iFormulate

Particle Engineering using Spray Drying and Fluid Beds

iFormulate Webinar

26th February 2020

iFormulate *Skills*

In association with:

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
- Complementary network of Associates

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Our Services

iFormulate Consult

iFormulate Strategic

iFormulate Skills

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Overview:

1. **Introduction**
2. **Particle Engineering using Spray Drying:** Jim Bullock
3. **Particle Engineering using Fluid Bed:** David York
4. **Wrap up and Q&A**

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 40 minutes

Your Speakers

Dr Jim Bullock
iFormulate Ltd

Professor David York
FREng, FIChE
University of Leeds

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Particle Engineering

- Making particles with the right…
  - Shape
  - Size
  - Structure
  - Surfaces
- To give you the product properties you want

Spray Drying or Fluid Bed Processing

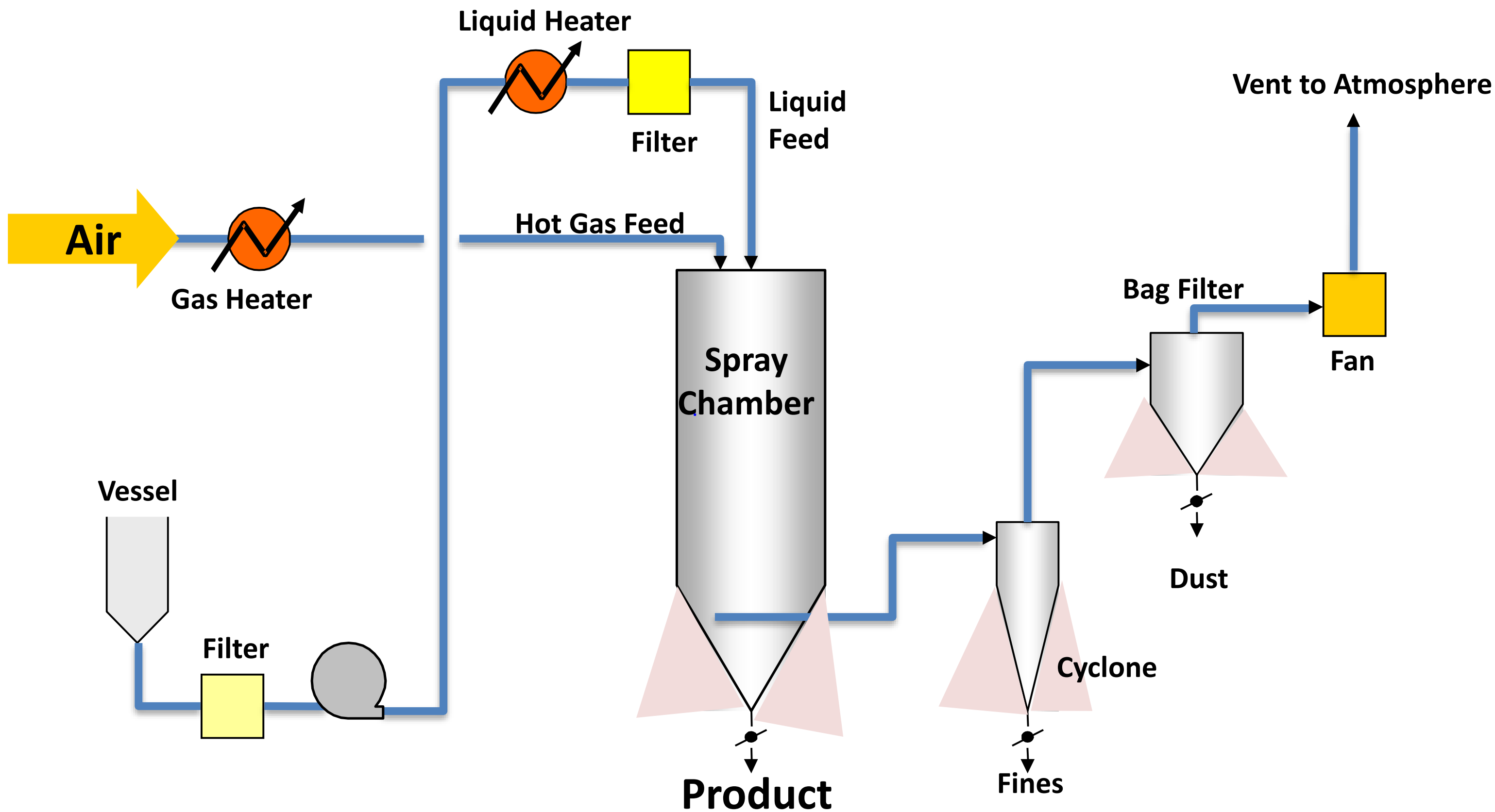
Alternatives to engineering particles

Spray Drying – Basic Principles

- **Fluid feed is pumped through an atomiser nozzle to form droplets**
  - Feed can be solution, emulsion or suspension in water or other solvents
  - Wide range of viscosities possible
- **Droplets enter chamber together with a separate stream of heated gas (usually air)**
