EXPERIMENTAL INVESTIGATION OF PARTICLE SEGREGATION IN HOPPER DISCHARGE

By

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To my family and friends

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LIST OF ABBREVIATIONS

d particle diameter [mm]

σ standard deviation [mm]

ρ density [g/cm^3]

x_i volume fraction of fines in a given sample [−]

x_f volume fraction of fines initially in hopper [−]

$x_{f,L}$ fines limiting fraction [−]

x_i/x_f normalized fines volume fraction [−]

Φ_D particle diameter ratio [−]

Abstract of Thesis Presented to the Graduate School
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Many industrial processes including food, mining, and pharmaceuticals involve handling mixtures of granular materials. The tendency of granular materials to segregate due to differences in particle properties such as, size, shape, and density negatively affects the process efficiency and product quality.

Our study focused on segregation of particles in hopper flow with the following conditions: well-mixed initial fill, funnel flow, and cylindrical hopper geometry. Glass beads and steel shots in three particle sizes each are used to investigate the effect of size ratio and density difference on particle segregation. Also, to study the effect of particle shape on segregation of particles, crushed glass in two sizes is used.

Results show that size-segregation patterns are initially fines-rich and then fines-depleted at the end of discharge. Segregation increases for larger particle size ratios and decreases for fines fractions that are above 10%. The extent of segregation for mixtures of crushed glass-crushed glass is not significantly different from spherical glass mixtures. Given the same size ratio, mixtures consisting of smaller particles show less segregation. The segregation trend due to density effects is initially rich in particles with the higher density and then rich in the particles with the lower density at the end of the discharge.

CHAPTER 1
INTRODUCTION

A fascinating characteristic of granular materials is their tendency to segregate due to difference in particle properties such as size, density, and shape. When a container of different liquids is shaken, they will mix, but in a blend of particles, they will segregate. A famous example is the “Brazilian Nut Effect” [1, 2] where a bed of large particles rise above smaller particles upon vibration. Segregation is of importance in a variety of industries that involve the handling, processing, or manufacturing of particulate materials. Some examples are mining [3], food [4, 5], and pharmaceuticals such as in the production of pills and tablets [6]. Segregation can adversely affect the efficiency of processes, as well as the quality of final products which is dependent on maintaining a homogeneous blend.

Segregation has been studied over many years by researchers from various disciplines due to its importance and ubiquitous occurrence. Despite the efforts to explain this phenomenon through various methodologies such as experimentation, simulation, and modeling, there are still many unanswered questions.

The segregation of particles in hopper discharge is the focus of this thesis. Specifically, a series of controlled segregation experiments are conducted, changing one variable at a time. These experiments provide insight into the fundamental mechanisms influencing segregation of particles in gravity driven flow. These experiments also serve as a basis for validation of analytical models and numerical simulations of particle segregation.

A hopper geometry is chosen for the segregation experiments since it is a standard geometry used in particle processes with many practical industrial applications. In addition, segregation experiments are relatively simple and straight forward in a hopper geometry. Furthermore, when segregation occurs in a large-scale particle processes which include many devices such as mixers, fluidized beds, and hoppers, it is important to characterize the amount of segregation that occurs in the hopper alone versus the other units. For example, if a perfectly mixed blend is fed to a hopper, how much segregation is to be expected from particle flow in that device?

Following this introduction, chapter 2 will review literature that is relevant to the current work. Then in chapter 3 experimental materials and procedures will be explained. The results from the experiments are then presented and discussed in chapter 4. Chapter 5 summarizes the thesis work and makes recommendations for further studies.

CHAPTER 2
LITERATURE REVIEW

This chapter provides an overview of previous experimental research that is relevant to the present study. Experiments related to segregation in hopper discharge are the main focus of this review. Section 2.1 illustrates several classification methods of particle segregation. Section 2.2 reviews literature concerned with funnel flow and mass flow followed by section 2.3 which looks into previous work related to fines fraction. Then, in section 2.4 literature about particle properties that influence segregation is discussed. Finally, section 2.5 reviews previous work that has investigated other factors that affect segregation.

2.1 Classification of Segregation

Tang and Puri [7] categorized particle segregation depending on several variables. Segregation may be classified by physical properties of particles, such as size, density, or shape. Another way of categorizing segregation is by energy input; vibration [8], gravity [9, 10], or shear [11] segregation. Segregation can also be classified depending on particle movement direction; vertical or horizontal. Finally, the device in which particle segregation occurs, such as hoppers [9, 12-16], drums, and chutes, can be another classification method.

The most common method of classification of particle segregation is by the fundamental mechanism which gives rise to segregation. As many as thirteen mechanisms by which particles segregate have been identified and reviewed by researchers [17-21]. The thirteen mechanisms summarized by de Silva *et al.* [19] are:

- **Trajectory**: caused by a greater speed reduction for smaller particles due to air drag
- **Air current**: fine particles are deposited at silo walls by air currents created by falling particles
- **Rolling**: large or rounded particles roll down the surface of a particle heap in formation.

- **Sieving**: smaller particles flow downward through a sliding or rolling layer of larger particles.

- **Impact**: a segregation mechanism where more bouncy particles are found further away from the center of a heap in formation

- **Embedding**: larger or denser particles penetrate the surface layer of a heap and become locked in position there

- **Angle of repose**: components with lower angle of repose flow more easily toward the edges of a heap

- **Push-away**: lighter particles are pushed towards the edge of a heap by equally sized heavier particles falling on the apex of the heap.

- **Displacement**: larger particles rise above smaller particles as a result of vibrations

- **Percolation**: smaller particles fall through void spaces between larger particles, sometimes as a result of localized shear

- **Fluidization**: fine or lighter particles are kept fluidized at the surface of the particle mixture

- **Agglomeration**: very fine particles form larger aggregates with greater mobility

- **Concentration driven displacement**: occurs in rotating devices where fine particles concentrate in zones due to higher mobility

In addition to the mechanisms listed above, electrostatic interactions of particles may also influence segregation. Of the thirteen mechanisms, percolation and sieving are hypothesized to be the primary means by which segregation occurs. According to Samadini *et al.* [13], segregation primarily occurs near the ‘V’-shaped granular free surface where shearing of particles occur. The other eleven mechanisms should be negligible since the particles used in the present work are relatively large, free-flowing, and spherical with uniform characteristics. Also, there is no projectile motion of particles during discharge from the hopper or inside the hopper.

2.2 Funnel Flow and Mass Flow

According to Tang and Puri [7], the terms funnel flow and mass flow were first developed by Jenike in 1954. Funnel flow has a first-in, last-out flow pattern due to stagnant materials at the hopper walls. Mass flow has a first-in, first-out flow pattern since materials at the hopper wall are in motion. Sleppy and Puri [4] performed experiments with binary equal weight fraction mixtures of granular sugar of size ratios, $\Phi_D = 2$ and $\Phi_D = 5.7$ for both funnel flow and mass flow conditions. Their results showed that a mixture segregates more in funnel flow than in mass flow. Markley and Puri [22] conducted similar experiments with identical particle size ratios and observed that less segregation occurs for scaled up hoppers which have the same orifice diameter. The generally accepted segregation pattern for funnel flow is that during the first half of the discharge, fines are predominant and the second half of the discharge is fines-depleted [3, 17].

2.3 Fines Fraction

The concentration of fine particles is another factor that affects segregation. Typically, more segregation occurs when the fines fraction is lower [7]. This is because there are more void spaces that the number of fine particles that can move through. Arteaga and Tuzun [15] developed a term called fines limiting fraction, $x_{f,L}$.

$$x_{f,L} = \frac{4}{4 + \Phi_D}$$

This model is applicable for relatively large, free-flowing, nearly spherical particles. Above the fines limiting fraction, very little or no segregation occurs. Research results from Sleppy and Puri [4], and Kettergagen *et al.* [12] are in qualitatively agreement with this model.

2.4 Particle Properties that Influence Segregation

Properties of particles contributing to segregation include particle size, shape, density, elasticity, cohesitivity, surface roughness, friction, and size ratio. However, some particle properties show a more significant impact on segregation than others do. Particle diameter ratio, density, and particle shape effects will be discussed in the following sections.

2.4.1 Particle Diameter Ratio

Most research on hopper flow segregation has focused on the effect of particle size ratio since this is considered to be the most dominant factor. Standish [3] examined size segregation in a Paul-Wurth hopper, a hopper with inclined, rotating pipe at the orifice which is typically used to feed blast furnaces. Samadini *et al.* [13] studied segregation in the discharge of bidisperse glass beads from a transparent, quasi-two-dimensional silo through an orifice. Results from both of these studies showed particle segregation occurring for a particle size ratio as low as 1.2. However, for lower size ratios, the extent of segregation was reduced. The results of Ketterhagen *et al.* [12] with glass bead mixtures of $\Phi_D = 2$ and $\Phi_D = 4.3$ show the same pattern. In contrast, Sleppy and Puri [4] concluded that there was negligible disparity in the extent of segregation between the $\Phi_D = 2$ and $\Phi_D = 5.7$ mixture of granular sugar. This may be due to the cubic shape of the granular sugar used in their experiments.

2.4.2 Particle Density

There are no published works investigating density effect alone on segregation in hopper discharge. Shi *et al.* [8] observed segregation trends with experiments in a vibrating glass cylinder containing equally sized particles of different densities. Their results showed that the less dense particles gradually formed a layer on top of the denser particles. The thickness of the layer increases as the density ratio increased. Shinohara *et al.* [22] investigated segregation patterns during the filling of a hopper. Denser components settle towards the center of the hopper and are surrounded by lighter particles. Although the most dominant factor affecting segregation is particle size ratio, the effect of density difference is still not negligible [23].

2.4.3 Particle Shape

There are no published works investigating shape effect alone on segregation in hopper discharge. According to Tang and Puri [7], mixtures with different particle shapes are easier to segregate than mixtures consisted of particles of the same shape. The greatest segregation occurs when the coarse particles are angular and the fine particles are spherical. This could be due to the increased void space formed from the irregularly shaped coarse particles or because irregularly shaped particles may fit into interstitial void spaces more easily. However, they indicated that the extent of segregation caused by particle shape is less than the effect of particle size ratio.

