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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

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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

Complementary network of Associates

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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

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So You Want To Dry: What Choice Have You Got?

Source: http://www.solidsforum.com/Drying

Absorption Dryers
Active Freeze Dryers
Adsorption Dryers
After Cooling Dryers
Agitated Dryers
Air Classifier Dryers
Air Dispersion Dryers
Air Dryers
Air Impingement Dryers
Airswept Turbo Dryers
Apron Dryers
Aseptic Filter Dryers
Auto Dryers
Automated Batch Dryers
Back Mix Dryers
Back Mix Feeding Dryers
Back Mix Flow Fluid Bed Dr
Ball Dryers
Band Dryers
Batch Agitated Dryers
Batch Dryers
Batch Rotating Dryers
Batch Tray Dryers
Belt Dryers
Bench Top Dryers
Brazing Furnaces
Calciner Dryers
Calciners
Carousel Dryers
Cascading Rotary Dryers
Centrifugal Dryers
Centrifugally Agitated Bed I
Classifying Dryers
Closed Loop Dryers
Compressed Air Dryers
Conduction Dryers
Conical Dryers
Conical Screw Dryers
Contact Fluidizer Dryers
Continuous Band Dryers
Continuous Dryers
Continuous Flow Dryers
Continuous Gravity Dryers
Continuous Tray Dryers
Continuous Tunnel Dryers
Convection Dryers
Conveyor Dryers
Counter Current Dryers
Counterflow Dryers
Cross Flow Dryers
Crystallizing Dryers
Dehumidificators
Dehydration Systems
Deliquescent Dryers
Delta Dryers
Desiccant Dryers
Direct Fired Dryers
Direct Heated Dryers
Disc Dryers
Dispersion Dryers
Drum Dryers
Dry Roasters
Dryers
Drying Cabinets
Drying Installations
Drying Ovens
Dual Plenum Dryers
Dual Plenum Roasters
Dust Tight Dryers
Even Flow Dryers
Festoon Dryers
Filter Dryers
Flaker Dryers
Flash Dryers
Flat Bed Through Air Dryers
Flotation Dryers
Fluid Bed Dryers
Fluidized Bed Calciners
Fluidized Bed Dryers
Freeze Dryers
Gas Dryers
Gas Tight Dryers
Heat Pump Dryers
Heated Dryers
Heatless Dryers
High Frequency Dryers
High Velocity Air Impingem
Highly Turbulent Bed Dryer
Hopper Dryers
Horizontal Band Dryers
Horizontal Paddle Dryers
Impingement Dryers
Impingement Ovens
Indirect Heated Dryers
Infrared Dryers
Infrared Zone Dryers
Inline Dryers
Jacketed Dryers
Kilns
Laboratory Dryers
Laboratory Spray Dryers
Loop Dryers
Lyophilizers
Membrane Dryers
Microwave Dryers
Mill Dryers
Mixer Dryers
Mobile Dryers
Moving Bed Dryers
Multi Pass Dryers
Multi Stage Dryers
Multi Tier Fluid Bed Dryers
Multi Zoned Dryers
Paddle Dryers
Pan Dryers
Paste Dryers
Pharmaceutical Dryers
Plate Dryers
Plug Flow Dryers
Plug Flow Fluid Bed Dryers
Pneumatic Dryers
Post Bake Equilibration Dry
Predryers
Press Dryers
Pressure Swing Dryers
Pulse Jet Dryers
Pulverised Air Dryers
Radiant Heating Dryers
Rapid Dryers
Refractance Window Dryer
Refractory Lined Dryers
Refrigerated Air Dryers
Refrigerated Dryers
Regenerative Desiccant Dry
Regens
Ribbon Dryers
Ring Dryers
Roaster Dryers
Roasters
Roller Dryers
Rolling Bed Dryers
Rotary Calciners
Rotary Drum Dryers
Rotary Dryers
Roto Cone Dryers
Rotor Dryers
Scraped Surface Dryers
Screw Dryers
Second Stage Dryers
Single Pass Dryers
Single Stage Dryers
Sludge Dryers
Slurry Dryers
Solids Drying Equipment
Spin Dryers
Spin Flash Dryers
Spiral Dryers
Spouted Bed Dryers
Spray Dryers
Stationary Bed Dryers
Steam Tube Dryers
Suction Drum Dryers
Suction Dryers
Supercritical Dryers
Superheated Steam Dryers
Superheated Vapor Dryers
Suspension Dryers
Thermal Disc Dryers
Thin Film Dryers
Through Air Dryers
Toasters
Tornesh Dryers
Tower Dryers
Tray Dryers
Truck Dryers
Tube Bundle Dryers
Tubular Dryers
Tumble Dryers
Tunnel Dryers
Turbo Dryers
Turbo Tray Dryers
Twin Tower Dryers
V-Cone Dryers
Vacuum Band Dryers
Vacuum Dryers
Vertical Dryers
Vertical Paddle Dryers
Vertical Ribbon Dryers
Vibrating Fluid Bed Dryers
Vibratory Dryers