- **Droplets fall and are dried to form solid particles as they reach the base of the drying chamber**
- **Particles are removed and separated usually using a bag filter and cyclone system**
- **Rapid and efficient process due to high specific surface area of droplets**
  - Allows rapid heat and mass transfer

Spray Drying – The Hardware

Example: Co-current Spray Dryer System



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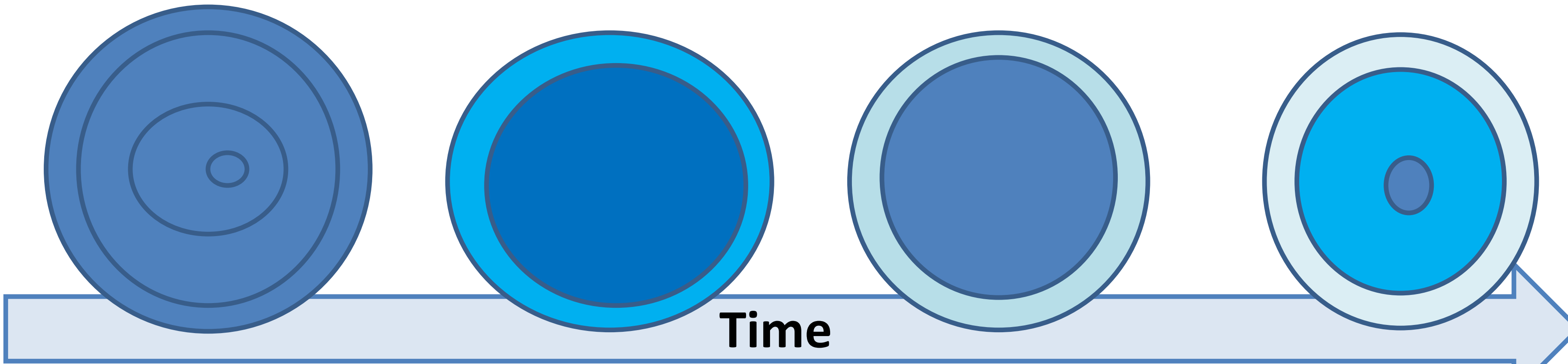
Particle Engineering Options in Spray Drying

- **Size**
  - Lower pressure atomisation → Bigger droplets
  - Bigger droplets → Bigger particles
  - But will they be dry by the time the reach the base of the tower?
- **Shape**
  - Often spherical
  - Drying conditions may cause other shapes to be formed
- **Structure**
  - Internal – e.g. core shell
  - External – e.g. granules formed by agglomeration

Engineering Product Properties

- **Granule strength**
  - e.g. robustness to dust formation
- **Controlled release**
  - e.g. core-shell encapsulation
- **Flow properties**
  - Size, shape, smoothness
- **Redispersibility**
  - Size, shape, density

What happens to droplets in a spray dryer?



Surface Drying

Surface Crust

Diffusion Control

1

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

2

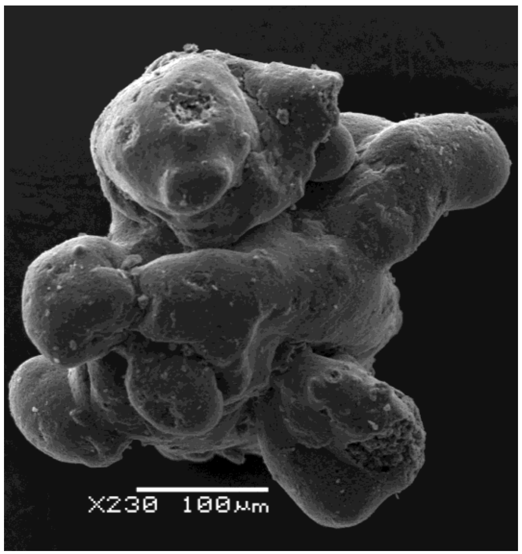
- After time, surface water level decreases
- Rate of transfer of water from inside not sufficient
- Surface starts to dry and rate decreases