2.4.4 Particle Friction

The influence of surface roughness on particle segregation behavior has received little attention in granular flow studies. In general, it has been assumed that surface roughness is an order of magnitude less important in influencing segregation than particle size or density [25]. Pohlman *et al.* [25] studied segregation patterns of a binary mixture of smooth and rough surfaced chrome steel beads in 2D and 3D rotating tumblers. Their experimental and simulation results showed that surface roughness affected the angle of repose. However, surface roughness did not cause radial segregation in 2D tumblers or banding in 3D tumblers.

2.5 Other Factors

Ketterhagen *et al.* [12] experimentally investigated the effect of initial fill conditions on segregation in hopper discharge. The fill methods that were studied were well-mixed fill and dual hopper fill. The dual hopper fill was achieved by discharging a well-mixed blend from a mass flow hopper into a funnel flow hopper directly below. The results from the dual hopper fill showed an extreme excess of fines towards the end of discharge – such segregation behavior is not observed in most well-mixed fill results.

Shah *et al.* [6] developed a testing method to predict the particle segregation potential using a hopper manufactured by Jenike & Johanson, Inc. Their method was consisted of four passes through the hopper and a pass through a 6 foot polycarbonate tube, approximately 1.5 inches in diameter.

Alexander *et al.* [26] filled a hopper with a mixture of glass beads or pharmaceutical excipients. The hopper was then discharged into an identical hopper below, which is then emptied into the first. Repeating this process several times resulted in an asymptotic segregation profile significantly different from the segregation profile of the first discharge.

CHAPTER 3
EXPERIMENTAL METHODS

This chapter discusses the details of the experimental equipment and procedure used in the research. In Section 3.1, the selection criteria and properties of the granular media will be explained. The sifting segregation tester is then described in Section 3.2 followed by the details of the antistatic bar in Section 3.3. Section 3.4 covers the procedure for conducting the experiments. Finally, the methods used to collect and analyze the data are presented in Section 3.5.

3.1 Granular Media

Three types of granular media were used in the experiments. Glass beads and through hardened steel shots were selected to investigate the segregation trends due to particle size ratio and density difference. Crushed glass was used to investigate the effect of particle shape on segregation patterns. The specific particle sizes were chosen so that some of the experimental results can be directly compared with results from Ketterhagen *et al.* [12]. The sizes of the particles comply with guidelines in the operating manual for the hopper as provided by the manufacturer, Jenike & Johanson Inc. These guidelines indicate that the maximum particle size should be limited to 1/8 inch and the total volume of materials to be tested should be at least 600mL.

The purchased particle mix was sieved to narrow the particle size distribution (PSD). Then the particles were placed in an oven for 24 hours and then stored in a desiccator, prior to experimentation, in order to keep them dry. This was done to prevent moisture on the surface of the particles from influencing the experimental results.

Particle size information was measured by image analysis via microscope or by Coulter LS 13320 particle size analyzer.

Table 3-1. Summary of mean diameter and standard deviation of granular materials

Material type	Mean diameter d [mm]	Standard deviation σ [mm]	RSD [%]
Glass beads	2.26	0.13	5.75
	1.06	0.06	5.66
	0.542	0.045	8.30
Steel shots	2.35	0.01	0.43
	1.13	0.045	3.98
	0.538	0.044	8.18
Crushed glass	0.549	0.29	51.4
	1.0	-	-

3.1.1 Glass Bead

Three sizes of glass beads listed in Table 3-1 (Union Process Inc., Akron, OH) with density of $\rho = 2.5$ g/cm^3 have been purchased. Photographs of the glass beads are shown in Figure 3-1. The glass beads have a relatively uniform diameter and spherical shape.

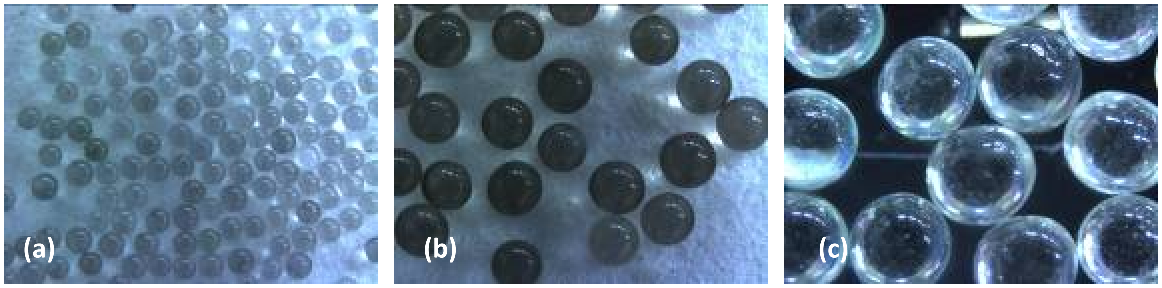

Figure 3-1. Glass beads with mean diameter <*d*> = (a) 0.542 ± 0.045mm, (b) 1.06 ± 0.06mm, and (c) 2.26 ± 0.13mm at 20 power magnification.

3.1.2 Steel Shot

Figure 3-2 shows photographs of through-hardened steel shots (Union Process Inc., Akron, OH, $\rho = 7.8$ g/cm^3) with similar sizes to those of glass beads. The steel shot PSD data are also listed in Table 3-1. The steel shots also have a relatively uniform diameter and spherical shape. Some rust is present on the surface of the steel shots.

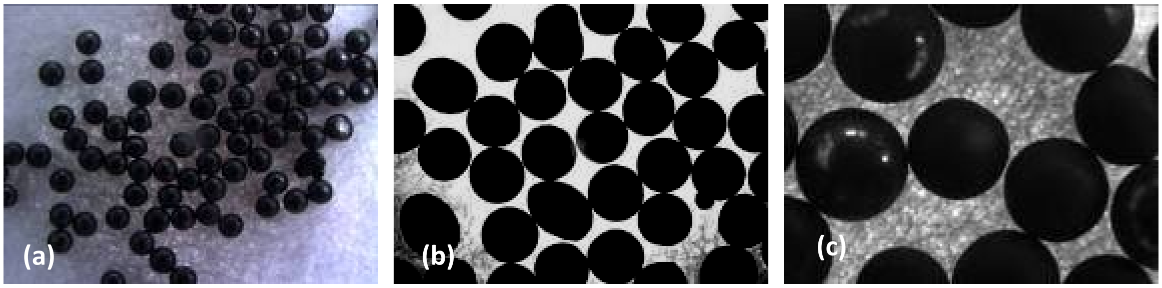

Figure 3-2. Steel shots with mean diameter <*d*> = (a) 0.538 ± 0.044mm, (b) 1.13 ± 0.045mm, and (c) 2.35 ± 0.01mm at 20 power magnification.

3.1.3 Crushed Media

Crushed glass (Strategic Materials Inc., Houston, TX, ρ = 2.5 g/cm3) with sizes matching those of <*d*> = 1.06mm and <*d*> = 0.524mm have been purchased. As shown in Figure 3-3, the shapes of the particles are very angular and deviate from spherical shape.

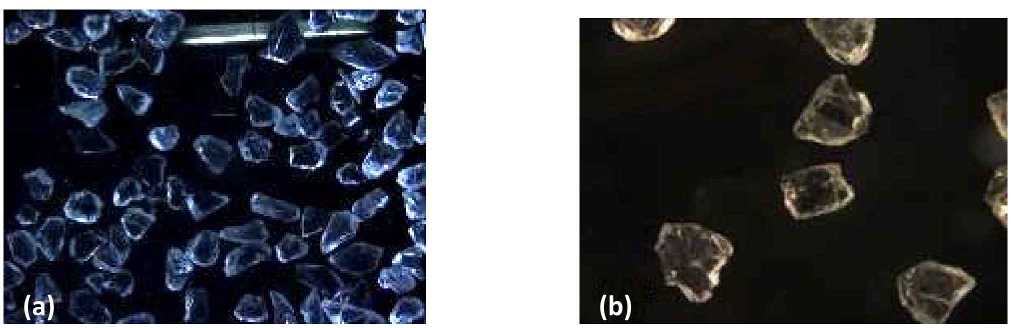

Figure 3-3. Crushed glass with mean diameter <*d*> = (a) 0.549 ± 0.29 mm, (b) 1.13 ± 0.045 mm at 20 power magnification.

3.2 Sifting Segregation Tester

A hopper (Jenike & Johanson Inc., Tygnsboro MA) with dimensions specified by the ASTM standard test for sifting segregation [27] was purchased for the experiments. The hopper is made of clear acrylic and has a half angle of 55° with respect to vertical. Detailed dimensions are shown in Figure 3-4. The slide gate in the outlet of the hopper can be opened and closed to collect samples discontinuously.

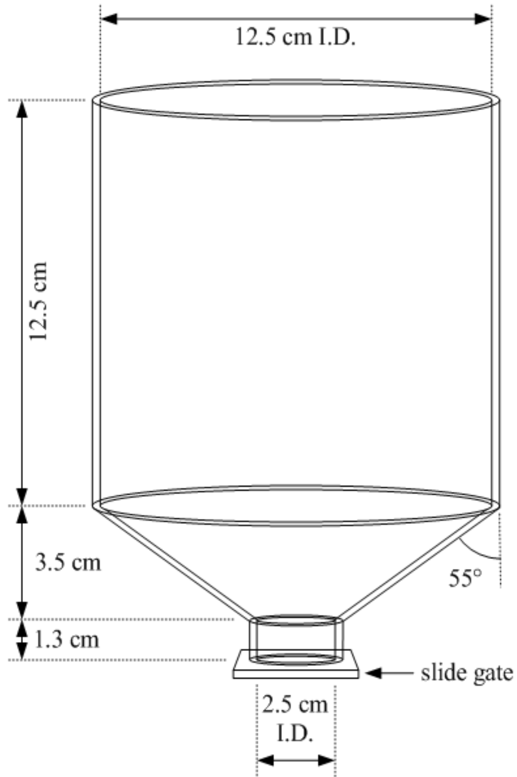

Figure 3-4. Schematic and dimensions of Sifting Segregation Tester [12]

3.3 Antistatic Bar

Glass beads, crushed glass and steel shots of smaller sizes become statically charged during discharge. This causes some particles to adhere to the hopper walls after the bulk of the material has discharged. Also, clustering of the particles occurs. To reduce this problem, an antistatic bar (TAKK Industries Inc., Cincinnati, OH) was used. A picture of the antistatic bar is shown in Figure 3-5. The antistatic bar is 3.5 inches in length and is mounted on the hopper opening to emit a field of positive and negative ions that neutralize the static electricity inside the hopper. The antistatic bar was easier to handle compared to the powder antistatic agent (Larostat HTS 905S, BASF Corp.) which was also used in some experiments. There was less work involved in filling the hopper and less potential for health hazards with the antistatic bar. In addition, Larostat showed some adhesive behavior when it was applied to the particles. The adhesive characteristic increased with the amount of Larostat applied.

