

## Article

# Mixing of Bi-Dispersed Milli-Beads in a Rotary Drum. Mechanical Segregation Analyzed by Lab-Scale Experiments and DEM Simulation

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**Abstract:** Mechanical flow and segregation phenomena within a bed composed of milli-metric size spherical beads rotated in a horizontal drum were investigated. The beads population was bi-dispersed, with two kinds of binary (half by half) compositions: a bi-size bed with two different sizes and a bi-density bed with two different densities. The distributions of the beads were observed optically on the front side of the bed by means of a lab-scale drum prototype. Different numbers and lengths of peripheral straight baffles were tested as well as different drum filling ratios. The photographic data were processed to obtain the front layer mechanical segregation index. This experimental index was compared to the simulated one, obtained by means of commercial discrete element software EDEM. The simulations were corroborated by the experiments provided that the friction coefficients of the discrete elements method (DEM) model were correctly adjusted. The global segregation index was also calculated from simulation data for all considered cases and its values were lower and less sensitive to baffles' configurations than those for the front layer.

**Keywords:** rotary drum; particulate solid; bi-dispersed bed; size and density segregation; optical observation; DEM simulation

## 1. Introduction

The segregation phenomenon in agitated particulate beds is a spontaneous process of separation and spatial redistribution of particles of different kinds. Particulate stirred materials may segregate because of differences in density, size, surface roughness, or shape. Industrial materials, even if carefully controlled before processing, are always slightly dispersed. Even a slight deviation may induce non uniform processing of the particulate material and heterogeneities of intra-batch final product properties. The most common initial material dispersion concerns size and density. The particle size or density variation may be induced by the process itself: agglomeration, coating, wet granulation or drying are all processes impacting strongly the particle features. Discrete elements method (DEM) modeling, which allows prediction of the interactions and position of each particle of the considered system is very appealing and promising to predict the homogeneity of the product. DEM simulations could provide valuable information about the particle trajectory and properties in locations not accessible by experiments.

According to the literature, rotary mixing of imperfectly monodispersed solid particulate beds unavoidably leads to mechanical segregation, i.e., to the accumulation of smaller or/and denser particles in the core of the bed. Boateng and Barr [1] analyzed thoroughly the segregation mechanisms for a

binary mixture of small and big particles. The main mechanism of transverse (radial) segregation is the percolation process when smaller particles will tend to filter downwards through a bed of flowing granular material while large particles will simultaneously tend to be displaced upwards. Accordingly, they developed an analytical and numerical model to predict the size and extent of the segregated core in relation with primary operating parameters such as drum diameter, bed depth and rotational speed.

Industrial-scale rotating drum mixers, granulators or dryers are usually operated in the rolling mode since it is supposed to ensure good particle mixing. In the rolling mode (called also continuous plane surface flow mode), the particulate bed volume can be simply divided into two zones: the active (also called flowing or shear layer) zone and the passive zone [1,2]. The particles in the passive zone are driven by the rotating drum wall, and particles in active zone fall down the slope after detaching from the wall. All the material in the passive zone rotates as bulk with the drum and the particles do not move in relation to one another. As the segregation phenomenon is based on the relative movement of the particles, segregation will occur mainly in the active zone [1,2]. The thickness or area of this zone are considered of prime importance for the mixing quality in the whole drum. As observed by Félix [2] during experiments with small glass beads, the thickness of the active zone increases with the drum/particle size ratio (drum diameter to particle diameter ratio). In the case of a bi-dispersed bed, it is then expected to obtain a shorter segregation time for a small drum/particle size ratio than for a large one. In the latter case, the distance the particle will have to cross in the active zone to be segregated will be longer and the segregation will progress slowly.

Xiao et al. [3] aimed at quantifying the relation between the active zone width and the segregation intensity. In their study, a DEM model was used to simulate the mixing process of equal sized plastic (ABS) beads (with two kinds of color) in a rotating drum at different rotational speeds and filling degrees. They used a mixing index based on number fractions of a given class of beads in control cells, which was in fact identical to the segregation index (or intensity) introduced before by Vargas [4]. They defined a mixing time which was the time when the mixing index reached its plateau. There was no significant change in final mixing index at different operating conditions. The mixing time was found to decrease with increasing rotational speed and decreasing filling degree. However, the ratio of the area of the active zone to the total bed transverse area showed exactly the opposite trend. Consequently, the mixing time was found to decrease with increasing active zone area, which is in contradiction with the suggestions made by Félix [2].

Liao and al [5] experimentally studied segregation induced by density in a thin rotating drum for high filling ratios. For a high filling ratio beyond 0.5 they observed a very picturesque segregation pattern in the drum transverse plane, the streak pattern, with streaks of the densier beads starting from the center of the drum and enlarging towards the periphery. The angular distance between two consecutive streaks increases considerably with the density ratio of the particles.