3

- For some materials evaporation starts inside particle - solvent vapour then diffuses through droplet solid wall
- Drying rate decreases rapidly
- Particle 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 droplets → higher relative surface area →faster drying
- Smaller droplets → less distance for water to travel to surface → less likely to have drying by diffusion from centre and particle heating
- Larger droplets fall faster → less time for drying → further for water to get to surface



- Larger particles often desirable for ease of handling, flow, dust - but drying rate may limit upper size of droplets
- Option to agglomerate fine droplets
  - Controlled collisions inside tower
  - Or by adding fluid bed at base of tower

Spray Dryer Geometry and Design Options

Atomisers

- Spinning disc atomiser → Short and wide tower
- Spray atomiser, longer residence time → Narrow tall tower

Co-current Design:

- More common: Product fluid and drying gas follow the same direction
- Lower viscosity product fluid → spherical particles
- Fine powder (100-200µm diameter)
- Can use for temperature sensitive product → Rapid drying, low times, hot/wet conditions
- Use for high water-content products with little resistance to internal diffusion

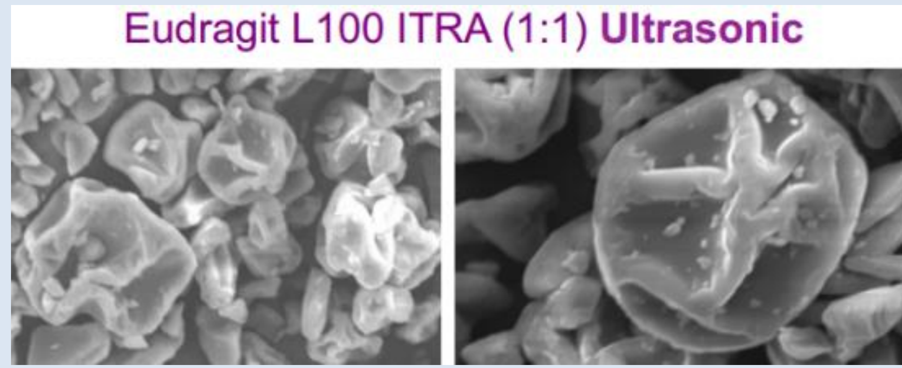
Engineering Particle Shape

Many possible morphologies e.g.:

- Small spheres
- Spheres with blowholes or collapsed spheres
- Fractured spheres → dust
- Large hollow spheres
- Encapsulates
- Agglomerated particles

Important to understand influence of:

- Material properties, formulation, water content
- Process conditions, tower design, nozzle type

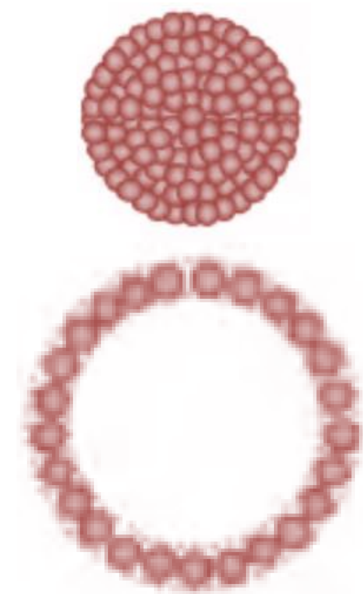


XEDEV Excellent in Development
ProCepT Particle Processing Equipment

Acknowledgements: Filip van Der Gucht, ProCept - Leeds Spray Drying Course 2014-2020

