Segregation Effects in Three Types of Mass Flow Hoppers By Dr. Kerry Johanson Material Flow Solutions, Inc.

Abstract

Mass flow is often suggested as a means of solving all segregation issues [1], [2], [3]. Although traditional mass flow may be a necessary condition to solve several segregation issues, it is not a sufficient condition. The solution to segregation in process equipment requires matching the segregation pattern and magnitude to the velocity profile in the specific mass flow bin or hopper. Many mass flow hoppers produce flow along the walls. However, not all of these designs are sufficient to mitigate segregation. This paper addresses segregation prevention in typical conical hoppers, cone-in-cone hoppers, and Diamondback® or plane flow hoppers. All of these bins can be designed as mass flow devices. Nonetheless, because of differences in velocity profiles in each of these bins, they offer differences in the ability to mitigate segregation. The focus of this paper is to compare the segregation prevention aspects of using these three types of mass flow bins with radial segregation patterns as well as axial segregation patterns. The radial stress theory was used to compute velocities in the bin. These velocities were then used to estimate the expected segregation mitigation for radial as well as axial segregation patterns. Six conditions were compared. These include conical hoppers, cone-in-cone hoppers, and Diamondback® hoppers. In each case, the effect of both radial and axial segregation patterns was examined. In each case, segregation mitigation plots were generated and compared to determine which hopper configurations yielded the best segregation prevention. It was determined that the cone-in-cone geometry offers the best segregation prevention, provided it is designed to have an overall velocity profile that is slower in the center than in the annular region.

1.0 Introduction

Two modes of segregation that are important to understand in processing situations. The first mode of segregation is the segregation that occurs when a piece of process equipment is filled through a batch filling mode and then emptied completely, or at least mostly, during the course of the processing. In this case, the goal is to assure that all of the material leaving the process equipment from a fill and then empty mode is within allowable content uniformity targets.



The second mode of segregation pccurs in a process that is mostly continuous in nature. In this case, we are concerned about the segregation that happens at a particular point in the process over some characteristic time frame. The goal is to minimize the variation of key components with time as material passes through the system in a continuous fashion. Ideally, all the material passing through the continuous process should be within the allowable content uniformity range.

In both modes of segregation knowing the velocity profile, process geometry, and segregation pattern in the process equipment can help determine the variation in concentration of key components in a segregating mixture. The velocity profile is dependent on the flow properties of the bulk material and the process geometry. The segregation pattern in the process is dependent on the measured radial segregation due to pile formation and the process geometry (i.e. where the piles form). There is a secondary relationship between the flow properties and segregation of the material that may be important in some cases. Local segregation could change the key flow properties that induce the velocity profiles in the first place. However, for the purposes of this paper, we will consider this effect a minor influence.

There is a tendency to believe that the best way to handle a segregation problem is to approach it from a brute force computational approach using DEM [4], FEM, CFD, and other calculation techniques to model the particle scale segregation, bulk scale flow, and process scale effects caused by operation parameters and geometry changes. This is actually a very complex problem and many numerical simulations would need to be done to correlate the effect of key variables in the process segregation. But at the end of the day, if we know the segregation pattern and the velocity profile in the bin and the process geometry we can estimate the segregation in the process and thereby determine the cause of the majority of the segregation of material leaving a process. We do not need to know the causes of particle scale segregation, just the pattern and magnitude to estimate what occurs in the process. Furthermore, this paper is limited to consider what process geometries would reduce the expected segregation due to fillthen-empty and continuous operations. This paper will consider three process geometries (simple cone, cone-in-cone, and plane flow hoppers) and rank their benefit as segregation reduction devices.

2.0 Methods

We will consider the velocity profile in a piece of process equipment where bulk solids flow through it. It is a well-established fact that the flow pattern in a piece of process equipment depends on the wall friction angle, the effective angle of internal friction, and the shape of the process geometry. This gave rise to the concept of a mass flow limit in the process geometry where the material ceases to flow along the walls in some geometry, with some solids possessing, and a set of adverse flow properties. It is well



established that there is a relationship between the friction angle at the hopper wall and the hopper angle that suggests that, as the friction angle increases, the flow in a conical piece of process equipment will eventually stop flowing at the walls (Figure 1). This effect describes the transition from mass flow to funnel flow where the flow at the walls ceases, resulting in a funnel flow active flow channel that expands at some critical angle up from the outlet.



