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Size Segregation of Ternary Mixtures in Inclined Chute Flows: an Experimental Study

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Granular materials can segregate spontaneously due to differences in particle properties when subjected to process vibration, shear strain or because of the equipment geometries. Among the different properties, difference in particle size is the most relevant factor that drives segregation. Although size-driven segregation has serious technical implications in a lot of industrial processes, a fundamental understanding of the phenomenon is still lacking. Furthermore, models and theories on segregation are often validated with DEM simulations as an alternative to experiments. This leads to a shortage of experimental data.
In this paper we experimentally investigate size-driven segregation of ternary mixtures of grains flowing down an inclined plane, for a range of mixture compositions. The segregation process is filmed through the transparent sidewall with a camera, and the evolution of the particle concentration is evaluated by means of post-processing image analysis. Since different-sized particles are of different colors, the post processing procedure consists in associating the color information of each pixel to the respective component. It is found that the segregation features are strictly related to the relative amount of the largest grains: a higher fraction of the larger particles improves mixing and reduces segregation.

1. Introduction

Granular materials composed of particles with different physical properties are notoriously prone to segregate when subjected to vibrations, shear strain or because of the equipment geometries. This heterogeneity in the powder represents an adversity on product quality and production costs and has serious technical implications in many industrial fields such as pharmaceutical, bulk chemical, food and agricultural (Gray and Ancey, 2011; Van Der Vaart et al., 2015). The development of tools for predicting segregation is therefore essential in order to control and minimize the occurrence.
Although there has been considerable recent progress in developing continuum based segregation models (Van Der Vaart et al., 2015), a fundamental understanding of the phenomenon is still lacking, and industrial applications still rely on empirical heuristics rules. Furthermore, because of the difficulty in measuring experimentally the evolving particle-size distribution (Gray, 2018), models and theories on segregation are often validated by means of DEM simulations that, however, are models in themselves. This leads to a shortage of experimental data.
In this paper we experimentally investigate size-driven segregation of ternary mixtures of grains flowing down an inclined plane, for a range of mixture compositions. We focus on gravity driven chute flow owing to its practical importance in granular processing (for instance, one could think to the transfer equipments in a steel industry) as well as its relative simplicity that allows for development and testing of new theories (Bhattacharya and McCarthy, 2014). A literature search reveals that many experimental investigations of the segregation phenomenon in chute flows involve binary (Savage and Lun, 1988) and polydisperse mixtures (Bhattacharya and McCarthy, 2014). Segregation in ternary mixtures has been experimentally studied in a lot of systems such as vibrated systems (Metzger et al., 2011), rotating drums (Gray and Ancey, 2011), down a pile (Johanson, 2014) or during hopper discharge (Yu and Saxén, 2010) but, to the authors' knowledge, never down inclined planes.

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The experiments are performed on a rough chute composed of a flow channel and a reservoir. The mixtures are made of three different-sized classes of cous-cous particles previously colored. To investigate the effect of particles concentration, we employ five different mixture compositions. The chute is initially filled with a homogeneously distributed and well-mixed layer of settled particles. By increasing the chute inclination, the powder starts to flow while an inflow feeds the system. The segregation process is filmed through the frontal transparent sidewall with a camera, and the evolution of the local concentration is quantified by means of post processing image analysis. To distinguish particles belonging to different-sized classes, the obtained images are subjected to image thresholding techniques. Then, the magnitude of segregation is evaluated through the statistic chi-square.

The paper is organized as follows. The experimental setup, the measurement method and the post processing procedure are discussed in Section 2; the experimental outcomes are reported in Section 3; the conclusions are drawn in Section 4.

