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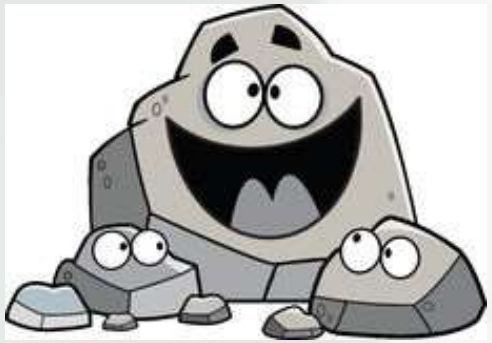
Introduces…

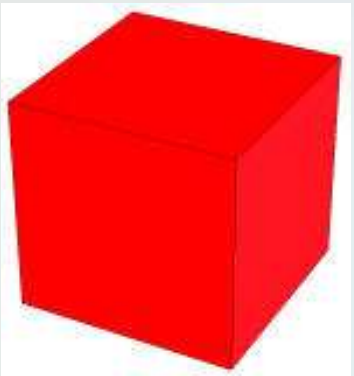
Powderology - An introduction to the mysteries of powders

Buckets, Boulders and Blocks

Ian Jolliffe & David Calvert

23rd June 2016





Overview:

1. *Introductions*
2. *What are Powders?*
3. *Challenges*
4. *Measurement and Control*
5. *Conclusions*

- **This webinar is being recorded and will be made available**

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

This webinar will last about 30 minutes

Your Speakers

Dr Ian Jolliffe
iFormulate Ltd

Dr David Calvert
iFormulate Ltd

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A Little About iFormulate

Founded in 2012 by two experienced industry professionals

Diverse experiences, knowledge and wide range of contacts

Polymers, materials science, chemistry, imaging, dyes, pigments, emulsion polymerisation, biocides, pharma, agchem, FMCG, food, anti-counterfeiting, environmental, formulation etc…

Consultancy, innovation, marketing, business development, strategy, regulatory, training, events, R&D

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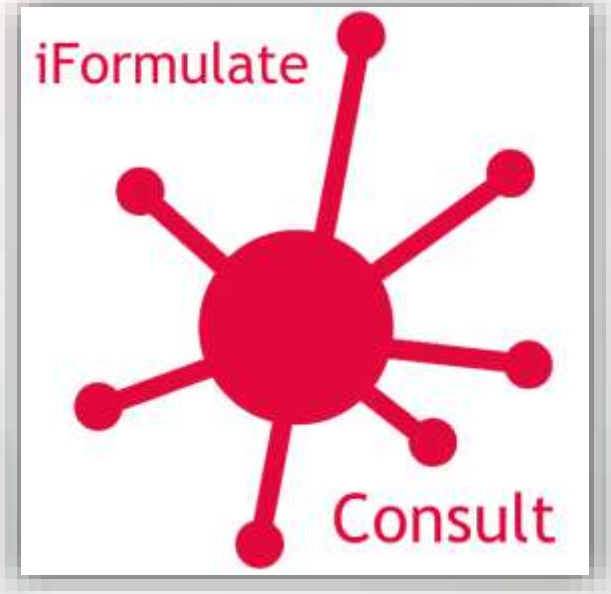
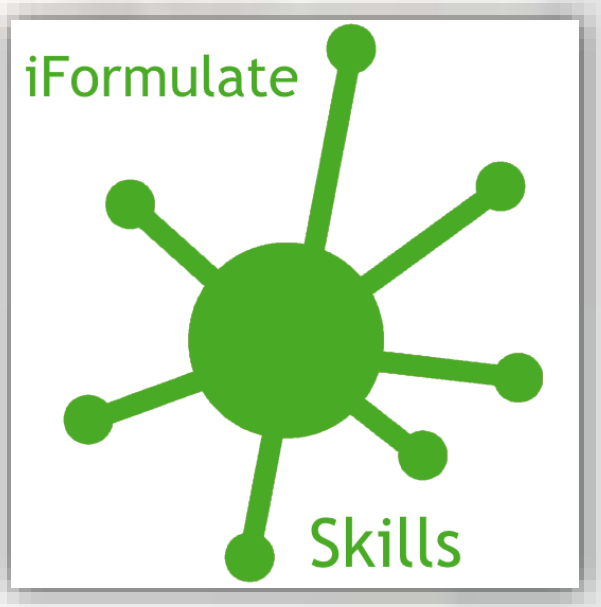
Our Services