Focus on a Few Types of Dryers

Dryer Type	Advantages	Disadvantages
Tray/Shelf Dryer	Low losses, versatile, small batches, uniform heating	Slow manual load/unload. Little opportunity for particle engineering
Rotary Dryer	Can uses for pastes, wet solids	
Freeze Dryer	Good for heat sensitive materials. Porous redispersible product	Slow, complex, expensive. Limited opportunity for particle engineering
Drum Dryer	Use viscous feeds. Relatively cost-effective and simple. Rapid drying	Maintenance requirement. Control of film thickness
Microwave Dryer	Dry sensitive materials, bulk, viscous, rapid	Less suitable for larger batches. Little opportunity for particle engineering
Vacuum Dryer	Low risk of oxidation, heat damage. Small batch sizes	Less suitable for larger batches. Little opportunity for control and particle engineering
Fluid Bed Dryer	High rate if drying. Uniform drying from particle surface. Free flowing particles	Particles at least 250µm to form controllable fluid bed. Process may break down particles and form dust.
Spray Dryer	…	…

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Typical Applications of Spray Drying

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

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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

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2. Spray Drying – The Basic Science

Spray drying is fast. Why?

- Atomisation results in much higher surface area/unit volume
- A 1m radius sphere of water has surface area $12 m^2$
- 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!

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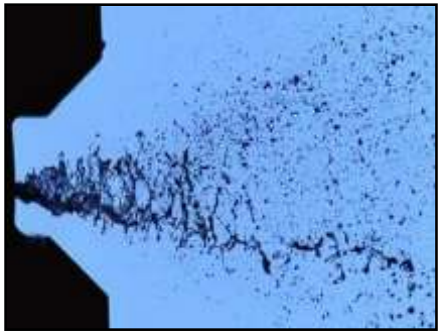
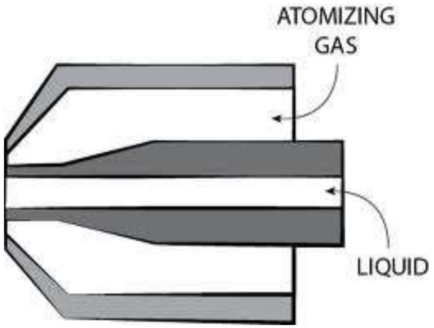
Atomisation