Figure 3-5. Antistatic bar with its power supply (left) and antistatic bar mounted on the hopper (right)

3.4 Details of Procedure

This section describes the experimental procedure. The filling process is explained in Section 3.4.1 followed by the hopper discharge and sample collecting procedure in section 3.4.2. For each experiment the hopper was cleaned and leveled. Experiments were repeated 3 to 6 times to minimize the effects of any regions in the initial fill that were not well mixed.

3.4.1 Filling

The well-mixed initial state was used for all the experiments reported in this paper. This filling method is harder to achieve compared to layered or dual hopper filling methods, especially when there is a large diameter ratio between the fine and coarse particles. The following procedure has been used to minimize segregation during filling and to obtain repeatable results.

First, the appropriate masses of coarse and fine particles are prepared. Particles are added in the hopper in 17~20 portions of approximately equivalent compositions. One portion contains

50mL of coarse particles and an appropriate amount of fine particles. The appropriate amount of fine particles for each portion is the total mass of fines divided by the number of portions. A portion of coarse and fine particles are then poured in a small plastic bag and are mixed by hand to achieve a homogeneous state. The bag is then carefully emptied into the hopper. This process is repeated for each of the remaining portions. It is important to minimize the amount of free fall of particles and heap formation to reduce the amount of particle segregation prior to the initiation of discharge. Disturbing the particles that are already filled in the hopper will also cause fine particles to percolate towards the bottom of the hopper.

3.4.2 Discharging and Sampling

Continuous and discontinuous discharge and sampling methods were used in the experiments. The continuous method is affected by opening the slide gate and collecting samples in a train of boxes without interrupting the discharge. This method is more relevant to industrial practices, but there is more particle loss compared to the discontinuous method. The discontinuous method is performed by opening and closing the slide gate each time a 55mL sample is collected. Approximately 18 samples are collected for each experiment. The discontinuous method is specified by the ASTM standard practice and is easier to carry out without loss of materials. Approximately 2~8% of the fine particles were lost for the continuous method and less than 1.5% of the fines were lost for the discontinuous method. Standish and Kilic [14] compared segregation results of continuous and discontinuous methods in a Paul-Wurth hopper using sinter mixtures of eight different size ratios within the range of 0.25mm to 6.0mm. The two methods were found to produce identical results. Ketterhagen *et al.* [12] also concluded that there was little difference in the segregation results between continuous and discontinuous methods. Most of the experiments done in this thesis work are using the discontinuous method due to the ease of its use.

3.5 Measurements and Data Analysis

For each sample that was collected, components were separated using a sieve. The weight of each component was measured using a balance. The weight data were then converted to volume fraction data, x_i using the particle density. Finally, the initial fines fraction, x_f was used to calculate the normalized fines volume fraction, x_i/x_f. The composition of the particle mix was expressed in terms of volume because some experiments involve mixtures of materials with different densities. Plots of normalized fines volume fraction as a function of cumulative volume discharged were constructed. A normalized fines volume fraction $x_i/x_f > 1$ implies that a sample is fines-rich and $x_i/x_f < 1$ denotes that a sample is fines-depleted. A normalized fines volume fraction $x_i/x_f = 1$ means that there is no segregation and the sample is of the same composition as the initial charge. The scatter bars represent the 95% confidence interval.

CHAPTER 4
RESULTS AND DISCUSSION

Table 4-1. Summary of experimental work

Size ratio	Particle 1	Particle 2	Particle 2 volume fraction (%)	Discharge method
4:1	Glass beads (2 mm)	Glass beads (0.5 mm)	5	Discontinuous
	Glass beads (2 mm)	Steel shots (0.5 mm)	5	Discontinuous, Continuous
	Steel shots (2 mm)	Glass beads (0.5 mm)	5	Discontinuous
	Steel shots (2 mm)	Steel shots (0.5 mm)	2.5, 5, 10, 20, 50	Discontinuous
2:1	Glass beads (2 mm)	Glass beads (1 mm)	5	Discontinuous, Continuous
	Glass beads (2 mm)	Steel shots (1 mm)		Discontinuous
	Steel shots (2 mm)	Steel shots (1 mm)		Discontinuous
	Glass beads (1 mm)	Glass beads (0.5 mm)		Discontinuous
	Crushed glass (1 mm)	Crushed glass (0.5 mm)		Discontinuous
1:1	Glass beads (2 mm)	Steel shots (2 mm)	50	Discontinuous, Continuous

This chapter presents experimental results of the thesis work. Section 4.1 describes the effect of various particle treatments done on a glass-glass mixture with $\Phi_D = 2$ and $x_f = 5\%$. Next, section 4.2 validates the use of the discontinuous discharge method. The effect of particle density on segregation is then covered in section 4.3 followed by the effect of fines fraction in section 4.4. Then, section 4.5 discusses how particle size ratio affects the segregation pattern in hopper discharge. Illustrated in section 4.6 is the effect of absolute particle size on segregation.

Section 4.7 then covers the experimental results from aspherical particle mixtures. Finally, a direct comparison with experimental data from previous literature is made in section 4.8.

4.1 Effect of Various Particle Treatments

Table 4.2. Treatment methods applied to glass beads with $\Phi_D = 2$ (2 and 1mm), $x_f = 5\%$

Treatment #	Antistatic bar	Larostat	Washed before experiments	Discontinuous discharge method	Continuous discharge method
1	√			√	
2				√	
3			√		
4		√		√	
5	√				√

The effect of various particle treatments have been investigated for glass-glass mixture with $\Phi_D = 2$, $x_f = 5\%$. Treatment methods used on the particle mixture are listed in Table 4-2. Figure 4-1 illustrates the experimental results for each treatment method. The segregation trend is very consistent for all treatment methods: fines-rich at beginning and fines-depleted at the end of discharge. This trend occurs because of the following reasons. As the hopper is discharged, a 'V'-shaped surface with an incline is formed. Flow down this incline causes fine particles to percolate downward into the stagnant of slow moving material below while larger particles roll down the incline to the hopper centerline. There is an excess of fine particles until the fines-depleted material accumulated at the hopper center line is discharged. Near the end of discharge the concentration of fines increases slightly as the material close to the hopper walls is discharged.

The consistency of the segregation pattern indicates that the various treatment methods for the particles do not significantly affect the results. Treatment 1 is the standard method for all the experiments in this thesis work unless specified otherwise.

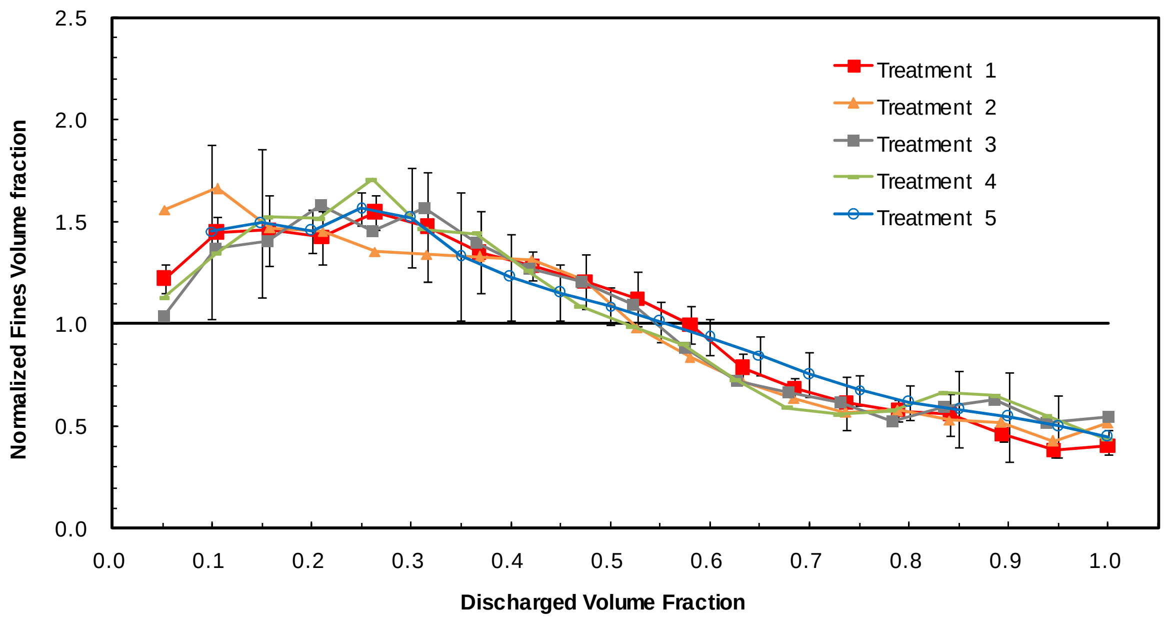

Figure 4-1. Experimental segregation results for variously treated glass-glass mixtures with $\Phi_D = 2$ (2 and 1mm) and $x_f = 5\%$

4.2 Continuous vs. Discontinuous Discharge

As mentioned in chapter 3, previous work [12, 14] suggests that the discharging and sampling method has a small effect on the segregation pattern. This can be confirmed through Figure 4-2 and 4-3. Figure 4-2 shows experimental results for glass(coarse)-steel(fine) system with $\Phi_D = 4$, $x_f = 5\%$, and the two discharge methods. This mixture was chosen because of all the mixtures investigated, it was predicted to show the greatest tendencies to segregate. The segregation profiles show the same trend. There is some difference in magnitude, but it is not too significant. Figure 4-3 shows segregation profiles for a glass-steel mixture with 50:50 volume ratio and $\Phi_D = 1$ and the two discharge methods. The y-axis in Figure 4-3 denotes the normalized steel volume fraction since there are no fines for this experiment.

