AVEKA Group

How Does Particle Characterization Enhance Research & Scale-Up, Validate Production, and Increase Your Bottom Line

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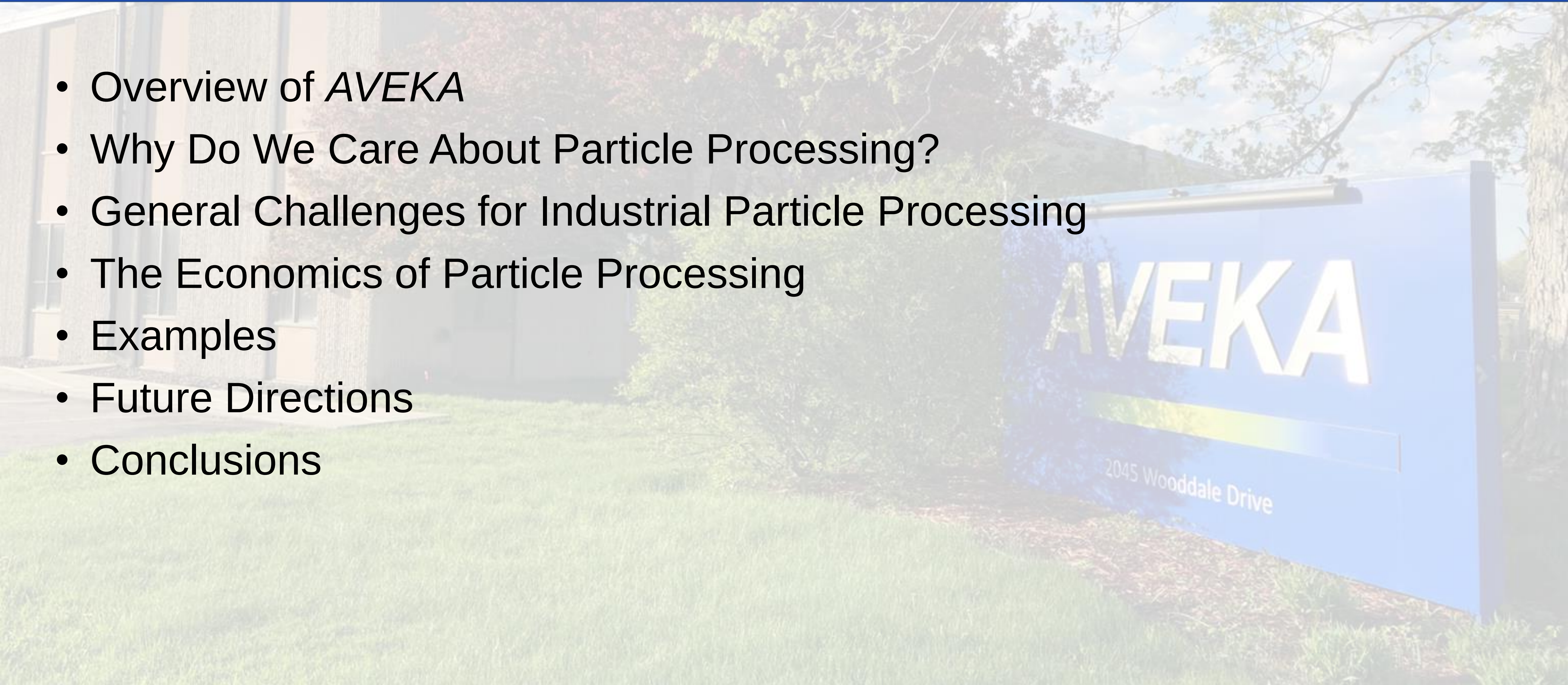
CEO & FOUNDER

Particle Processing Services
Toll Manufacturing
Research & Development
Innovative Solutions

Presentation Outline

- Overview of *AVEKA*
- Why Do We Care About Particle Processing?
- General Challenges for Industrial Particle Processing
- The Economics of Particle Processing
- Examples
- Future Directions
- Conclusions



AVEKA Group Overview

- Employee Owned and Operated
- Particle technology company focused on contract manufacturing and process development
- Spin-off of 3M in 1994
- Comprised of 5 Manufacturing Sites in Minnesota and Iowa
- ISO certifications / food-grade certifications
- Currently 280 employees

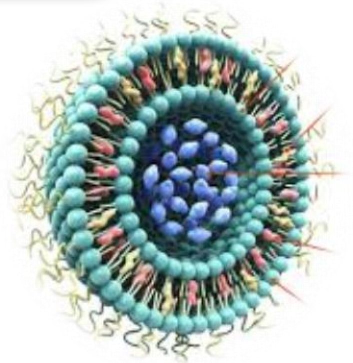
The *AVEKA* Group – A Different Type of CMO

- Broad Range of Unit Operations & Industry Areas
- Active Research & Development Group
- Extreme Technical and Collaborative Business Model

2000+	600+	300+
Inquires Per Year	Project Proposals Per Year	Customers Per Year

Who Cares About Particle Processing?

- 1930's Taconite Processing and its Effects
- Cement Manufacturing and Energy Costs
- Xerography and the Information Age
- COVID-19 and the Pandemic



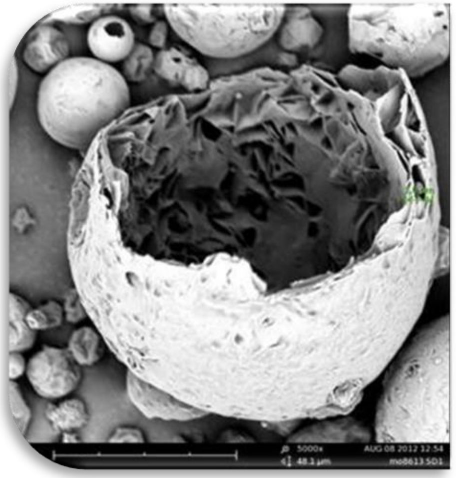
Big Challenges in Industrial Particle Processing

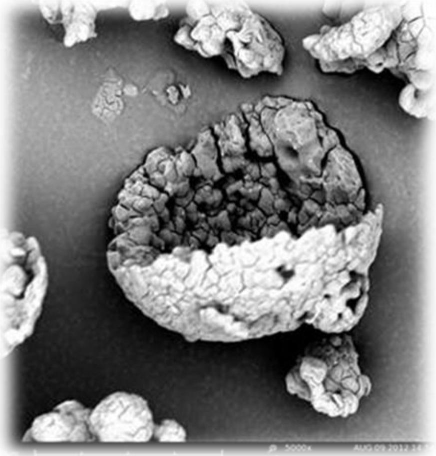
Operations

- Economics
- Technology Readiness Levels

Technology

- Water
- Powder Flow
- Grinding Efficiency

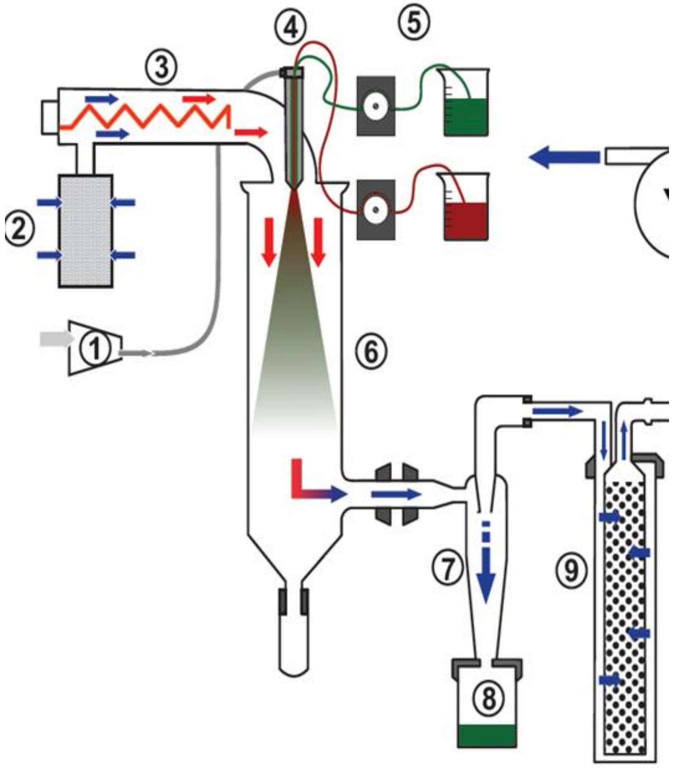




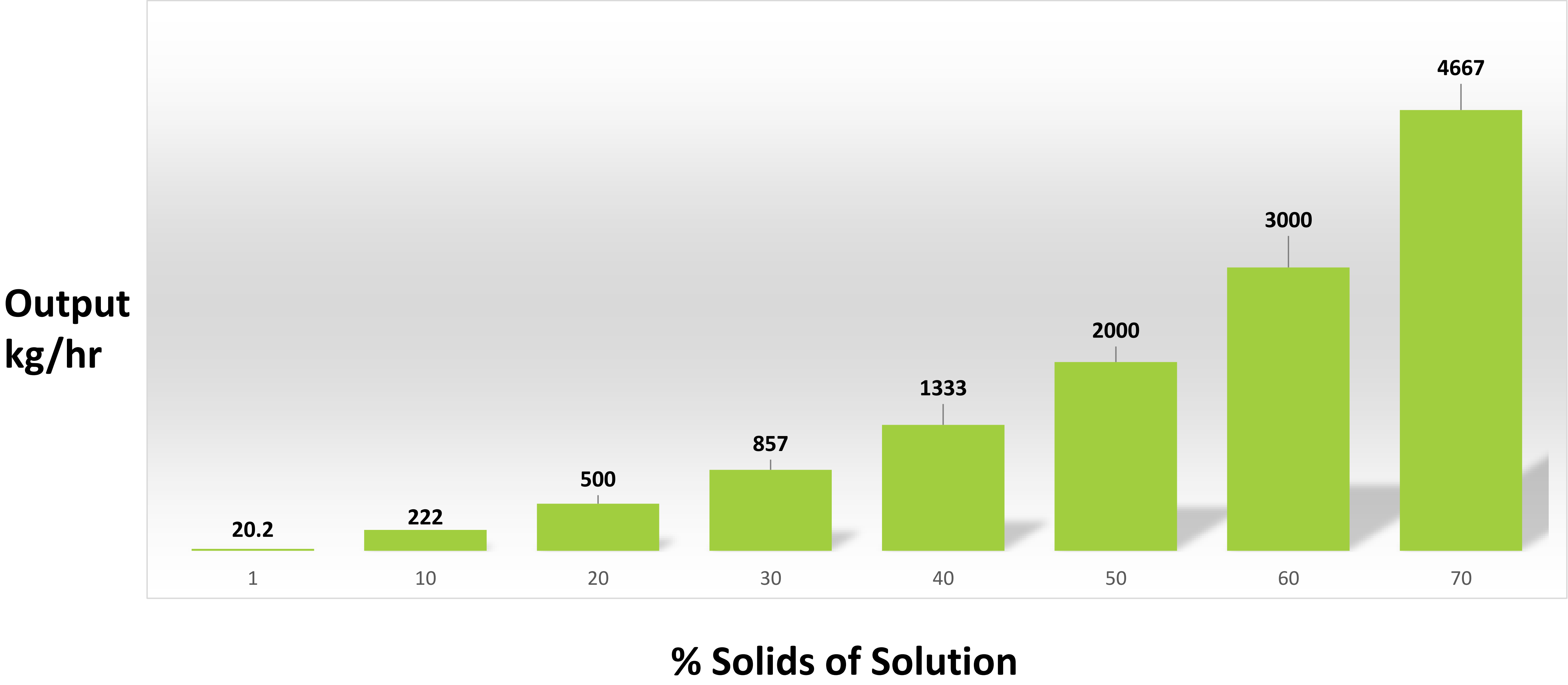
Economics of Particle Processing
Spray Drying - an Illustrative Example

Cost of Spray Drying Equipment: $30,000,000

- Process Parameters:
  - Input Solids
  - Drying Rate
- Yields: 98-99%
- Operating Income: $500-700/hour



Spray Drying Economics I



Spray Drying Economics II

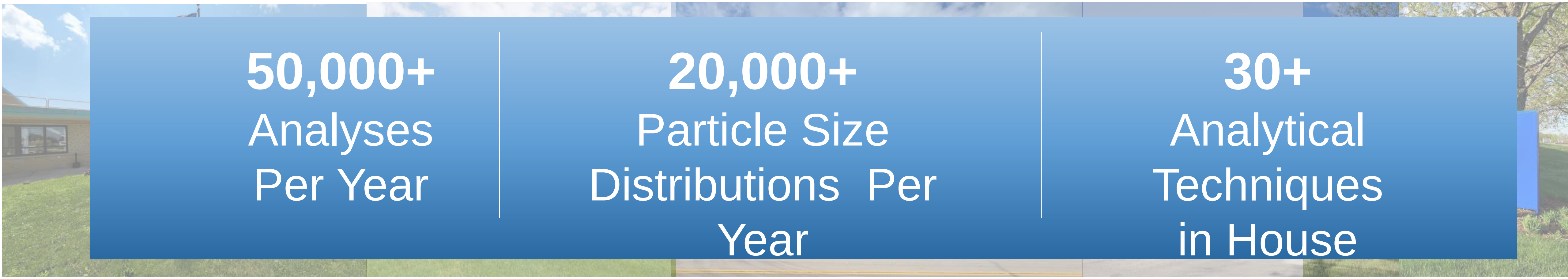
Assumptions

- 19%, 20%, and 21% solids
- 80%, 90%, and 95% Operational Time
- 2000 kg/hr Water Evaporation Rate

