduction to Harper

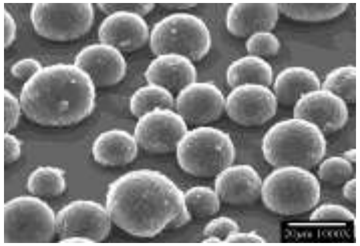
Focus on Processing System Solutions for…

<u>Advanced Materials</u>:

- Fibers & Filaments
- Metal Oxides & Powders
- Technical Ceramics
- Energy Materials
- Nano Materials
- Rare Earths
- Graphene

<u>Processes:</u>

- Sintering
- Drying
- Calcination
- Reduction
- Oxidation
- Carbonization
- Carburization
- Solid-solid reaction
- Gas-solid reaction
- Purification
- Metalizing
- Debinding
- Parts processing
- Phase transformation



Today's Agenda

- So You're Thinking of Scaling Up…
  - Planning for the ideal way to scale up
  - Managing risk – a step wise approach
- Thermal Processing Design Considerations in Scale Up
  - Material Flowability
  - Temperature Profile & Retention Times
  - Bed Depth / Percent Fill
  - Off-gassing
  - Co-current vs. Counter Current Flow
  - Cross Contamination

Case Studies & Examples

- Planning for the Unknowns & Stumbling Blocks
- Question & Answer Time