2. Materials and method

2.1 Experimental setup

The experiments reported in this paper are conducted on a rough chute 1.1 m long similar to that used in a previous work (Santomaso and Canu, 2001). The particles are confined between two vertical walls made of transparent plexiglass. The gap between the sidewalls is 2.0 cm wide. For the no-slip boundary condition to hold, the bottom wall is made rough by gluing sandpaper on it. To ensure a constant feed rate to the chute, its upper part is used as a reservoir of particles. The discharge orifice, and hence the feeding rate, are kept constant for all the experiments. At the beginning, the chute lies horizontally, the mixture is filled within the reservoir, and a layer of particles is deposited on the chute. The thickness of the deposited layer is 1.5 cm in depth, and, after an initial transient wave, it remains approximately constant until complete discharge. To avoid bias in the segregation evaluation, the filling procedure requires particular care to ensure a well-mixed initial state both in the deposited layer and within the reservoir. The granular flow is then triggered by inclining the chute until the critical inclination angle (i.e. the internal friction angle characteristic of the material employed that ensures homogeneous flow) of $\theta_c = 33°$ with respect to the horizon. Note that the angle of inclination controls the flow behavior: at smaller angles the powder would accumulates on the rough bottom leading to the formation of a granular pile; whereas at higher angles the flow would accelerate indefinitely (Santomaso and Canu, 2001). The flow is recorded with a 2-megapixel USB camera placed approximately 50 cm far from the feeding point. The videos are acquired at a rate of 120 frames per second and a resolution of 640x480 pixels. To ensure constant and reproducible illumination conditions, two sets of dimmable white and yellow LEDs are pointed directly towards the recording area.

2.2 Materials

As granular material, we need three classes of dry cohesionless spherical particle having same density but different size and different color. The latter is a fundamental requirement for distinguishing different-sized particles by means of image analysis. After having evaluated different candidates, we have employed ternary mixtures of three fractions of cous-cous particles obtained using sieving: a fine fraction, a middle fraction and a large fraction. Their mean diameters are equals to 0.90, 1.09 and 1.30 mm respectively. Table 1 shows the compositions of the five ternary mixtures employed in terms of mass fractions. In order to increase the accuracy of the results and estimate their variability, four replicates for each composition are performed.

Table 1: Composition of the 5 mixtures tested in terms of mass fractions of the small (w_S), medium (w_M) and large (w_L) grains. Each class is characterized by its own color.

	Colour	Mixture 1	Mixture 2	Mixture 3	Mixture 4	Mixture 5
w_S	Red	0.20	0.60	0.20	0.10	0.40
w_M	Green	0.30	0.10	0.50	0.10	0.40
w_L	Blue	0.50	0.30	0.30	0.80	0.20

Cous-cous particles are suitable also because easily colorable with color additives. Several trial experiments showed that the best combination of colors in order to have non-overlapping and narrow picks in the hue channel are red (for smaller particles), blue (for larger particles) and green (for middle-sized grains). Figure 1 shows the spectrum of each pure component. The red pick is identified between 0.98 and 1.02, the green pick spans between 0.18 and 0.28, whereas the blue one is in between 0.47 and 0.57. Since they are wide apart, the mixture components can be uniquely identified by simply looking at the hue value of each pixel. This is advantageous because, unlike other coloring procedures, it does not require a calibration curve (Dal Grande et al., 2008). Further details on the post-processing procedure are reported in Section 2.3.

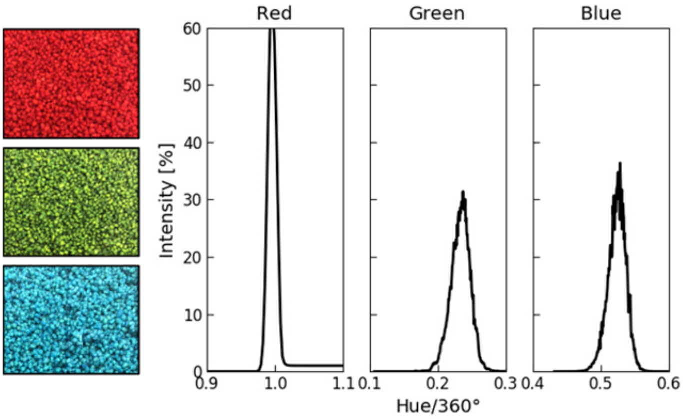

Figure 1: Hue values of the three different-sized and different-colored classes of cous-cous. The average class diameter of red, green and blue particles is equal to 0.90 mm, 1.09 mm and 1.30 mm respectively.