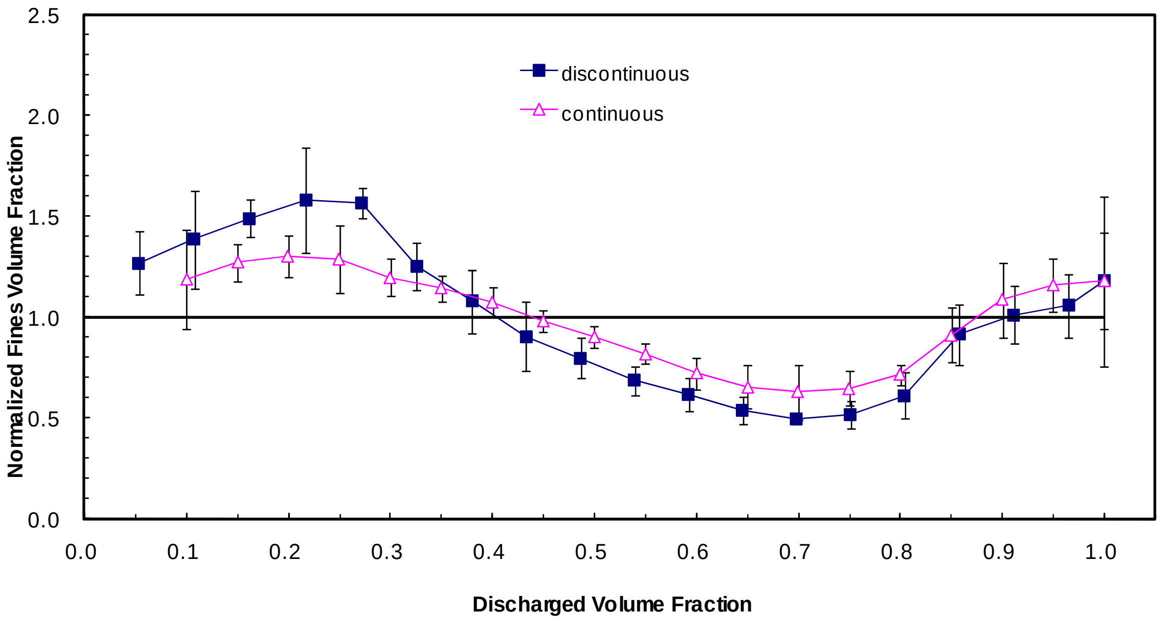

Figure 4-2. Glass(coarse)-steel(fine) mixture experimental results with $\Phi_D = 4$, $x_f = 5\%$, and continuous or discontinuous discharge methods

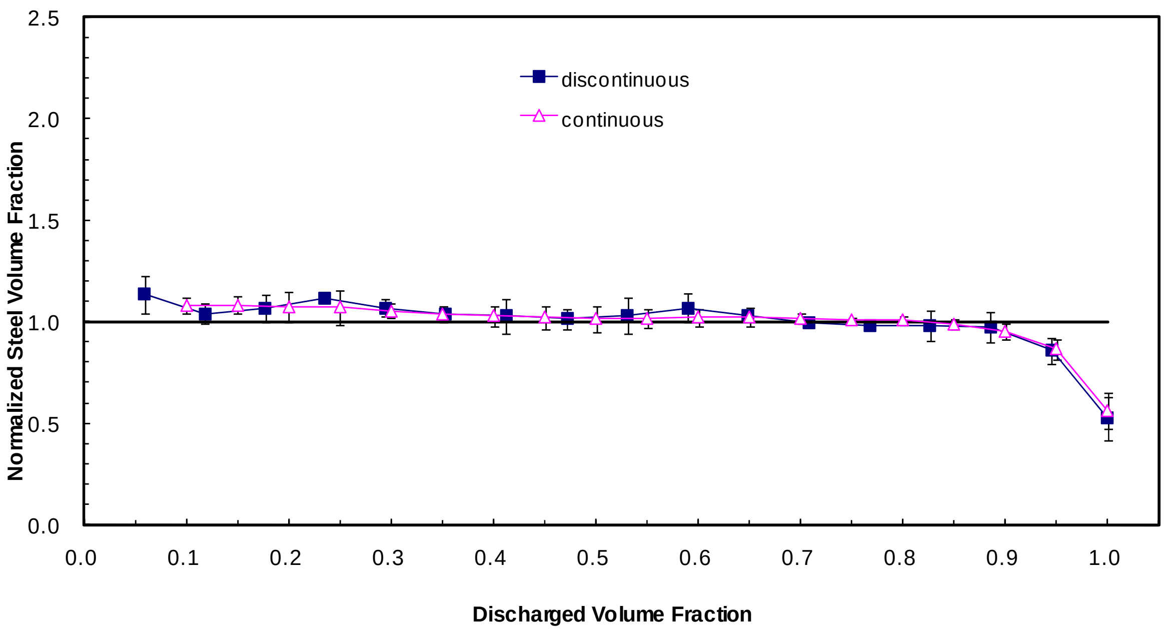

Figure 4-3. Segregation patterns for 50:50 glass-steel mixture $\Phi_D = 1$ and continuous and discontinuous discharge

4.3 Effect of Particle Density

The result shown in Figure 4-3 indicates that in a mixture of equally sized particles, the density difference will cause segregation. There is a slight abundance in steel shots until 90% of the discharge, while the last 10% of the discharge is rich with glass beads. Heavier particles moving downward more readily in the hopper than lighter particles is consistent with this segregation profile.

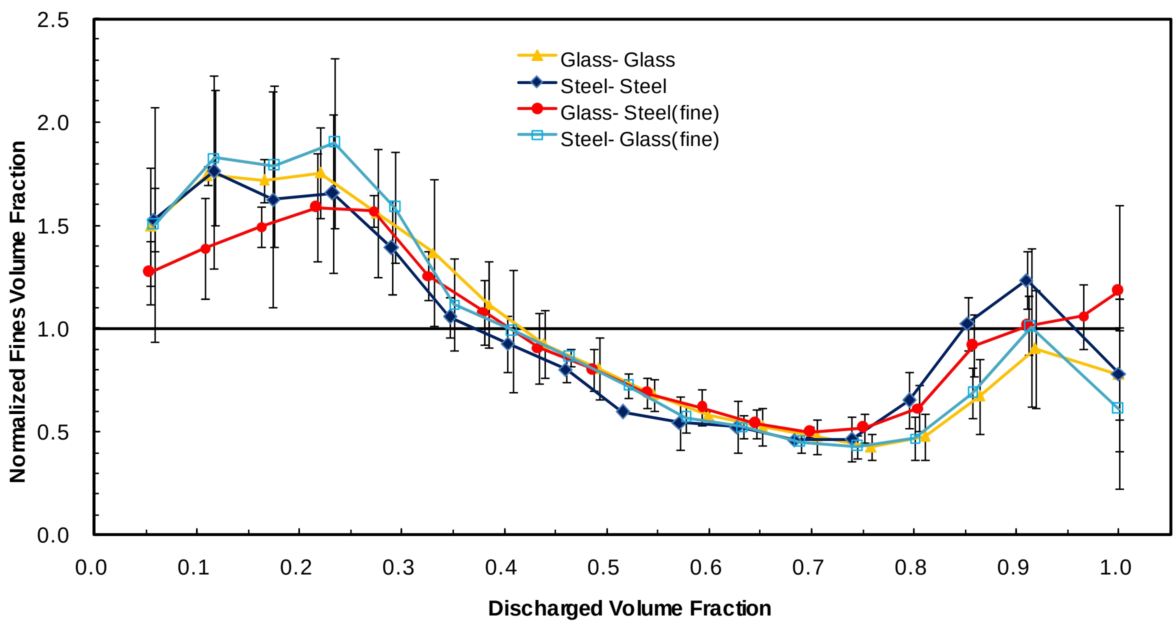

Figure 4-4. Experimental results for mixtures consisted of several combination of materials with $\Phi_D = 4$ and $x_f = 5\%$

However, when both the particle size and density are different, density became a secondary factor for segregation. Figure 4-4 shows experimental results for various mixtures with $\Phi_D = 4$ and $x_f = 5\%$. The magnitude of segregation in the glass-steel (fine) and the steel-glass (fine) is much greater than when density alone is the only variable influencing segregation (as in Figure 4-3). In fact, the density of the individual particles in the binary mix shows very little influence on the resulting segregation patterns when the particle size ratio is 4:1. These results indicate that the segregation behavior is dominated by geometric effects at this particle size ratio and that the effect of particle density or differences in the surface friction between glass and steel are not appreciable.

However, when the size ratio is reduced to 2:1, as shown in Figure 4-5, the qualitative shape of the segregation profiles of the various mixtures is consistent, but some quantitative differences in the segregation behavior are evident. These differences are most likely due to those differences in particle density and/or surface friction between glass and steel – these effects begin to compete with geometric effects as the particle size ratio is reduced.