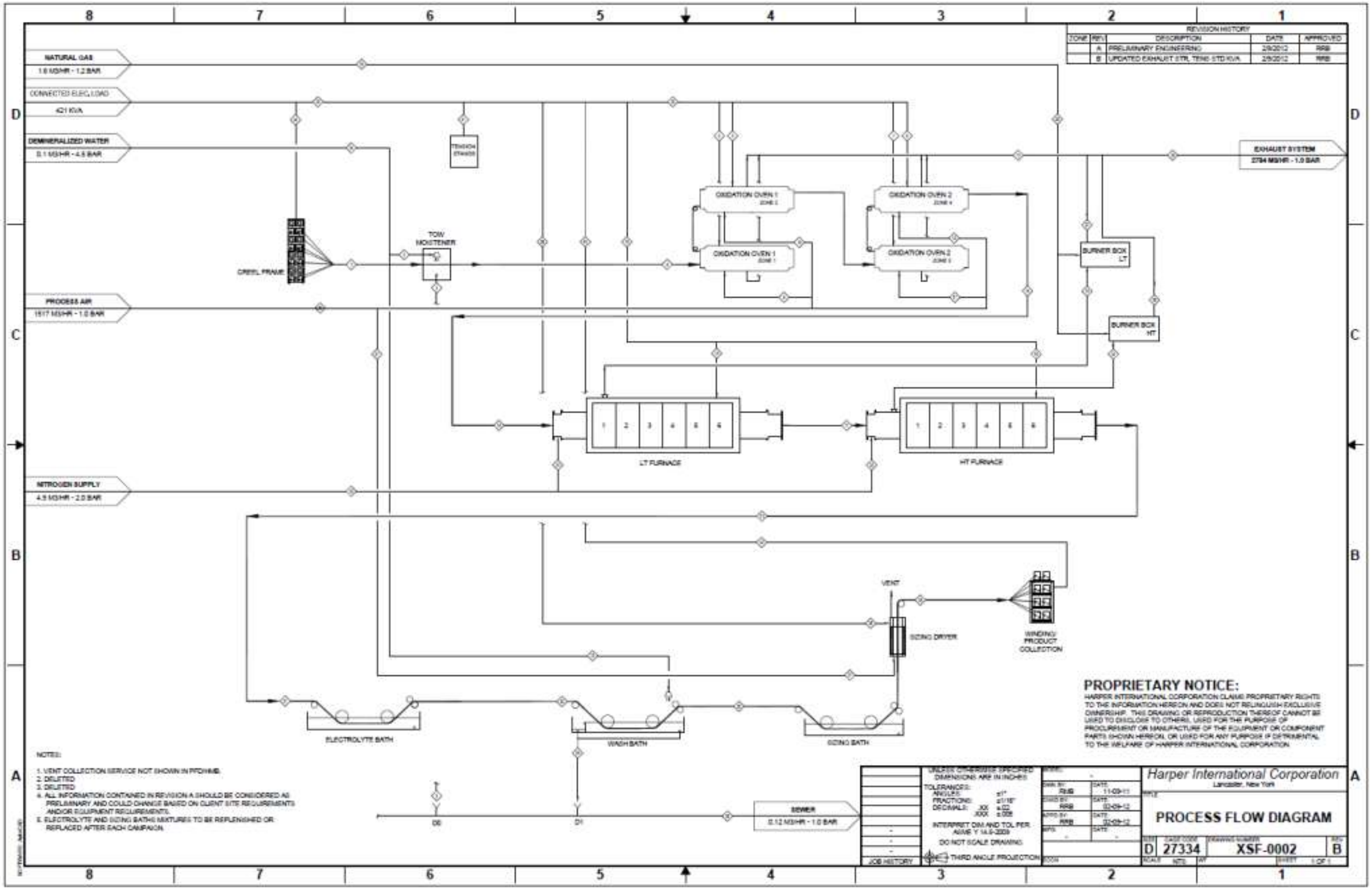
Planning for the Ideal Way to Scale Up

- Think through your bench top configuration
  - Today's Operation: strengths and weaknesses
  - Gram-scale production will not be a window into larger scale
    - Not limited by mass or heat transfer
    - Load and furnace configuration / geometry is irrelevant
    - All you have learned from your bench configuration is temperature requirement
  - You have likely performed thermal processes all in one step

Planning for the Ideal Way to Scale Up

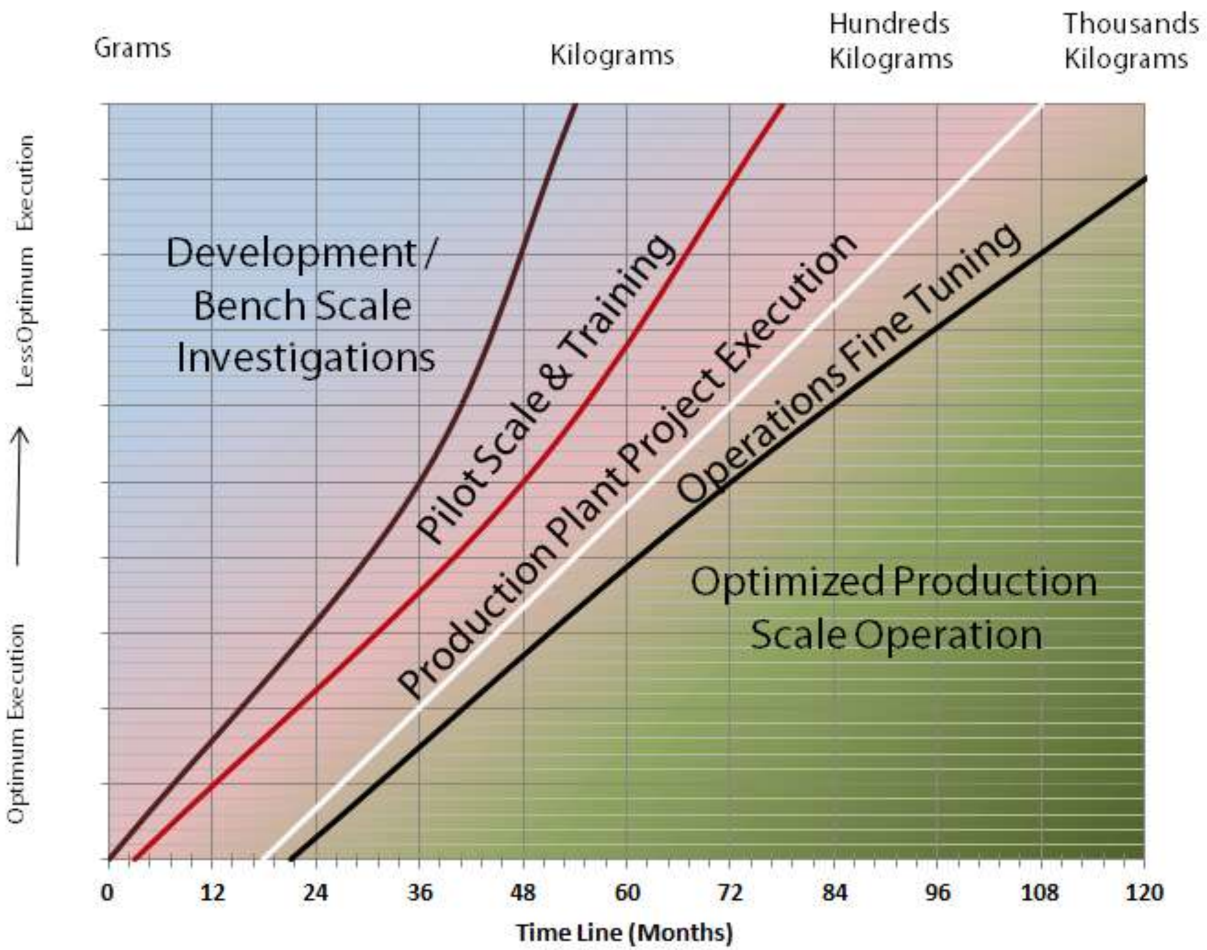
- Complete your Process Flow Diagram of Units Ops
  - Why?
    - Focuses efforts on key steps in the process
    - Defines material flow in and out of unit operations to assess future technical problems
  - A complex thermal process never stands alone
    - Upstream and downstream unit operations affect the optimum furnace design
    - Items to consider - cooling, gas separation, contact construction materials