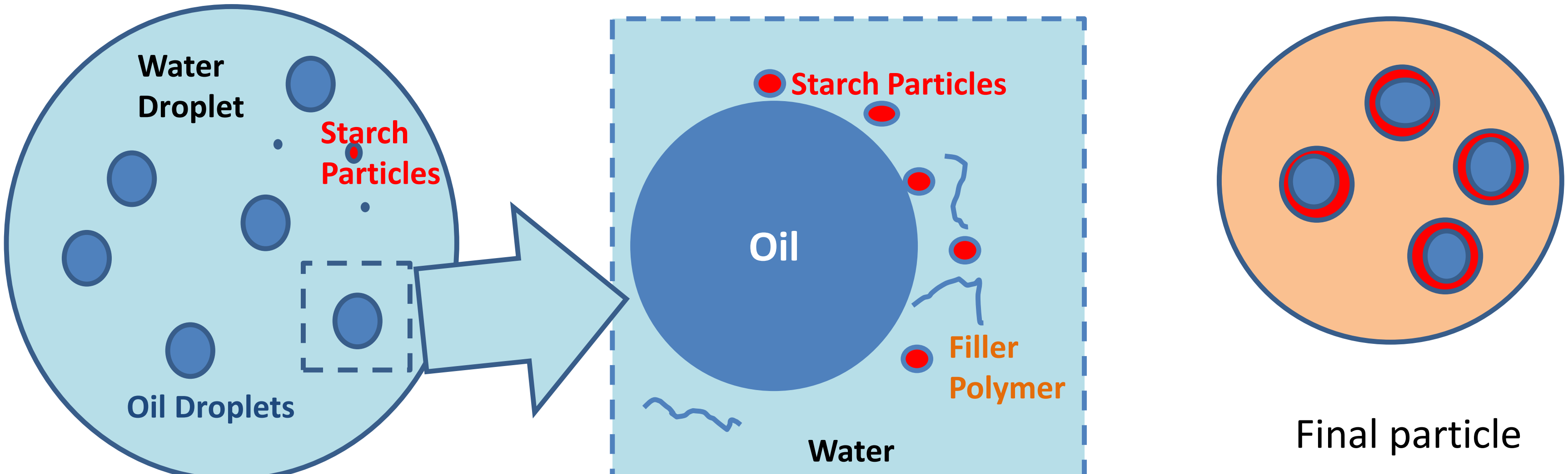
To Shell or not to Shell?

- **Peclet number** = ratio of solvent evaporation rate to solute diffusion rate
- **Low Peclet number** – no shell is formed because the solutes can diffuse faster than the droplet can shrink
- **High Peclet number** – shell is formed because the solutes diffuse slower than the droplet shrinks
- May have multiple solute species with different Peclet numbers
- For more on Peclet numbers and morphology, see e.g.
  - A.B.D. Nandiyanto, K. Okuyama/Advanced Powder Technology 22 (2011) 1–19
  - Vehring, R. Pharmaceutical Particle Engineering ***via*** Spray Drying. *Pharm Res* **25,** 999–1022 (2008).



Particle Engineering: Microencapsulation

- Enable spray drying of volatile materials with minimal losses
- Feedstock: Emulsify water-insoluble oil using high shear mixing and emulsify
- Include encapsulating material (e.g. protein or starch) which can change properties on heating