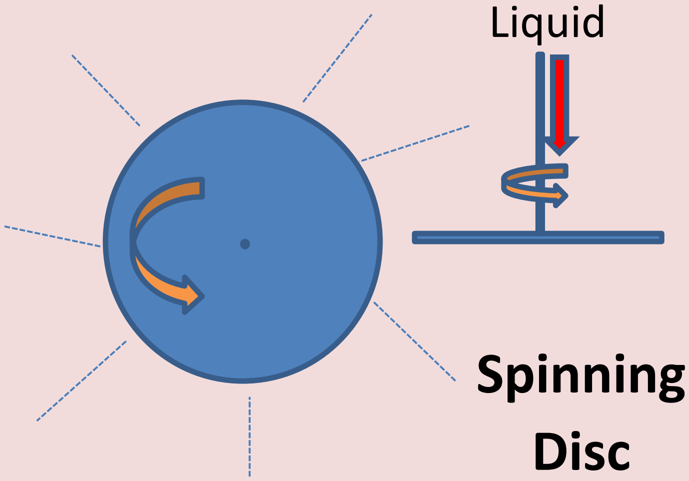
- Provide the energy to break liquid into small droplets
- Break liquid into thin sheets. This generates instabilities which then generate small droplets
- Most common types give a wide size distribution
  - Pressure nozzle
  - Air assisted nozzle
  - Spinning disc
- Special types give narrow size range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



Pressure Nozzle



Air Assisted Nozzle



Spinning Disc

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

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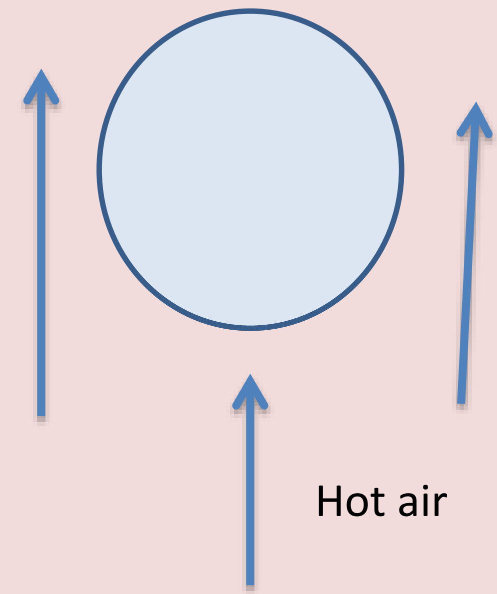
Drying and Air Humidity

- 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

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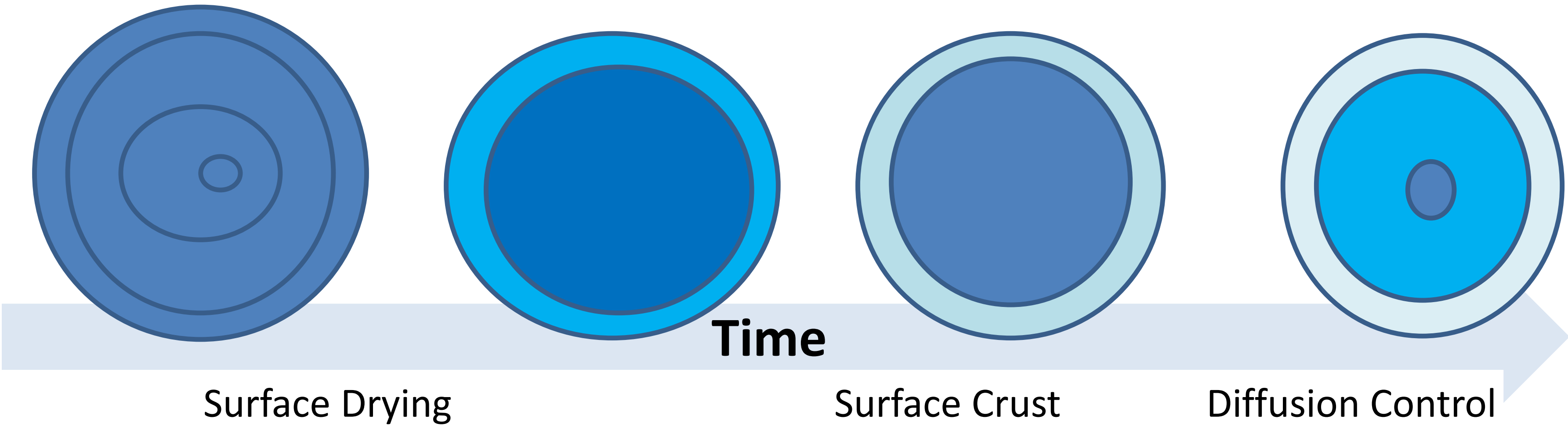
Typical Drying Mechanism