Planning for the Ideal Way to Scale Up



Managing Risk in Scale Up – a Step-wise Approach

Timeline to Production Scale Operations for Emerging Materials



Managing Risk – a Step-wise Approach

Why go through all these steps:

- Helps better define the process
- Will uncover unknowns and things that may have been overlooked
- Mitigates or minimizes risk

Downside:

- Development using this methodology will take time and increase development costs!

Furnace 101

Reactor Type	Typical Material Profile	Material Handling Transport	Volumetric Efficiency
Rotary Tube	Ideal for Powder and Bulk Materials	Via Rotating Tube and Angle of Inclination	Low (10% - 20% Filled)
Pusher / Roller Hearths	Used for Powders, Bulk Materials and Net Shapes	Via Transport in Saggers	Moderate
Mesh / Strip Belt	Used for Powders, Bulk Materials and Net Shapes	Via Mechanical Belt	Moderate
Vertical Tube / Slot	Ideal for Powders	Via Gravity or Set by Rotary Valve or Auger	High
Horizontal Slot	Ideal for Fibers, Filaments and Webs	Material Generally Not in Contact with Furnace	Low

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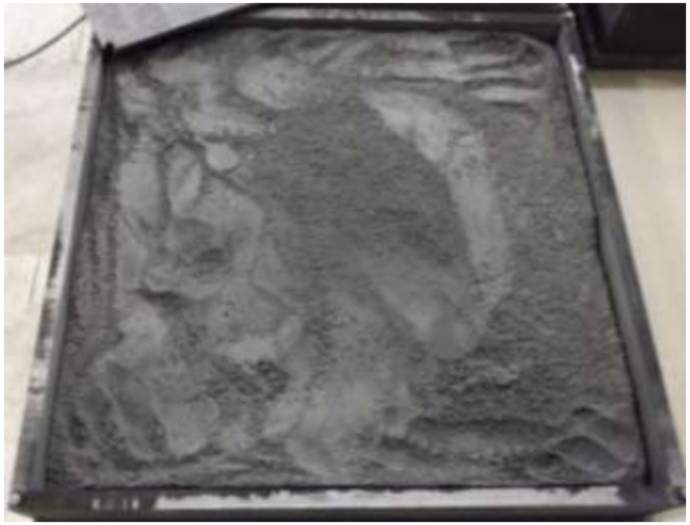
Thermal Processing Design Considerations in Scale Up

- Material Flowability
- Temperature Profile & Retention Times
- Bed Depth / Percent Fill
- Off-gassing
- Co-current vs. Counter Current Flow
- Cross Contamination