Figure 1. Typical mass-flow-limiting line for bulk material

This mass flow limit was the direct result of applying the radial stress theory to the flow of bulk material in conical hopper geometries [5], [6]. The radial stress theory is well established and a very brief summary of the method is given here for those less familiar with its use. The theory starts with the equations of motion with the acceleration terms neglected equations 1 and 2.

$$\frac{1}{r^{2}} \cdot \frac{\partial \left(r^{2} \cdot \sigma_{r}\right)}{\partial r} + \frac{1}{r \cdot \sin(\theta)} \cdot \frac{\partial \left(\tau_{r\theta} \cdot \sin(\theta)\right)}{\partial \theta} +$$

$$\frac{1}{r \cdot \sin(\theta)} \cdot \frac{\partial \tau_{r\phi}}{\partial \phi} - \frac{\sigma_{\theta} + \sigma_{\phi}}{r} = \gamma \cdot g_{r} - \frac{\partial P}{\partial r}$$
(1)

$$\frac{1}{r^{2}} \cdot \frac{\partial (r^{2} \cdot \tau_{r\theta})}{\partial r} + \frac{1}{r \cdot \sin(\theta)} \cdot \frac{\partial (\sigma_{\theta} \cdot \sin(\theta))}{\partial \theta} +$$

$$\frac{1}{r \cdot \sin(\theta)} \cdot \frac{\partial (\tau_{\theta\phi})}{\partial \phi} + \frac{\tau_{r\theta}}{r} - \frac{\cot(\theta)}{r} \cdot \sigma_{\phi} = \gamma \cdot g_{\theta} - \frac{1}{r} \cdot \frac{\partial P}{\partial \theta}$$
(2)

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The Mohr-Coulomb criteria, or constitutive relation, is then used to relate the stresses within the bulk material to each other as shown in Figure 2. The stress is assumed to be a linear function of the distance from the apex of the converging conical geometry with some unknown function $s(\theta)$ that describes how the stress changes with the theta position in the hopper. The analysis also includes the angle omega ($\omega(\theta)$) which is the direction of the major principal stress relative to the spherical coordinate system in a conical geometry.



 $\sigma = \gamma \cdot g \cdot r \cdot s(\theta)$ Radial Stress Assumption



Application of the Mohr Columb constitutive relation and the radial stress assumption gives rise to two ordinary differential equations (3) and (4) that when solved with boundary conditions equation (5) and (6) determines the stress profile in the conical hopper during steady flow.

$$\frac{d\omega}{d\theta} = \frac{1}{2} \cdot \frac{\cos(\delta)^2 - (\cot(\theta) \cdot \sin(2 \cdot \omega) + 1 + \cos(2 \cdot \omega)) \cdot \sin(\delta) \cdot (\sin(\delta) + 1)] \cdot s + \cos(\theta) + \cos(\theta) \cdot \sin(\delta) \cdot \sin(\delta) + \cos(\theta) - \sin(2 \cdot \omega) \cdot \sin(\delta) + \cos(\theta) - \sin(\delta) \cdot \sin(\delta) + \cos(\theta) - 1}{(\cos(2 \cdot \omega) + \sin(\delta)) \cdot s \cdot \sin(\delta)} - 1$$
(3)

$$\frac{ds}{d\theta} = \frac{\begin{bmatrix} \sin(2\cdot\omega) + \sin(\delta)\cdot\cot(\theta)\cdot\cos(2\cdot\omega) - \\ \sin(\delta)\cdot\cot(\theta) - \sin(\delta)\cdot\sin(2\cdot\omega) \end{bmatrix} \cdot s +}{\cos(2\cdot\omega)\cdot\sin(\theta) + \sin(2\cdot\omega)\cdot\cos(\theta)}$$
(4)

$$\omega = 0 \quad at \quad \theta = 0 \tag{5}$$

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$$\omega = \frac{1}{2} \cdot \left(\phi_{we} + a \sin\left(\frac{\sin(\phi_{we})}{\sin(\delta)}\right) \right) \quad at \quad \theta = \theta_{w} \quad (6)$$