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

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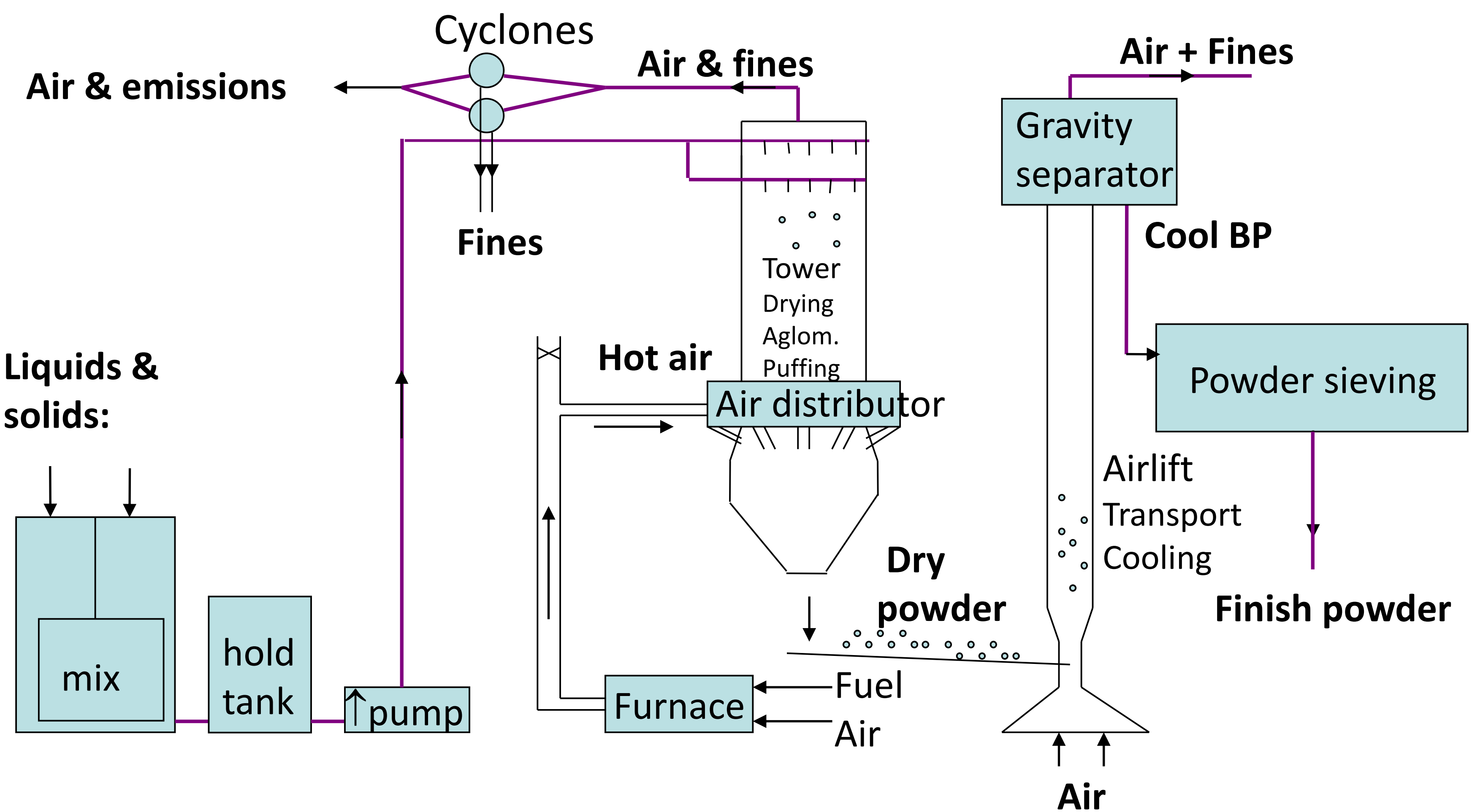
Agglomeration and Granulation

Counter-current Design:

- Product fluid enters at top, hot air flows up from below
- Greatest driving force for drying at bottom of tower
- Can agglomerate droplets in tower
- Tower design to extend residence time and maximise probability of collisions
- Material properties for particles to adhere
- Particle size 150-600μm
- Use for e.g. detergents and slow drying slurries, can make low density and highly porous product

Counter Current Spray Dryer



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Agglomeration and Granulation

Agglomeration in Fluid Bed after Spray Drying

- Usually use with co-current design spray dryers which produce finer primary particles
- Relatively fine spherical primary particle
- Fluid bed at base of dryer

Spray Drying for Particle Engineering

Advantages:

- Very rapid drying due to large surface area and good contact, high mass and heat transfer
- Single stage (semi)continuous process → flexible and reduces handling
- Heat used to vaporise solvent and not heat the solid → can use for sensitive materials
- Good method for removing water at the end of a process
- High potential for particle engineering in the dryer (primary particle size and morphology, agglomeration, encapsulation)

Disadvantages:

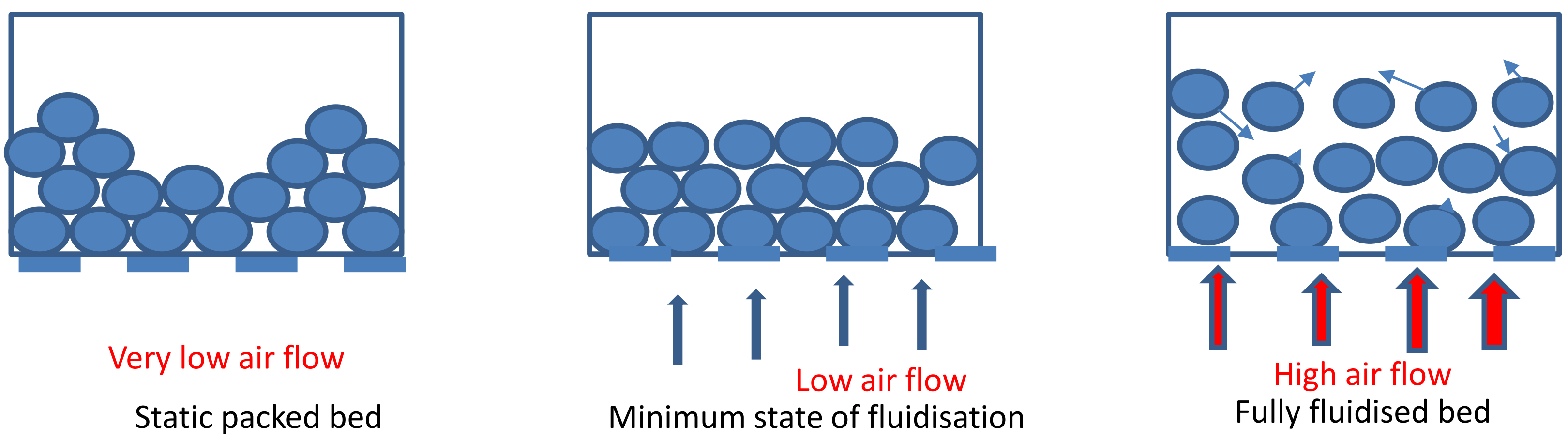
- Large, relatively complex and expensive equipment
- Must be able to pump and atomise starting suspension/solution
- Need good understanding of properties and process to control for optimum results
- Scale-up may require care