% Solids	Dried Product kg/hr	80% (7008 hr/yr)	90% (7884 hr/yr)	95% (8322 hr/yr)
19	469	3,286,752	3,697,596	3,903,018
20	500	3,504,000	3,942,000	4,161,000
21	532	3,728,256	4,194,288	4,427,304

The *AVEKA* Group – A Characterization Focused CMO

- Broad Range of Characterization Methods
- Characterization for Process Control, Quality, Materials, Understanding & Development
- Focus on Data Development & Analysis



50,000+ Analyses Per Year	**20,000+** Particle Size Distributions Per Year	**30+** Analytical Techniques in House

Particle Characterization at AVEKA

Particle Size Analysis

- Particles 1 nm to 2+ mm
- Particle size distribution (PSD)
- Sonic sieving
- Rototap

Electronic Imaging

- Optical microscopy
- Scanning electron microscopy (SEM)

Surface Area Analysis

True Density Analysis

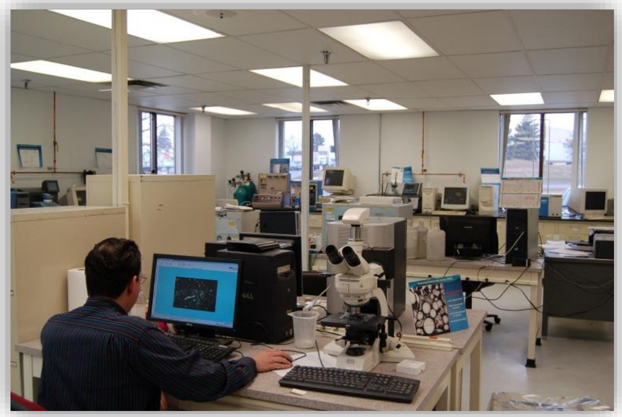
- Helium pycnometry

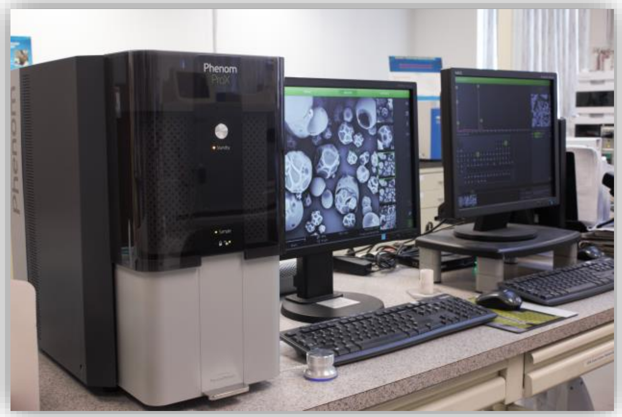
Formulation Analysis

- High performance liquid chromatography (HPLC)
- Thermogravimetric analysis (TGA)
- Spectrophotometer
- Differential scanning calorimetry (DSC)

Flow Characteristics

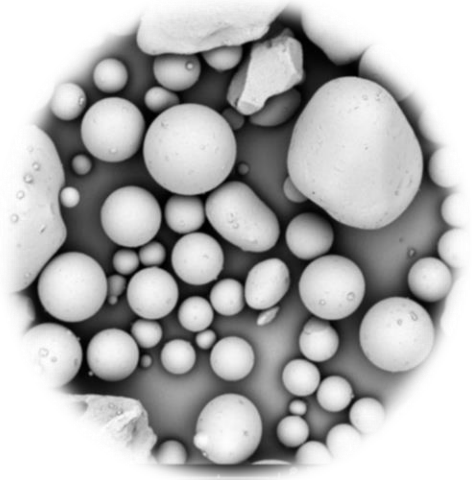
- Freeman FT4
- Zeta potential analysis (ZP)
- Rheological analysis
- Moisture and solids analysis (MSA)
- Karl Fisher





The Examples…Finally

Spray Drying Optimization	Density, Shape, Final Understanding
Classification Efficiency	Maximizing Yields
Grinding	Controlled D_{50} Characterization Methods
Beyond Particle Size Distribution	New Methods and Uses

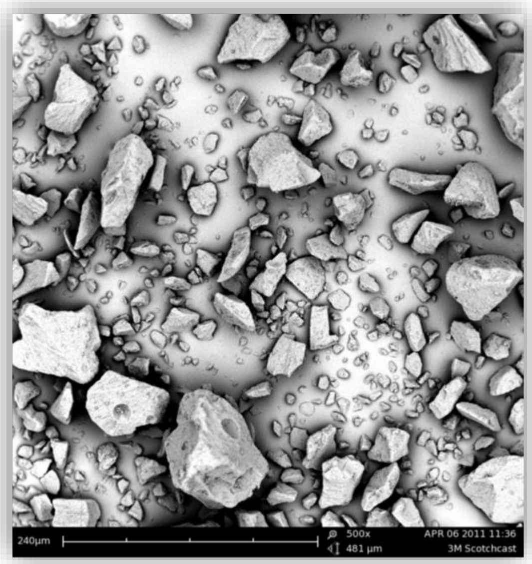


Statement of Need

How We Approached the Problem

What Went Right (or Wrong)

Opportunities



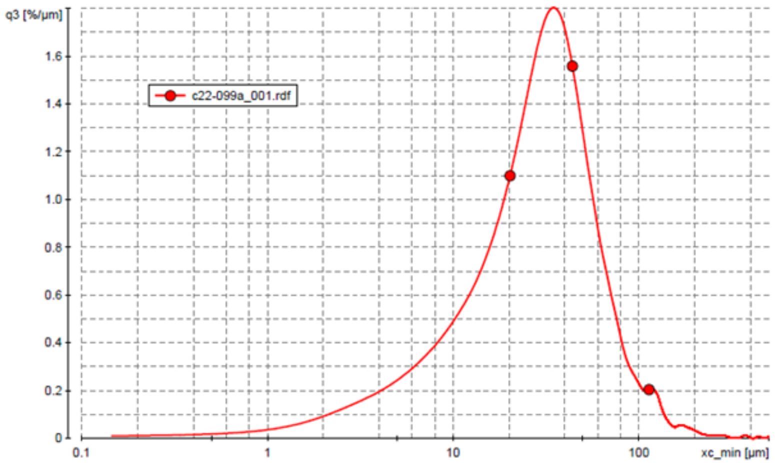
Spray Drying Density

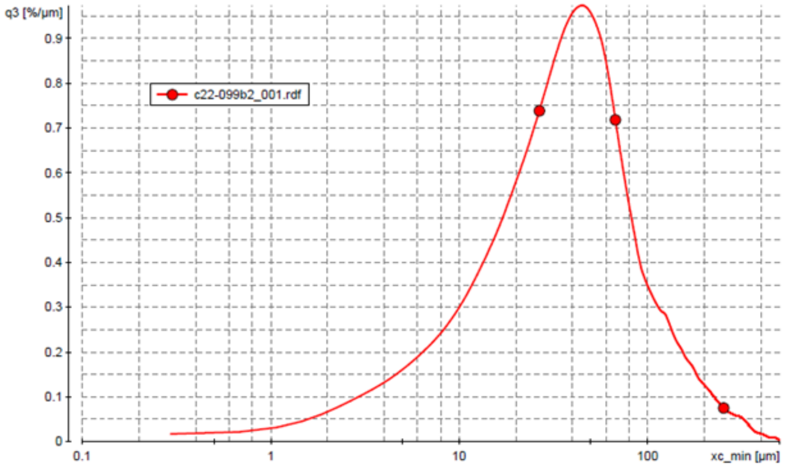
Statement of challenge

Depending on Dryer used Product Would Not Fit in Bag

- Sodium Acetate
- Large Scale Optimization Understanding

The Result



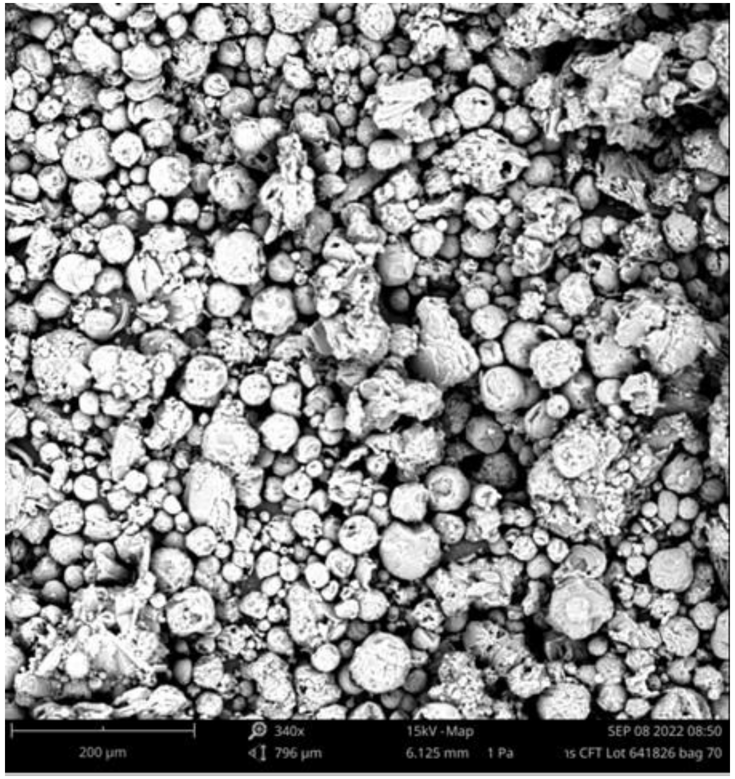


Dryer 1	
D_{10}	20.3 µm
D_{50}	43.8 µm
D_{90}	112 µm
Sphericity	0.895
Tap $Density_{0}$	0.44 g/cc
Tap $Density_{30}$	0.48 g/cc
Tap $Density_{60}$	0.50 g/cc

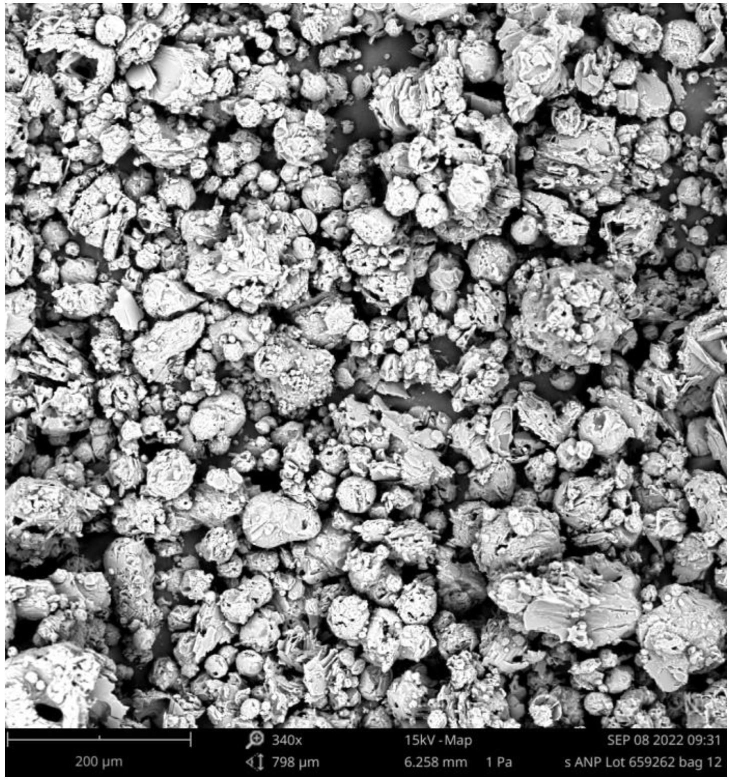
Dryer 2	
D_{10}	26.3 µm
D_{50}	65.1 µm
D_{90}	224 µm
Sphericity	0.857
Tap $Density_{0}$	0.35 g/cc
Tap $Density_{30}$	0.39 g/cc
Tap $Density_{120}$	0.40 g/cc