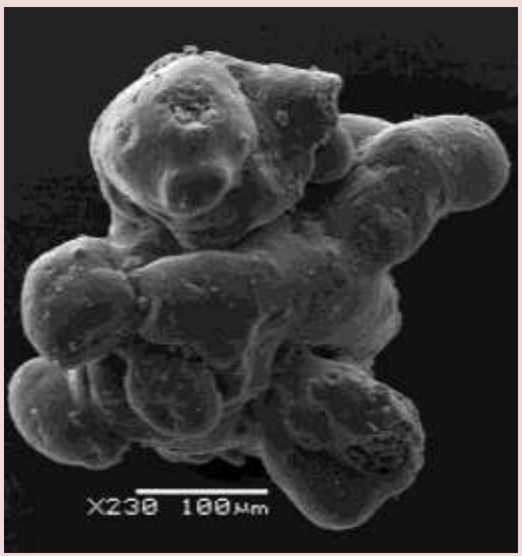
- Initially surface is saturated with solvent
- Drying takes place by diffusion of solvent across boundary layer around droplet
- Here the relative humidity is important

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

- 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 → higher relative surface area →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

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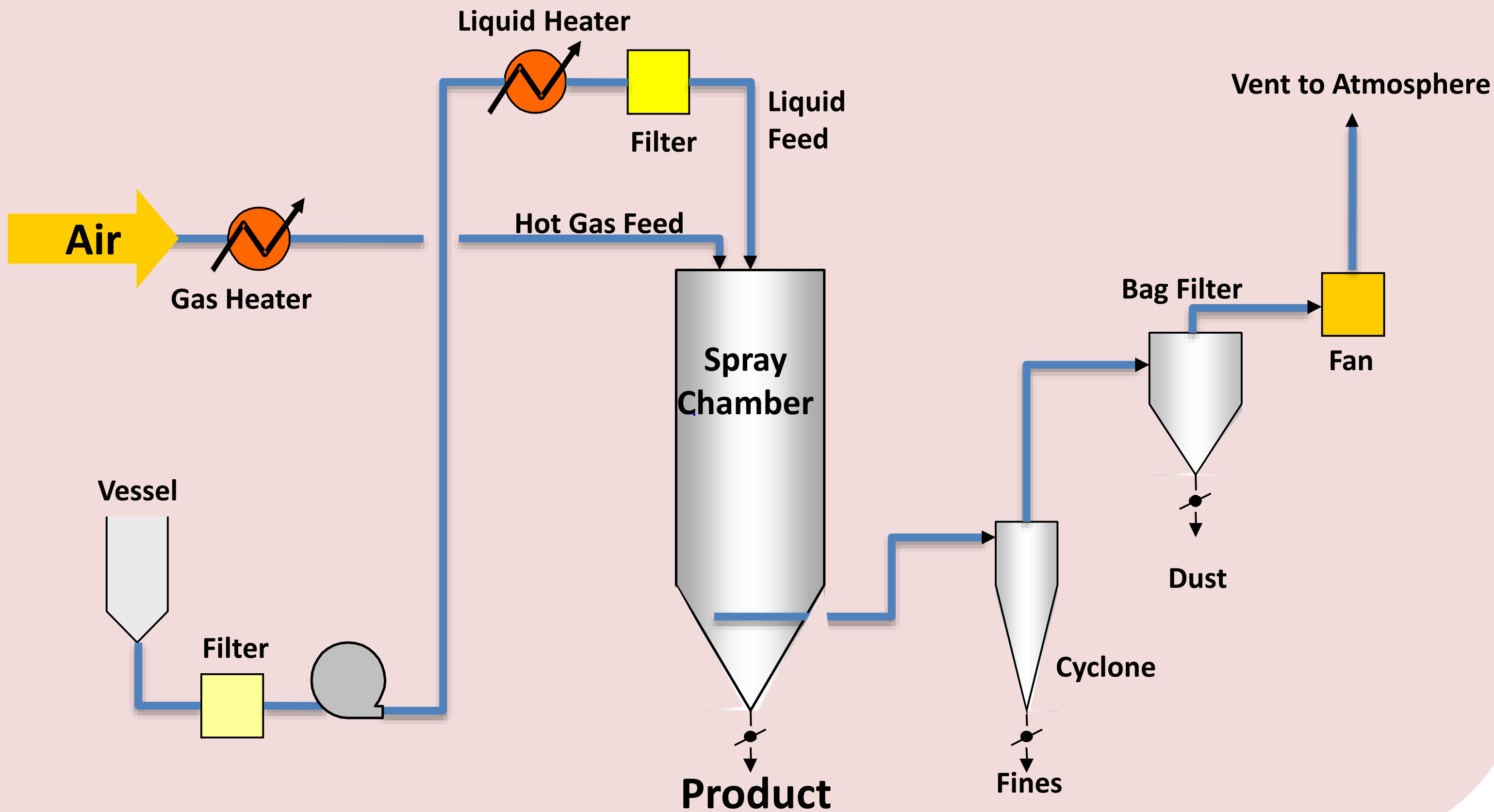
3. Spray Drying – The Hardware