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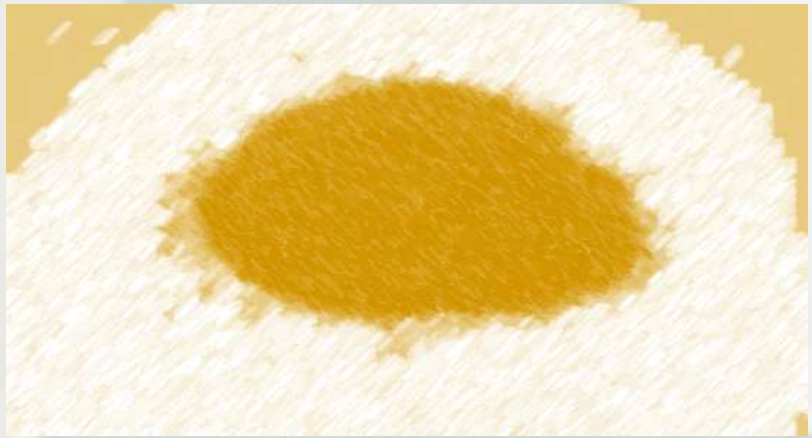
Powder Relevance and Applications

- Pharmaceuticals
- Healthcare
- Home Care
- Cosmetics
- Inks and Coatings
- Agrochemicals

- Raw Material
- Intermediate
- Final Format



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TODAY: What a state; bulk particles we call powders

- There's liquids
- There's solids
- There’s gases
- And there are particulates or powders
  - When particulates dispersed in liquids we call them suspensions (covered in iFormulate Suspensionology Webinar and one day course)
  - If dispersed in a gas - usually air - we call them ...er powders!
- Plus there are things we want to make from powders
  - Granules
  - Tablets
  - Powders packed in unit doses e.g. sachets, capsules , bespoke containers
- How do we meet the challenges of these formulation formats?

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Design for Formulation

- Previous webinar and course talked about building in quality from the design stage
  - knowing what you are making and why
  - Understanding and putting in place the appropriate controls to achieve the product promised to your “customers”
- Most products start with at least some ingredients that are powders
- Powders are difficult to handle either in processing or as finished products
- Powders are often dusty, don’t always flow, often unpredictable and affected by the environment and surfaces they come into contact with
- Even Muesli is now advertised as low dust!

Formulators are innovative in finding solutions to making powder ingredients easier to use *eg banishing the Dust of Muesli* *[another of the world's big issues!]*

Granola! the powdered ingredients stuck together in agglomerates that flow + all the dust mopped up

Better still- stick all the granules together into a bar for total particle free solution for the consumer

In a state ? If you think solids ,liquids and gasses are a challenge then powders will really get you in a state!

Bucket of SOLID: Concrete

Bucket of FLUID Water

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Buckets of powder

Bucket of Powder: Maize starch

Add the weight…

Not at the bottom – nor at the top!

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But not all powders/groups of particles behave the same….

Bucket of Powder: Maize starch

Bucket of Powder: pea gravel

Generally we can say powders made of larger particle size are less compressible
…..and flow better

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In a state ? If you think solids ,liquids and gasses are a challenge then powders will really get you in a state!

- *What state are powders?*
- *Particles dispersed in gas*
  - *Gas is usually air*
  - *PLUS they are under the influence of gravity*
  - *Interparticle forces and interparticle friction*
- *They pack down ie can exist with different volumes/ porosities*
- *They can flow*
  - *Powder flow recently described by a speaker at APS symposium as the "missing link" in the evolution of powder characterisation**

* Arjen P. Tinke APS course “Powder Flow 2015” 09Jun2015

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More challenges of powders

- Products based on powders are rarely one component
  - Mixing
  - Segregation (Muesli again)
- Surface changes eg moisture adsorption, surface charge
- Stability
  - Caking – vibration and gravity cause particles to pack closer and closer together such that the inter-particulate forces for fine particles make it difficult to induce flow again
    - Minor moisture changes of cycling of humidity can weld these in to solid cakes that need a lot of force to break up
      - Pulverised sucrose – and flow inducing mallet!
  - Separation
    - Segregation
      - Processing movement of powders causes particles with different sizes and shaped particles to move at different rates causing separation
        - Mixed powders are further complicated by varying in density and surface roughness
      - Need for transport test vs Muesli nuts and fruit at the top - i.e. large particles rise to surface
    - Charged particles
      - Different degrees of electrostatic charge or affinity for surfaces can cause some particles to be separated from others in a mix

