Segregation of Cohesive Particles in Granular Flows

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What is the problem? In processing granular materials such as particles, beads, grains, and powders, small particles fall between larger ones and de-mix, or "segregate," as the particles flow. The physics behind this problem and physics-based models for this situation are nearly solved for mm-sized non-cohesive large and small particles. But cohesive particles are common in many practical particle systems. In some cases, the "stickiness" of cohesive particles can prevent unwanted segregation, but in other cases, large and small particles still segregate, or clumps of particles can form that can themselves segregate. This segregation results in poor product quality (poorly dispersed colorant in plastics processing, non-uniform distributions of binder and active pharmaceutical ingredients in tablets, etc.), waste (segregated processed materials may not meet requirements and are discarded), safety concerns or health risks (resulting from poor quality mixtures, particularly in pharmaceutical cases), and fouled equipment (cohesive particles clog equipment and coat equipment and sensor surfaces). While particle cohesion has been studied in detail, there has been little study of how particle cohesion alters segregation. There is no physics-based model or accurate empirical model for cohesive particle segregation, so predicting and modeling segregation in particle processing systems is often *ad hoc* for cohesive particles.

Segregation and cohesive particles: Predicting the motion of flowing particles is critical in many chemical and pharmaceutical processing operations. Yet this is particularly difficult for cohesive particles. The cohesiveness between the particles can be a consequence of liquid bridges between particles, material surface properties (stickiness), electrostatic forces, or other factors. The cohesive forces are particularly problematic for smaller particles. Examples include the clumping of powders (Fig. 1a), a powder coating a chute (Fig. 1b), or a powder cake adhering to an inverted container (Fig. 1c).[1-3] Flow of cohesive particles differs substantially from free-flowing non-cohesive particles, an example of which is flow in a rotating tumbler, shown in Fig. 1d for free-flowing non-cohesive particles (left) and clumps of cohesive particles (right).[4]

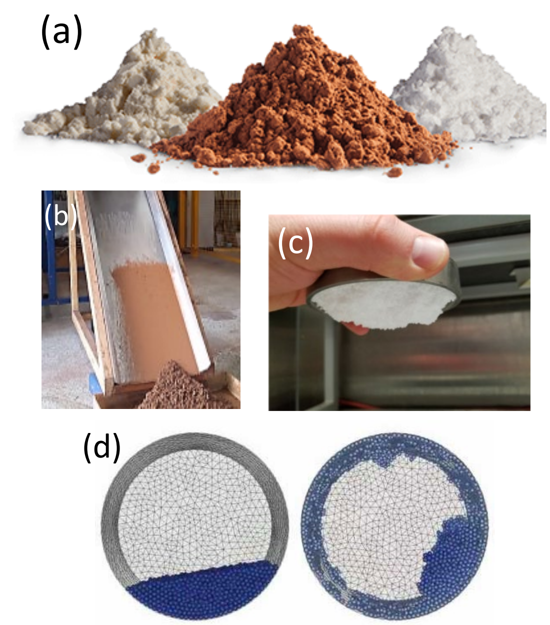


Fig. 1: a) Clumps of cohesive materials in a heap; b) cohesive powder coating a chute; c) cohesive material in inverted container; d) simulation of non-cohesive particles (left) and clumping cohesive particles (right) in a rotating tumbler (particles are blue).

Despite the ubiquity of cohesive particles in industrial processes, research on the segregation of cohesive particles is far behind that of non-cohesive particles, primarily because the cohesion between particles complicates the situation substantially. Particle de-mixing, or segregation, for flowing non-cohesive particles of different sizes is shown schematically in Fig. 2. Small particles fall into the interstices between large particles, a process known as "percolation," due to gravity. The accumulating small particles in the lower portion of the flow push the larger particles upward, quickly leading to a segregated flow of large particles above small particles. The problem is made more complicated by the different physical mechanisms that lead to segregation. For large-to-small particle size ratios, $R = d_l/d_s < 6.5$, small particles are in continuous contact with large particles and all particles must be flowing for any segregation to occur. For larger size ratios, $R > 6.5$, the small particles, which we refer to as "fine" particles to differentiate this case, are so fine that they can pass through the interstices between large particles even when the large particles are not flowing. These two scenarios (small size ratio and large size ratio) can both result in demixing of the two particle species, but with very different physical mechanisms. The understanding of and mechanisms for segregation at small size ratios has been put on a firm physical foundation over the past decade with reasonable capability for modeling and predicting segregation. On the other hand, only recently has there been any study of fine particle segregation in flowing granular materials.[5] This recent research suggests that fine particle segregation is strongly dependent on the kinematics of the granular flow, specifically the shear rate in the flowing material, the large-to-fine particle size ratio ($R = d_l/d_f$), and the particle properties (specifically, the coefficient of restitution). In fact, a high enough shear rate or restitution coefficient causes the fine particles to energetically ricochet off large particles, which significantly slows segregation.