2.3 Image analysis

First, the recorded videos are converted into sequences of images. Next, the RGB images are converted to grayscale and used for foreground–background segmentation. Preliminary experiments on many videos showed that good thresholding limits for distinguishing the particles on the foreground (i.e. those at the sidewalls) from the ones on the background are 0.12 and 0.70. Once the background is removed, the particles are identified according to their own color, likewise the human eye does. To discriminate against components, we use the color thresholds reported in Figure 1. Note that, we retrieve color information from the hue channel of the HSV (Hue, Saturation and Value) color space, however one could prefer to work in HLS (Hue, Lightness and Saturation) or HIS (Hue, Saturation and Intensity) color spaces. The entire procedure for the post-processing image analysis is summarized in Figure 2. The procedure leads to four segmented images: one for the background and one for each class of particles. The effectiveness of the procedure can be qualitatively assessed by comparing the original image with the reconstructed one.

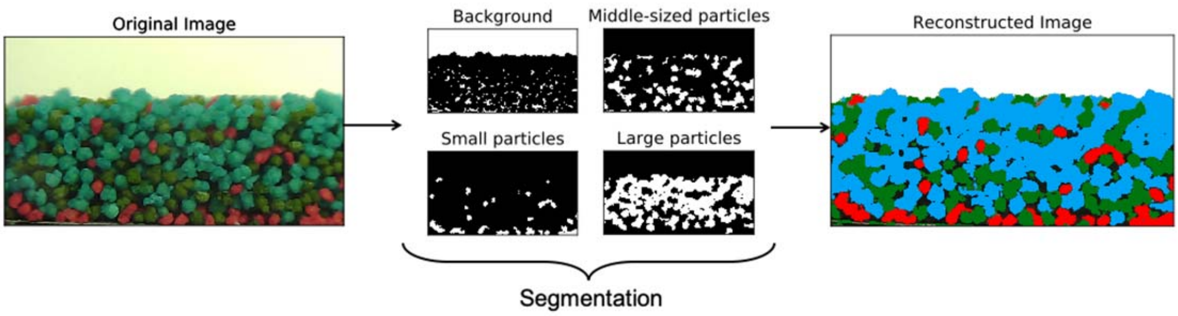

Figure 2: Steps toward images reconstruction. The original frame is converted both to gray scale and HSV color space to obtain background segmentation and components segmentation respectively. The reconstructed image, compared with the original one, shows the goodness of the approach.

2.4 Degree of segregation

To evaluate the magnitude of segregation along the bed depth, the images are split into non-overlapping superimposed layers of 5 pixels high. The number of layers is not fixed but changes according to the evolution of the bed thickness, however it is almost always equal to 45-50 pixels. In each *j-th* layer, the solid area fraction of the *i-th* sized class is calculated as:

$$c_j = \frac{px_{i,j}}{\sum_{i=1}^{n} px_{i,j}} , \quad (1)$$

where px_i is the number of pixel belonging to the *i-th* class. The solid area fraction is then converted into mass fraction and used to asses segregation.

The intensity of segregation in binary mixtures is usually evaluated by means of segregation indices obtained by combining standard deviations. However, calculating variances for ternary or multicomponent mixtures is much more complicated (Rosas and Blanco, 2012). In this work, we use the segregation index proposed by Gayle et al. (1958). This index is suitable for ternary mixtures, it is based on chi-squared statistics and reads (Gayle et al., 1958):

$$S_\chi = \frac{\chi^2 - \chi_R^2}{\chi_S^2 - \chi_R^2}, \tag{2}$$

where χ_R^2 is the expected chi-square for random mixtures, χ_S^2 is the expected chi-square for segregated mixtures and χ^2 is the observed chi-square. The former is equal to the overall number of degrees of freedom, namely the number of degrees of freedom in each sampled layer multiplied by the total number of layers (note that here there are 2 degree of freedom in each sampled layer since the sum of all mass fractions is equal to 100% and the fraction of the third component can be uniquely determined by difference). χ_S^2 is instead the overall number of degrees of freedom multiplied by the total mass fraction in each sample, which is fixed at 100% (Gayle et al., 1958). The observed chi-square χ^2 is instead (Gayle et al., 1958):

$$\chi^2 = \sum_j \left(\sum_{i=1}^{n} \frac{(O_i - E_i)^2}{E_i} \right). \tag{3}$$