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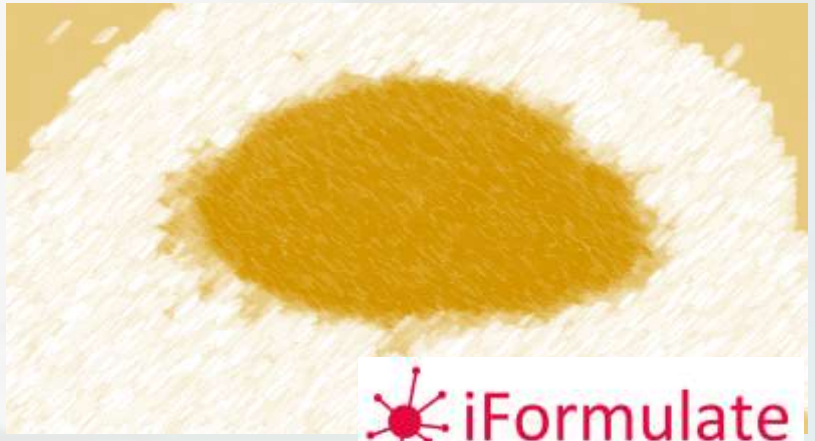
How do we handle these differences in properties and understand powder ingredients ?

- Why are powders different ?
- They can have differences in particle
  - Size
  - Shape
  - Surface effects....Charge, roughness, adsorbed materials
- First step is to *Understand what you want* from your powder to reliably make a product that fulfils the promises made to your customer
- What Material Attributes are critical to the Performance of your Product (Critical Quality Attributes - CQA)? Things to consider:
  - Flow – particles size, shape, density
  - Packing properties – bulk density
  - Segregation propensity – matching sizes, shape or density
  - Surface characteristics: roughness, moisture, electrostatic charge



What determines which material attributes of your powder are critical to your product? - 1. *Customer promise*

- Customer promise – what we say that the product will do
  - Basis of Critical Quality Attributes (along with industry/regulatory standards)
- Examples
  - Sensory feel
    - Gritty if particle size is not carefully controlled
    - Cream cleansers, orally dispersible powders, oral suspensions , skin creams
    - Visible residues
  - Rate of solution
    - Slow dissolution of pain relief ingredients in tablet
    - Slow hydration - large particles of hydrocolloid/gum may take so long to hydrate that the consumer gives up and ends up with a product full of “fish-eyes”
  - Separation
    - Expectation that the product is the same every time you take a sample from it
    - i.e. not the first spoonful is full of one component and the last is rich with another component – neither spoonfuls perform as intended
  - Processability
    - Segregation results in variable product with different amounts of ingredients

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What determines which material attributes of your powder are critical to your product?

2. What needs to *be controlled!*

- All parameters that affect the critical Quality Attributes of the product (which may be manifested through critical effects on the process)
  - Particle size
    - May affect release rate of an active ingredient through dissolution
      - Larger particles take longer to dissolve
    - May affect the ability to flow or susceptibility to picking up moisture
  - Particle shape
    - Spray dried vs crystalline shape vs flow e.g. lactose
    - Crystal habit versus the ability to form tablets
    - Mixing/stability of mixtures ordered mixing adhesion of fine particles onto rough larger particles
    - Packing properties - see below