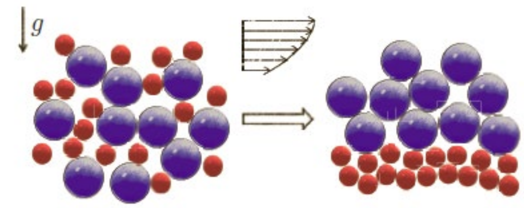


Fig. 2: Small particles percolate through flowing large particles to segregate below the large particles in non-cohesive mixtures.

The combination of segregation and particle cohesion is even more complicated and can lead to some situations that are beneficial and some that are problematic. For instance, cohesive fine particles can stick to the surface of large particles, which uniformly distributes the fine particles among the large particles and prevents segregation of the fine particles. On the other hand, if the fine particles stick to each other and form clumps, they will not be uniformly distributed and the clumps themselves may segregate. A further complication is that cohesive properties of particles tend to be accentuated for fine particles, where the cohesive forces can be large compared to the weight of the particles. Again, this can be beneficial or problematic. Fine particles that are cohesive are likely to stick to large particles regardless of the flow conditions (shear rate) such that they are uniformly distributed. But if the fine particles are too cohesive, they may clump together or cause large particles to clump together.

While cohesive particles are commonplace, most attempts to predict or control cohesive particle segregation in process industries are *ad hoc* and based on experience, not science. Of course, this is a difficult problem, particularly for small particle sizes and non-spherical particle shapes typical of powders and grains central to the production and processing of many pharmaceutical, chemical, and consumer products. Further complicating the situation is that cohesiveness between particles can result from intentional addition of agents (moisture) to keep particles mixed (Fig. 3a[6]), adhesion (Fig. 3b[7]), electrostatic charging (Fig. 3c[8]), particle shapes (interlocking particles), particle surface roughness, and other mechanisms. However, it is likely that many problems related to segregation could be prevented altogether or better managed if there was a better understanding of and models for cohesive particle segregation in the first place.

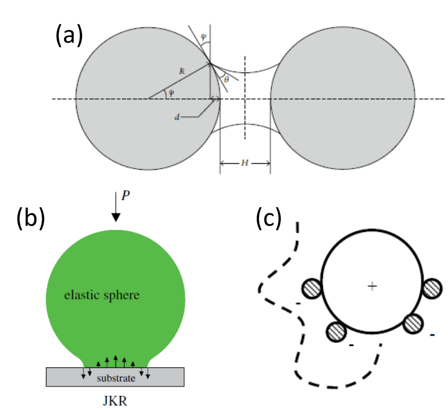


Fig. 3: a) Liquid bridge cohesion between particles; b) Cohesion modeled with the JKR model; c) Electrostatic cohesion for fine particles sticking to a large particle.

A key parameter in the study of cohesive forces on particles is the relative strength of the cohesive force and the gravitational force. For cohesion due to liquid bridges between particles (see Fig. 3a[6]), this can be represented by the Bond number, the ratio of gravitational to capillary forces, which incorporates the particle size and density as well as the density and surface tension of the liquid. However, here we intend to consider a generic cohesive force, the JKR (Johnson, Kendall and Roberts) model[9] (see Fig. 3b[7]), to simplify the problem, rather than separately considering liquid bridges or other types of cohesive forces, each of which have their own complications. The generic form of the Bond number reflects the ratio of the gravitational force to the "pull-off" force, although an alternative parameter called the cohesive number has been proposed based on the ratio of the work of cohesion to the gravitational potential energy.[10,11]