In Equation 3, O_i and E_i are respectively the observed and expected frequencies of the *i-th* component in the *j-th* layer. As frequencies, we used mass fractions: the expected mass fraction is the initial mixture composition, whereas the observed mass fraction is the local one. The segregation index, S_χ, such obtained can vary from zero for a well-mixed system to unity for a complete segregated system.

3. Results and discussion

3.1 Raw results

We have reconstructed the evolution of the small, medium and large particles in time and throughout the dimensionless depth of the flowing layer (i.e. H/d_s where d_s is the small particles mean diameter). The resulting contour maps are reported in Figure 3 and Figure 4 respectively for mixtures 1 and mixture 5. The contour maps for the other mixtures are omitted for space reasons.
The contour maps are characterized by 100 contour regions and, to reduce the noise, concentrations are smoothed over time by applying a moving average with a rolling window of 60 frames. This is the reason why there is a time-lag of 0.25 s. As Figure 3 shows, as soon as the flow begins, the finer particles move downwards, the larger grains rise on top, and middle-sized grains collect in between. This happens because small particles are statistically more likely than larger grains to fit into gaps that open up beneath them (Savage and Lun, 1988). This leads to inversely graded layers with coarser particles on top (Gray, 2018). We can also see that the interface delimiting the finer particles is much more defined than the one delimiting the larger one. The situation is a bit different in Figure 4. In that case, fewer large particles were present in mass, and hence much smaller in number. It happens that, middle grains collect on top, whereas the few large particles are dragged in the middle of the flow. This evidence proofs that segregation is affected by the relative amount of each component. It is also interesting to note that, in that case, the rate of segregation is slower.

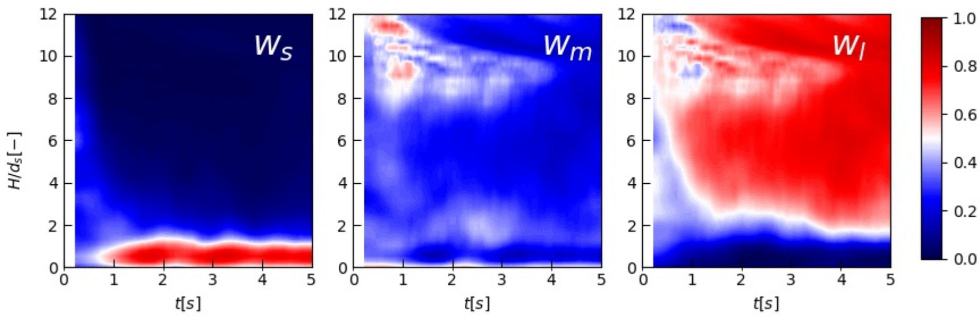

Figure 3: Experimental measurements of small (on the left), medium (in the middle) and large (on the right) particles in time and throughout the dimensionless depth of the flowing layer (i.e. H/d_s). The mixture under analysis is mixture 1, which is characterized by $w_S = 20\%$, $w_M = 30\%$ and $w_L = 50\%$.

Since the segregation process can be influenced also by the pore size distribution, we have estimated the initial packing density (i.e. in static conditions) as a function of the fractions of our three classes of cous-cous particles. This estimation was done with the 3-parameter model proposed by Wong and Kwan (2014). We found that, the packing density changes in between 0.51 and 0.53. Because this negligible variation, and since we do not know how packing density distributes during flows with respect to the initial packing density, we could not appreciate its influence on the segregation features.