SEM Images

Spray Dryer 1



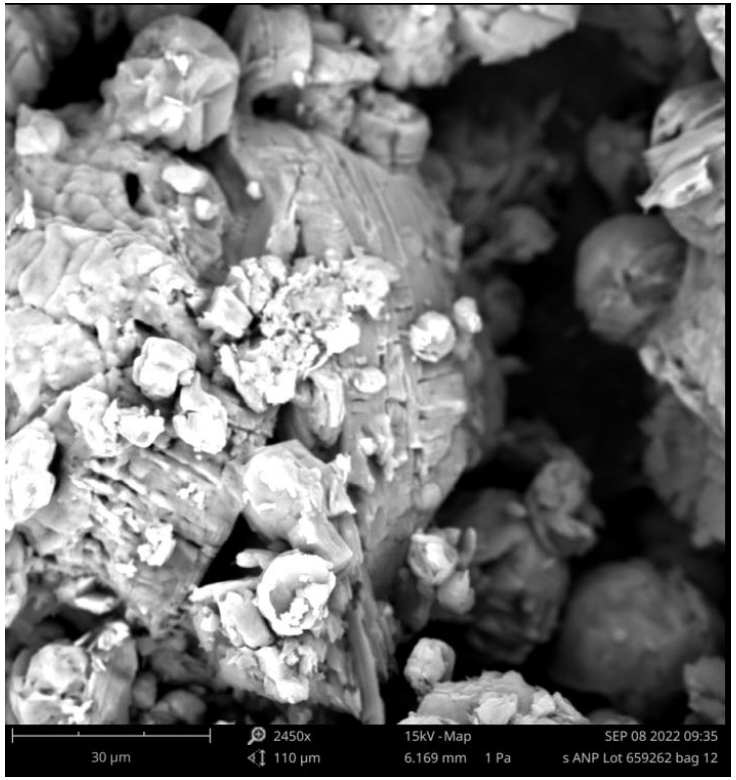
Spray Dryer 2



High Magnification SEM Images

Spray Dryer 1

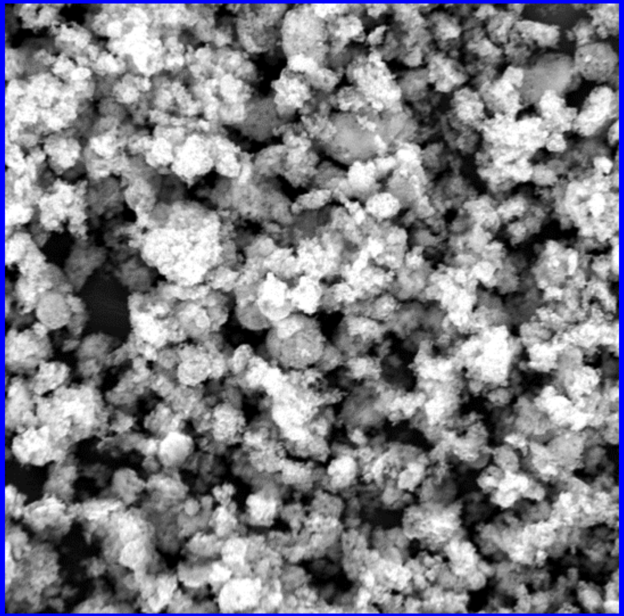
Spray Dryer 2



Spray Drying Spherical Powder

Statement of challenge

Spray Drying produced correct size, but not desired sphericity

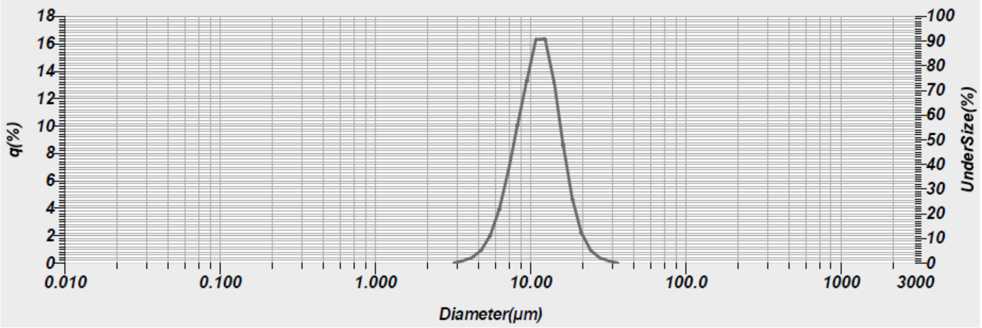


The Result

Spay drying after bead milling produced the desired material

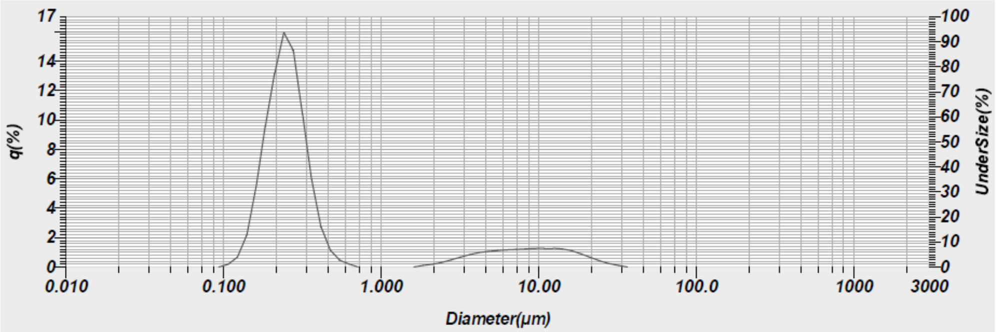
Starting Material

Mean PSD = 11.7 µm

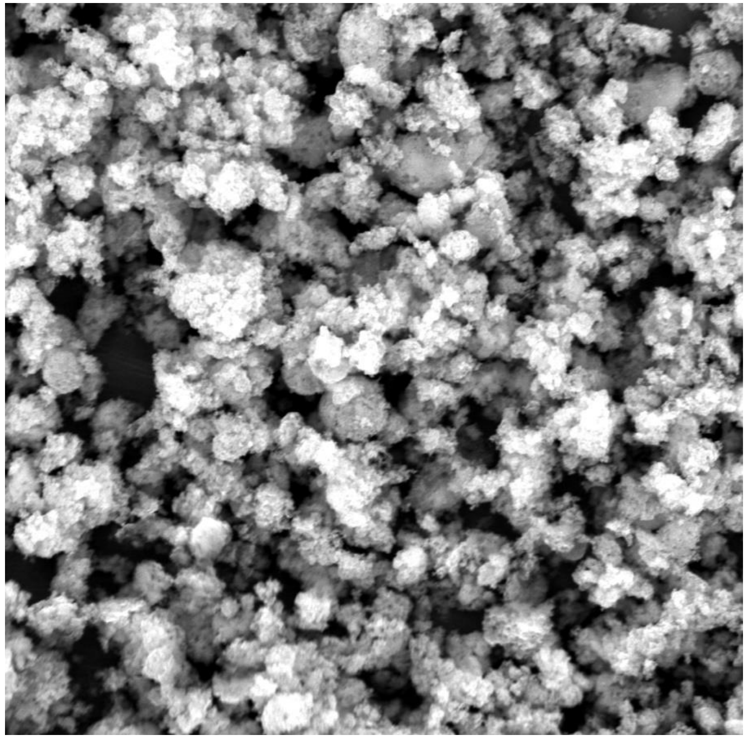


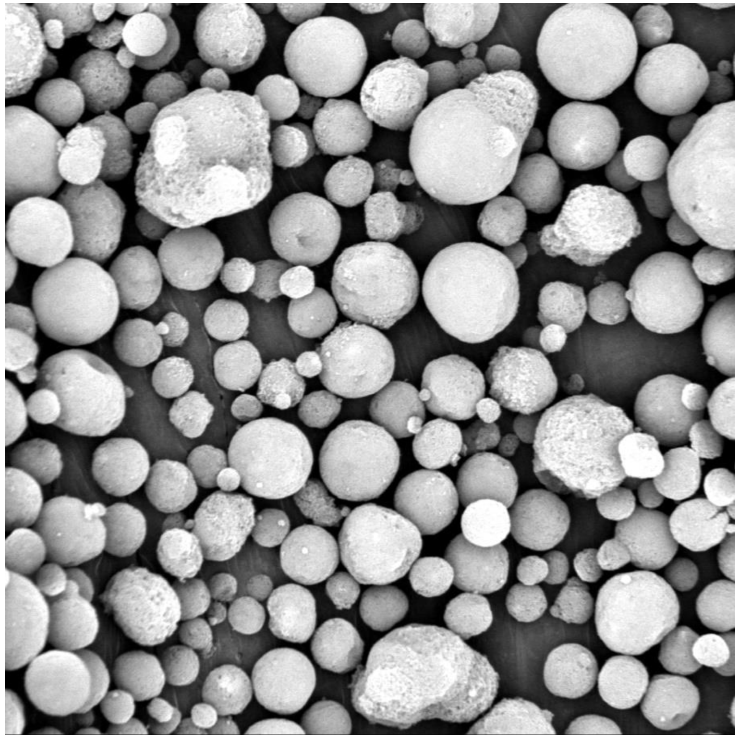
Milled Material

Mean PSD = 1.9 µm



SEM Spray Drying Milled Starting Material





SEM (5000x) of spray-dried silica without (left) and with (right) prior bead milling

Spray Drying Density

Statement of challenge

Maximize Tap Density With Small Changes in PSD

- Inorganic Material
- Spray Drying Methods

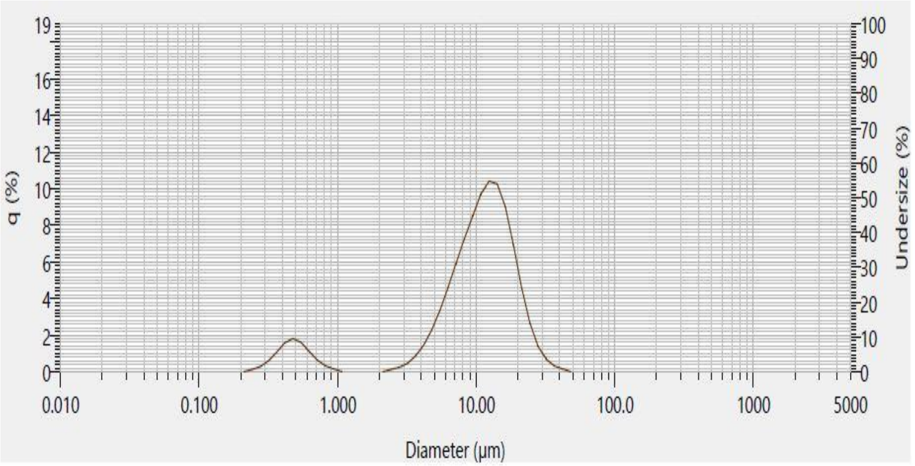
The Result

Most process changes did not work. % Solids in slurry did work.

