

Segregation of powders during gravity-driven flow through ducts

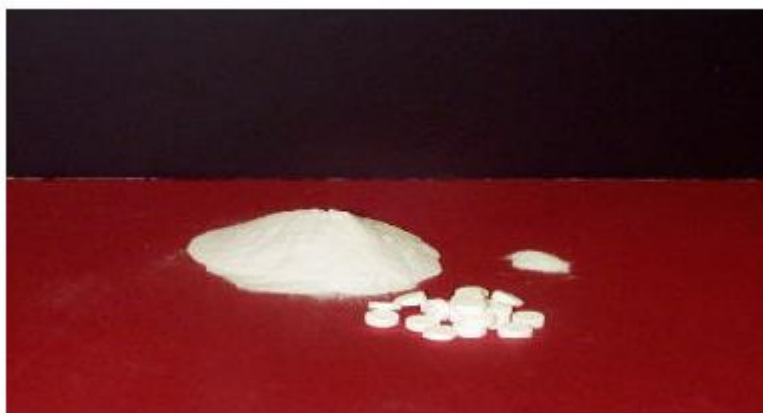
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Abstract

During certain pharmaceutical operations, such as encapsulation and tableting, problems arise due to the handling, transport, and storage of powders. Considerable effort is often expended to blend or mix several different species of powders (of various sizes, shapes and densities), only to have the mixture de-mix or segregate when transported. A well-mixed system may segregate during transport to the tablet press and this may lead to pills that do not fall within Food and Drug Administration specifications. At present there is a lack of understanding of the relationship between the collective motion of the particles, and the particle properties, boundary conditions and the history of the system. The goal of this work is investigate the physical mechanisms of segregation during gravity-driven flow of powders. Sedimentation or dropping experiments have been carried out in vertical ducts (or pipes) for different particle systems and various process conditions. Glass beads and pharmaceutical excipients have been used as the initial test materials. The glass beads of different size were dyed different colors to allow one to observe the flow and segregation. The experiment involved dropping a fixed amount of powder in a pipe with a sealed bottom end. Once the powder had come to rest at the bottom of the pipe, it was analyzed for segregation as a function of height. The degree of segregation was quantified using sieve analysis of the material. During a vertical drop, the air drag force influences the particles according to their size, density and shape, causing

different settling velocities, which ultimately leads to a distribution of settling velocities in the mixture. The results of these experiments are being used to develop an understanding of how the different magnitudes of the air drag and gravity forces can lead to segregation of powders during vertical drops.

Introduction

Pharmaceutical companies make pills by mixing powder ingredients. In the case of a tablet or pill, a small portion of it is the active medicine. It is important to maintain the percentage of each component in the formulation from pill to pill, to achieve effectiveness of the drug and to avoid possible increases in side effects, especially in cases where any small variation could be significant to the patient's health.

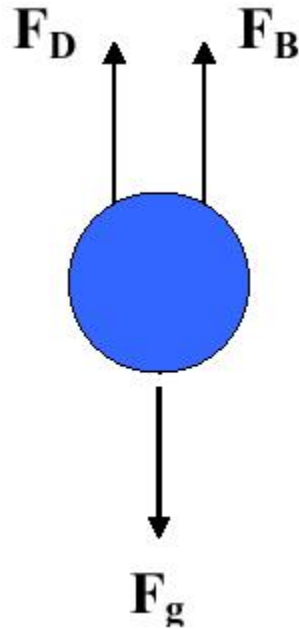
Much effort is invested to mix the different components of a pharmaceutical formulation which later may segregate in processes that involve handling, transport and storage. By definition, segregation is the tendency of particles to separate according to their individual physical properties. The purpose of this work is to develop an experimental procedure to detect and quantify the degree of segregation of powders during vertical drops.

Factors influencing powder segregation

Segregation of powders can arise due to differences in physical and mechanical properties of the constituent particles. The physical properties that are known to induce segregation are: size, shape, density, cohesivity, friction, surface texture, modulus of elasticity and coefficient of restitution. The mechanisms of segregation are associated with different process conditions, such as, vibration, fluidization, pouring in a heap, fall height, rate of feed, moisture and mixing ratio. The main mechanisms of segregation are: displacement, sieving, percolation, sifting, rolling, angle of repose, trajectory, impact, air current, fluidization and push-away effects.

Vertical drops - effects on a single particle

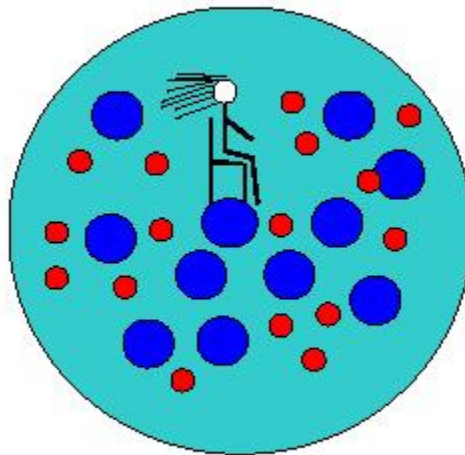
- Force of gravity, F_g
Increases with density and size of the particle (e.g. For a sphere, F_g is proportional to R^3)
- Buoyancy force, F_B
Increases with the density of the surrounding fluid and the particle volume (e.g. For a sphere, F_B is proportional to R^3)
- Drag force, F_D
Increases with particle size (e.g. For a sphere in a fluid with a low Reynolds number, F_D is proportional to R)