Fluid Beds Operation

- Start with a bed of particles which is porous to let a gas pass through
- As the gas flows across the particles a pressure drop occurs due to resistance
- As flowrate increases the pressure drop equals the pull of gravity on the particles and they “escape gravity” and become fluid like
- As long as the gas flowrate is maintained the fluid bed is maintained

What is a Fluidised Bed?

- Basically a packed bed of particles that are brought to a state of random movement by pressurised air.
- Behaves like a fluid
- Simplified to particles suspended in air.



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Properties

- Has a defined pressure drop across the height of the bed
- Lots of movement in random directions
- Has a bubbling surface like boiling water.

- **Lots of mixing.**
  - **Particle/air and particle/particle**
- **Good mass transfer**
- **Good heat transfer**
- **So as long as the flowrate is maintained the fluid nature continues**
  - **Provides time for many transformations with respect to particle engineering**

Uses of Fluidised Beds

1. **Heterogeneous reactions- due to good mass transfer**
   Coal burning in power stations
   Catalytic reactors-hydrocarbon cracking

2.Drying – due to good heat transfer and time window
   Good control of residence time and temperature.
   Sometimes used with spray drying to increase capacity or temp sensitive materials

3 Cooling- as above
   After fluid bed drying

4.Coating- provides time for good contact and drying
   Encapsulation using polymeric solutions

5. Agglomeration- as above but additive is designed to stick particles instead of spreading
   Low density particles with raspberry structure
   Used to "instantise" food such as coffee and milk powders

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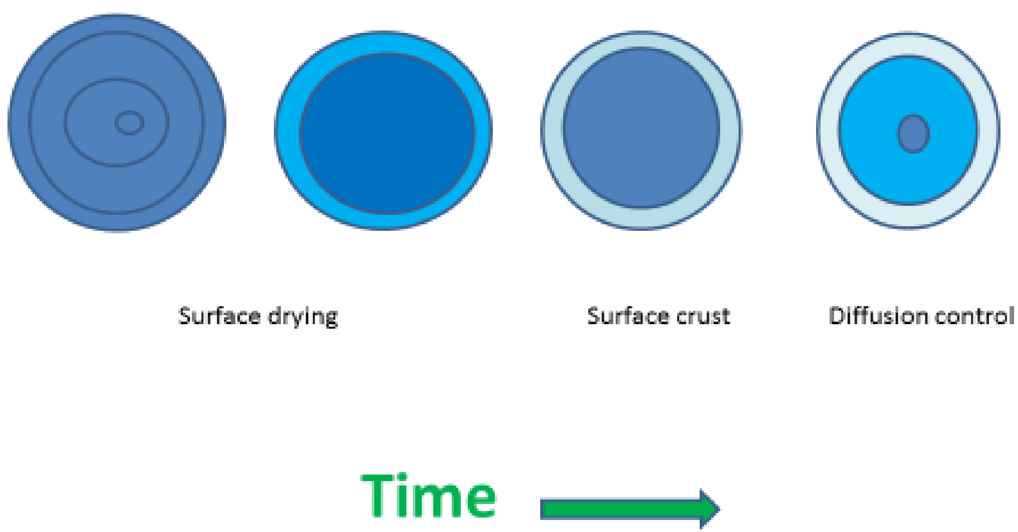
Drying and Cooling

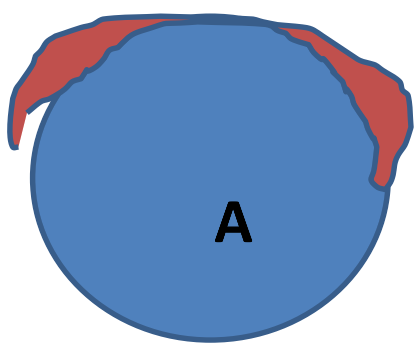
- Often spray drying does not give enough time to remove all the solvent due to slow diffusion at the end.
  - Harder to get very dry particles
- Fluid beds can give longer time, often at lower temps.