Slurry 23% Solids

Mean PSD = 11.4 µm

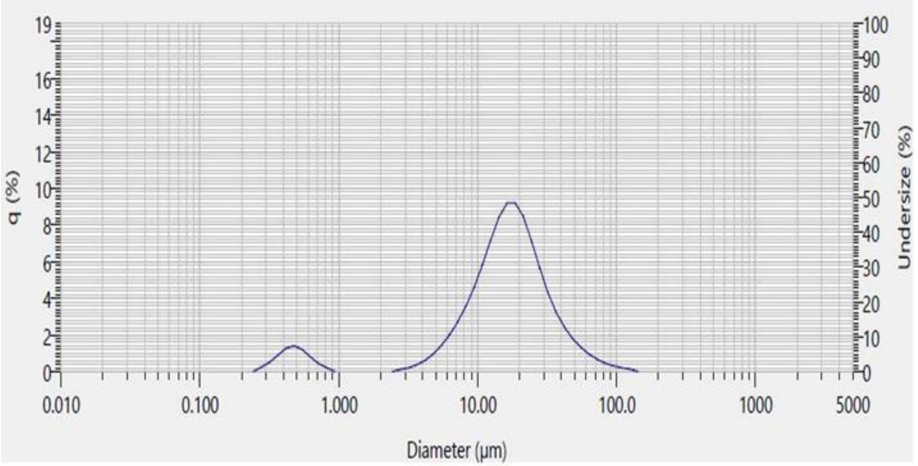
Tap Density = 0.28 g/cc



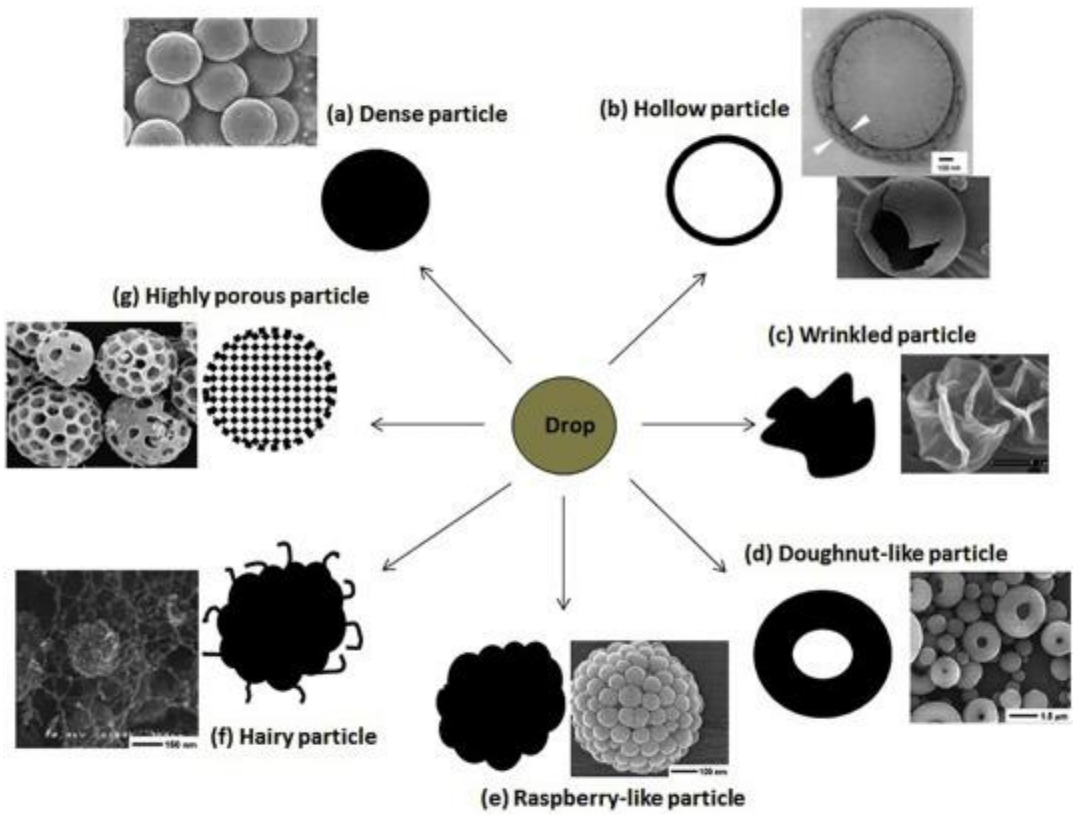
Slurry 30% Solids

Mean PSD = 19.5 µm

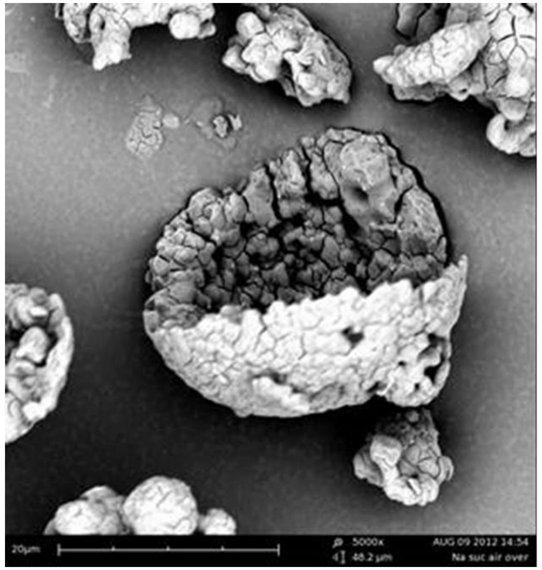
Tap Density = 0.37 g/cc

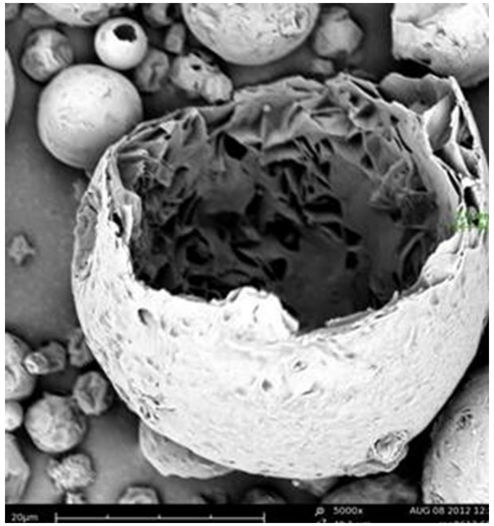


What is Really Happening Here?



De Souza Lima, Powder Technology 359, 161-171, 2020.





Characterization and Analysis can be Misleading Without an Understanding of the Process and the Materials

Classification Optimization

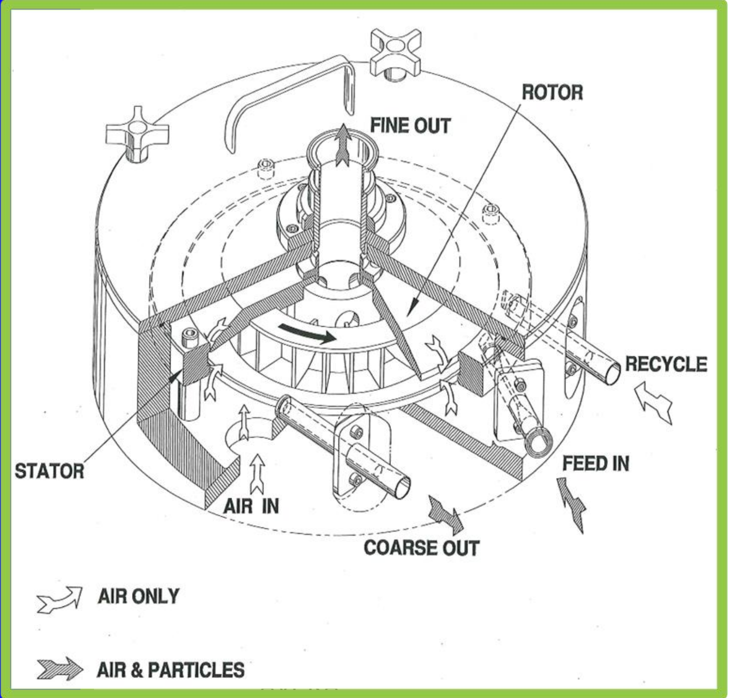
Statement of challenge

Maximize yields during classification

- Glass beads
- Air classified

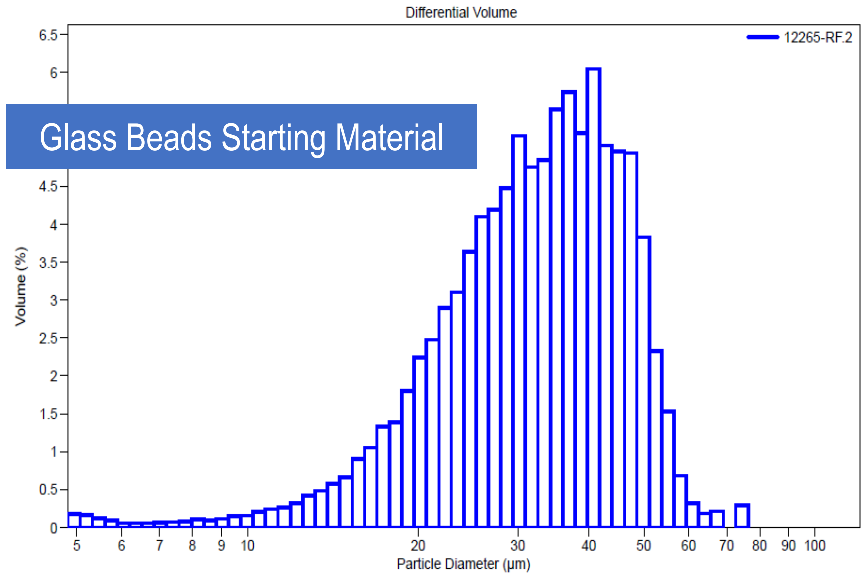
The Result

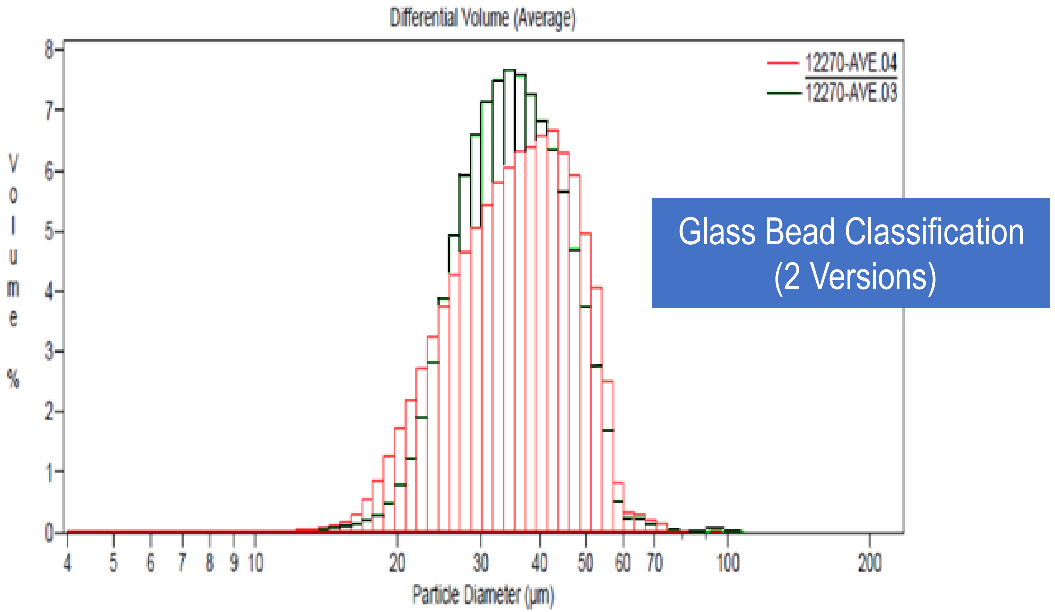
Small cut points had huge yield results



Glass Bead Classifications & Yields

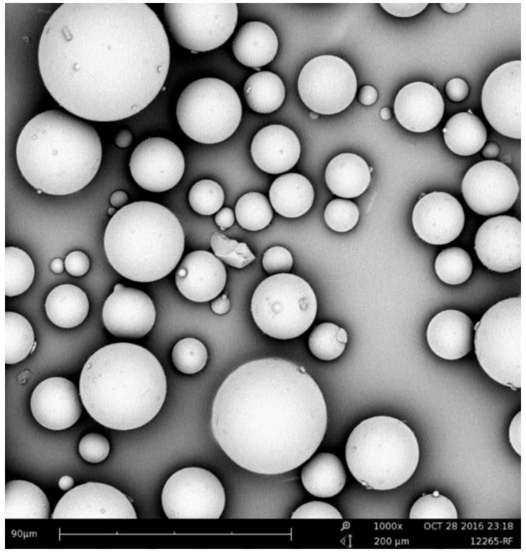
Objective: Increase yield after classification





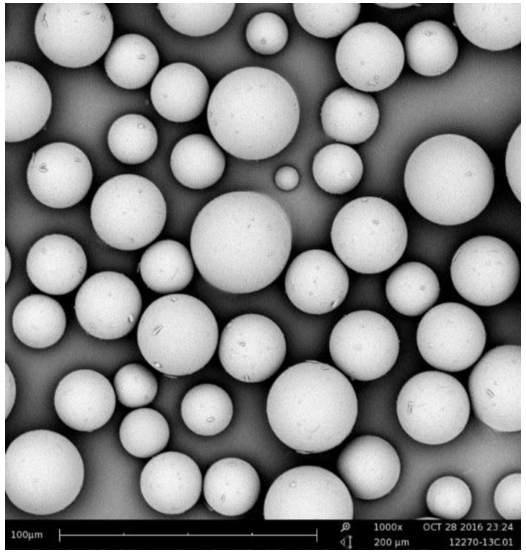
	<10%	<25%	<50%	<75%	<90%
Starting Glass	19.01 μm	25.08 μm	33.10 μm	41.56 μm	48.29 μm
Classification (red)	23.95 μm	28.22 μm	35.82 μm	43.94 μm	50.41 μm
Classification (green)	24.95 μm	29.05 μm	34.89 μm	41.96 μm	48.47 μm

