iFormulate

Introduces...

David York on Spray Drying for Formulations

Jim Bullock & David York
26th February 2016

In association with

UNIVERSITY OF LEEDS

Spray Drying and Atomisation of Formulations, 12th-14th April 2016

www.engineering.leeds.ac.uk/short-courses/particles/SprayDrying.shtml
Overview:

1. Why Choose Spray Drying?
2. Spray Drying – The Basic Science
3. Spray Drying – The Hardware
4. Material and Particle Engineering
5. Summary and Learning More

This webinar is being recorded and will be made available.

The audience is muted and you may ask questions using question function in GoToWebinar.

This webinar will last about 30 minutes.

Your Speakers

Dr Jim Bullock
iFormulate Ltd

Professor David York
University of Leeds
A Little About iFormulate

A company founded in 2012 by two experienced industry professionals...

Combining diverse experiences, knowledge and wide range of contacts:

...polymers, materials science, chemistry, imaging, dyes, pigments, emulsion polymerisation, biocides, anti-counterfeiting, environmental, formulation, consultancy, marketing, business development, strategy, regulatory, training, events, R&D, innovation

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1. Why Choose Spray Drying?

Start with drying in general...

- usually comes at the end of a production process
- entails removal of solvent, often water
- can reduce transportation cost
- can make materials more suitable for handling
- helps avoid moisture that could lead to corrosion
- can be used to mix ingredients in solution or slurry and so make consistent products
- can increase shelf life of products
Drying and Other Things

• If the **only thing** you want to do is to dry your product then probably don’t choose spray drying
• But getting a dry product is probably not the only thing you’re trying to do

Some Other Things You Might Want From Drying
• A “gentle drying” process.
• Good yield and economics
• Product that flows well or product that isn’t dusty
• A product with fine particles – or large particles
• A product with smooth particles – or rough particles
• A product with strong particles – or weak particles
• Particle engineering
What Is Particle Engineering?

• Designing and creating the particles you want to have at the end of the process
• Giving the final product the desired properties
• By controlling:
  – Particle size and size distribution
  – Particle morphology
  – Surface roughness of particles
  – Mechanical strength of particles
  – Internal structure of particles
## So You Want To Dry: What Choice Have You Got?


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<th>Drying Type</th>
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<td>Ribbon Dryers</td>
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<td>Through Air Dryers</td>
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</table>
# Focus on a Few Types of Dryers

<table>
<thead>
<tr>
<th>Dryer Type</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tray/Shelf Dryer</td>
<td>Low losses, versatile, small batches, uniform heating</td>
<td>Slow manual load/unload. Little opportunity for particle engineering</td>
</tr>
<tr>
<td>Rotary Dryer</td>
<td>Can use for pastes, wet solids</td>
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</tr>
<tr>
<td>Freeze Dryer</td>
<td>Good for heat sensitive materials. Porous redispersible product</td>
<td>Slow, complex, expensive. Limited opportunity for particle engineering</td>
</tr>
<tr>
<td>Microwave Dryer</td>
<td>Dry sensitive materials, bulk, viscous, rapid</td>
<td>Less suitable for larger batches. Little opportunity for particle engineering</td>
</tr>
<tr>
<td>Vacuum Dryer</td>
<td>Low risk of oxidation, heat damage. Small batch sizes</td>
<td>Less suitable for larger batches. Little opportunity for control and particle engineering</td>
</tr>
<tr>
<td>Fluid Bed Dryer</td>
<td>High rate if drying. Uniform drying from particle surface. Free flowing particles</td>
<td>Particles at least 250µm to form controllable fluid bed. Process may break down particles and form dust.</td>
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<tr>
<td>Spray Dryer</td>
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### Typical Applications of Spray Drying

<table>
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<tr>
<th>Application</th>
<th>Examples</th>
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<tbody>
<tr>
<td>Remove water from aqueous solutions or dispersions → fine powders</td>
<td>Silicates, polymers, pigments</td>
</tr>
<tr>
<td>Chemical conversion</td>
<td>Conversion of sodium ortho- to tripolyphosphate (e.g. detergents)</td>
</tr>
<tr>
<td>Rapid drying of thermally sensitive materials</td>
<td>Pharmaceuticals, antibiotics</td>
</tr>
<tr>
<td>Provide easily dispersible powders</td>
<td>Milk, coffee</td>
</tr>
<tr>
<td>Make free flowing, consistent formulated products</td>
<td>Food, drink mixes, flavourings</td>
</tr>
<tr>
<td>Make structured particles</td>
<td>Porous particles: detergents, catalyst support</td>
</tr>
<tr>
<td></td>
<td>Microencapsulates: perfumes, flavours</td>
</tr>
<tr>
<td>Increase storage stability, reduce shipping weight</td>
<td>Milk, foodstuffs, enzymes</td>
</tr>
</tbody>
</table>
Spray Drying

Advantages:

• Very rapid drying via large surface area and good contact, high mass and heat transfer
• Single stage (semi)continuous process → flexible and reduces handling
• Suitable if non-aqueous solvents or inert atmospheres are needed
• Heat used to vaporise solvent and not heat solid → use for sensitive materials
• Particles are generally uniform and disperse and flow well
• High potential for particle engineering in the dryer (primary particle size and morphology, agglomeration, encapsulation)

Disadvantages:

• Large, relatively complex and expensive equipment
• Need starting material to be pumpable and atomisable suspension or solution
• Need to be able to understand and control each stage for optimum results
• What you get in the lab may look different from what you get on production scale
Spray drying is fast. Why?