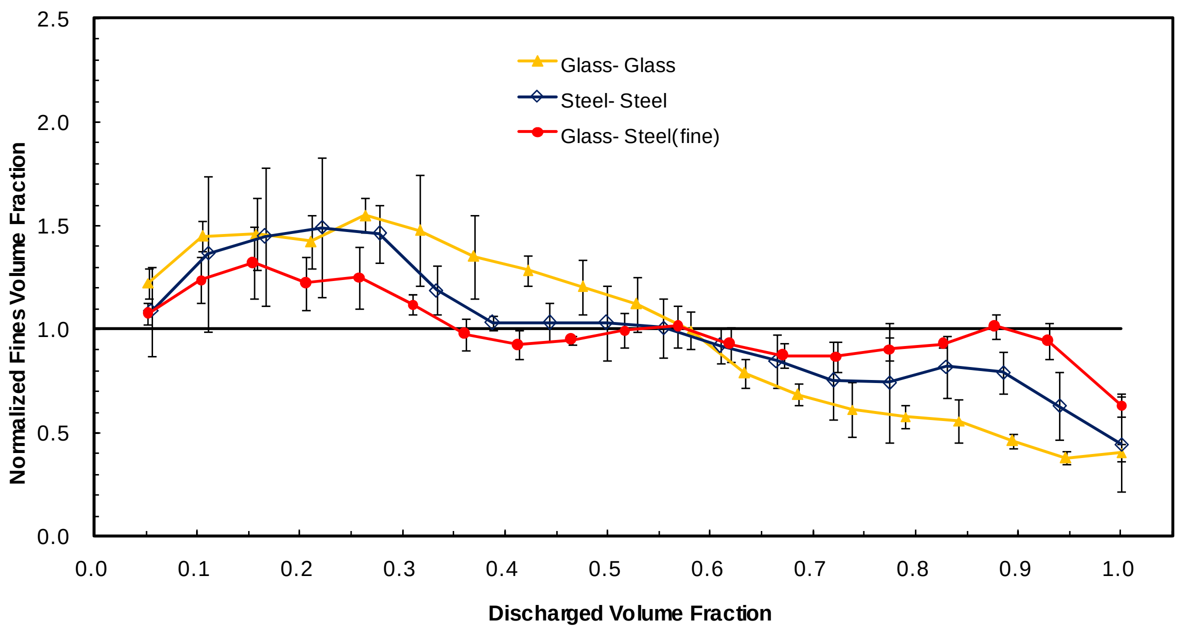

Figure 4-5. Experimental results for mixtures consisted of several combination of materials with $\Phi_D = 2$ and $x_f = 5\%$

4.4 Effect of Fines Fraction

It was concluded in previous experimental works [4, 7, 15] that increasing fines content decreased the magnitude of particle segregation. They hypothesized that as the fines content increases, the number of available void spaces for fines to percolate to decreases – hence, reducing the ability of the mixture to size segregate. These results were reproduced by Ketterhagen *et al.* [12] in Figure 4-6. The segregation patterns for the fines fraction of 5% and

10% are not significantly different. However, as the fines fraction is increased to 20% and 50%, there is noticeable decrease in segregation.

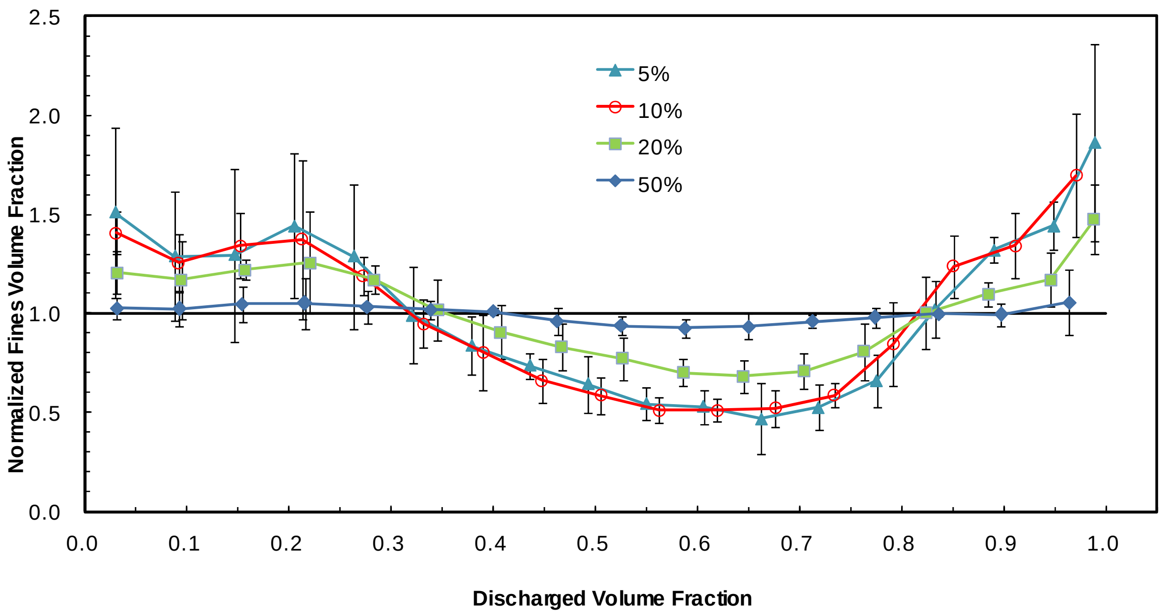

Figure 4-6. Experimental segregation results from Ketterhagen *et al.* [12] for glass-glass mixtures with $\Phi_D = 4$ and given fines fractions

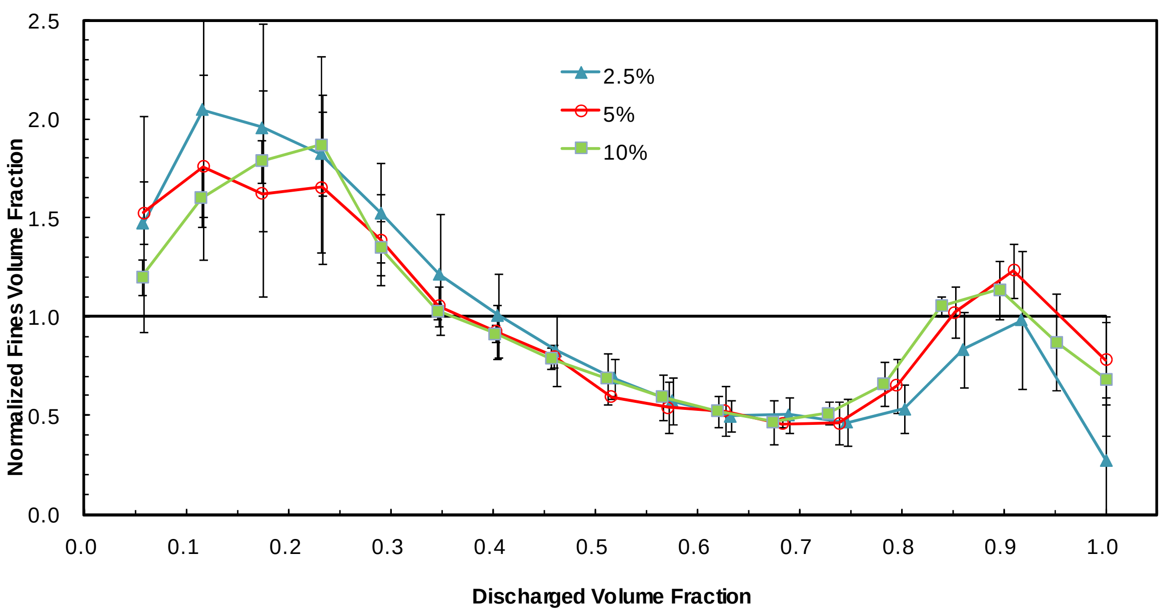

Figure 4-7. Experimental segregation results for steel-steel mixtures with $\Phi_D = 4$ and $x_f = 2.5\%$, 5%, and 10%

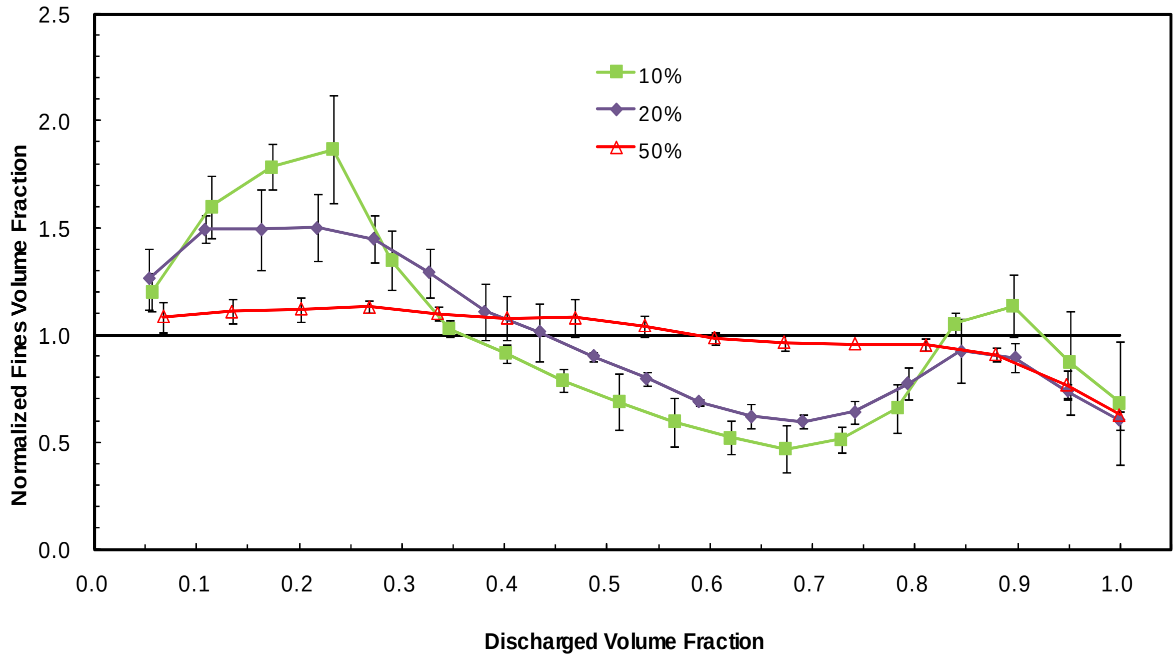

Figure 4-8. Experimental segregation results for steel-steel mixtures with $\Phi_D = 4$ and $x_f = 10\%$, 20%, and 50%

In this work, experiments were conducted with steel-steel mixtures with $\Phi_D = 4$ and various fine fractions, including a 2.5% fines fraction (see Figures 4-7 and 4-8). As in the previous work, the removal of available void spaces with fines fractions less than 10% does not noticeably inhibit segregation. The segregation patterns for the 2.5%, 5% and 10% mixtures are similar. However, as the fines concentration is increased above 10%, significant mitigation of segregation is observed. The 50% fines mixture shows minimal segregation. These results further substantiate the geometric argument for the effect of fines concentration on segregation.