Key Design Considerations

Thinking about **Material Flowability** as you scale up

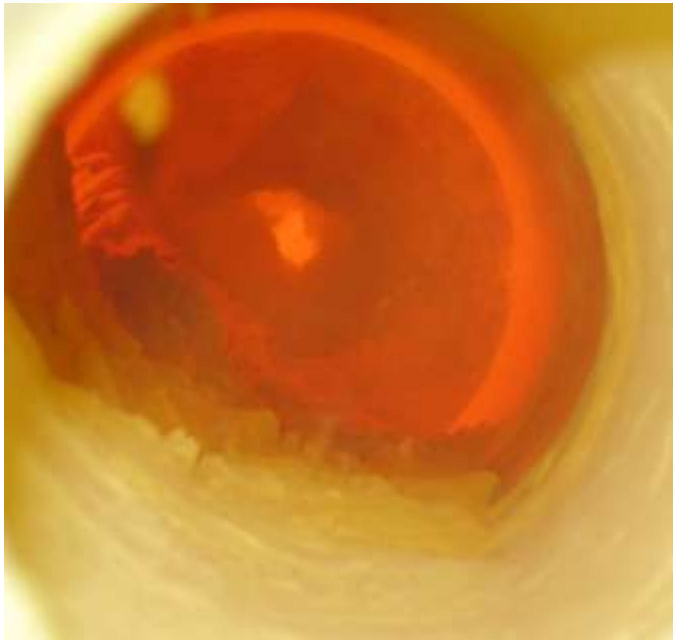
- Ideal system will heat just the process material - minimize kiln furniture
  - Static powder in trays vs. free flowing powder



Key Design Considerations

Thinking about **Material Flowability** as you scale up

- Look out for sticking / caking issues, and design enhancements that can be added to ensure flowability



Key Design Considerations

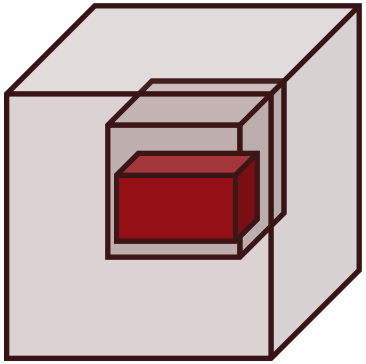
Thinking about **Material Flowability** as you scale up

- Knowing your material's properties: function of temperature
- Once you understand flowability, you should be able to remove some Furnace types from consideration in your scale up!!!

Key Design Considerations

Thinking about **Temp Profile & Retention Times** as you scale up

- How was time-temperature profile developed in lab scale?
  - Strong function of mass being treated and load configuration



Furnace: 12 in.^{3} - 6 in.^{3} @ 0.4 kg/L = 10 kg

Load: 4 in. x 4 in. x 1in. @ 1.0 kg/L = 260 g

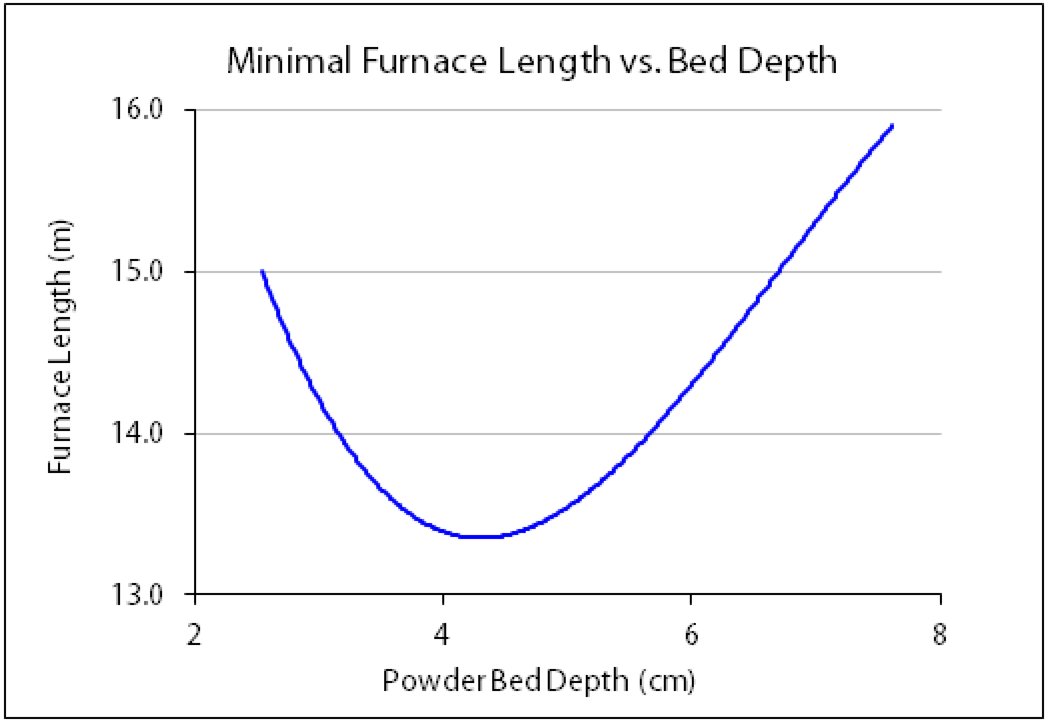
Overwhelming furnace mass… 40X!