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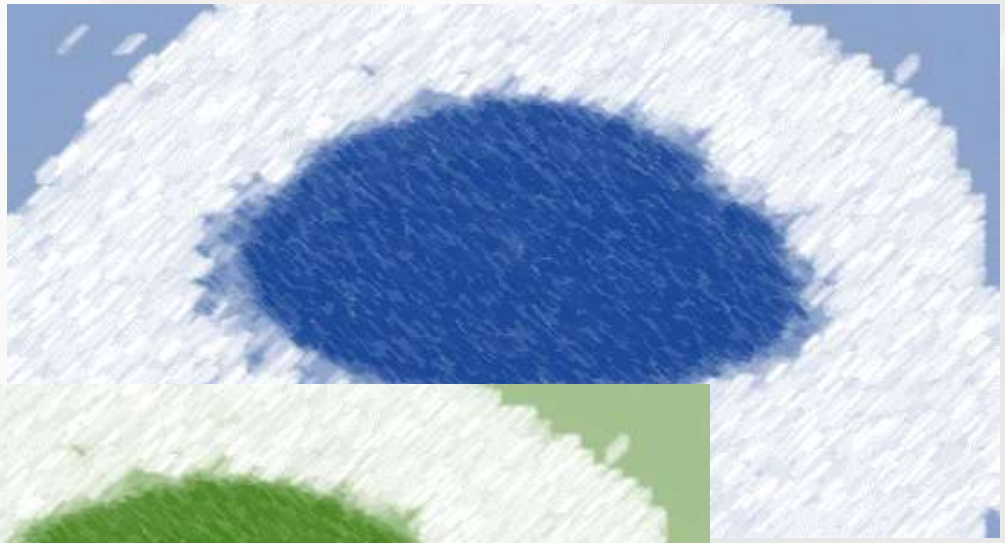
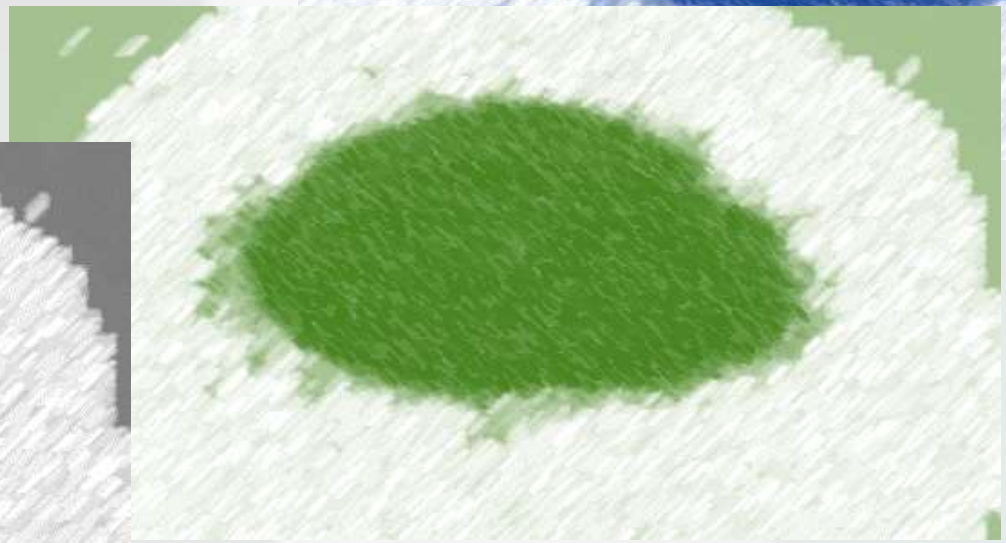
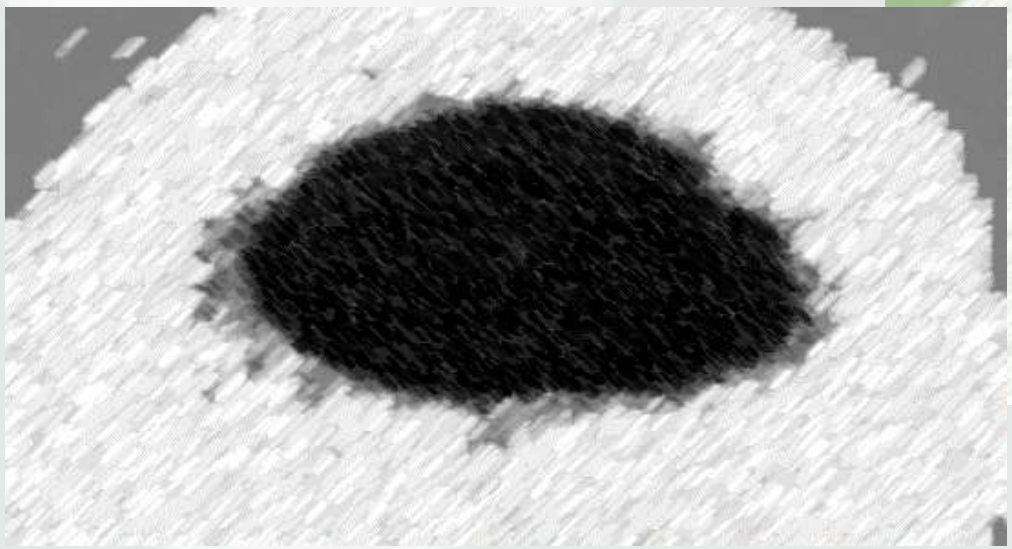
How can you evaluate and control powders?