How will we study cohesive particle segregation? We will use computer simulations, validated with experiments, to develop a physical understanding of the flow and segregation of cohesive particles at the flow level. This will lead to a physics-based understanding of the segregation of cohesive particles much like the models and approaches we have developed for the segregation of non-cohesive particles.[12,13]

> ***Research Goal:*** *To develop fundamental knowledge about cohesive particle segregation that will lead to an understanding of the key flow and particle parameters that influence segregation as well as insights into how to predict and control the segregation of cohesive particles.*

I. Background: Segregation in Dense Flowing Granular Materials

The problem of segregation has been studied in great detail for non-cohesive particles — segregation can be readily predicted for a wide range of flow conditions[12,13] and types of particles.[14,15] Even the forces acting on individual particles have been isolated.[16-20] Before considering segregation of cohesive particles, it is instructive to review the case of non-cohesive particles, which is also quite relevant in industrial flows. An understanding of both the particle-level physics and means for modeling the segregation have emerged over the past decade. The key innovation is a methodology to characterize the velocity at which small particles percolate through large particles based on the local shear rate and local relative concentrations of the two species of particles.[13,21] The most successful continuum-framework approach to modeling particle segregation in dense flowing mixtures of particles is the advection-diffusion-segregation transport model for the spatial and temporal evolution of the concentration field for species c_i ,[22-28] which for a bidisperse mixture can be written in the form:[13,24]

$$\frac{\partial c_i}{\partial t} + \boldsymbol{\nabla} \cdot (\boldsymbol{u} c_i) + \frac{\partial (w_{p,i} c_i)}{\partial z} = \boldsymbol{\nabla} \cdot (D \boldsymbol{\nabla} c_i). \qquad (1)$$

Here $\boldsymbol{u}$ is the velocity field, D is the collisional diffusion coefficient, $w_{p,i}$ is the segregation velocity of species i, and the z-coordinate is normal to the shear direction. To solve Eq. 1, $\boldsymbol{u}$, D, $w_{p,i}$, and appropriate boundary conditions must be known. The velocity field can be determined from Discrete Element Method (DEM) simulations, theory, or experiments and only weakly depends on the concentration field. The diffusion coefficient is a function of the local mean particle size, d, and the local shear rate, $\dot{\gamma}$, computed using the relation $D = Cd^2\dot{\gamma}$, where C is of order 0.1 and depends on particle properties.[29-31]

Although the advection-diffusion-segregation continuum transport approach (Eq. 1) was conceived decades ago,[22] the key ingredient that makes this approach useful for practical application is the third term on the LHS of Eq. 1, expressing the segregation flux as $w_{p,i} c_i$, where $w_{p,i}$ is the "percolation velocity" (hence, subscript p), also called the "segregation velocity." The percolation velocity is simply the average velocity at which species i moves upward (large particles) or downward (small particles) relative to the bulk flow. Although several forms have been suggested,[25,32,33] a successful segregation flux model based on "kinetic sieving" of non-cohesive small particles through a bed of flowing large particles,[21] is[13,24,34]

$$w_{p,i} = S\dot{\gamma}(1 - c_i), \qquad (2)$$

where S is a segregation parameter (dimensions of length; positive for rising large particles, negative for sinking small particles), and (1-c_i) is the concentration of the other species. Intuitively, for a particle to segregate, it must differ in size (characterized by S, which depends on particle size ratio R) and be surrounded by particles of the other particle species (1-c_i) that are flowing under shear ($\dot{\gamma}$) to open voids for segregation to occur. The segregation parameter S for specific pairs of non-cohesive particle species can be found from DEM simulations[34] or experiments[35,36] for both spherical and non-spherical[15,37] particles. For free surface flows, S is independent of the details of the flow, whether it be chute flow, heap flow, rotating tumbler flow, or hopper surface flow.[13]

II. Proposed Research

We propose three thrusts for our study of the segregation of cohesive particles:

- DEM simulation of cohesive particle segregation for different size ratios and degrees of cohesion.
- Experiments with cohesive particles primarily focused on validating the DEM simulation results.
- Development of the theory underlying the behavior of cohesive particles as they segregate.