What happens as a drop dries?

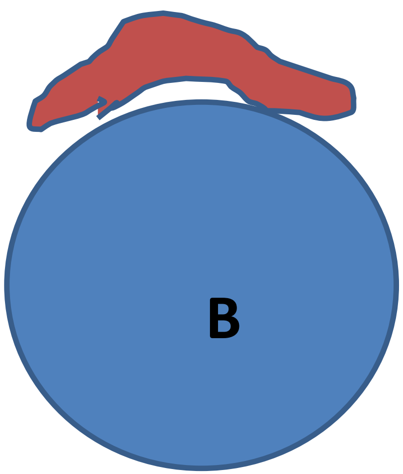


Coating and/or Agglomeration

- If liquid hits a particle surface and spreads and dries, it will gradually become fully coated with repeat hits - **A**
- However, if liquid makes the surface sticky it can glue two or more particles together to agglomerate- like a raspberry - **B**



Dried film



Undried sticky liquid

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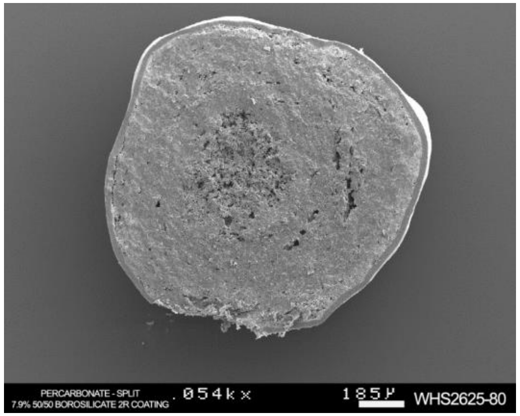
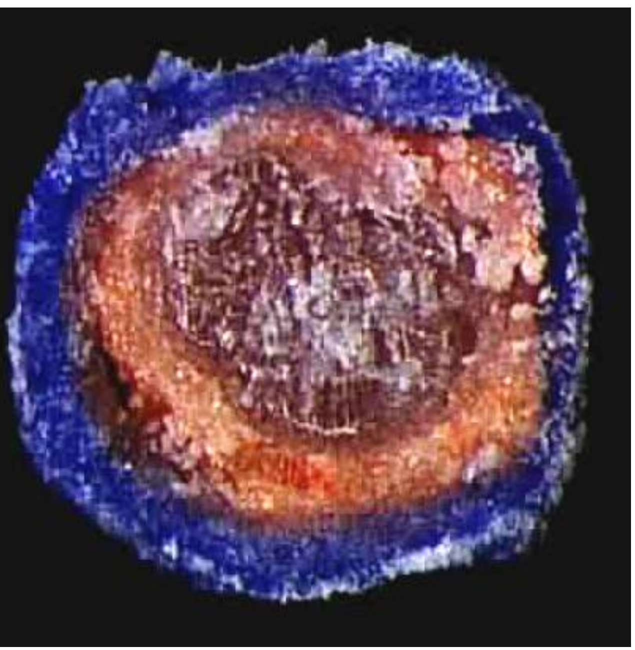
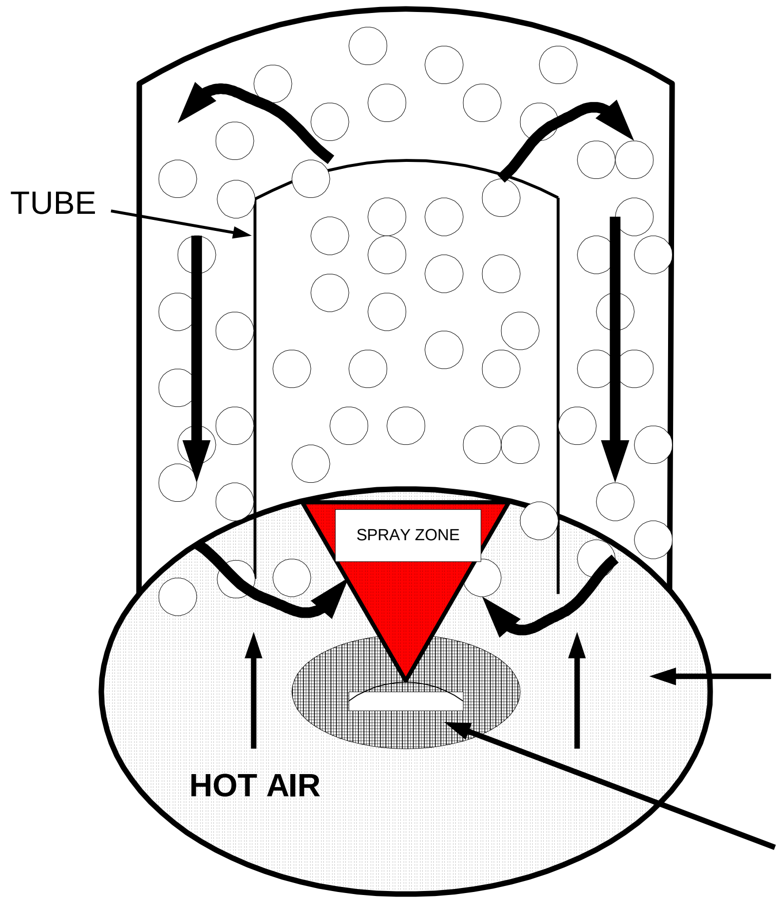
Wurster Coater Chamber