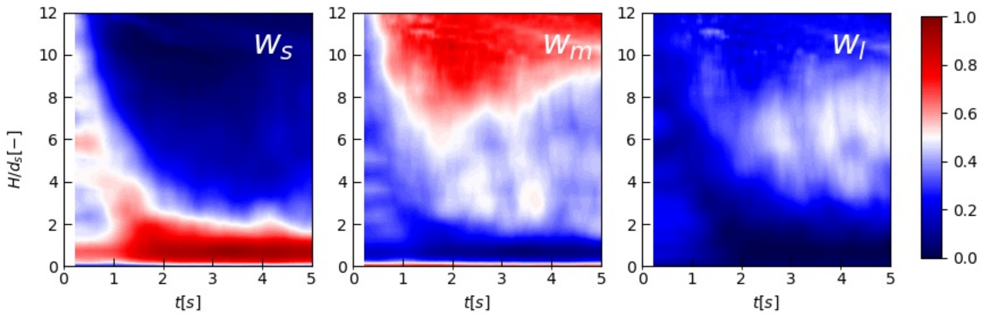

Figure 4: Experimental measurements of small (on the left), medium (in the middle) and large (on the right) particles in time and throughout the dimensionless depth of the flowing layer. The mixture under analysis is mixture 5, which is characterized by $w_S = 40\%$, $w_M = 40\%$ and $w_L = 20\%$.

3.2 Segregation evaluation

Figure 5 shows quantitative measures of the segregation index (see Equation 1) as a function of time for the five mixtures tested. The experimental outcomes, which are noisy and fluctuating, are fitted with the following sigmoidal function:

$$y = L \tanh(kt) \ , \tag{4}$$

where L is the asymptotic value reached for $t \to \infty$ and k is the velocity required to achieve L. The parameters such obtained are reported in Table 2. As expected, the segregation potential is different according to the relative amount of each component. For instance, mixture 2, which is the one characterized by a higher concentration of fines, displays a higher segregation intensity. On the other hand, mixture 4, that is the one with higher mass of larger grains, segregates less. Moreover, if the relative amount of small and large particles is kept constant (see Mixtures 2 and 5), an increase in the number of middle-sized grains decreases the extend of segregation (Metzger et al., 2011).

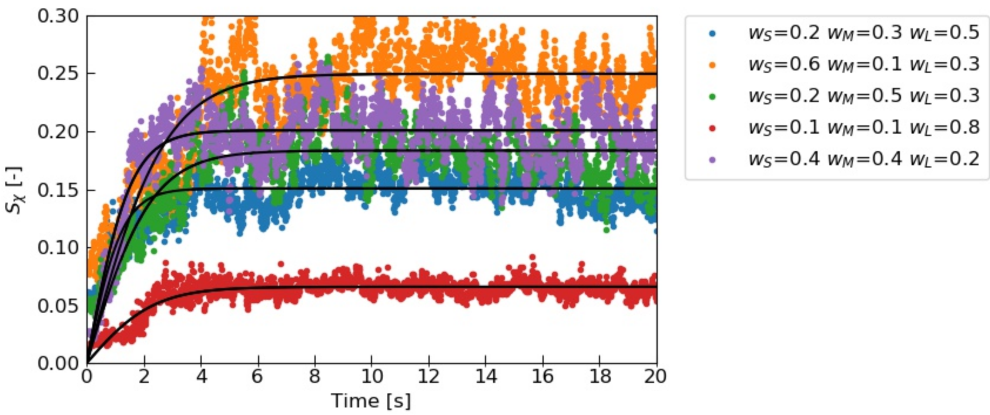

Figure 5: Evolution of the segregation index. The experimental data (points) are fitted with Eq. 4 (solid lines).