It is clear that the scope of the above approach is quite broad, so we intend this to be a preliminary study that provides a basic understanding of the problem. We will apply resources available to us from Northwestern University to leverage those provided by IFPRI. Within 2 years, we will apply for additional support from the National Science Foundation via its GOALI (Grant Opportunities for Academic Liaison with Industry) program, based on preliminary results obtained through this study. Ideally, IFPRI and its member companies will be GOALI partners for this larger-scale proposal for ongoing support of this research.

There are many variations on the problem of segregation of cohesive particles including the origin of the cohesive forces, sizes of particles, the granular flow geometry, and which particles are cohesive (both large and small particles or just one or the other). In addition, segregation can occur for even monodisperse cohesive particles -- agglomeration of cohesive particles forms clumps between which individual particles segregate while at the same time shear due to the flow can result in the breakup of large clumps. All of these are important and worthy of study, but based on discussions with colleagues from IFPRI, we will focus on the segregation of two sizes of particles both of which are cohesive, avoiding the clumping segregation problem as well as situations where only one species is cohesive. We will consider a canonical granular flow geometry, that of a bounded heap, because of our experience with this flow, its geometric simplicity, our ability to perform matching DEM simulations and experiments, and the ability to obtain maximal segregation date with the fewest simulations. We may also consider pure shear flow between differentially moving planes, a useful simplified flow geometry. We can only consider a limited number of size ratios, but we will begin with small particles ($R < 3$), and then extend the study to the different segregation mechanism of fine particles in the free-sifting regime ($R > 6.5$). Finally, we will consider a generic model for cohesion between particles, the JKR (Johnson, Kendall and Roberts) model,[9] rather than specific cohesion models for electrostatic charging, van der Waals forces, liquid bridges (moisture), interlocking particle shapes or surface roughness, or other particle cohesion mechanisms.

<u>Thrust 1 — DEM simulations</u>: Discrete Element Method (DEM) simulations will be the focus of the proposed work. In DEM simulations, motions of and forces between individual particles are computed simultaneously for tens to hundreds of thousands of particles. The flow of two particle species down a bounded heap, shown in Fig. 4, will be considered because this flow geometry is easily simulated, has a thin flowing layer where segregation occurs, and has simple kinematics (velocity field is easily characterized). Perhaps more importantly, we have found that the bounded heap flow naturally provides a range of local shear rates and particle species concentrations all within a single simulation. To explain, as particles flow down the heap, particles at the bottom of the thin flowing surface layer deposit on the heap, which changes the local concentration with position along the heap and in the flowing layer. This provides the variability in concentration that is crucial in understanding segregation, as is evident from the (1-c_i) term in Eq. 2 for non-cohesive particles. At the same time, because particles are being deposited on the heap, the velocity in the flowing layer decreases with distance down the slope of the heap and depth below the free surface. This provides a range of shear rates ($\dot{\gamma}$), which is also crucial to segregation as evident in Eq. 2. As we found with non-cohesive particle segregation with varying particle size and density,[14,38] performing DEM simulations over a range of conditions in this simple geometry provides a vast amount of data that is relatively easily broken down into the key aspects important to understanding the underlying physical mechanisms of segregation as well as the dependence of the segregation on flow and particle conditions.

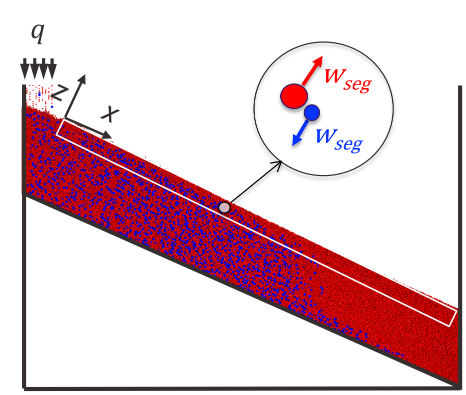


Fig. 4: Bounded heap flow.