Example: Co-current Spray Dryer System



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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

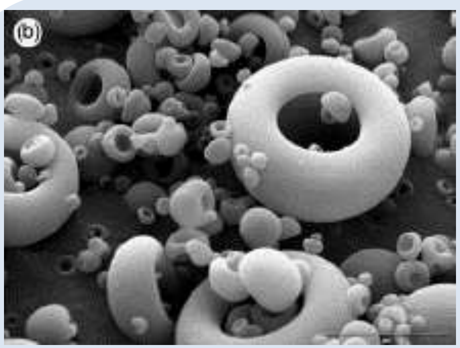
Counter-current: Slurry enters at top, hot air flows up from below

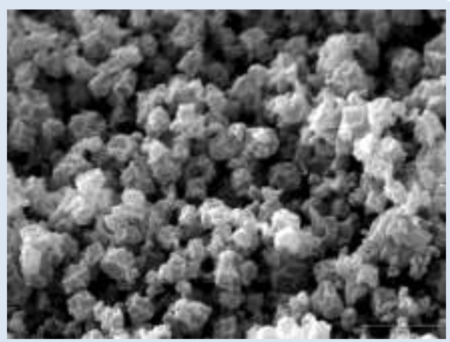
- Greatest driving force at bottom
- Often droplets agglomerate in tower → particle size 150-600µm
- Use: Detergents and slow drying slurries
- Can make low density and highly porous

4. Material and Particle Engineering

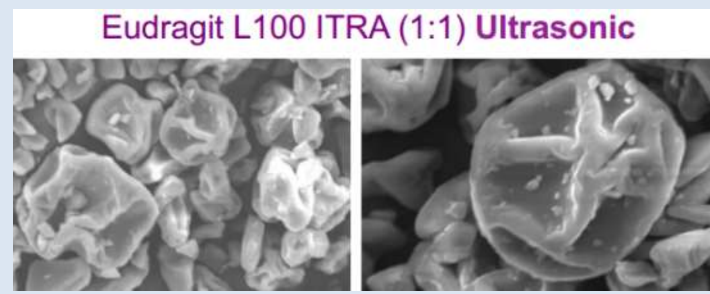
Spray drying can produce..