4.5 Effect of Size Ratio

Figures 4-9, 4-10, and 4-11 show the segregation patterns for glass-glass, steel-steel, and glass-steel mixtures respectively with $x_f = 5\%$. These results for spherical particles indicate that, irrespective of material composition, increasing particle size ratio increases particle segregation. The larger the particle size ratio, the larger is the size of each void space between the particles.

And, fines can much more easily percolate into a larger-sized void space between particles – hence, a greater tendency for a mixture to size segregate.

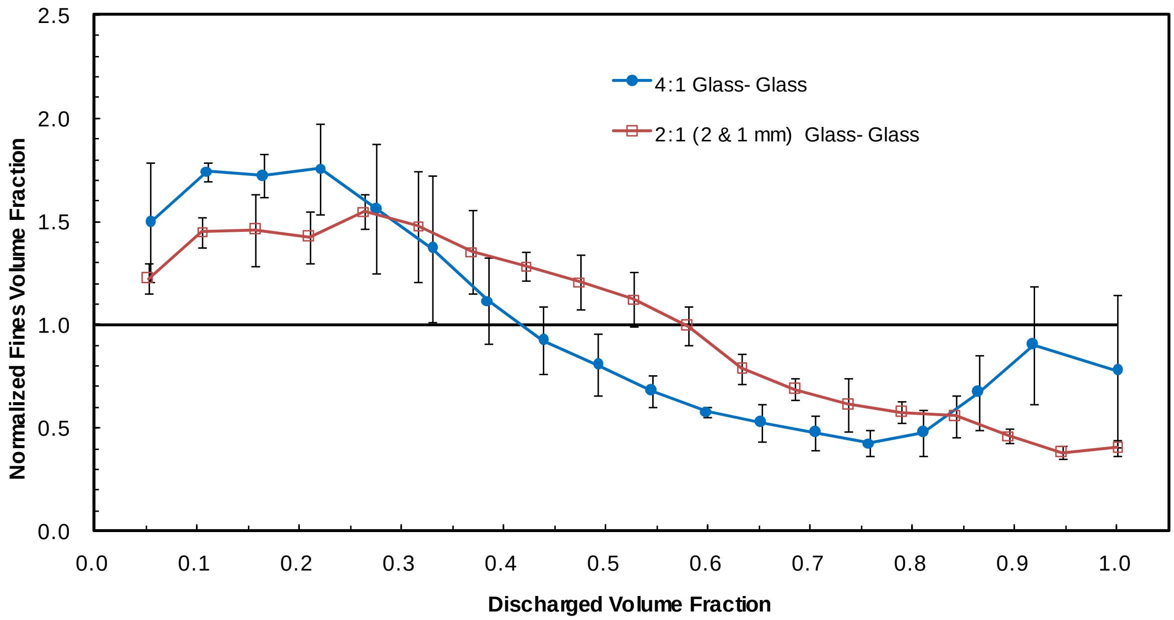

Figure 4-9. Glass-glass mixture experimental results with given size ratios and $x_f = 5\%$

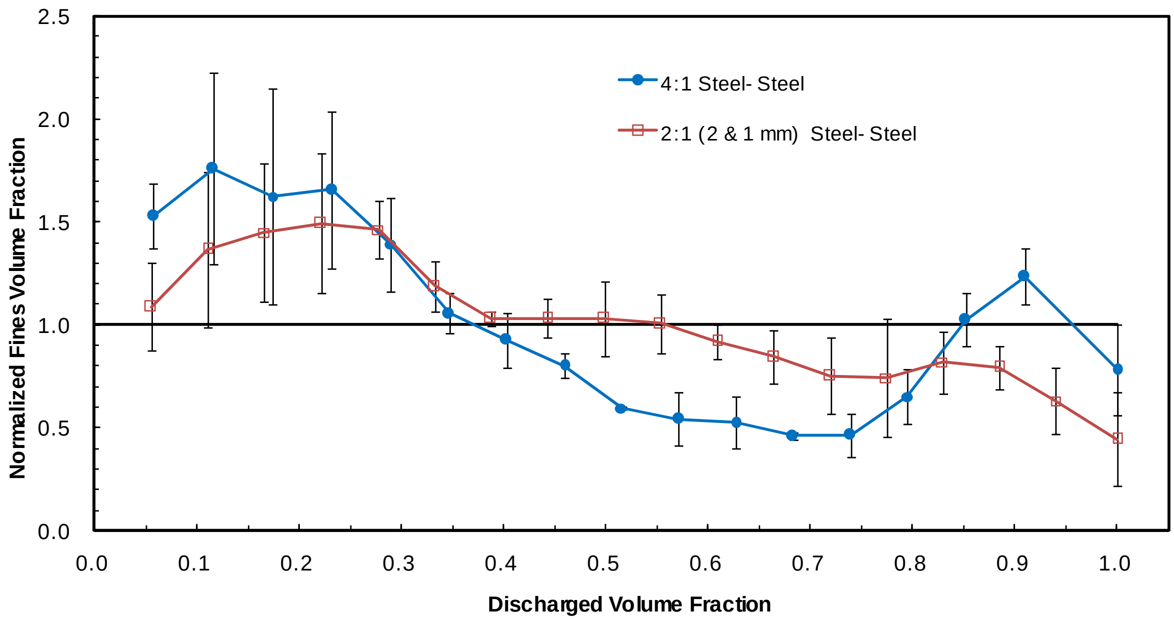

Figure 4-10. Steel-steel mixture experimental results with given size ratios and $x_f = 5\%$

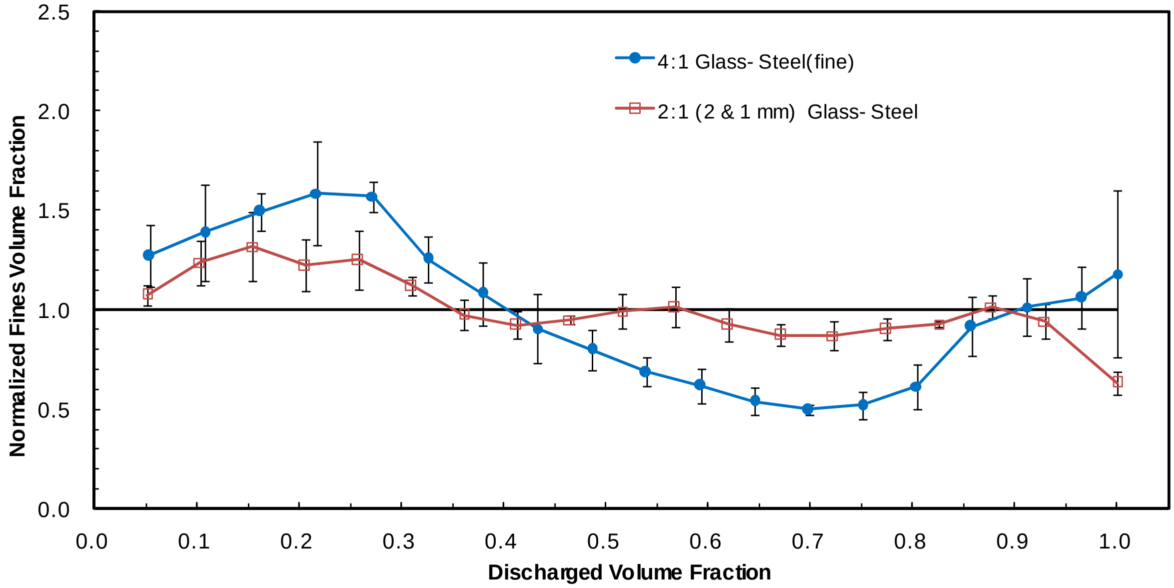

Figure 4-11. Glass-steel mixture experimental results with given size ratios and $x_f = 5\%$

4.6 Effect of Absolute Particle Size

Figure 4-12 shows experimental segregation results for glass-glass mixtures with the same size ratio but different absolute particle sizes. The 2mm/1mm mixture displays more segregation than the 1/0.5mm mixture. Since the 1mm/0.5mm mixture has a higher number of particles and a lower absolute particle size, surface forces are more important than in the 2mm/1mm mixture. More particles increases the number of particle-particle frictional contacts and smaller particles increases cohesive effects. Increasing surface forces tend to inhibit particle migration and mitigate segregation.

4.7 Effect of Particle Shape

Figure 4-13 shows segregation results with a mixture of two sizes of crushed glass compared to spherical glass beads. A marked increase in the angle of the particle free surface during discharge was observed with the crushed glass. Nevertheless, only a minor increase in particle segregation is observed with the crushed glass. It could be that the angular nature of the particles preferentially alters the shape of the void spaces such that, in some cases, the fines can not easily move downward into those available spaces.

