Sampling and mixing

Mixtures

- Type of mixtures
  - Positive mixtures- spontaneously homogenously - solutions
  - Negative mixtures- Spontaneously separate-emulsions
  - Neutral mixtures- neutral -powders
- Neutral mixtures
  - Homogeneity depends on handling and process conditions
  - Type of neutral mixtures
    - Random mix
    - “Perfect mix”
    - Ordered mix
Sampling of powders

- Why do we take samples?
- How many samples?
- Where and with what technique?
- How much?

Why do we take samples?

- To detect variation
  - validate processes
  - Product quality variations
- To describe the sample - gross sample “general prov”
- To control processes
How many samples?

- Depends on what information you want
- Remember the statistics
  - Accuracy
  - Precision
  - Variance

Variance

- Example
  - 400g A with a weight of 0.05g
  - 400g B with a weight of 0.1g
  - Sample weight 50 g
  - What is the Variance

- Effect of sample size on Variance

<table>
<thead>
<tr>
<th>Sample</th>
<th>σ</th>
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<tbody>
<tr>
<td>100</td>
<td>1.3%</td>
</tr>
<tr>
<td>50</td>
<td>1.88%</td>
</tr>
<tr>
<td>25</td>
<td>2.7%</td>
</tr>
<tr>
<td>10</td>
<td>4.3%</td>
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</tbody>
</table>
Variance as a tool to estimate number of samples

- How representative is a sample
  \[ true \ value = x_{\text{true}} \pm \frac{ts}{\sqrt{n}} \]

- Normal distribution
- 95% conf. Interval \( t=1.96 \)
- \( t \)-distribution
- 95% conf. Interval \( t=2.14 \) for \( n=15-1 \)

Number of samples
- Depends on wanted precision and standard deviations of samples
  \[ n = \left( \frac{ts_t}{E} \right)^2 \]

Where and with what technique?

- Discuss the following situations
  - A lorry comes in with grains to a mill
  - Validation of a tray dryer
  - Process sampling from a mill stream
  - Sampling from an oral dispersion in a bottle
Golden rules of sampling

- A powder should be sampled when in motion
- The whole stream of powder should be taken for many short increments of time in preference to part of the stream being taken for the whole time

How much?

- Best rule adjust to the situation (scale of scrutiny)
  - Product characteristic
  - Demand on accuracy
- However
  - To few particles will give to much variation
  - Low amount of one component increase the need of large samples for detection
Effect of scale of scrutiny

<table>
<thead>
<tr>
<th>Sample number</th>
<th>N=1000</th>
<th>N=10 000</th>
<th>N= 100 000</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>7</td>
<td>108</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>10</td>
<td>91</td>
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<tr>
<td>3</td>
<td>2</td>
<td>8</td>
<td>105</td>
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<tr>
<td>4</td>
<td>1</td>
<td>15</td>
<td>116</td>
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<tr>
<td>5</td>
<td>0</td>
<td>13</td>
<td>84</td>
</tr>
<tr>
<td>6</td>
<td>1</td>
<td>10</td>
<td>93</td>
</tr>
<tr>
<td>7</td>
<td>1</td>
<td>6</td>
<td>113</td>
</tr>
</tbody>
</table>

average /1000
0,85714286 0,98571429 1,01428571
s
0,69006556 0,32366944 0,12149858

True value 1/1000

Mixing

- Purpose: To obtain as homogenous bulk as possible
- Common problems
  - Mix in a small amount of one substance in a large bulk
  - Segregation and over mixing
  - Mixing that effects the size of the particles
Quality of a mix

- A mix is evaluated from standard mean and standard deviation (of appropriate kind)
- Problem
  - Segregated materials are not following a normal distribution
  - Is dependent on sample size
- Variance might give a better picture
- Mixing index also an alternative

\[ M = \frac{\sigma_{\text{randommix}}}{\sigma_{\text{sample}}} \]

Mixing mechanisms

- Convection
  - Due to circulating flow of powder during mixing
- Shear Mixing
  - The momentum exchange between the powder particles having different velocities
- Diffusion
  - The random motion of powder particles
The mixing curve

From Powder technology handbook

**FIGURE 6.2** Characteristic curve of mixing process (schematic).

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**Mixing**

- **Standard equipment**
  - High share mixers
  - Tumbling mixers
    - Y-cone
    - Rotating cube
    - Double cone
  - Fluidized bed
  - Agitator mixers
- **Things to consider**
  - Homogeneity
  - Risk for overmixing
    - Leading to demixing
    - Influence properties of particles
  - High share rates
    - Influence particle size for week powders
  - Scaling up
    - Not always linear
Mixer types

- Segregating
  - Rotating drum
  - V-blender
  - Double cone blender
  - Cubic blenders
- Non segregating
  - Ribbon blender
  - Nauta blender
  - Lödiger
  - Fluidizing blender (Forberg)

Characterized by Froudes number

- $Fr < 1$
  - Thrust mixers - Ribonblender, Nauta mixer
  - Free fall mixers - V-blender
- $Fr \approx 1$
  - Fluidized beds
- $Fr > 1$
  - Centrifugal mixers
  - Intensive mixers

How do they look
What to consider when designing a mixing process

- What's needed for homogenous mixer
- Mixing time
- Batch size
- Degree of filling
- Energy need
- Temperature
- Mild or shearing mixing
- Deagglomeration
- Segregation
- Handling of powder
- Cleaning
- Worker protection
- Explosion risks
- Prize
- Material
- Etc etc etc
Segregation mechanisms

- Percolation - slip through the holes
- Trajectory Effect - size or density segregation due to air drag during filing or feeding
  \[ L = \frac{v_c \rho d^3}{18 \mu} \]
- Rolling Effect - Due to friction and gravity
- Stumbling Effect, Push-Away Effect
- Elutriation effects - dusting segregation - dust layer formed on top of particle bed
- Densification

Segregation

- Can occur
  - During mixing
  - Storage
  - Transport

- Can be counteracted by
  - Narrow particle size
  - Ordered mixtures
  - Irregular and cohesive powder
  - Granulation
  - Ordered mixtures
  - Reduce vibrations