- 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



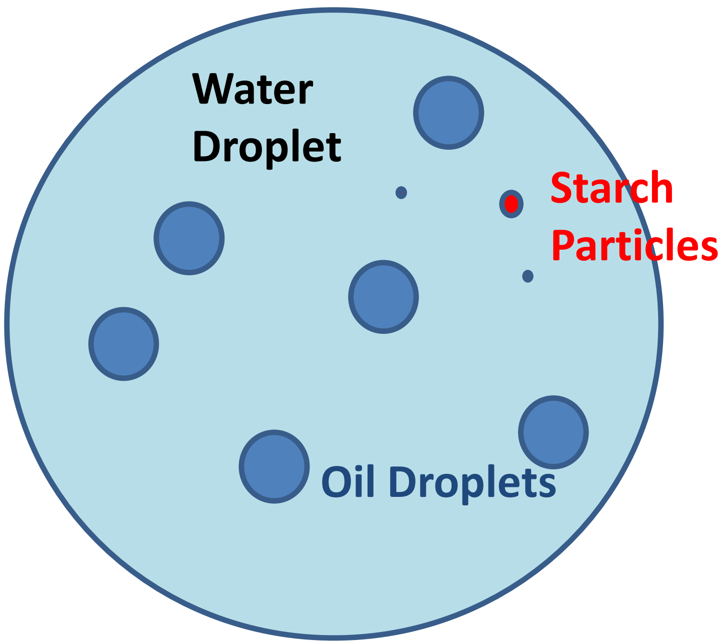
Acknowledgements: Filip van Der Gucht, ProCept - Leeds Spray Drying Course 2014-2016

XEDEV Excellent in Development

ProCepT Particle Processing Equipment

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



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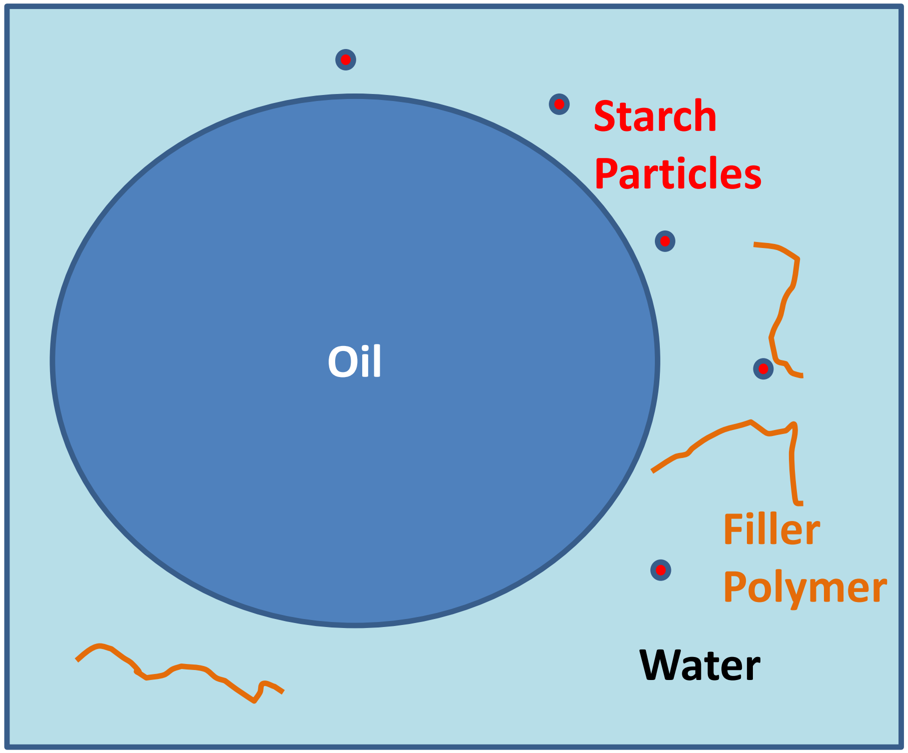
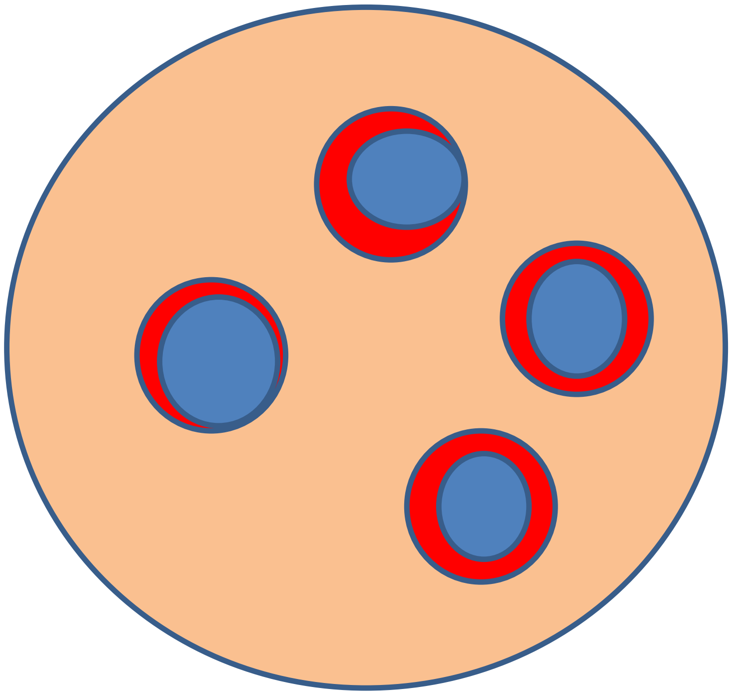
Particle Engineering: Microencapsulation