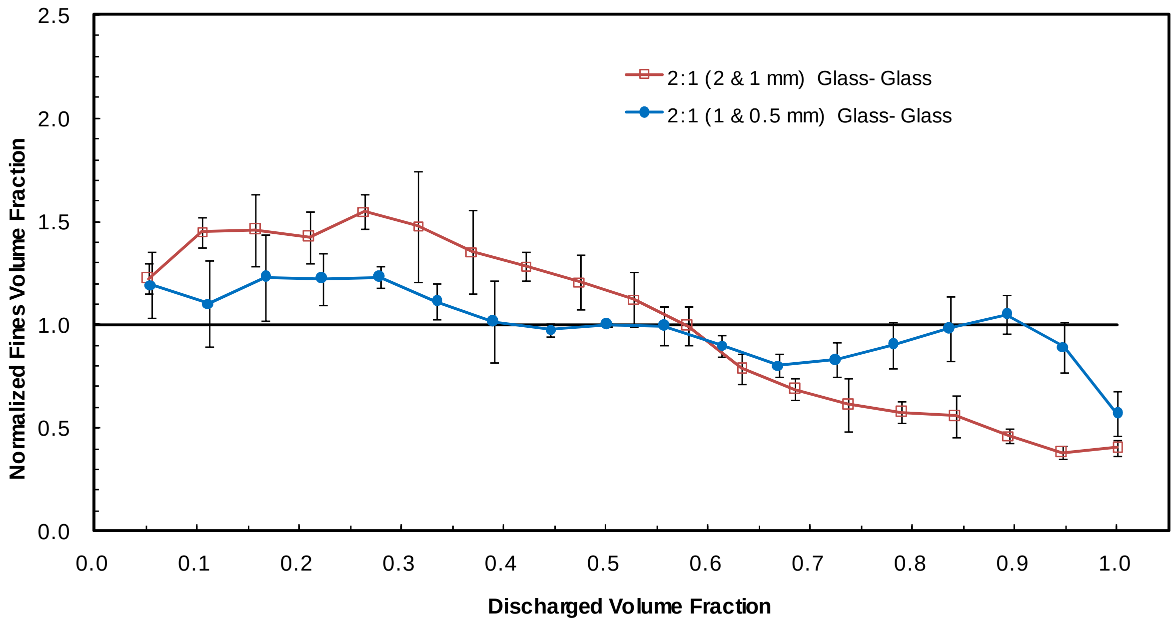

Figure 4-12. Glass-glass mixture experimental results with $\Phi_D = 2$ for different sized particles and $x_f = 5\%$

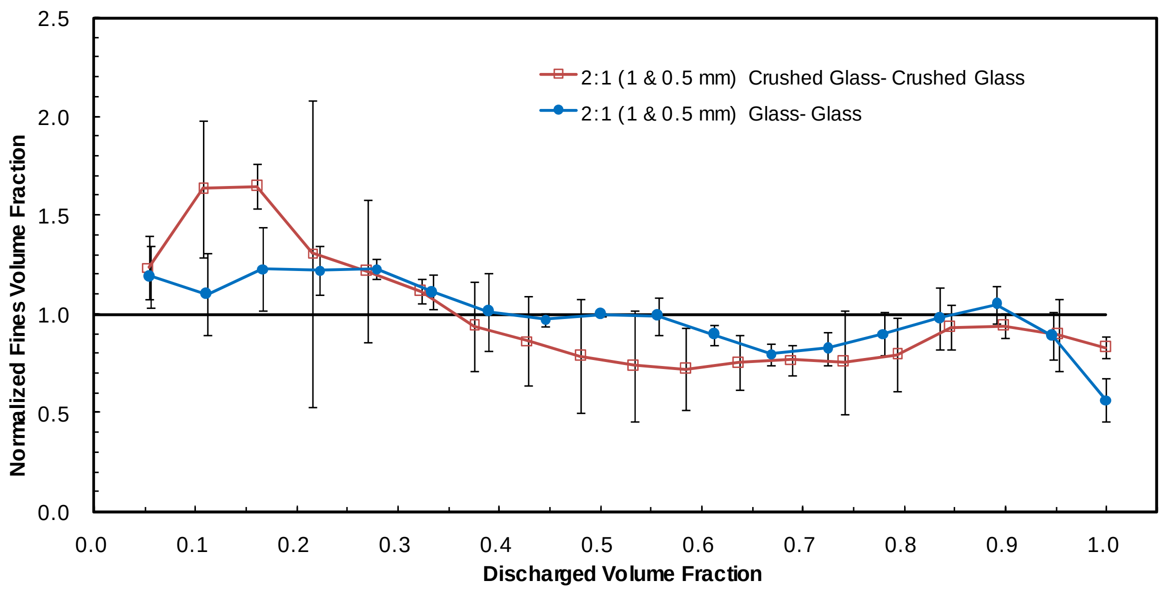

Figure 4-13. Experimental segregation results for crushed glass mixture and glass bead mixture with $\Phi_D = 2$ and $x_f = 5\%$

4.8 Comparison with Previous Experimental Results

Figures 4-14, 4-15, and 4-16 show direct comparisons of the present experimental work with the results obtained by Ketterhagen *et al.* [12]. For particle mixtures involving glass and steel, the shape and magnitude of the segregation profiles are similar. In both sets of experiments, there is a fines rich region in the first half of the discharge, followed by a fines-depleted region.

However, when the results involving the glass/glass mixtures are compared, significant differences in the shape of the segregation profile are observed. These differences are observed at both the 2:1 and 4:1 particle size ratios. In the previous experimentation, after a fines-rich and then fines-depleted region during the discharge, a fines rich region is again observed at the very end of the discharge. This final fines-rich region is not observed in the present experiments. The concentration of fines does increase at the very end of the discharge in the present experiments but overall the composition of the mixture is still slightly fines-depleted.

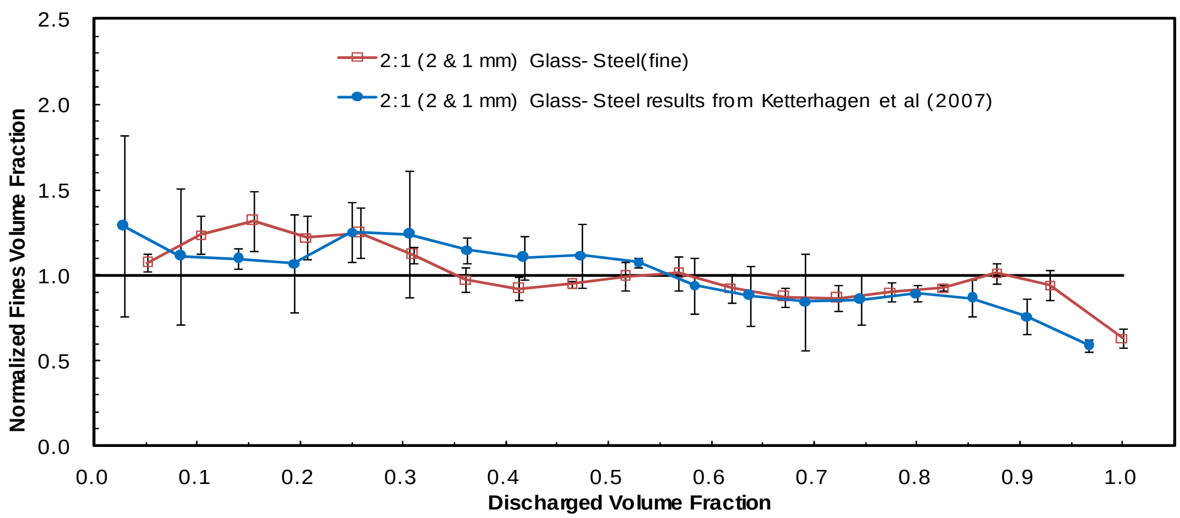

Figure 4-14. Comparison of results for glass-steel mixtures with $\Phi_D = 2$ and $x_f = 5\%$

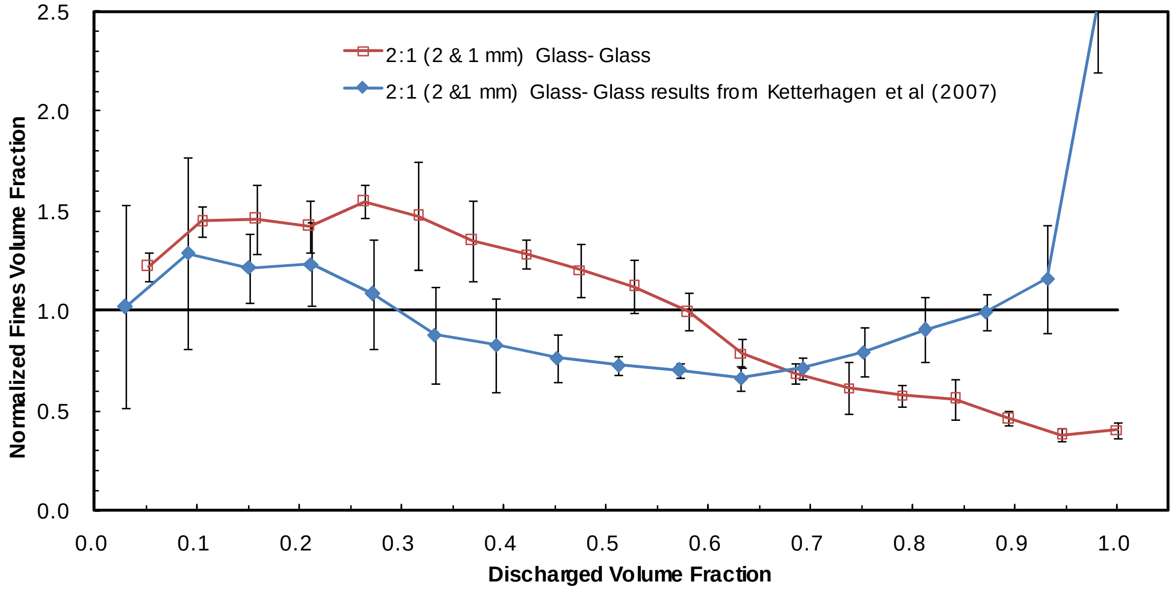

Figure 4-15. Comparison of results for glass-glass mixtures with $\Phi_D = 2$ and $x_f = 5\%$