To probe the impact of cohesion on segregation in the two different segregation regimes, that of small particles ($R < 3$) and fine particle ($R > 6.5$), we will consider two size ratios, $R = 2$ and $R = 7$, chosen based on computational efficiency and our previous experience with these size ratios. For $R = 2$, we will use a 50-50 concentration of the two particle sizes. For $R = 7$, it is likely that it will be necessary to use a much larger volume concentration for the large particles than the fine particles, for computational efficiency and to better understand particle-particle interactions. Cohesion will be varied by adjusting the effective surface energy per unit area parameter in the JKR model, which will be the same for both particle species in the simulations. The cohesion will be varied to reflect several different conditions (mildly cohesive to strongly cohesive) based on the Bond number or cohesion number.

For the research proposed here, we will likely use the open source DEM codes LAMMPS, which was originally developed for molecular dynamics simulations and later adapted for particle dynamics simulations. We already have experience with LAMMPS running on Northwestern University's high performance computing system, QUEST. LAMMPS has the capability to handle large particle size ratios (typically limited to a size ratio of about 5 or 6 for most DEM codes, but up to 100 for LAMMPS). Even more importantly here, LAMMPS has the built-in capability to simulate cohesion via the JKR model in addition to the classic cohesionless Hertzian contact model.[9] The JKR model is based on the balance between the stored elastic energy and the loss in surface energy based on the contact pressure and contact area that results in a tensile "pull-off" force when two contacting particles separate (see Fig. 3b[7]). While other models exist for cohesion related to liquid bridges between particles[6] and even using van der Waals forces between very fine particles[39], the JKR model represents a generic cohesive force that has been used successfully in DEM simulations of cohesive particles and even calibrated to experimental results.[40] Similarly, we will compare our DEM results to experiments with cohesive particles as described below to validate and calibrate the simulations.

Based on these simulations, we expect to connect the percolation velocity model of Eq. 2 to the cohesion, size ratio, and other parameters of the problem. Because we cannot explore a broad parameter space under the constraints of this research, we expect that these results will provide insight and guidance as we prepare an NSF GOALI proposal on the topic of segregation of cohesive particles.

Specific tasks for this thrust are as follows:

- Implement and debug the JKR model in LAMMPS for the bounded heap flow.
- Simulate bounded heap flow with size-bidisperse cohesive particles measuring the local percolation velocity, shear rate, contact duration, and particle concentration for two size ratios and three degrees of cohesion (small, similar, and large cohesion force compared to the particle weight).
- Analyze DEM results in terms of the local percolation velocity, concentration, and shear rate to connect those results to Eq. 2.

Thrust 2 — Validation Experiments: A limited number of bounded heap experiments for cohesive size-bidisperse particles will be performed to validate the DEM simulations. For ease of handling and measurement, we will use mm-size particles. However, mm-size particles that are typically used for granular flow research (glass, ceramic, or cellulose) are typically non-cohesive. Furthermore, the precise control of cohesive properties of particles can be difficult. Nevertheless, researchers have successfully generated cohesive 90–150 μm glass beads by using a silanization process in which organosilyl groups are attached to the surface of glass beads using Sigmacote®, which is frequently used to coat glassware or plasticware for increased hydrophobicity in biomedical applications.[41] An even more advanced approach has been developed in which the coating material is a polyborosiloxane (PBS) for which the coating thickness, which can be controlled, determines the cohesiveness of the particles.[42] An example of a pile of cohesive glass beads generated using this technique is shown in Fig. 5.[42]

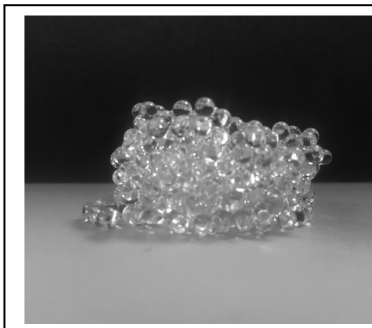


Fig. 5: Pile of 3 mm cohesive glass beads with a 2.2 μm PBS coating.