Final particle

- As water dries, particles move to oil droplet
- As temperature increases, particles sinter to give solid coating

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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

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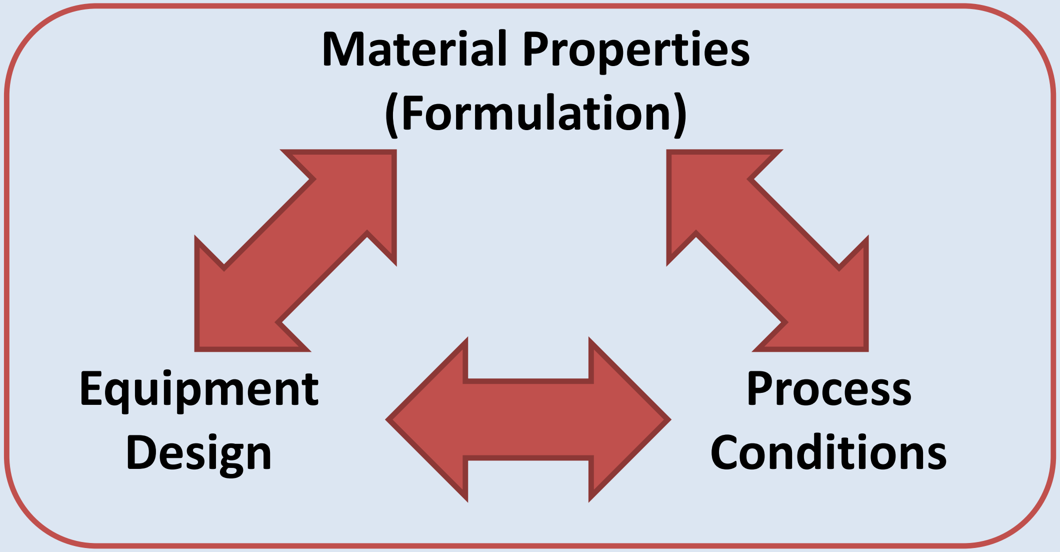
5. Spray Drying – Summary and Learning More

- 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

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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

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