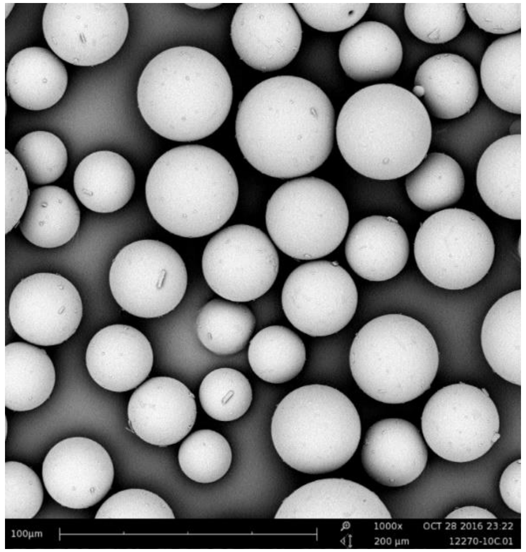
Glass Bead Classification & Yields



Starting Beads

Broad Cut (24-50 µm)
73% Yield



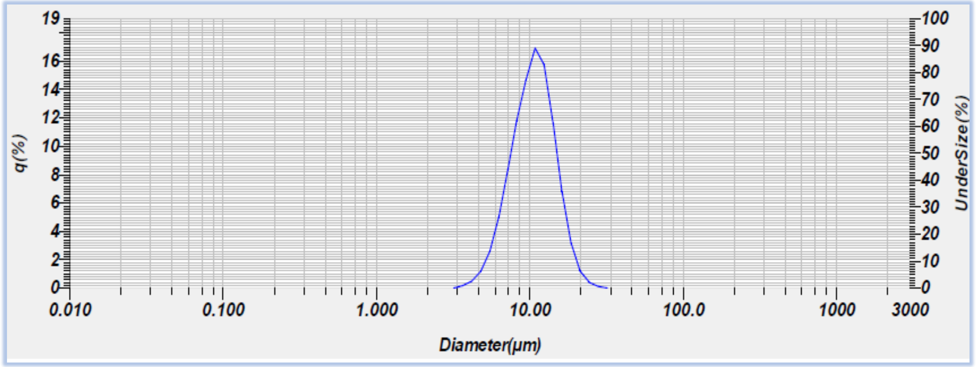


Narrow Cut (25-48 µm)
43% Yield

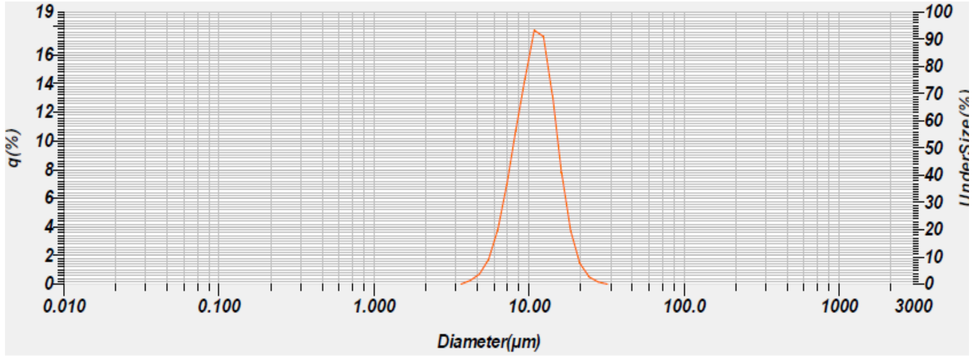
Opportunity: Increased yields using characterization

Two Step Classification - Silica

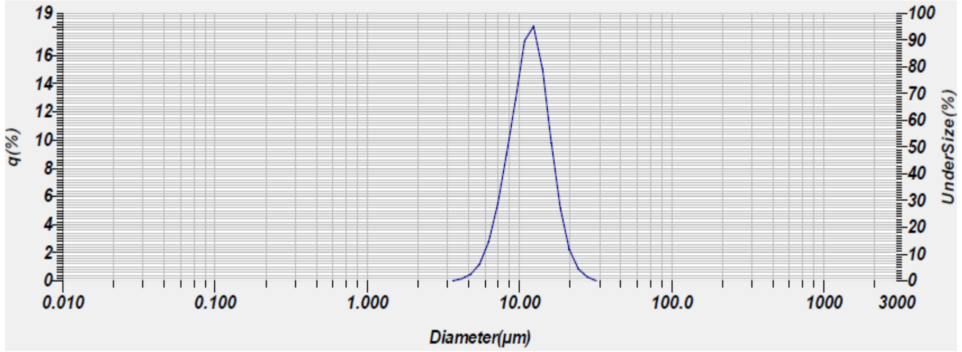
Raw Material - 10.97 µm



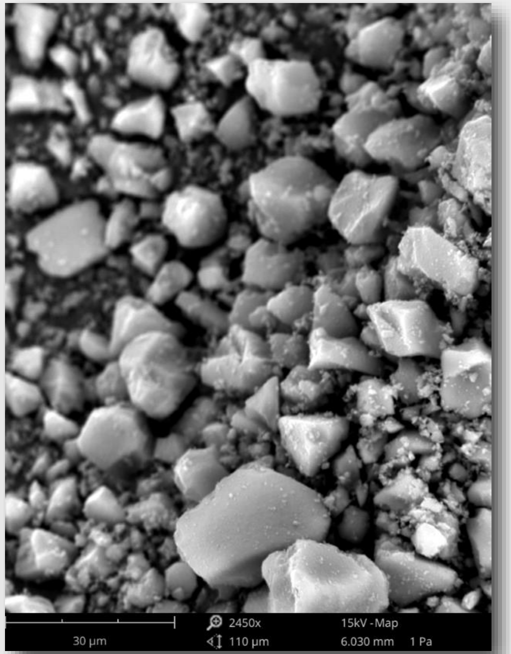
First Pass Classification – 11.43 µm



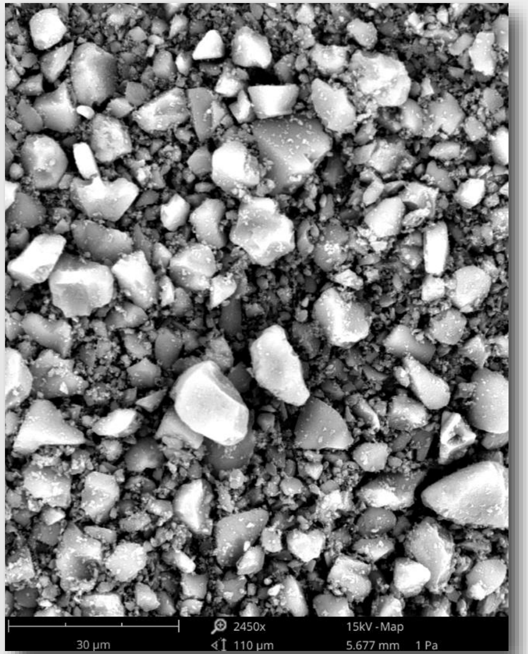
Second Pass Classification – 12.08 µm



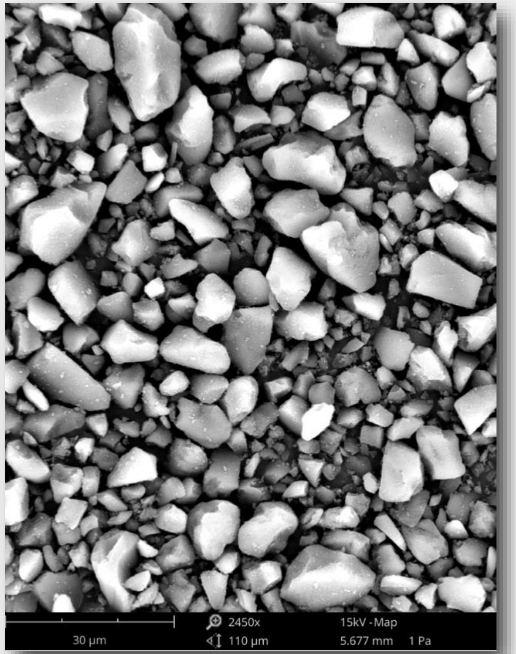
Two Step Classification - Silica



Raw Material
10.97 µm



First Pass Classification
11.42 µm



Second Pass Classification
12.08

Milling and Classification Optimization

Statement of challenge

Control PSD to enhance flow for Additive Manufacturing

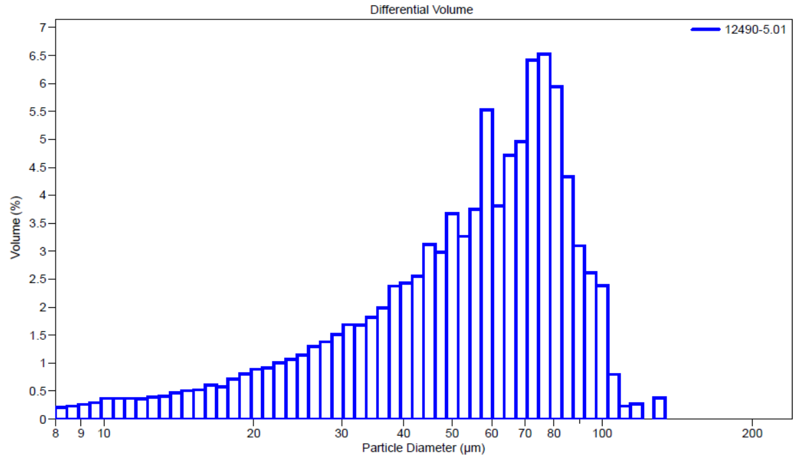
- ➢ Polymer Powder
- ➢ Jet Milled
- ➢ Air classified

The Result

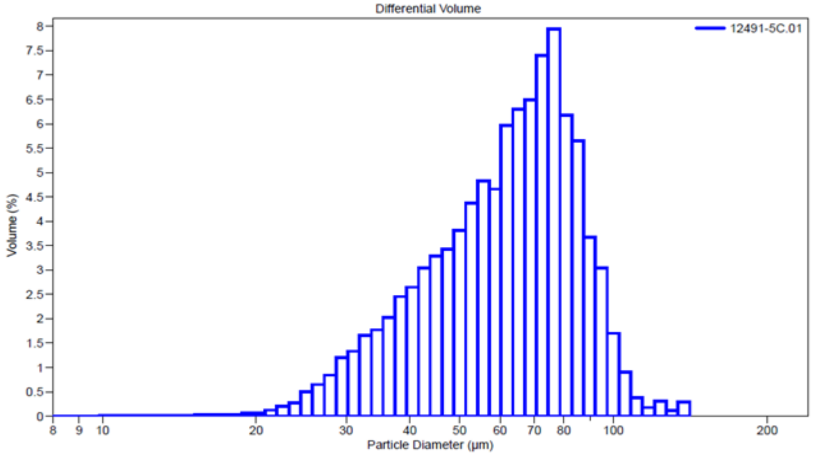
Jet Milling step followed by air classification step. Particle size measurement using number not volume distribution

Jet Milling and Classification of Polymer

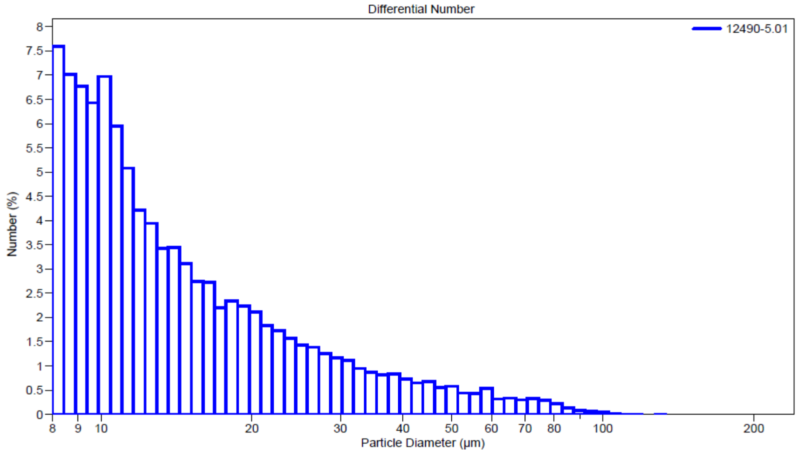
Jet Milled Polymer – Volume Distribution



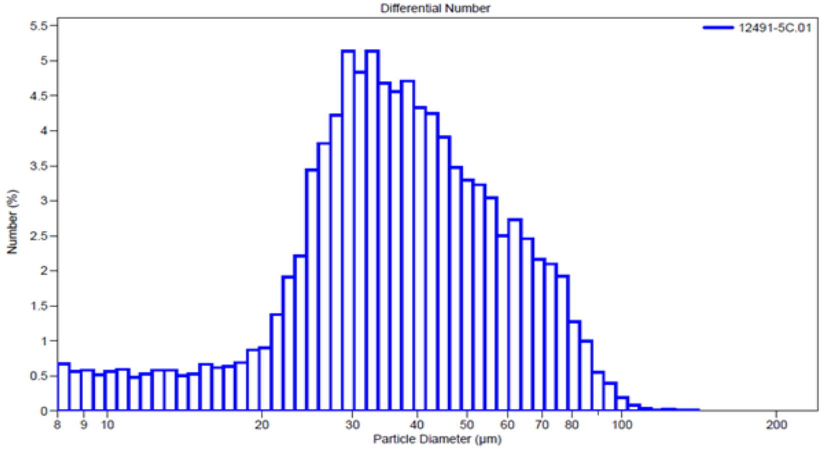
Jet Milled and Classified Polymer – Volume Distribution