Key Design Considerations

Thinking about **Temp Profile & Retention Times** as you scale up

- How was time-temperature profile developed in lab scale?
  - Strong function of mass being treated and load configuration
  - Thin bed depths
  - → **heat transfer**

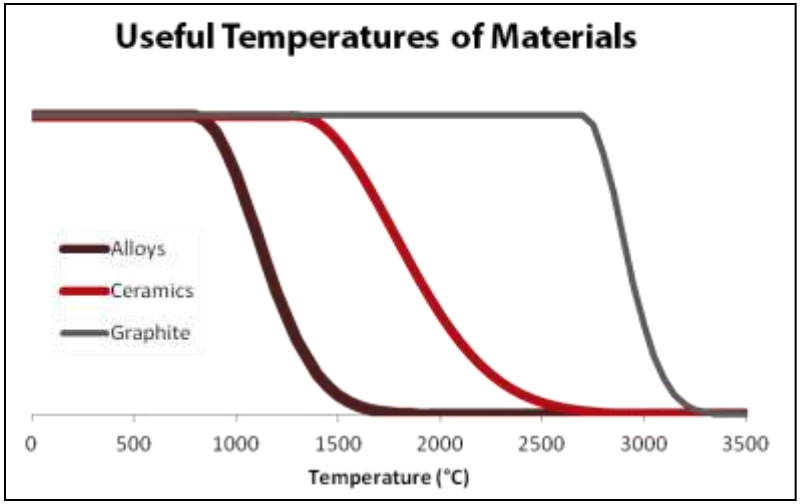
  - <u>**Fixed Production**</u>
  - Insulating material
  - Ramp rate *f*(D)
  - 0.5 hour soak



Key Design Considerations

Thinking about **Temp Profile & Retention Times** as you scale up

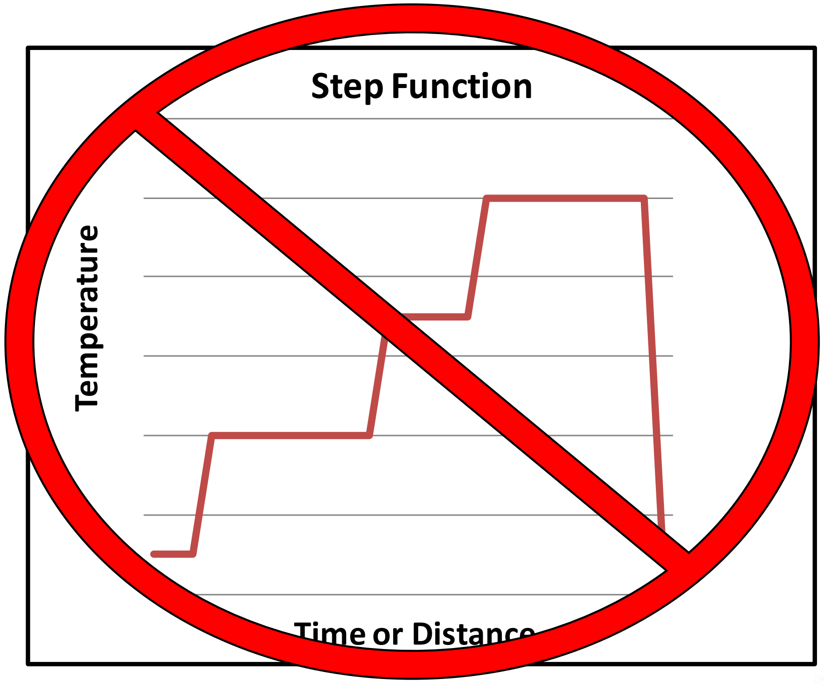
- Over-heated vs. under-heated
  - In smaller configurations, you might not see effect of non-uniform heating
  - Small window for soak time?
- Optimal process temperatures and limit of various materials of construction
  - As temp profile changes with steps upwards, materials of construction will almost certainly be different