Vertical drops-collective effects

Particles experience the following forces:

- Forces due to presence of other particles.
Collisions or contacts
Hindered motion
Free/excluded volume effects
Particles must diffuse (past others)
- Cohesion
Relative magnitudes of particle-particle cohesive forces and body forces affect segregation. Body forces are the sum of the drag, gravitational and buoyancy forces.
- Wall effects

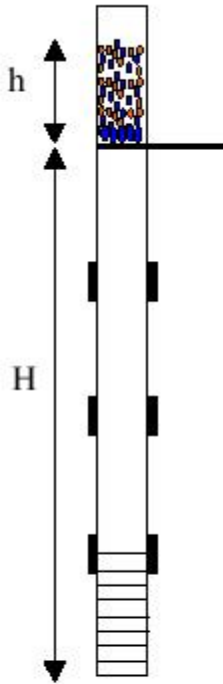


Experimental methods

The apparatus is composed of a vertical pipe with a collection unit at the bottom. The collection unit consists of sequential rings, which allow for a size distribution analysis by layer after the material is dropped.

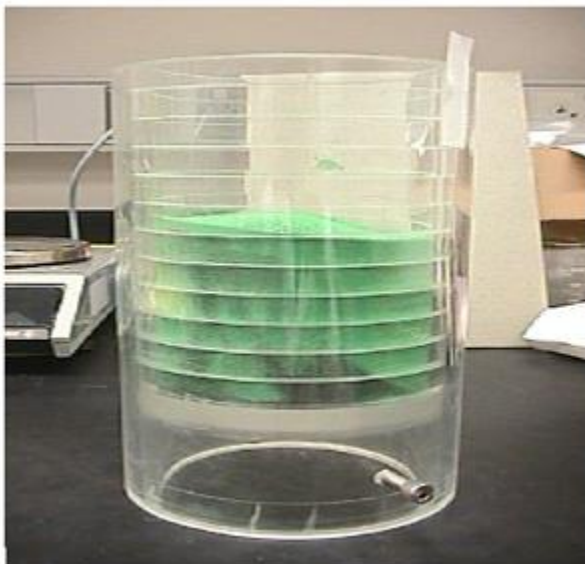
The main characteristics of the apparatus used to quantify particle segregation during a vertical drop are:

- Internal diameter of 4 inches
- Drop height, H , can be set from 6 inches to 6 feet.
- Initial condition, well mixed system
- Collection unit, can separate samples by height



Collection unit

The collection unit permits separating the powder by height in 0.5 inch increments. This consists of a section of Plexiglass pipe which has been cut into half inch ring sections. These are held together before the dropping experiment and then separated afterwards. The rings are pushed off one another from the top, in order to collect samples on each ring. The samples are sieved for size analysis.



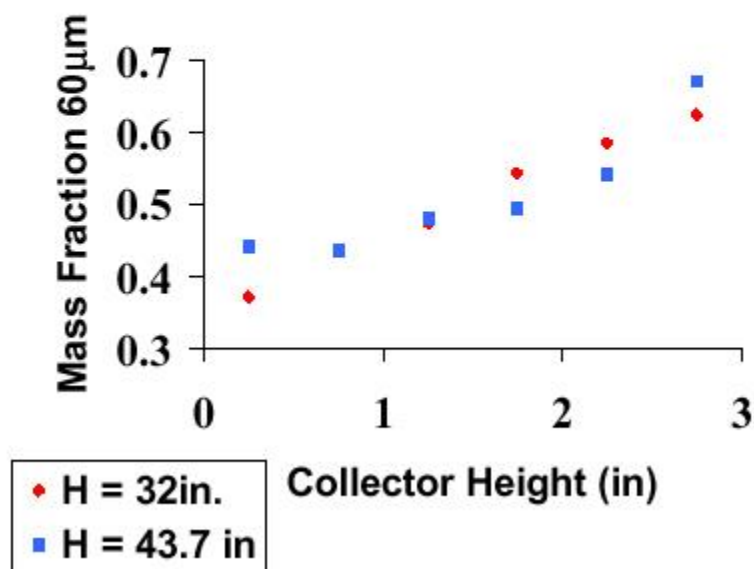
Above: Top view of the collection unit.

Left: Side View of the collection unit.

Results

One mixture consisted of equal masses of two different types of glass beads, one with an average diameter of $60\text{ }\mu\text{m}$, with a diameter range between $30\text{ }\mu\text{m}$ to $80\text{ }\mu\text{m}$, and the other one with an average diameter of $330\text{ }\mu\text{m}$, with a diameter range between $290\text{ }\mu\text{m}$ to $420\text{ }\mu\text{m}$. The density of both type of beads were the same and equal to 2.2 g/cm^3 .

60 μm and 330 μm Glass Beads $d_1/d_2=5.5$



Conclusions

The results obtained, demonstrate the feasibility of using the apparatus to examine segregation. It can be concluded that during vertical drops, the air drag force influences the particles according to their size, density and shape, causing different settling velocities. As a consequence, the distribution in settling velocities leads to segregation during the vertical drop of the mixture.

Future work

In future work, this experimental procedures developed will permit us to examine the effect of system parameters on the degree of segregation. The parameters that will be studied include drop height, amount of material dropped, average particle size, and particle density. The results of these experiments will be used to develop a heuristic model to explain segregation during the vertical drop. A dimensional analysis will be carried out in order to interpret the results and investigate which effects are most important. The first parametric studies will be carried out for glass beads, with later work focused on pharmaceutical excipients.

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