The validation experiments will be similar in nature to the methodology that we developed to measure the segregation parameter, S in Eq. 2, for practical particle systems.[35,36] In this approach, a bidisperse mixture of particles flow down the slope of a heap and deposit on the heap. The heap is physically partitioned into bins along its length, and the species concentration in each bin is measured. The difference between measured species concentration profiles and that predicted from Eq. 1 is minimized by varying *S*, thereby providing an experimental estimate of *S* for the mixture, which can be compared to DEM simulation results. It may also be necessary to measure the velocity in the flowing layer using particle tracking velocimetry, a technique that we played a role in developing.[43,44] Furthermore, in the event that the JKR contact model in the DEM simulations cannot adequately model real cohesive particle systems, a contingency approach to the proposed research would be to focus more heavily on this experimental approach to studying cohesive particle segregation.

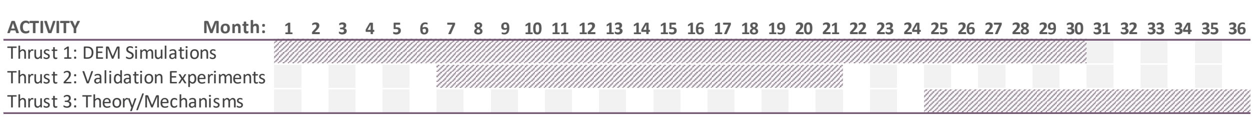
Specific tasks for this thrust are as follows:
- Implement a particle coating process to generate particles with controlled cohesiveness.[42]
- Perform bounded heap experiments for size-bidisperse cohesive particles with size ratio $R = 2$.
- Compare experimental results to DEM results in terms of segregation (*S*)[35,36] and velocity fields.[43,44]

<u>Thrust 3 — Underlying Theory and Mechanisms</u>: This thrust is primarily exploratory in that it is quite unlikely that a full theory or complete understanding of segregation mechanisms will result. Instead, we hope to analyze the DEM simulation and experimental results in a way that guides future work. In particular, we will identify how the key aspects of cohesiveness interact with particle size segregation in terms of the physical parameters of the problem. For instance, is the Bond number the appropriate dimensionless parameter to consider (e.g., should shear stress as well as particle weight be considered)? How does the cohesiveness interact with the particle size ratio in affecting segregation? Can the segregation parameter, *S* in Eq. 2, be modified to account for the cohesiveness of the particles as well as the particle size ratio? What are the differences in the impact of cohesion on segregation for small size ratios (continuous ongoing particle contacts) and large size ratios (tending toward free-sifting if there were no cohesion)?

Specific tasks for this thrust are as follows:
- Compare DEM and experimental results for cohesive particles to results for non-cohesive particles.
- Based on the DEM simulation results, connect the degree of segregation to the percolation velocity and determine if the model of Eq. 2 can be adapted to cohesive particles for Bond numbers below a certain value, perhaps by including a dependence of *S* on cohesion in addition to the particle size ratio.
- Extract the key physical mechanisms at play in segregation (or mixing) for cohesive particle flows.

III. <u>Research Plan and Resources</u>

The plan outlined above is quite ambitious and would not be feasible except for the possibility of additional internal funding to support the effort. We expect that Thrusts 1 and 3 will be performed by a post-doctoral researcher with about half of the researcher’s salary paid from this gift. Thrust 2 will likely be performed by undergraduate or MS students as a research project for academic credit. The work will be done under the direction of Principal Investigator Richard M. Lueptow, Professor of Mechanical Engineering and former Charles Deering McCormick Professor of Teaching Excellence. He is an expert on granular flows, flow modeling, and experimental methods. Other co-investigators are Julio M. Ottino, Robert R. McCormick Institute Professor in Chemical and Biological Engineering and Walter P. Murphy Professor, who is one of the world’s leading experts on mixing and chaos as well as granular flows and Paul B. Umbanhowar, Research Professor, who is an expert in the physics of granular flow and related computational methods. A high-level plan for the project is indicated below.



We have already engaged IFPRI representatives in developing the focus for the initial research and providing advice on experimental methods with cohesive particles. We will continue to involve IFPRI representatives as we progress with the research program, providing updates and access to our research group Zoom meetings reporting on progress (as we currently do with Dow and P&G for our NSF GOALI projects). In our experience, these interactions are crucial to understanding the problem from an industry perspective, overcoming hurdles in the research, and obtaining feedback from experienced industry researchers on how to assure that our research is optimally relevant to the most pertinent problems in industrial processing of cohesive particles.

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