Key Design Considerations

Thinking about **Temp Profile & Retention Times** as you scale up

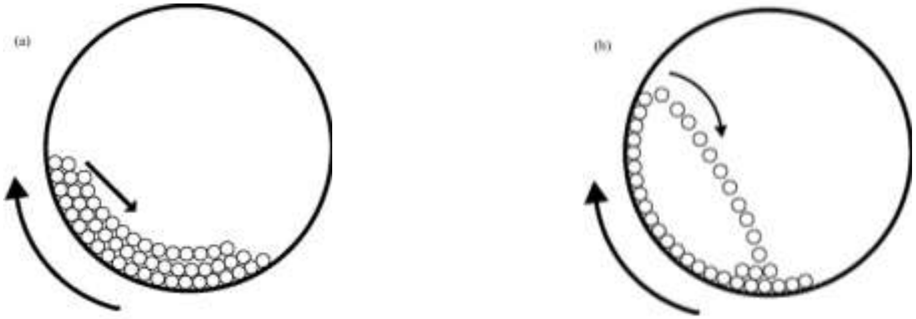
- Crazy temperature profile – conservative or unrealistic.
  - Heat transfer limited?
  - Mass transfer limited?
- Profile is **never** a step function
- Cooling Required?



Key Design Considerations

Thinking about **Bed Depth / Percent Fill** as you scale up

- Percent fill can be a deceiving design aspect, One has to align % fill (dimensionless) with actual bed depth to fully embrace heat penetration
- Heat penetration through bed depth in lab / pilot processing (likely a static tray, batch)
- Unless the bed is being enveloped (mixed) frequently, bed depth can become a factor in the design