Fluidized Bed

Particles Circulate repeatedly through the Coating Zone defined by the Tube

Particle Temperature close to exhaust temperature

6-10 second cycle

15 min to several hour batch time

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Lots of Possibilities for Engineering Particles by Fluid Bed

- Can make different structures
- Can enhance the structure of spray dried particles
- Can make particles more free flowing with particle growth or surface coating
- Can add coatings for delayed release/increased stability
- HOWEVER…

Limitation of Fluid Beds

- Particles need to be free flowing
  - Harder with lots of sticky particles
- Particle properties will determine how easy they can fluidise

A. Too fine, difficult to keep in the bed (like dust)

B. Too heavy, difficult to get air flow fast enough to lift the particles

C. Too fragile, can fracture particles as particles collide

Simple Comparison
Fluid Bed versus Spray Dryer

- Both involve making particles by passing air over the increased surfaces inherent in their processes
  - Spray drying involves making liquid drops which form particles on drying.
  - Fluid beds require solid particles at the start of their processes
- Spray drying is very fast, and provides many opportunities
- Fluid beds are much slower but this can provide other structures
- The benefits of each are due to how they function

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LEARNING MORE

Spray Drying and Atomisation of Formulations, 31st March – 2nd April 2020

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https://eps.leeds.ac.uk/dir-record/short-courses/1004/spray-drying-and-atomisation-of-formulations

Day 1. Spray Drying and Atomisation Basics

- Industry and academic experts provide the essential scientific background
- Practical hands-on laboratory demonstrations

Day 2: Industrial Formulation Case Studies

- Experienced specialists will show how the science of spray drying has been applied to influence the properties of real formulated products across a wide range of business sectors
- Further laboratory demonstrations

Day 3: Powder finishing, modelling and future developments

- Further case studies
- The course finishes with a trouble shooting forum

Fluid Bed Processing and Formulation

11th-13th May 2020

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https://eps.leeds.ac.uk/dir-record/short-courses/989/fluid-bed-processing

Day 1. Basic Science and Understanding

- Fluidisation basics
- Mass transfer in the fluid bed
- Particle agglomeration in fluid beds
- Fluid atomisation; twin fluid nozzles
- Powder material properties
- Lab demonstrations

Day 2. Applications and Case Studies

- Basics of fluid bed design
- Modelling and scale-up
- Spreading, coating and agglomeration
- Continuous and batch operation
- Powder morphology and performance
- Particle engineering and characterisation
- Problem solving forum

Day 3. Case Studies and New Developments

- Fluid bed granulation modelling and scale-up
- Particle coating and controlled release
- Combining spray drying with a fluid bed
- Structured fluid beds
- Instrumentation and control
- High gravity fluidized beds

Any Questions?

- Participants remain muted
- Please use the GoToWebinar question/chat boxes
- Any follow up questions or other enquiries:

  E: info@iformulate.biz
- Participants will be sent details of how to access a recording of this webinar
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