The application of the radial stress theory can also be used to estimate the velocity profile in the hopper but requires the use of a flow rule which states that the direction of major principal stress coincides with the direction of major principal strain rate and gives rise to equations (7) and (8).

$$\tan(2 \cdot \omega) = \frac{\frac{1}{r} \cdot \frac{\partial u_r}{\partial \theta} + \frac{\partial u_{\theta}}{\partial r} - \frac{u_{\theta}}{r}}{\frac{\partial u_r}{\partial r} - \frac{u_r}{r} - \frac{1}{r} \cdot \frac{\partial u_{\theta}}{\partial \theta}}$$
(7)
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$$u_r = \frac{-V(\theta)}{r^2} \qquad u_{\theta} = 0 \tag{8}$$

This flow rule can be solved to yield the velocity profile in a conical hopper during a steady flow, equation (9). Please note that this velocity is a function of the direction of principal stress relative to the coordinate direction.

$$V = V_0 \cdot \left(\frac{r_0}{r}\right)^2 \cdot \exp\left(-3 \cdot \int_{\theta_0}^{\theta_w} \tan(2 \cdot \omega) \cdot d\theta\right)$$
(9)

The application of the radial stress and velocity theory to hoppers leads to the computation of velocity profiles as a function of the wall friction angle and effective angle of internal friction as shown in Figure 3. Please note that all of these velocity profiles are mass flow profiles, but that the velocity ratio between the center and the side depends very strongly on the wall friction angle. If the wall friction angle increases, the velocity profile tends to become much steeper across the hopper. This difference in mass flow velocities is one key reason why just using a mass flow hopper is not sufficient to prevent the segregation of powder and granular materials.





Figure 3. Typical mass flow velocities in conical bins given by radial stress theory

The radial stress theory can also be applied to the cone-in-cone geometry to compute the mass flow limit for that geometry [7]. Figure 4 shows a typical result for the case where the inner cone is at 10 degrees and the outer cone slopes at 20 degrees measured from the vertical. The solid line is the traditional mass flow line for the inner cone. The dotted line is the solution of the radial stress theory for the case where the inner cone is 10 degrees. Note that the dotted line approaches an asymptotic solution which is the boundary between mass flow and funnel flow behavior in the annular region. Combining the two radial stress solutions gives rise to the new mass flow limit for flow in a cone-in-cone device. It is clear that this device can extend the mass flow behavior to flatter hoppers than a simple cone can obtain.





4. Typical radial stress solution for cone in cone geometry

The radial stress and velocity theory can also be used to compute the velocity profiles in cone-in-cone hoppers. However, the dimensions of the inner and outer cones can be adjusted to create overall velocity profiles where the flow in the inner cone equals the flow in the annular region or where the flow in the inner cone is some multiple of the flow in the annular region as shown in Figure 5.



Figure 5. Potential flow profiles in cone-in-cone geometries

The velocity profiles can be computed from the application of the radial stress and velocity theory for plane flow devices such as the Diamondback® hopper. This hopper is a combination of two plane flow hoppers placed on top of each other. The first plane flow hopper is chisel-shaped with a circular cross-section and transitions down to an oval cross-section with convergence in only one direction. The second hopper is an oval hopper that transitions down to a circular cross-section with convergence in only one direction. In this case, the velocity in each hopper can be computed and then combined to give a velocity in the composite geometry. It is important to note the overall velocity above a Diamondback® hopper can be computed by first normalizing the velocity in the chisel-shaped and oval hopper section such that the average area

Figure 4.



under the velocity curve equals 1.0. The velocity profile in the oval hopper propagates up from the hopper outlet in accordance with the dimensionless velocity profile, the average flow rate, and the cross-sectional area, yielding fast flow in the center of the oval bin and a slower flow at the edge. However, this induces a nonuniform velocity along the length of the oval outlet at the bottom of the chisel-shaped outlet which propagates up through the chisel-shaped hopper. The velocity in the chisel-shaped hopper is normalized and applied to the overall hopper geometry by multiplication of two normalized velocity profiles by first rotating the chisel-shaped hopper velocity by 90 degrees and multiplying it by the velocity of the oval hopper. This yields a velocity profile that has the combined effect of both hopper sections (Figure 6).