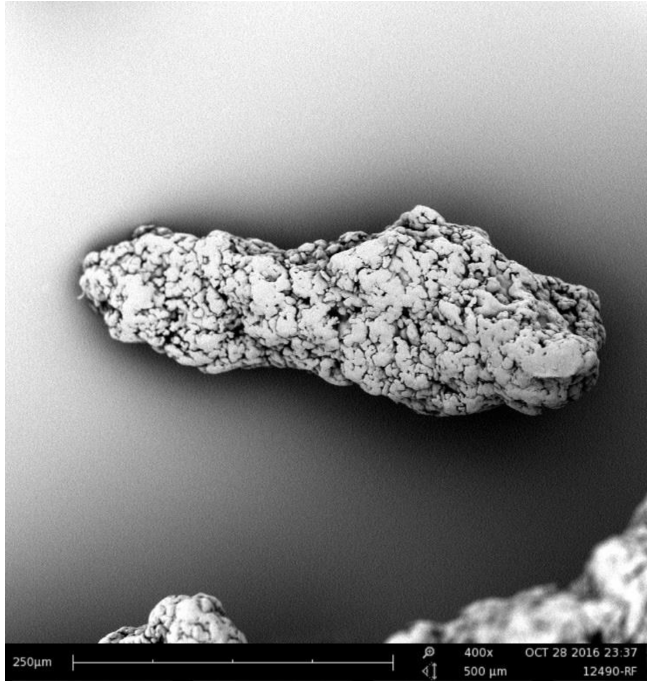
Jet Milled Polymer – Number Distribution



Jet Milled and Classified Polymer – Number Distribution

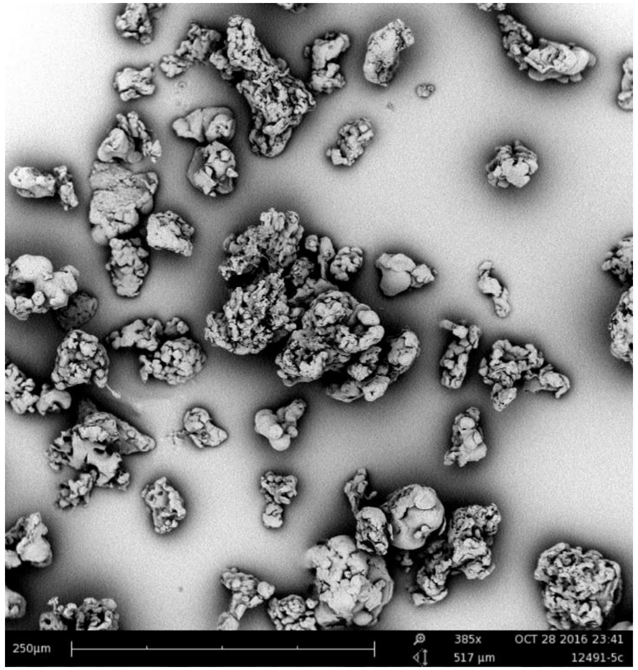


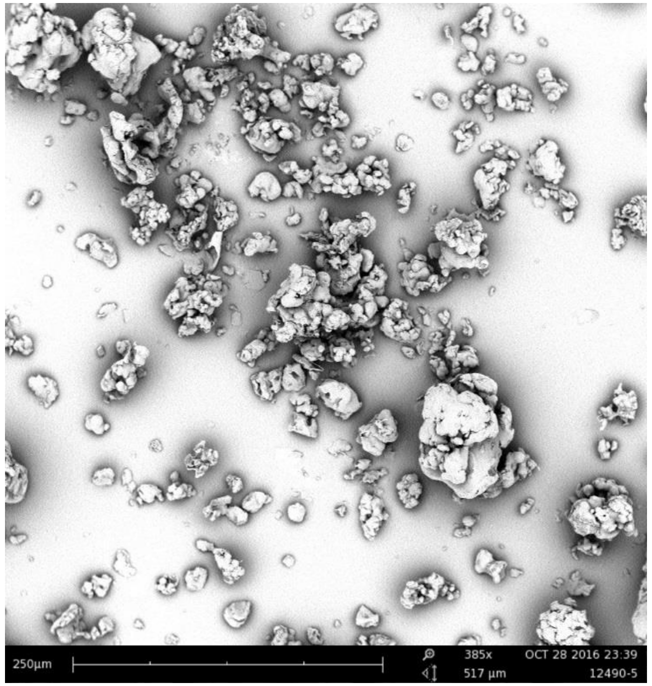
Jet Milling and Classification of Polymer



Starting Material

Jet Milled and Classified Polymer





Jet Milled Polymer

Grinding Optimization

Statement of challenge

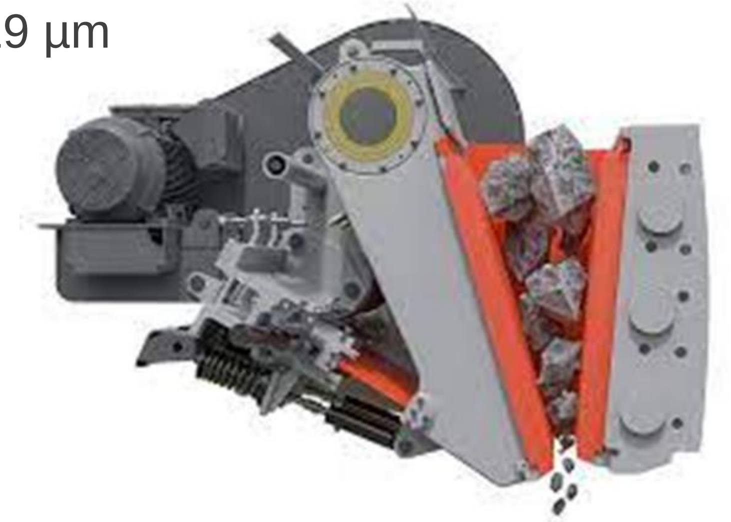
Tighten Distribution and Reduce Processing Time of Ceramic Substrates

- Multi-step grinding
- Grinding aid addition

The Result

3x reduction in grinding time

D_{50} tightened from 15-35 µm to 17-19 µm



Multi-Step Grinding Line

- Tote Tipper Feeder
- Komar Industries auger crusher
- Pneumatic conveying to screener
- Ball Mill and final screening

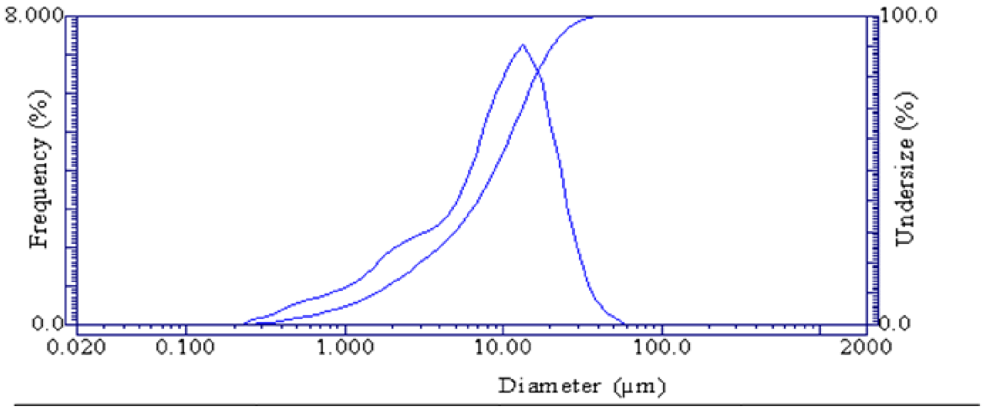
Multi-Step Grinding Line

2003	D_{50} = 15-35µm	Crushing and Ball Milling
2006	D_{50} = 18-24µm	Hammer and Ball Milling
2013	D_{50} = 16-19µm	Crushing, Hammer Milling and Ball Milling
2015	D_{50} = 17-19µm	Added Grinding Aid

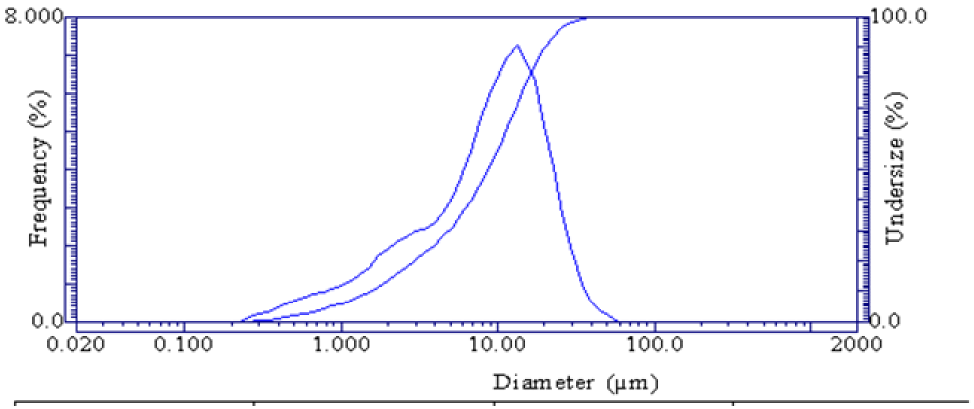
Opportunity: Process and yield optimization

CAVEAT, CAVEAT, CAVEAT

You are Never Really Done



PSD of Sample that Passed Final Requirement



PSD of Sample that did not Pass Final Requirement

Small number of large particles not measured with laser diffraction method

Wet Screening with visual inspection required to count large particles

Process and Material Unknowns

Statement of challenge

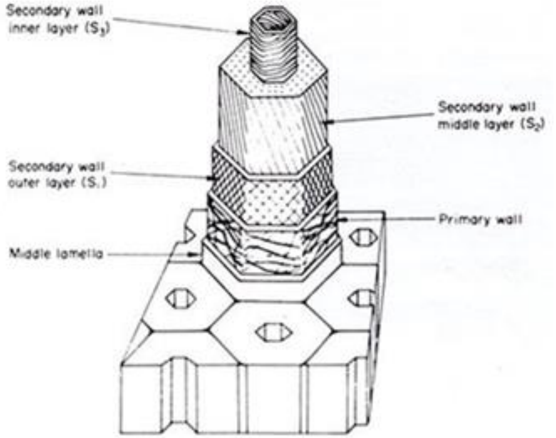
Produce Cellulose Fiber with High Water Holding Ability (20X weight)

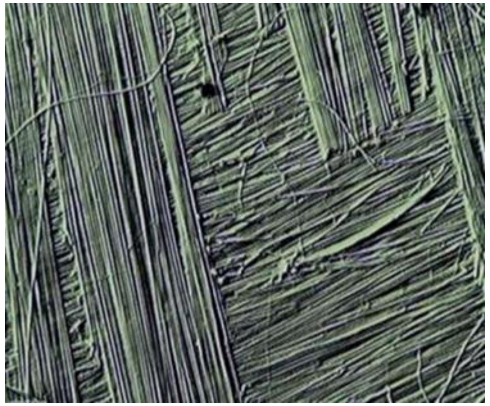
- Removal of lignin and hemicellulose from corn bran
- Caustic Washes and Concentration
- Drying

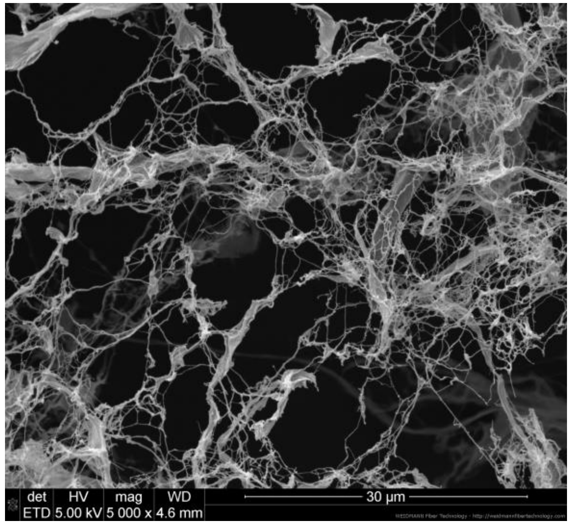
The Result

Process not optimized as received from customer

Desired product not purified cellulose, but rather microfibrilated bran





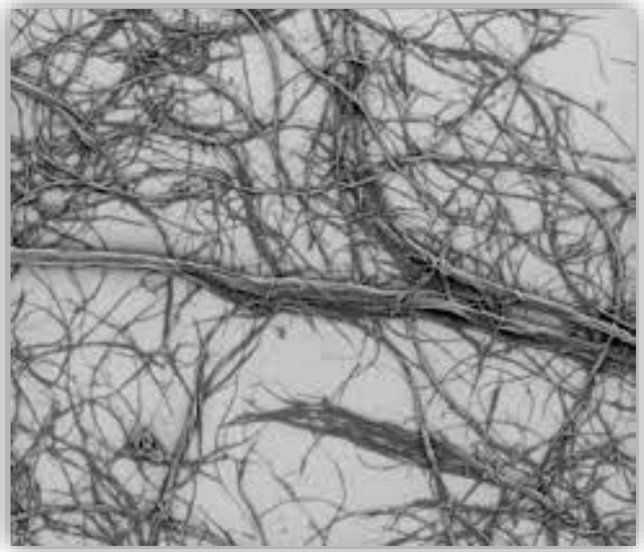


What Went Wrong

- Yields were poor
- Process Incredibly Inconsistent
- Water Holding Results were inconsistent