- Particle size and shape
- Flow and Packing properties
- Segregation







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1.Particle size

- Visualisation eg microscopy
  - “you can see a lot by looking” Yogi Berra
  - Analgesic tablets that suddenly wouldn't tablet – same suppliers of materials all in spec
    - Microscopy showed change in particle shape – due to a change in site by manufacturer
    - Importance of specification: specification says less than 100µm, new suppliers material met the spec but instead of median practice size of 50-70µm of the existing supplier new potential supplier was median particle size of 10-20µm.
- Particle size or particle size distribution?
- Methods – another day
- Presentation of distribution
  - simple bar chart of weight on each sieve isn’t that helpful
- Watch outs
  - Number or weight/volume distributions
    - “I’ll have 2 Kg of potatoes and make them all small ones so I get more”
  - Mean, Median, Mode
  - Particle size distributions
  - & REMEMBER As particle size decreases, powders become more difficult to handle

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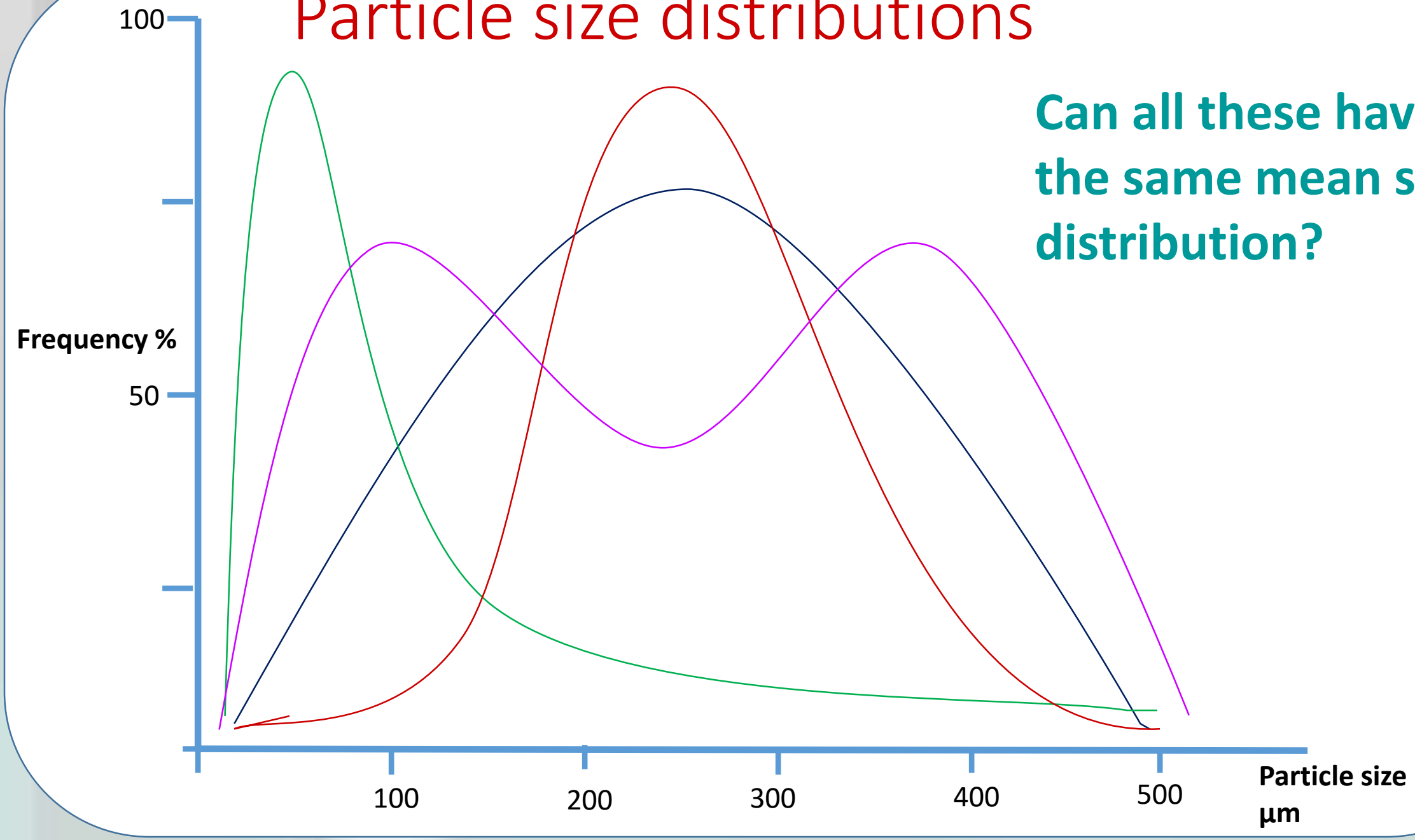
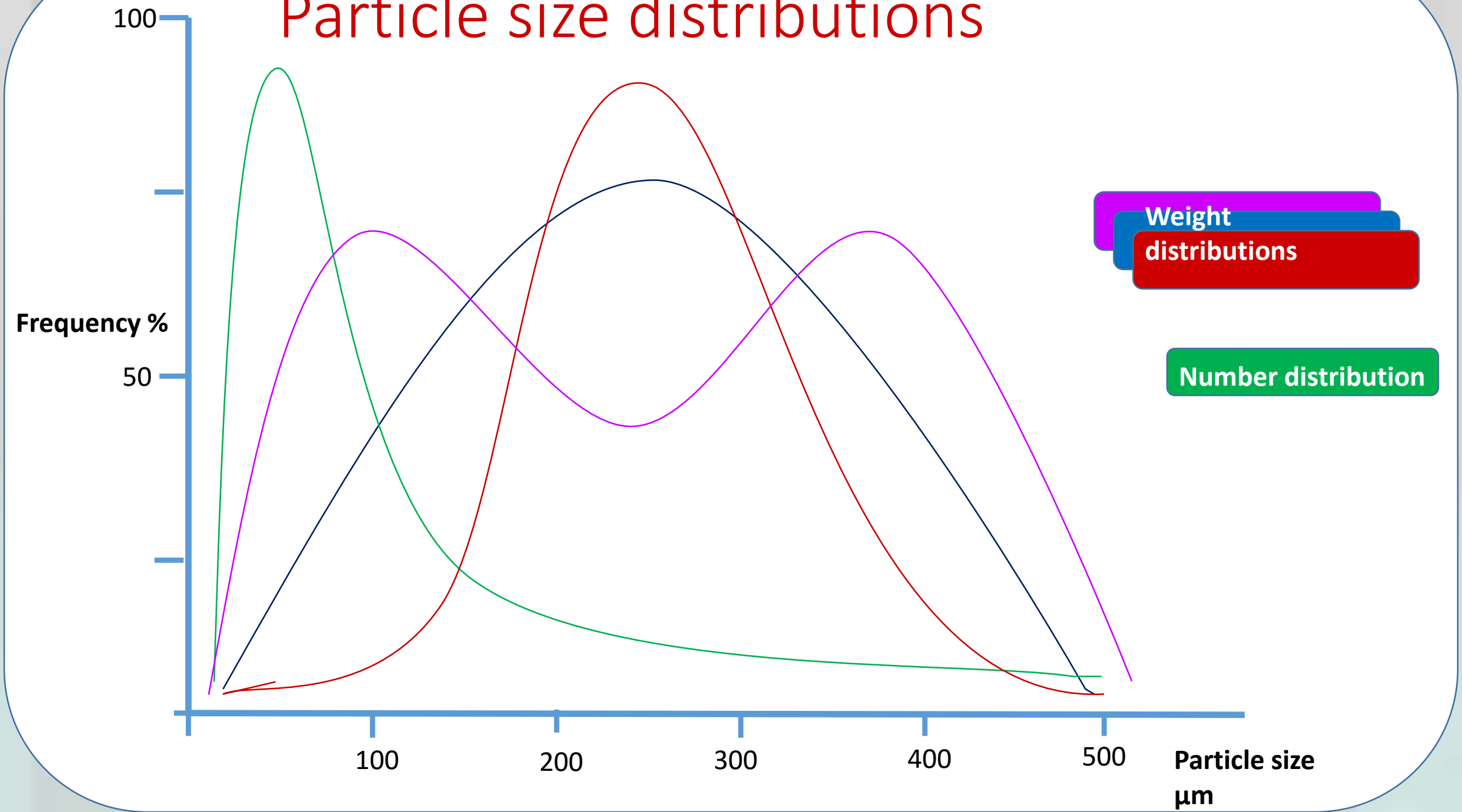
Particle size distributions



Can all these have the same mean size distribution?