Figure 6. Typical velocity profile in Diamondback® hopper





SPECTester[®] Measures Segregation Potential of Multi-component Mixtures Using Reflectance Spectra

- 1. Feed material into cell to form a pile
- 2. Measure the NIR spectra of regions along the pile
- 3. Fill component trays with pure components and measure the NIR spectra of pure components
- Using nonlinear spectral mixing rules, compute the concentration of key components along pile to yield segregation profile

Figure 7. Segregation tester used to measure the segregation pattern of the material

The next piece of information required is to understand how a particular material might segregate when charged onto a pile. We measured that profile using the SPECTester® available commercially from Micromeritics [8]. This tester forms a pile and then uses differences in reflectance spectra [9] to compute the concentration profile of key components along the length of the pile and expresses the segregation pattern as a function of dimension radius where 0.0 is the top of the pile a 1.0 is the bottom of the pile. Please note we have not said anything about the cause of segregation. Fines can sift down through the material of coarse particles causing segregation. Differences in frictional characteristics between particles can cause particles to slide down the pile at different relative velocities, resulting in segregation. Air currents induced in free fall streams can be liberated during pile impact and carry fine particles down the pile in the freeboard space, resulting in segregation. Differences in coefficients of restitution can cause particles to bounce to different relative locations down the pile. All of these effects combine to give the segregation pattern for a particular material. Some effects happen within the thin shearing layer on the top of the pile, some effects happen in the freeboard space above the pile, and some effects happen right on the pile surface. Regardless of the reason, each material will have a characteristic segregation pattern that can be measured. A typical API mixture consisting of MCC, lactose, API, glycolate and magnesium stearate was used to evaluate the segregation in conical, cone-in-cone, and Diamondback® hoppers as shown in Figure 8.





Figure 8. Segregation profile measured for a typical pharmaceutical mixture

3.0 Results

The measured segregation profile was used, along with the calculated hopper velocities given by radial stress and velocity theory, to estimate the segregation leaving hoppers when the hopper was first filled and then emptied. This was done numerically by deviding the hopper into zones of equal volume and placing a marker randomly within that small differential volume at some x, y, and z location (Figure 9). The dimensionless position from the top of the charge point to the location of interest was computed and the concentration of key materials from the segregation test was assigned to this spatial coordinate. The radial stress and velocity theory was then used to compute the local velocity in the hopper and the markers were moved using particle tracking methods to generate the flow of segregated material through the hoppers. However, in some cases during discharge the velocity profile would generate a top surface that sloped at an angle steeper than the angle of repose. In this case the material should cascade down the pile and not follow the typical mass flow velocity profile. Thus, whenever this situation was detected a cascade velocity in the direction of the repose angle was employed to move the marker position to a new location [10]. The position of the markers leaving the bin was monitored and the average concentration in a preselected time exit range was computed.





Figure 9. Position of markers in bin during flow based on radial stress theory and cascade velocity

The result of this analysis is generation of concentration profiles as a function of discharge time resulting from an operation scenario where a powder mixture was first charged into a bin, segregated during filling, and then completely emptied from the bin. This senerio is common practice in batch processes where a drug formulation is created, placed in a surge bin, and then transported to a tablet press and used completely. All the material flowing from this process must be within allowable content uniformity ranges for the process to be validated. Typically acceptable content uniformity ranges are between 95% and 105% of the mean concentration for European standards and between 90% and 110% of the mean concentration for American standards. Thus, a content uniformity standard can be selected and then the analysis performed to determine when and how often the material leaving the feed system is outside the bounds of the content uniformity range (Figure 10). It is important to note that the selection of allowable content uniformity range is an itergral part of determining if a particular process is acceptable to prevent segregation of a mixture. This method provides a systems approach to estimate the benefit of using a mass flow design to mitigate segregation. Prior art and design among particle science and technology practitioners would rely on the general comments stating that segregation can be best helped by using mass flow designs but did not offer any guidance as to what type of mass flow is best. This approach will answer that question.