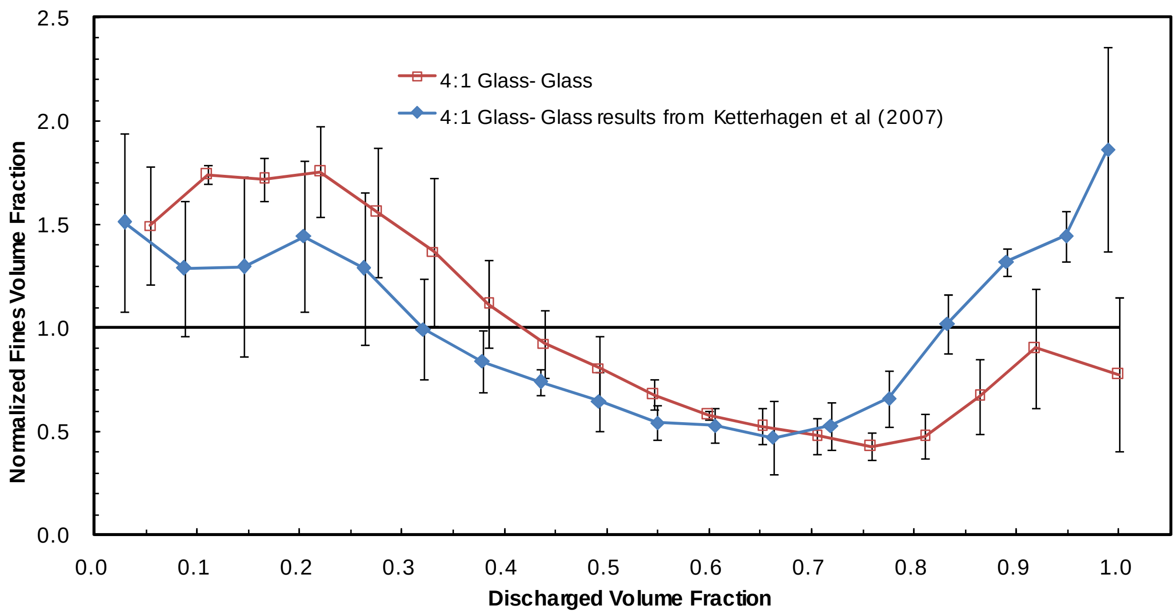

Figure 4-16. Comparison of results for given mixtures with $\Phi_D = 4$ and $x_f = 5\%$

Given that spherical particles with the same density and size were used in both sets of experiments, the most likely cause of this difference between the two sets of results is interparticle forces or particle-wall forces. However, it is unlikely that *electrostatic* interparticle effects are responsible for the differences in the results. While the antistatic bar was used in the current experimentation to reduce/eliminate electrostatic effects whereas the Larostat powder was used previously, the results shown in Figure 4-1 indicate that the method of electrostatic treatment does not play a significant role in the segregation behavior. It is more likely that differences in particle-particle and particle-wall frictional forces are responsible for the variations in the segregation behavior. The glass beads used in the two sets of experiments were purchased from different suppliers and there could be variations in their surface characteristics.

The simulation results of Ketterhagen *et al.* [12] substantiate this hypothesis. Figures 4-17 and 4-18 show the predicted segregation profiles for discharge from a wedge-shaped hopper in which the particle-wall friction coefficient (Figure 4-17) and the particle-particle friction coefficient (Figure 4-18) were varied. Decreasing particle wall friction creates a more mass flow-like behavior in the hopper, minimizing the tendency for fines to be retained in the hopper until the very end of the discharge. With low particle-wall friction a fines-rich region at the end of the discharge does not exist. Also, predictions from the simulation indicate that increases in particle-particle friction produce the same effects as decreasing particle-wall friction. Increasing particle-particle friction inhibits the percolation of fines through the mixture to the bottom of the hopper. Hence, a fines-rich region is not present with increasing particle-particle friction.

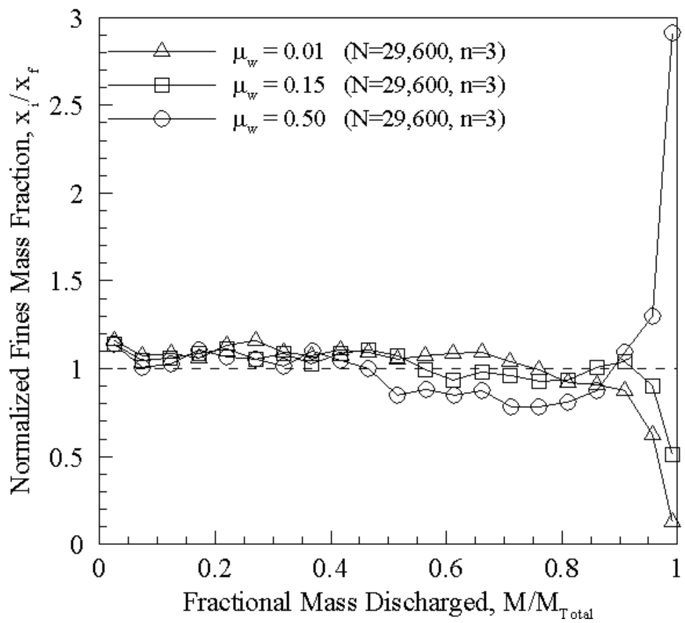

Figure 4-17. Effect of varying particle-wall friction coefficient on predicted segregation profiles for discharge from a wedge-shaped hopper with $\Phi_D = 4$ and $x_f = 5\%$

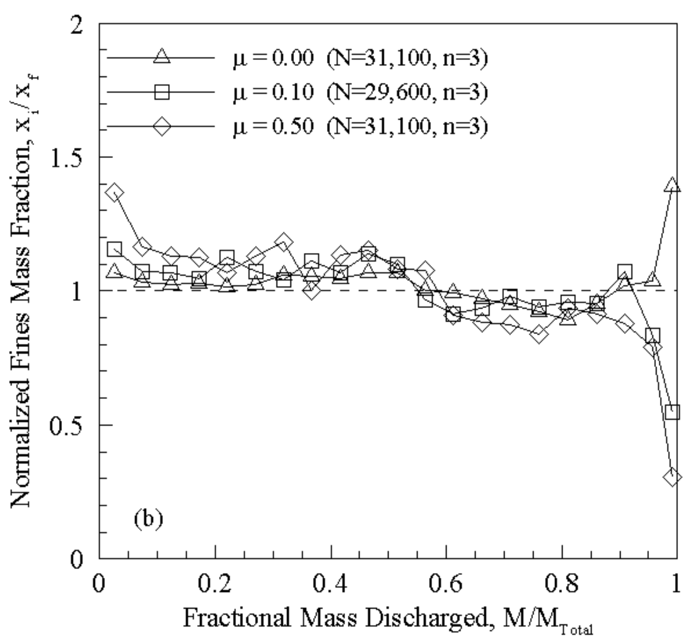

Figure 4-18. Effect of varying particle-particle friction coefficient on predicted segregation profiles for discharge from a wedge-shaped hopper with $\Phi_D = 4$ and $x_f = 5\%$

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

This work examined the segregation pattern of granular materials during discharge from a hopper. The experiments were carried out in an ASTM standard Sifting Segregation Tester. The effects of particle treatment, mode of discharge, concentration of fines, particle size and size ratio, and particle density and shape on segregation were investigated. Various mixtures of glass beads, steel shots, and crushed glass of several sizes were used. Based on the experimental results, the following conclusions are made:

- The method of antistatic treatment or particle washing does not significantly influence the segregation results.
- Segregation increases with increasing particle diameter ratio.
- In general, size-segregation patterns are initially fines-rich and then fines-depleted at the end of discharge.
- Segregation patterns due to density effects are initially rich in particles with the higher density and then rich in the particles with the lower density at the end of the discharge.
- The segregation profiles asymptote for fines fractions less than 10%. When $x_f \geq 10\%$, mixtures with increased fines displays less segregation.
- Given the same particle size ratio, mixtures with smaller absolute particle sizes show less segregation than mixtures with larger absolute particle sizes.
- The extent of segregation for mixtures of crushed glass-crushed glass is not significantly different from spherical glass mixtures.
- The segregation behavior for spherical glass mixtures at the end of the discharge is markedly different than the results reported by Ketterhagen *et al.* [12].

As discussed in chapter 4, the disparity between the segregation trends in this thesis work and the work of Ketterhagen *et al.* [12] is most likely due to the surface effects such as friction. In order to verify this, experiments need to be conducted which spherical glass beads from the same supplier as in the work of Ketterhagen *et al.* [12] to check the reproducibility of their results. The purpose of these experiments is to verify that there are no systematic differences in procedure such as method of particle mixing, hopper filling, particle preparation (e.g. antistatic treatment), etc. If the results from the previous study are reproduced, it is assumed that differences in surface effects are the cause of the discrepancy between the two sets of data. In this case, a surface treatment will then be applied to the two sets of glass beads from the two suppliers. The proposed surface treatment will make the particles hydrophobic and should create consistent surface characteristics and consistent segregation behavior between the two sets of particles. If the results from the previous study are not reproduced, any differences in hopper filling method, method of particle mixing, particle preparation and other particle properties (such as sphericity) needs to be investigated in detail.

The effect of particle shape should be further explored. In the present work crushed glass mixtures (2:1 size ratio) did not show much difference in segregation behavior from their spherical mixture counterpart. However, previous studies of mixtures of non-spherical and spherical particles did show a difference in segregation behavior from a purely spherical particle mixture. Experiments with crushed glass mixtures of size ratio 4:1 should be conducted to see if particle shape effects become more pronounced with increasing size ratio. Additional experimentation with mixtures of non-spherical and spherical particles, varying size ratio, fines concentration, and fines material, would give enhanced insight into the effect of particle shape.

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BIOGRAPHICAL SKETCH

Byung-Hwan Chu was born in Seoul, Korea, in 1983. He enrolled at New Mexico State University beginning in 2002. After receiving his Bachelor of Science degree in chemical engineering in 2006, the author started his graduate study in chemical engineering at the University of Florida. Upon receiving his Master of Science degree, the author plans to continue his education to earn a Ph.D.