General informative article

Segregation of Powders

Dry powders or mixtures of powders may segregate during e.g. handling, mixing or transportation. Segregation will definitely influence the quality of the final product like tablet-to-tablet variation of medicines, taste experience of foods, reactivity of chemical mixtures, and effectiveness of nutritional supplements. As a consequence, it is crucial to investigate a powders' tendency to show segregation. Many industrial processes are based on blending of powder or granular components, for example:

- ◊ Pharmaceutical formulations
- ◊ Dry food blends
- ◊ Dry detergent blends
- ◊ Multi vitamins
- ◊ Plastic compounding
- ◊ Flavours

Figure 1. Segregated poweder blends

Considerable efforts are often devoted to the mixing of several different species of powders in order to create a homogeneous mixture of the intermediate or final product. Nevertheless, a well mixed system may easily segregate during handling, e.g. when charging or discharging a bin or big-bag, transportation by truck or by rail, via conveyers or by pneumatic transport in pipe-lines, but this could also happen when loading the powder in e.g. a tablet press for the production of pharmaceutical tablets. Not only mixtures of different components but also chemically homogeneous powders may segregate if the material has a broad particle size distribution. Segregation can create regions of coarse and fine particles, which could have different properties.

Segregation Mechanisms

The extent to which segregation will occur in an industrial situation depends on the physical characteristics of the particles, handling equipment, process, and environment.

Figure 2. Segregated powder blends

Varieties of mechanisms which can cause segregation are fluidization, sifting, and dusting which are identified as:

- ♦ Trajectory segregation when particles are caused to move through air e.g fall from the end of a conveyer belt.
- ♦ Percolation of fine particles when a mixture is disturbed, causing rearrangement of particles, finer material travels down filling inter-particulate gaps and forcing larger particles upwards e.g. stirring, shaking, vibration or pouring into a heap.
- ♦ Rise of coarse particles on vibration when a mixture of particles of different sizes is gently shaken encourages larger particles to rise to the surface.
- ♦ Elutriation segregation in an aerated powder, especially close to the point of fluidisation, coarse and or dense particles settle to the bottom of a container leaving fines disproportionately distributed in the upper layers.

Testing Procedure

Figure 3. Miniaturised riffler for sub-diving small sample sizes

The fluidisation segregation tendency of powders is assessed in accordance with the ASTM D 6941 standard. By means of a rotary riffler the sample is divided in representative lots and a fixed representative volume of powder is placed in the Fluidisation Tester. The powder is fluidised with a series of air flow rates for specified times, creating horizontal layers of particles with different sizes and or densities. Coarse and high-density particles settle or are driven into the bed while small and light particles remain fluidised near the surface.

Figure 4. Delft Solids Solutions' test rig for fluidization testing

The top and bottom fraction is collected and sub-divided by a rotary riffler. In the so-obtained representative lots the particle size distribution and skeletal density in both top and bottom fraction is determined.

These parameters are used to quantify the segregation degree by means of a segregation factor. Additional representative lots can be returned to the customer for chemical assay of e.g. active ingredients (API).

Typical particle size distribution of fresh sample and the obtained top and bottom fraction evidencing the segregation tendency.

The sifting segregation tendency of powders is assessed in accordance with the ASTM D 6940 standard.

Figure 5. Set-up for sifting segregation testing

A representative sample of a bulk solid is placed in the upper hopper and discharged to form a heap within the lower hopper, allowing segregation to take place.
The segregated powder is discharged in a funnel flow pattern intended to recover zones of segregated powder. Specimens are collected from the dis charge stream and sub-divided by a rotary riffler to be tested relevant to the application.

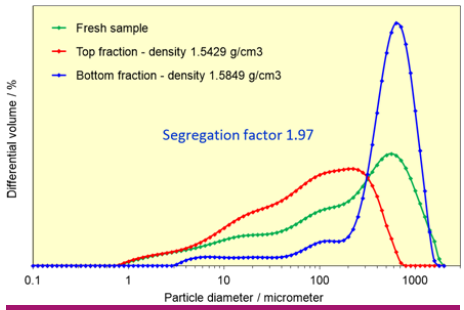

Figure 5. Typical particle size distribution of fresh sample and the obtained top and bottom fraction evidencing the segregation tendency.

Solving problems

Different ways exist to reduce or eliminate segregation problems: modification of the feed (e.g. particle size distribution, additives), adaption of existing equipment (e.g. bin or hopper design), or alteration of the current process (e.g. transfer rates). Problem solving should be an interactive process between Delft Solids Solutions and the customer.