To describe how L relates to the mixture composition, we derive a multiple linear regression model. As independent variables we set the mass fractions of the small, medium and large particles. We obtained:

$$L = 0.14 + 0.22\, w_S + 0.04\, w_M - 0.12\, w_L \ . \tag{5}$$

Despite the limited number of experiments, the R^2 is equal to 0.954 thus, we can say with good accuracy that the segregation potential growths with the increase of the small particles and reduces with the increase of the large particles. Furthermore, the mass fraction of middle-sized grains has lower impact of the final degree of segregation. This, however, is true only for the range of concentration tested. To extend this result, one should consider more mixture compositions, including also the limiting case of binary systems. As concerns the parameter k, there is not significant evidence that it is related to the mixture composition. Nevertheless, deeper studies are required to check the generality of this new method.

Table 2: Parameters of the sigmoidal function used to fit the experimental segregation index.

	Mixture 1	Mixture 2	Mixture 3	Mixture 4	Mixture 5
L	0.151	0.250	0.183	0.066	0.201
k	0.795	0.363	0.442	0.424	0.652

4. Conclusions

Size-driven segregation in ternary granular mixtures flowing down inclined planes is experimentally studied for a range of mixture compositions. As suggested by the experimental data, the relative amount of each component influences the segregation potential. In particular, the effect of segregation is reduced as the number of large particles increases. Furthermore, if there are only few larger particles in the mixture, they do not reach the free surface but remain trapped in between a lower layer of fines and an upper layer of middle-sized grains. As concerns the rate of segregation, we did not find significant relations.

To the author's believes, this work has a huge potential. First, it presents a fast, effective and cheap method for measuring the evolving particle-size distribution. Data such obtained could be used to determine the optimal formulation required to prevent segregation and improve process performances. Then, it fills the shortage of experimental data. To our knowledge, no one has experimentally studied segregation by size in ternary systems during chute flow. Last but not least, the provided results are usable for developing and validating multi-component theories describing segregation.

References

Bhattacharya T., McCarthy, J. J., 2014, Chute flow as a means of segregation characterization, Powder Technology, 256, 126-139.

Dal Grande F., Santomaso A.C., Canu P., 2008, Improving local composition measurements of binary mixtures by image analysis, Powder Technology, 187, 205-213.

Gayle J.B., Lacey O.L., Gary J.H., 1958, Mixing of Solids. Chi Square as a Criterion, Industrial and Engineering Chemistry, 50, 1279-1282.

Gray J.M.N.T., Ancey C., 2011, Multi-component particle-size segregation in shallow granular avalanches, Journal of Fluid Mechanics, 678, 535-588.

Gray J.M.N.T., 2018, Particle Segregation in Dense Granular Flows, Annual Review of Fluid Mechanics, 50, 407-33.

Johanson K., 2014, Review of new segregation tester method by Dr. Kerry Johanson, P.E., Powder Technology, 257, 1-10.

Metzger M.J., Remy B., Glasser B.J., 2011, All the Brazil nuts are not on top: Vibration induced granular size segregation of binary, ternary and multi-sized mixtures, Powder Technology, 205, 42-51.

Rosas J.G., Blanco M., 2012, A criterion for assessing homogeneity distribution in hyperspectral images. Part 1: Homogeneity index bases and blending processes, Journal of Pharmaceutical and Biomedical Analysis, 70, 680-690.

Santomaso A.C., Canu P., 2001, Transition to movement in granular chute flows, Chemical Engineering Science, 11, 3563-3573.

Savage S. B., Lun C. K.K., 1988, Particle size segregation in inclined chute flow of dry cohesionless granular solids, Journal of Fluid Mechanics, 189, 311-335.

Van der Vaart K., Gajjar P., Epely-Chauvin G., Andreini N., Gray J.M.N.T., Ancey C., 2015, Underlying Asymmetry within Particle Size Segregation, Physical Review Letters, 114, 1-5

Wong V., Kwan A.K.H., 2014, A 3-parameter model for packing density prediction of ternary mixes of spherical particles, Powder Technology, 268, 357–367.

Yu Y., Saxén H., 2010, Experimental and DEM study of segregation of ternary size particles in a blast furnace top bunker model, Chemical Engineering Science, 65, 5237-5250.