- Atomisation results in much higher surface area/unit volume
- A 1m radius sphere of water has surface area $12m^2$
- As 200µm droplets it has $10^4$ times more surface
- Therefore dries a ten thousand times faster
- Increase in surface area so needs energy to create it
- For above example first sphere has surface energy of 0.864 J. converted to 200µm droplets you’d need 864 kJ
  - For 4 tonnes water.
- Therefore you need some energy to atomise!
Atomisation

- Provide the energy to break liquid into small droplets
- Break liquid into thin sheets. This generates instabilities which then generate small droplets
- Most common types give a wide size distribution
  - Pressure nozzle
  - Air assisted nozzle
  - Spinning disc
- Special types give narrow size range but low throughput
  - Acoustic
  - Electrostatic
The Importance of Viscosity

- Viscosity has a critical influence on the ease of pumping and atomising the fluid.
- However, for most liquids the viscosity changes as the shear rate increases.
- The critical viscosity is value at the shear rate of the nozzle or at edge of spinning disc.
  - Usually very high, e.g. 1/100000 secs
  - Ideal is shear thinning fluids
- Detergent slurries have consistency of thick porridge but are extremely shear thinning.
- Nozzles need to be big enough not to block.
Drying and Air Humidity

- Flow rate: A function of driving force/resistance
- Driving force: A function of moisture difference across surface of droplet
- Higher temperature means air can take more water before saturation (0.0038% at 0°C to 2.3% at 95°C)
- Once hot air is saturated no further drying can take place
- As heat is given to the droplets the air temperature goes down
- So droplet temperature rarely reaches the temperature of the inlet air whilst there is still moisture to remove
- Rate of heat going into droplets depends on temperature between drop and the air

Resistance to mass transfer:
- Surface area of droplet
- Internal diffusion to surface
- Removal by dryer air
Typical Drying Mechanism

Surface Drying
- Initially surface is saturated with solvent
- Drying takes place by diffusion of solvent across boundary layer around droplet
- Here the relative humidity is important

Surface Crust
- As time passes, surface water level decreases
- Rate of transfer of water from inside is not sufficient
- Surface starts to dry and rate decreases

Diffusion Control
- For some materials evaporation starts inside granule - solvent vapour has to diffuse through droplet solid wall
- Drying rate decreases rapidly
- Droplet temperature increases as heat energy is not converted into vapour fast enough
- The material properties of the droplet are critical to this stage
Why is Droplet Size Important?

- Smaller droplet $\rightarrow$ higher relative surface area $\rightarrow$ faster drying
- In smaller droplets the water has less distance to travel to surface
  - Thus chance of “stage three” drying is reduced
- Large droplets fall faster so have less time for drying and have further for water to get to surface

- Often large particle sizes are desirable for ease of handling, flow, dust - but drying rate may limit upper size of droplets
- Option to spray fine droplets and then agglomerate
  - Either controlled collisions inside tower or by adding fluid bed at base of tower
3. Spray Drying – The Hardware

Example: Co-current Spray Dryer System
Spray Dryer Shape and Design Options

- Choose chamber shape according to product and atomiser
- Spinning disc → “Short and fat” tower
- Spray atomisers and longer residence times → Narrow tall tower

Co-current design: Most common

- Lower viscosity → spherical particles
- Fine powder (100-200µm). May post-agglomerate in fluid bed
- Use: Temperature sensitive solutions → Rapid drying, low times at hot/wet conditions
- Use: high water content products that have little resistance to internal diffusion

Counter-current: Slurry enters at top, hot air flows up from below

- Greatest driving force at bottom
- Often droplets agglomerate in tower → particle size 150-600µm
- Use: Detergents and slow drying slurries
- Can make low density and highly porous
4. Material and Particle Engineering

Spray drying can produce..

- Small spheres, large hollow spheres, highly agglomerated particles, fine dust, high aspect ratio particles, encapsulates, dried heat sensitive particles
- What you get depends on:
  - Material properties, formulation, water content
  - Process conditions, tower design, nozzle type
- Therefore it’s best to start with the end in mind and work backwards!

Acknowledgements: Prof Geoff Lee, University of Erlangen - Leeds Spray Drying Course 2014-2016

Eudragit L100 ITRA (1:1) Ultrasonic

Acknowledgements: Filip van Der Gucht, ProCept - Leeds Spray Drying Course 2014-2016
Particle Engineering: Microencapsulation

- Volatile materials can be spray dried with minimal losses
- Encapsulating a flavour oil which is insoluble in water
- Make an emulsion using high shear mixing and an emulsifier
- Include a material that changes properties on heating such as protein or starches
As water dries, particles move to oil droplet
As temperature increases, particles sinter to give solid coating
5. Spray Drying – Summary and Learning More

- Spray Drying
  - is highly flexible
  - is cost effective
  - can be used to fine tune the material and particle properties of the final product

- Industrial applications are very varied but the basic science is the same
  - Small volumes – biopharma
  - Huge volumes – detergents, milk powder
5. Spray Drying – Summary and Learning More

- Understanding material properties and the influence of operating parameters is critical for good applications.

- Spray drying has a scientific basis.
- It can be complex but it is understandable.
5. Spray Drying: Learning More

Spray Drying and Atomisation of Formulations, 12th-14th April 2016

www.engineering.leeds.ac.uk/short-courses/particles/SprayDrying.shtml

Fundamental principles:
• Fluid properties, rheology and atomisation
• Influence of drying parameters on product microstructure, materials properties and quality
• The hazards of spray drying and how to ensure safe operation
• Laboratory demonstration of key steps
• Choice and design of appropriate equipment at all scales

Practical application at all scales:
• Scale-up and the possible pitfalls
• Applying principles to the manufacture of real industrial formulated products
• Case studies from different industries
• The benefits and opportunities of modelling
5. Spray Drying: Learning More

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• Coming up: Ink-jet Basics – 14th April

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