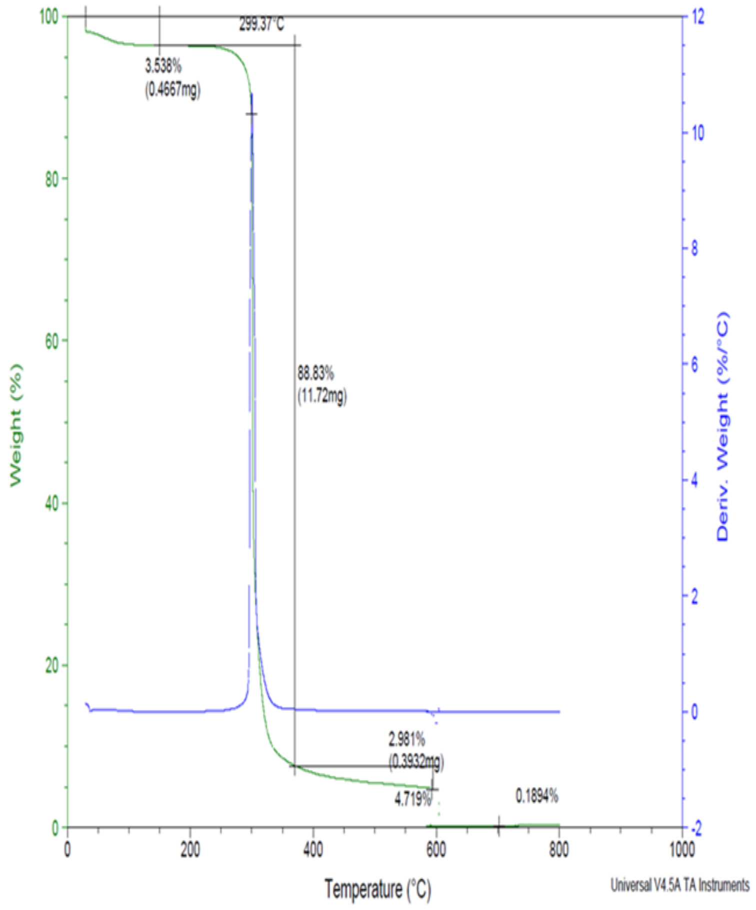
Solution

- Analyze
- Understand

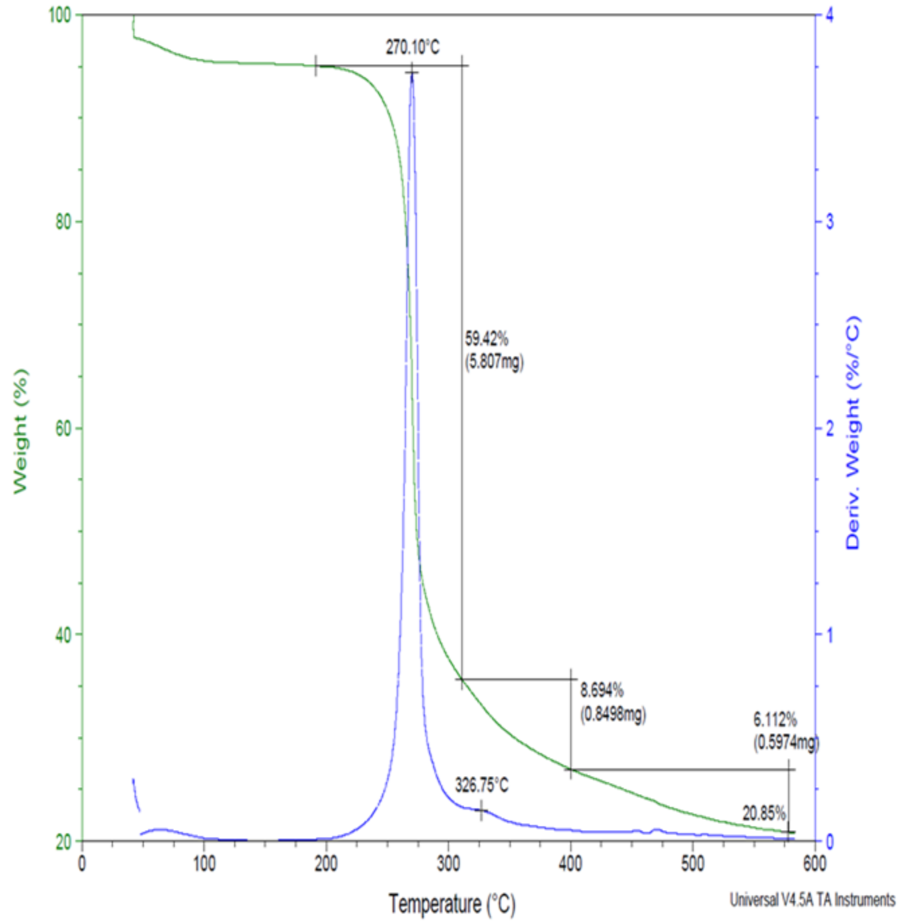


TGA of Cellulose and Hemicellulose

Wood Cellulose

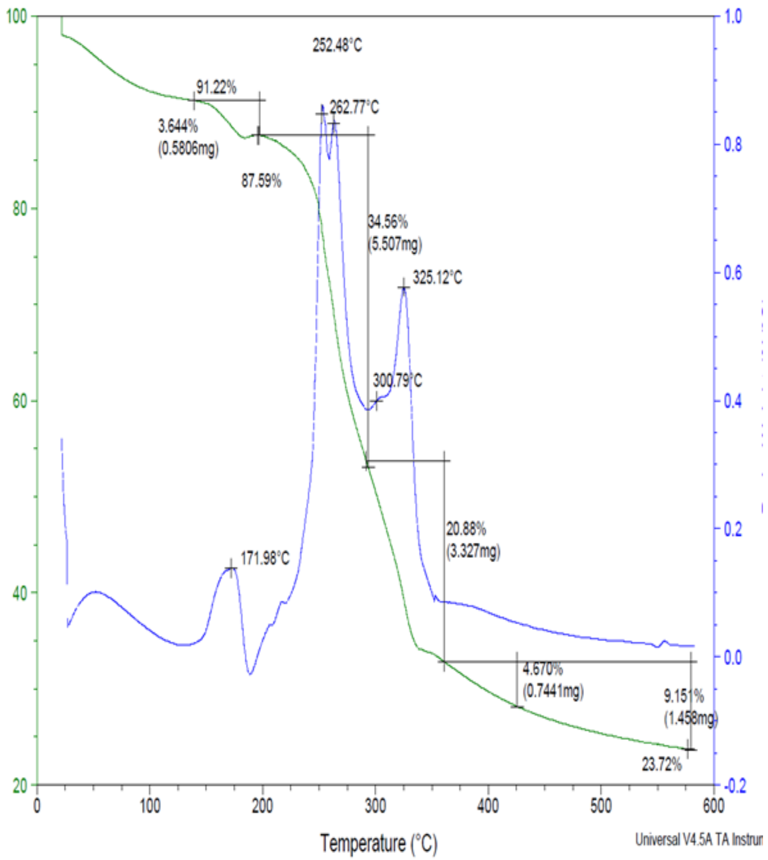


Hemicellulose

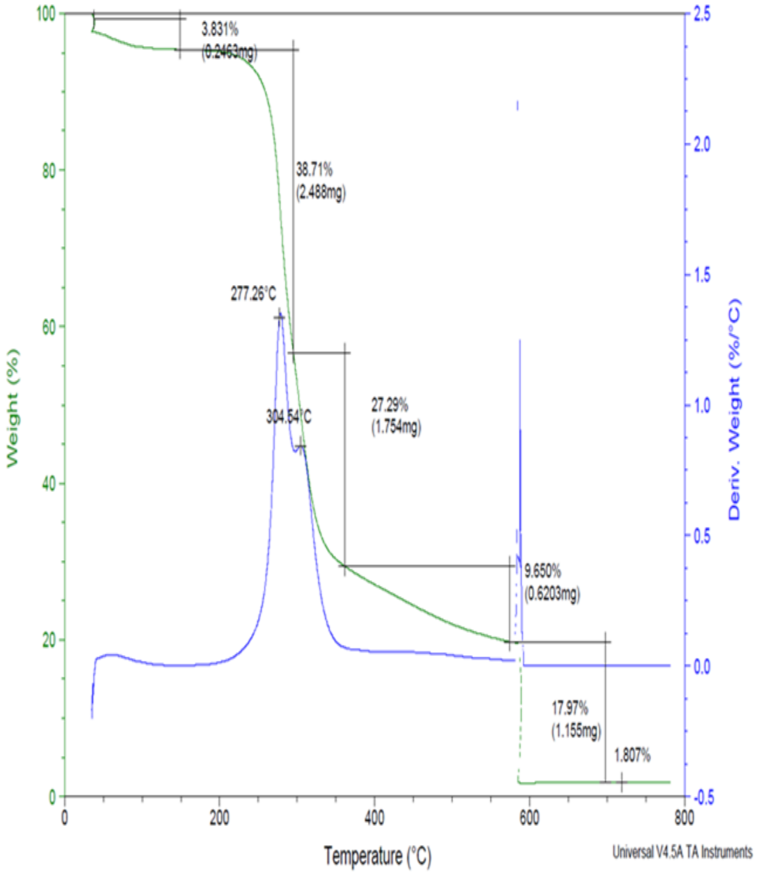


TGA of Corn Bran and Purified Cellulose

Corn Bran



Processed Cellulose



New Characterization Method Required for Process Understanding

Process Scale-Up Analysis

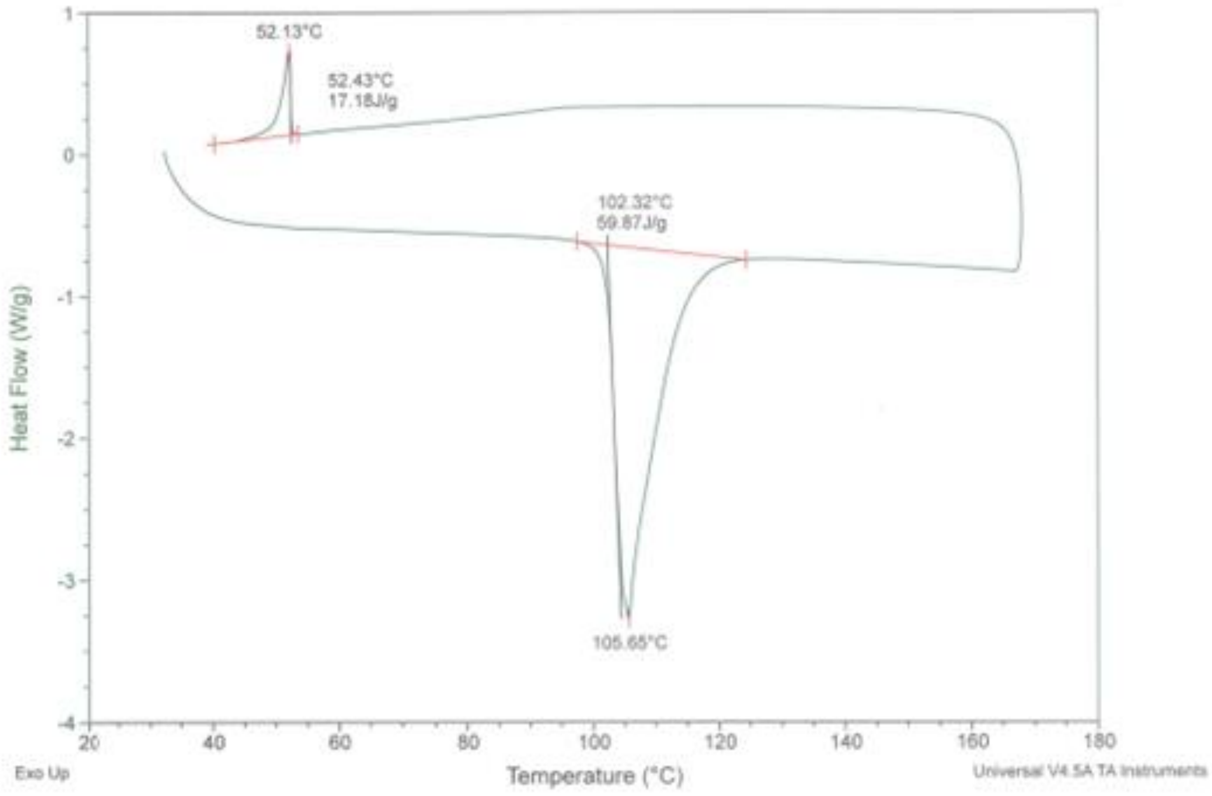
Statement of challenge

Define equipment parameters for heating

- Organic Materials
- Alloyed
- Melted and atomized

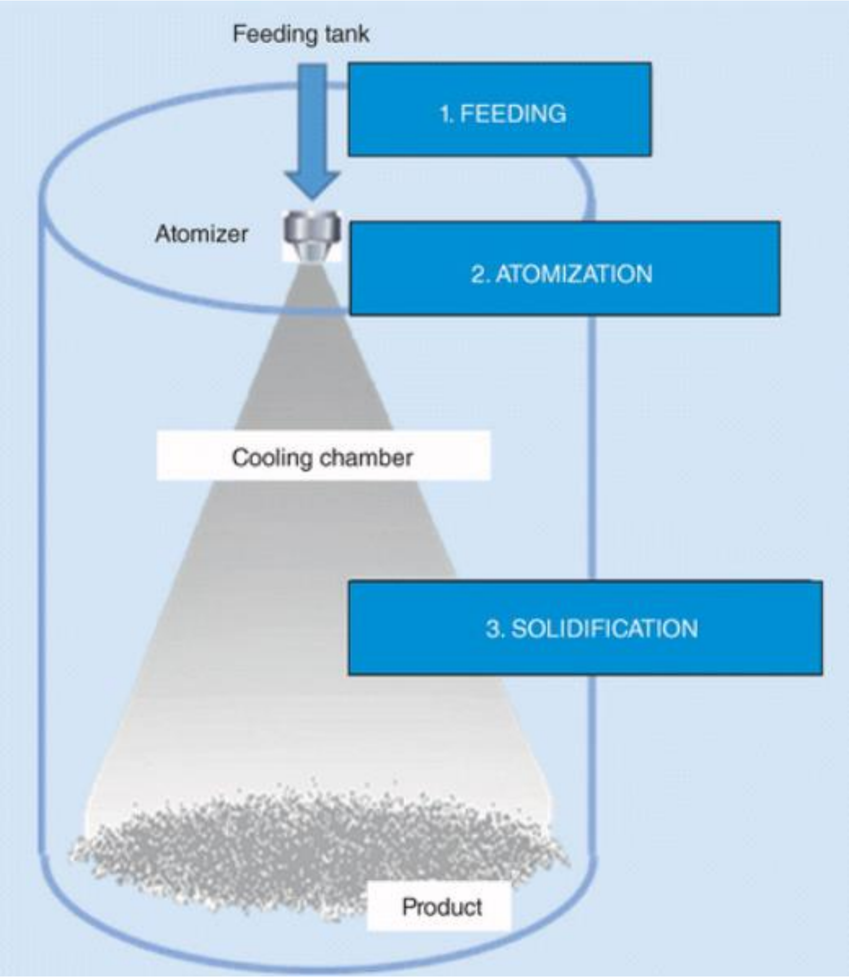
The Result

Scale-up of prilling process using DSC (Differential Scanning Calorimetry)

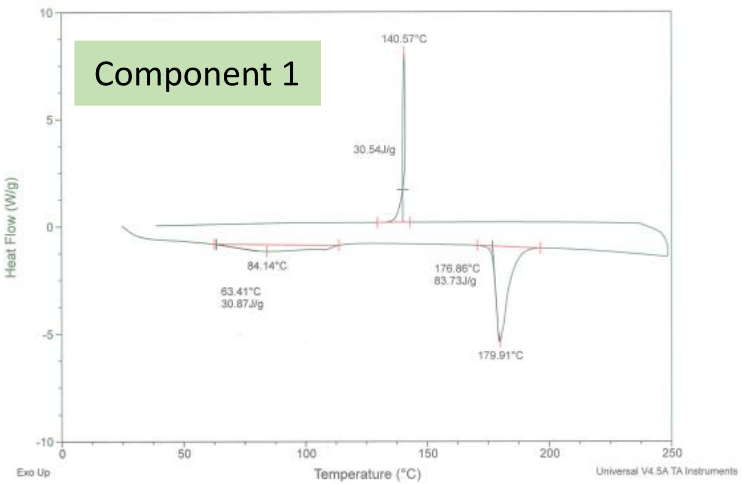


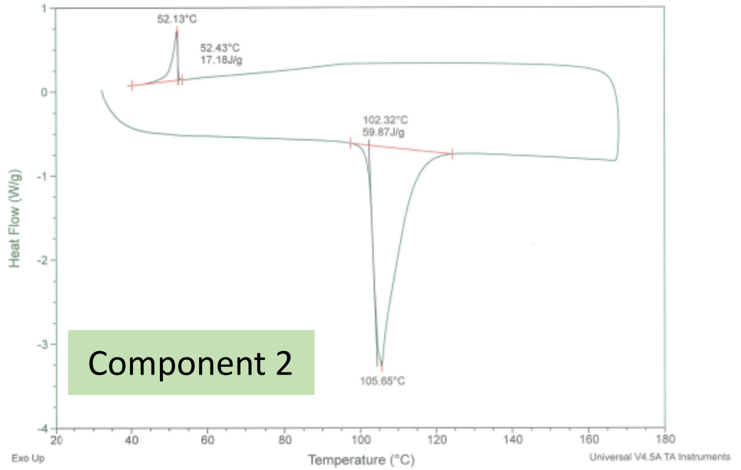
Determination of Process Heating Requirements using DSC

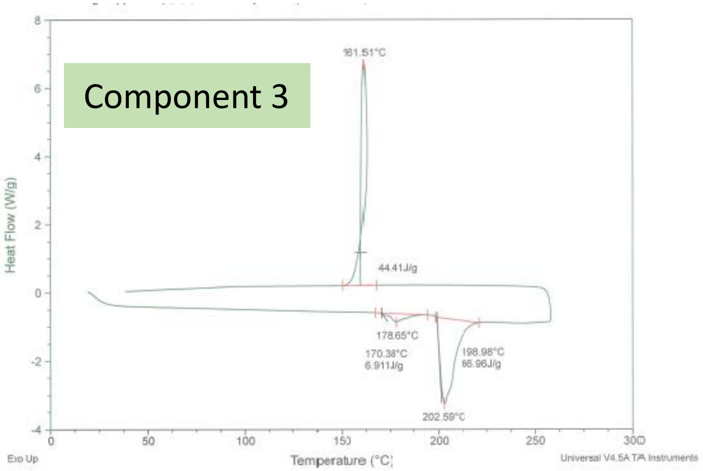
Process for reacting & prilling a plurality of components to form an alloy

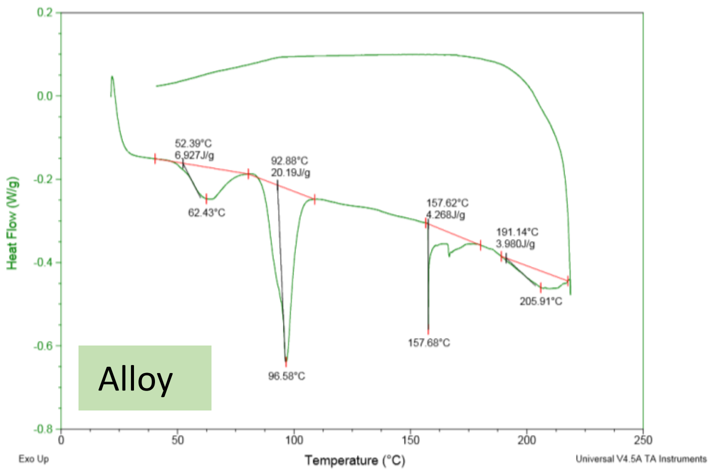


Representation of Prilling Process

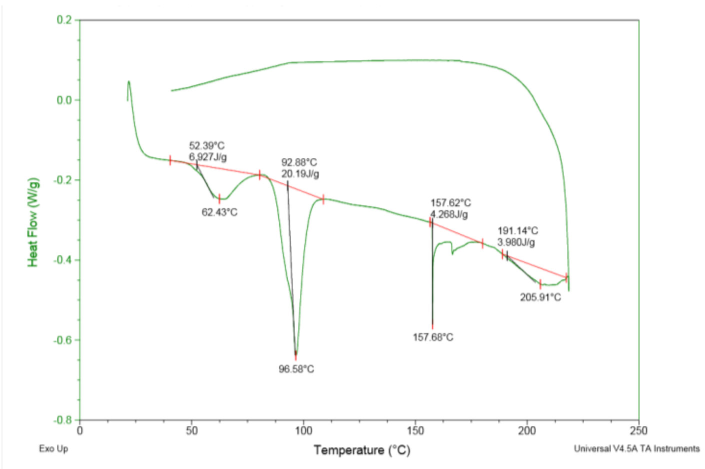




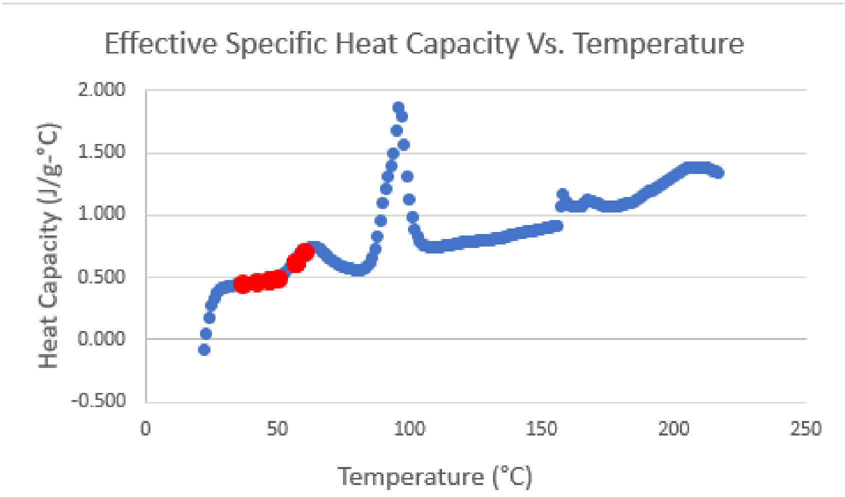




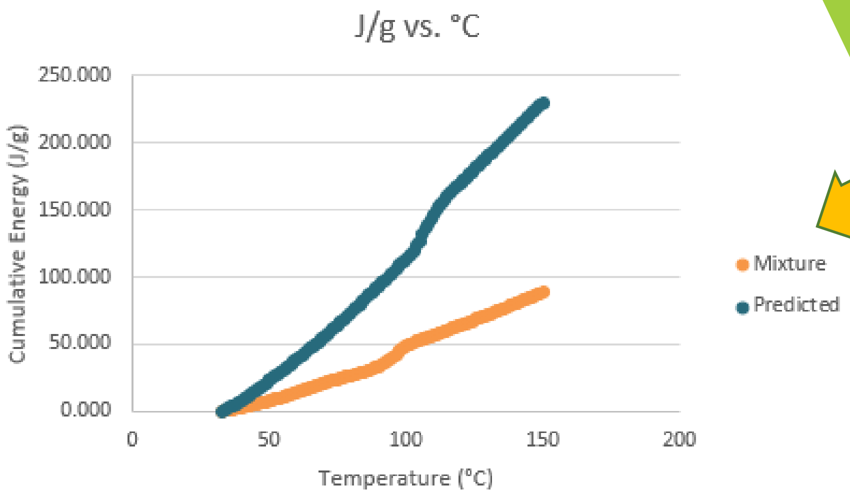
DSC data to calculate heating requirements
For each component and the alloy



Calculate effective specific heat capacity for each components plus the alloy.



Generate curves of cumulative energy input.



It will require significantly less heating power to heat the reacted alloy than predicted by the heat capacities of the individual reactants.

Characterization and Particle Processing

- It’s always the economics
- Small changes in processing can have profound effects on profitability
- How do you optimize your processes or develop new materials without extensive characterization

Thoughts

Characterization

- Never use only one technique
- Always consider your process and the materials in your analysis
- Images are great, but they can be misleading

E. W. Merrow Chem Eng 24, 89-92, 1988

“Estimating Startup Times for Solids-Processing Plants”

Does industrial, academic, and funding management understand the critical value of particle processing in profitability and how characterization improves profitability?

Where We Go from Here?

Horiba/*AVEKA* Webinars - 2023 (proposed)

- Spray Drying
- Agglomeration
- Industrial Challenges of Particle Processing
- Contract Manufacturing
- Jet Milling

Do you have any interests that we can address?

International Fine Particle Research Institute (IFPRI)

- New Merrow Report Commissioned by IFPRI
- Current and Past Projects on Most Areas Mentioned in this Talk
- Forum for Learning Best Practices from Some of the Best Practitioners

IFPRI

Summary

- **We are in the broadest and most exciting field in the world**
- **This is a challenging field, but characterization can help with understanding and profitability**
- **Let us know if we can help**

***AVEKA* Group**

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