Particle size distributions

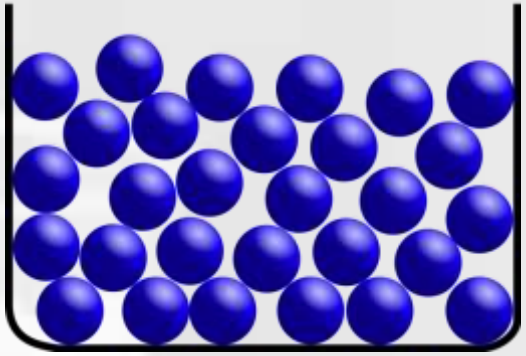


Flow and packing

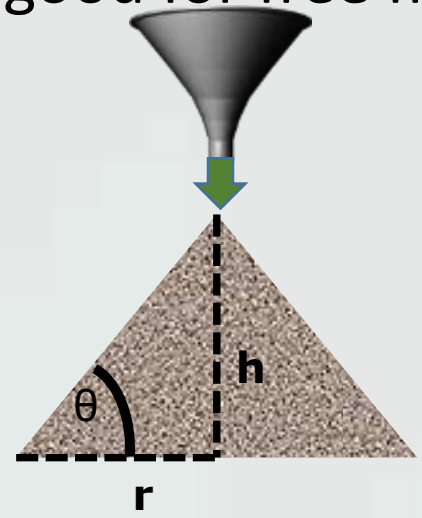
Bulk density

Weight of powder/ Volume of powder usually in g per cm^3

Ie the weight of powder particles divided by the volume of particles and air spaces between them .



- Bulk density
  - FROM: Are you likely to get your product into the container = packing properties
  - TO: Good indicator of flow – e.g. Carr's compressibility Index, Hausner ratio= flow properties Derivative of particle size, shape, surface characteristics e.g. adsorbed moisture, surface roughness
- Flow rate
  - Pouring through a funnel – good for free flowing powders ...but are they a problem?!
  - Ditto angle of repose
  - Shear cells
  - Powder rheometers



Angle of repose = θ

Tan(θ) = h/r

Excellent flow: $\theta < 25^{o}$
Good flow: $25^{o} < \theta < 30^{o}$
Adequate flow: $30^{o} < \theta < 40^{o}$
Very poor flow: $\theta > 40^{o}$

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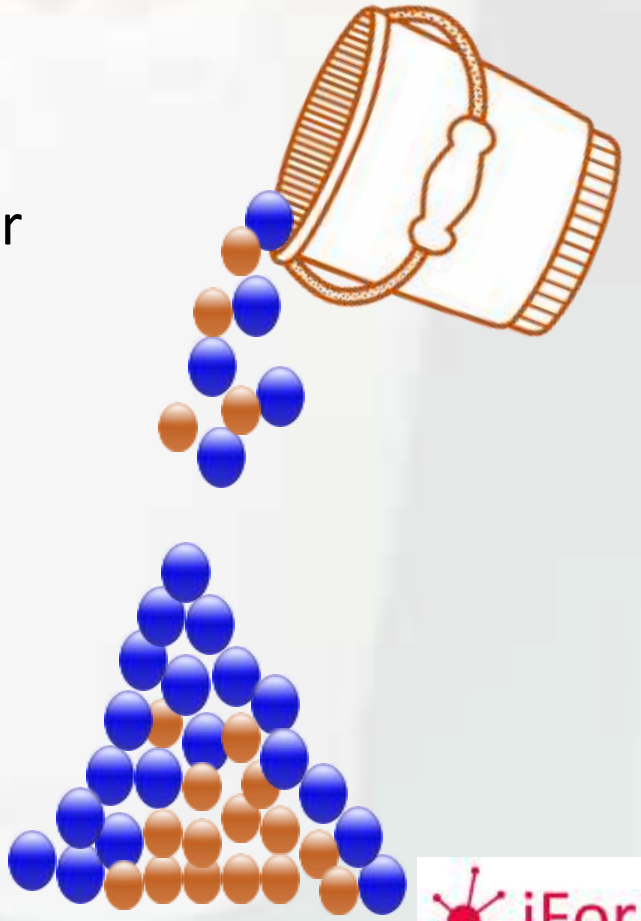
Separation/Segregation : How does it occur?