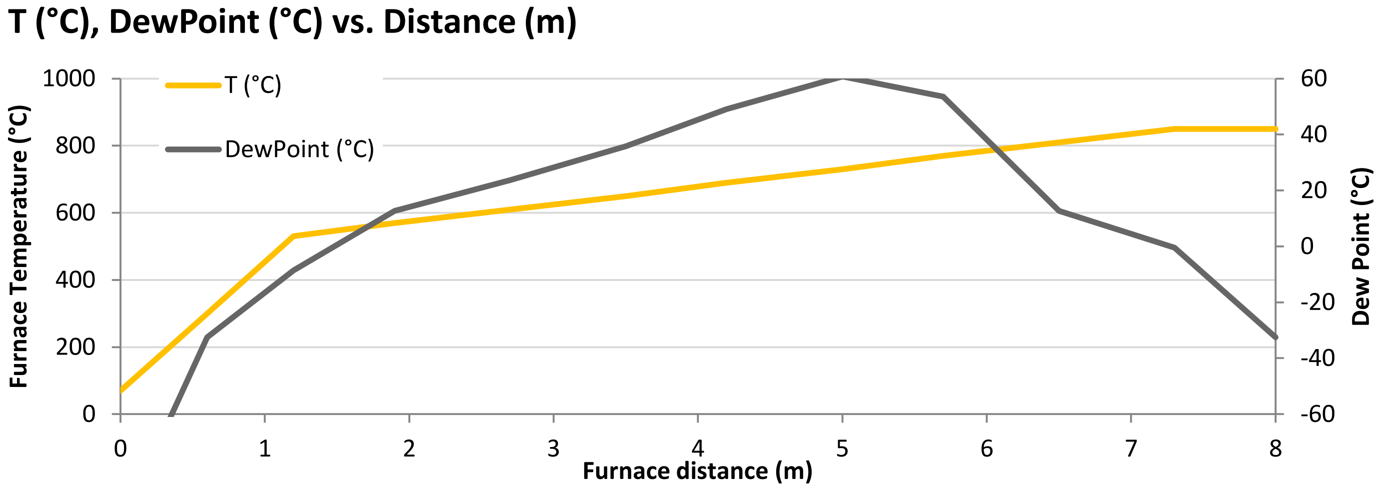


Key Design Considerations

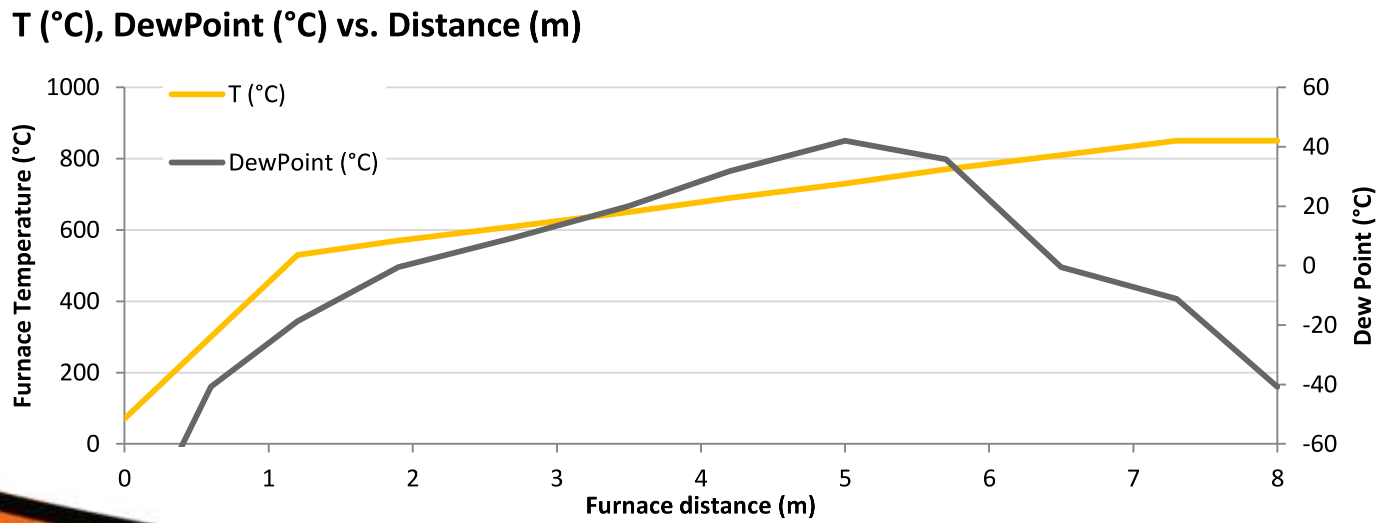
Thinking about **Off-gassing** as you scale up

- Why?
  - Removal of **condensable** gases - Pyrolysis
  - **Driving** the gas-solid reaction - Reduction
  - Preventing **re-adsorption** - Calcination

Driving the Gas-Solid Reaction



15 kg/h
15 m³/h ent
15 m³/h exit
vent zone 4



15 kg/h
38m³/h ent
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Key Design Considerations

Thinking about **Off-gassing** as you scale up

- Why?
  - Removal of **condensable** gases - Pyrolysis
  - **Driving** the gas-solid reaction - Reduction
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- Removing these byproducts will become more difficult as you scale up
- What about **preheating** process gas?

Key Design Considerations

Thinking about **Co-current vs. Counter Current Process Gas Flow**

- Counter Current
  - Most thermally efficient
  - Acts like a heat exchanger
  - Moves condensables away from product
- Co-Current
  - Fine material that can be entrained
  - Reaction gas? Concentration of gases along furnace length
  - Want volatiles to stay IN or ON product surface

Key Design Considerations

Thinking about **Material Compatibility** as you scale up

- Cross Contamination with the Equipment Materials of Construction:
  - Most important to materials where low impurity levels are desired:
    - Battery Materials
    - Rare Earths
    - High Purity Ceramics
  - Corrosive off-gasses are evolved

- Has a strong effect on the general furnace type and final scale of the furnace due to size limitations of many non-alloy components. Examples:
  - Rotary/Vertical tube size
  - Ceramic muffle & trays – take a long time to heat up, can't shock or it will break

Planning for the Unknowns & Stumbling Blocks

- Merging unit operations – is it really worth it?
  - Make sure you adequately flow chart your unit operations
- Your process might be best served with multiple unit operations
  - What you might be doing in single-step Batch process may not work as a single step Continuous process
  - Example: A furnace should function as both a dryer and a heat treatment step in the same unit operation. While investment may be more, may serve the process better as 2 operations.
- Adaptation to the upstream and downstream equipment can be a challenge
  - Consider material delivery and removal methodologies for getting it into a specific unit operation
- Consider atmospheric control when integrating upstream and downstream processes.

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