Figure 10. Typical concentration profiles resulting from radial stess velocity and cascade velocity profiles in conical hoppers

The concentration profiles were computed for a condition where the wall friction was a particular value (25.4 degrees) and the angle of the hopper was changed to give different velocity profiles. The concentration of API was used as the segregation indicator for this example. Note that the funnel flow profile causes the concentration of API to initially drop below allowable bounds, then increase above allowable bounds, and finally decrease again to below allowable bounds. The amount of material that was within allowable bounds increases as the hopper becomes steeper. However, even if the conical hopper was 5 degrees measured from the vertical, there is still some material at the end of the discharge cycle that falls outside the allowable content uniformity bounds. If we take the amount of material that is within the allowable content uniformity range as the segregation indicator, then 100% would be a perfect operation. However, in the case of placing this drug mixture in a conical hopper with an allowable content uniformity range of 95% to 105% and operating in first fill and then empty mode, the best we can expect is that 87.2% of material will be within acceptable limits.





Figure 11. Segregation limits for drug mixture place in a conical hopper subject to first fill and then empty mode of operation

This type of behavior is also found with other types of materials that have a nearly linear segregation profile of key components. Consider the segregation index as described above as the fraction of material within acceptable range, 1.0 being perfect operation and 0 being fully segregated. In this case, the segregation profile is a linear function of position on a pile where the fraction of good material was plotted as a function of the wall friction angle and the angle of the conical hopper as shown in Figure 12. The point where the contour lines bunch up is the funnel flow limit. Obviously, funnel flow designs are very poor in segregation prevention. However, there is still significant segregation in conical hoppers that are first filled and then emptied, even in very steep conical hoppers.





Figure 12. Typical segregation in conical hoppers that operate in a fill then empty mode: 1.0 is a perfect operation and 0.0 is fully segregated material

This paper also considers the flow in a cone-in-cone geometry where the outer cone is 20 degrees measured from the vertical and the inner cone is 10 degrees measured from the vertical. In this case, the analysis was carried out to investigate the effect of the ratio of the average velocity in the inner cone and the annular region. Velocity ratios between 0.5 to 1.5 were investigated using the radial stress and velocity theory to determine the best configuration. The results of this analysis are summarized in Figure 13. It is clear that operation in a funnel flow regime is not a good idea to prevent segregation. If the velocity profile between the inner and outer cones is 1.5, then the degree of segregated material leaving the system is also very bad and is actually slightly worse than operation in funnel flow. However, as the ratio of velocities between the inner and outer cone approachs 1.0, the amount of material leaving the bin that is outside the allowable content uniformity range is very low. The best segregation profile occurs when the ratio of the velocity is 0.9 and this results in 98.3% of the material leaving the bin to be within the allowable content uniformity limits (Figure 14). Operation at velocity ratios much less than 0.9 also causes bad segregation during a fill then empty mode of operation.





Figure 13. Segregation of drug mixture leaving cone-in-cone hopper as a function of the velocity ration between the outer and inner cones for bins that operate in a fill then empty mode: 1.0 is perfect operation, 0.0 is fully segregated material



Figure 14. Segregation index of drug mixture leaving cone-in-cone hopper as a function of the velocity ratio between the outer and inner cones for bins that operate in a fill then empty mode: 100% is a perfect operation, 0% is fully segregated material