- Segregation occurs when particles in a mixture have a different size shape (density is a minor cause)
- 1. Larger particles have more momentum and travel further (Trajectory segregation)
- 2 . “Sieving” segregation Fine particles will fall into gaps or even through gaps between larger particles.
- 3. Fluidisation / Dusting -
- 4. Rise of coarse particles on vibration of a powder bed



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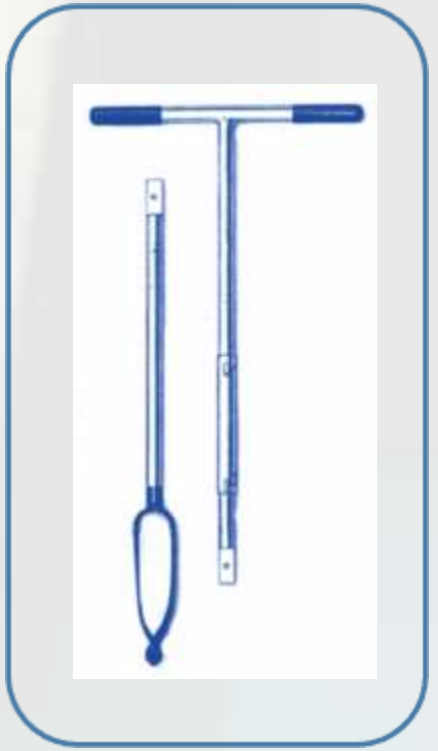
Separation/segregation: Are there established methods?

- Segregation Measurement Tester [Material Flow Systems Inc]
- Methods looking at concentrations in air entrained fines vs bulk of powder
  - eg Fluidisation Segregation Test Equipment [Jenike and Johanson Inc]
- Vibration
- Angle of repose type piles
- Important to chose a method that reflects what happens in your process!



- PLUS...Challenge of sampling and sample splitting!

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When particles you buy aren't how you want them

- Change particle size - comminution/ milling
- Classify e.g. sieving to remove fines or coarse particles (but what do you do with those!)
- Modify surfaces-
  - remove surface adsorbed moisture
  - Neutralise electrostatic charge
- Add flow aids
- Add moisture scavengers
- Further processing...

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Concluding

- Making formulations from particulates is a challenge
- Properties of powders not easy to define but there are some key parameters that can be quickly and easily measure to help us understand them
- Understanding what you are making and why to fulfil your customer promise- in particular what Quality Attributes are Critical to fulfilling your product promise- every time!
- Understand what Material Attributes are required in your raw materials and intermediates to achieve this promise CQAs
- Work out what needs to be controlled and how it is to be controlled
- Using the right tools at the right time to build product understanding will result in a controlled robust process which makes the desired product
- Application of a structured approach to product formulation will Enhancing your and your businesses productivity

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Ways We Help Companies

Technology Profiling Assignments

- “we need some new ideas for our development programme” (scouting)
- “we want to know about the pros and cons of technology x, and who we should talk to”
- “what markets could/should we enter with our technology?”
- iFormulate Consult flyer: http://bit.ly/iFo-Cnslt

Problem Solving assignments

- “we want some help solving a problem with our formulation”
- “we want some help developing a new formulation”

Training in Formulation

- Custom “in-house” training designed with client
- Open short training courses

iFormulate Consult

How It Works

- Client provides outline brief
- Discussion (under NDA if needed) to ascertain more details and refine brief
- iFormulate provides proposal and quotation
  - Proposed approach, deliverables, timescale and fees
- Staged in many cases
- Often mid-term review
- Final report, supporting information and meeting

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Any Questions?

- Participants remain muted
- Please use the GoToWebinar question boxes
- Any follow up questions or other enquiries: info@iformulate.biz
- Participants will be sent details of how to access a recording of this webinar
- Design for Formulation Webinar : http://iformulate.biz/training-and-events/iformulate-introduces-ian-jolliffe-formulationdesign/
- Next webinar: "Adhesion : A Sticky Problem" Thursday 15th September 2016
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