The method used above is a reasonable method based on a systems approach using the radial stress and velocity theory to characterize segregation of material leaving the bin during a fill then empty mode of operation. However, in some cases, the plant operates the process in a mostly continuous mode where material is made in a batch mode then dumped into a surge bin where the level in the surge bin is maintained at a more or less consistent height. The material leaving the batch system can segregate as indicated in the analysis above but the main surge bin rarely operates in a complete discharge mode. This mode of operation requires a different analysis to estimate the segregation leaving the system. The goal, in this case, is to maintain the content uniformity to some allowable value during all operation times where the input concentration may vary from batch dump to batch dump. In this case the computed radial stress velocity profiles can be used to estimate the residence time distribution for continuous operation through the bin (Figure 15). This residence time distribution function can be used to compute the expected concentration profile leaving/passing through the bin and then examined to determine when and if the concentration profile is outside allowable content uniformity ranges. The continuous flow mode analysis requires that the concentration during a fill and then empty batch mode be used as an input concentration to the continuous surge bin. The analysis above was done for the drug mixture segregation profiles for a conical hopper where the feed location was offset from the centerline of the hopper, but the bin was filled and then completely emptied. It was assumed that as soon as the feed bin was emptied another identical feed bin of the same size and geometry was placed above the surge bin in such a way as to maintain the level in the surge bin to be relatively constant. This gave a cyclic input concentration profile that had a frequency equal to the size of the batch feed hopper. One of the important parameters was found to be the relative size of the batch bin to the surge bin. The concentration of API leaving the surge bin was plotted as a function of the number of surge bin volumes that were passing through the process. The size of the surge bin was divided by the size of the batch feed bin to give a parameter that described the number batch cycles passing through the surge bin. If this ratio was 1.0, then the batch bin was the same size as the surge bin. If this ratio was 2.0, the batch bin was $\frac{1}{2}$ the capacity of the batch bin. The output concentration was then calculated given the cyclic input batch discharge concentration and residence time distribution. The ratio of the maximum concentration fluctuation ($C_{max} - C_{min}$) between the input and output concentrations was used to determine the effectiveness of the continuous operation on reducing the concentration. Based on the assumed input concentration variation, the maximum concentration span (C_{max} -C_{min}) would need to reduce to 31% of the current value to assure that the output concentration profile was always within the 95% to 105% content uniformity range (Figure 16).





Figure 15. Typical residence time distribution functions and output concentration profiles computed from the radial stress velocity profiles



Figure 16. Expected reduction in the concentration variation for flow of assumed input concentration through a cone-in-cone bin as a function of the number of input cycles per bin volume

In the case of the cone-in-cone, changing the velocity profile to create a more uniform velocity actually reduces the ability of the bin to mitigate the segregation passing through the surge bin. Thus, the segregation velocity profile that helps prevent



segregation of a fill and then empty mode of operation is much worse when applied to the case of trying to mitigate segregation during continuous flow modes of operation. Segregation mitigation in this mode of flow requires significant differences in velocity within the bin.



Figure 17. Expected reduction in the concentration variation for flow of assumed input concentration through a cone-in-cone bin as a function of the number of input cycles per bin volume showing the effect of velocity profile in cone-in-cone bin

The same is true when the segregation model is applied to flow in a conical hopper. The situation where the velocity profile across the bin is relatively steep (high friction angle) gives better segregation mitigation than the case were the velocity profile is more uniform (low friction angle). This is summarized in Figure 18. It is important to note that, depending on the number of input concentration cycles per bin volume, the flow through the conical hopper with a large friction angle may result in better segregation mitigation in continuous mode than a cone-in-cone hopper with a large velocity ratio between the inner and outer cones.





Figure 18. Expected reduction in the concentration variation for flow of assumed input concentration through a conical bin as a function of the number of input cycles per bin volume showing the difference in the wall friction angle

4.0 Conclusions

The radial stress and velocity calculations computed from standard flow property measurements, when combined with measured segregation patterns, can provide a reasonable system approach to determine if a particular bin can successfully mitigate Two modes of segregation mitigation must be segregation during processing. considered. First is the segregation that occrs when a piece of process equipment is filled and then completely emptied. In this mode of operation, the cone-in-cone bin shows a significant ability to mitigate segregation where conical or plane flow hoppers would still posses significant segregation. Second is the case where segregation occurs during a repeated batch operation passing through a surge bin in a continuous manner. In this mode of operation, the best velocity profile is one that shows significant velocity difference in the velocity across the bin. This will occur in conical or plane flow bins with a large friction angle or in cone-in-cone bins with a large difference between the average flow between the inner and outer cones. The best choice bin seems to depend on the number of concentration variations in the surge bin relative to the volume of the surge bin. If the batch size variations are large relative to the surge bin, the conein-cone geometry appears to give better segregation mitigation. However, if the batch size variation is small relative to the surge bin (i.e. many variations in a surge size volume), then the conical bin with a large friction angle may be a better choice to mitigate segregation in a continuous mode of operation.



5.0 References

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