The obvious way to reduce segregation seems to be the insertion of baffles (fins) inside the rotary drum. However, the location and design of the baffles is an issue far from being simple. Vargas et al. [4] studied the bi-disperse (size or density) granular materials in a quasi-two-dimensional (2-D) rotating drum by means of both simulations and experiments. Two basic baffle configurations were considered: central (axial) placement and peripheral placement. A segregation intensity parameter based on local number fractions of a given particle class was defined in order to quantify globally the mixing quality. This segregation index was the standard deviation of local bed compositions with respect to theoretical (perfectly mixed bed) average composition. Several baffles attached at the periphery, unless very long, were ineffective in reducing segregation, while even one flat baffle located axially induced a huge drop of both density and size induced segregation intensity. These results were interpreted by the fact that axial baffles introduced periodic flow inversions within the active (shear) layer. This effect was called “flow modulation” by the authors. According to this interpretation, the peripheral baffles could have an effect only when their radial size was large enough to cross the active region during mixing. That means that the length of a radial peripheral baffle should be at least adapted to the bed height and thus the filling ratio.

The investigations of Vargas et al. [4] were continued by Bhattacharya et al. [6]. In this new study, still combining DEM simulation and experiments with a 2-D drum, the start point was that axially located baffles can drastically reduce segregation and different novel baffle designs were considered: C-shaped, S-shaped, reverse S-shaped and flat baffle. The S-shaped one was proven to be the most efficient in reducing segregation, but the exact location of the baffle was shown to be also of utmost importance. The S-shaped baffle had to be placed near the free surface within the shear layer (active zone) of the bed. Moreover, the authors confirmed that an unbaffled tumbler has a comparable or better mixing (anti-segregation) performance than a tumbler with baffles attached radially to the wall, what is still a common practice in industry.

Maione et al. [7] used also DEM modeling and simulation to study the segregation of dispersed beds during rotary mixing. The originality of their work was to consider a mixture of two types of real life particles with different shapes: steel balls and wood chips. The non-spherical particles were represented by multisphere templates or by equivalent size spheres. The number and type of baffles was constant. The drum mixer had a considerable axial length (3-D kind) and the segregation index (same as with Vargas [4]) was evaluated all over the length. It may thus be supposed that the friction effects on the drum ends had a negligible impact on global segregation rate and final level. For this particular material and drum geometry, the drum equipped with baffles exhibited less segregation intensity than the drum without baffles.

Very recently, Zhang et al. [8] investigated the mixing and heat transfer process of particulate solid in an externally heated rotary kiln by using EDEM software with user-defined routines. Instead of a segregation index based on number fraction of a given bead class in arbitrary chosen control volumes, used by all the authors cited above, they used a new mixing index based on a number of contacts between the beads. At a given mixing time, this index was defined as the number of contacts between beads of the two different classes divided by the total number of contacts (including the contacts between beads of the same class). This definition is very suitable for DEM simulations because all contacts are inherently counted by the software. The higher the mixing index, the better the mixing quality (degree). The number and the physical parameters of the two classes of particles were exactly the same and there were no baffles inside the drum. They showed that after just three revolutions, the mixing index leveled off at the same value for all operating conditions. The index rise was considerably faster for high revolutions speeds and low filling ratios. These results corroborated the previously published ones, pointing out the occurrence of a plateau (asymptotic value) for the mixing or segregation indexes. It must be remembered however, that this plateau value depends strongly on bed composition (beads population dispersion) and the baffles' configuration.

The aim of this paper was to show the influence of the baffles' configuration and drum filling ratio on the segregation intensity of bi-dispersed spherical milli-beads during rotary mixing. The analysis was based on lab-scale experiments and DEM simulations of flow in a rotating 'slice' type (nearly bi-dimensional) drum. The DEM model was validated on the basis of the bed front layer segregation index and then applied to investigate the segregation intensity all along the depth of the bed. In this way, it was possible to determine to what extent the frontal segregation index is informative about the segregation intensity in the entire bed.

As our goal was to stick as much as possible to industrial practice, peripheral (radial) straight baffles were considered. This kind of baffles does not provide a good mixing efficiency but provide a big extension of the internal drum wall surface which is important in cooling or heating applications. The drum was very shallow ('slice' form), in order to limit its volume and keep the number of particles reasonable in regard to the simulation time. The drawback of this choice was the results bias due the friction at the front and rear ends of the drum. The corollary result was that friction was very influent.

In a parallel work by the same authors, the mechanical DEM model developed and validated in this study was coupled with a thermal DEM model providing temperature distributions within bi-dispersed beds during mixing and contact heating. The impact of mechanical segregation on thermal homogeneity of the bed was analyzed and the results have been already published [9].

## 2. Materials and Methods

### 2.1. Experimental Device and Operating Conditions

The drum used in this study was a ‘slice’ type one with a large diameter to depth ratio (close to 7), with internal diameter of 300 mm and an internal depth of 42 mm. The depth was intended to be small for bead quantity saving and thus simulation time saving. It was finally fixed at around 20 times the beads mean diameter in order to avoid the curvature of the free surface of the material in the axial plane induced by the front and rear panels, as recommended in the literature [2].

The front panel was made of glass, the rear one and the circular peripheral wall were made of stainless steel. Five baffles configurations were considered: a drum with no baffles, a drum equipped with 4 straight equidistant baffles with a height of 15 or 40 mm and a drum with 8 straight equidistant baffles of 15 or 40 mm. The default revolution speed was 3 rpm which corresponded to very slow rotation speeds often encountered for the industrial drying processes of pharmaceuticals. The rotary drum Froude number was equal to  $1.5 \times 10^{-3}$  corresponding to a ‘rolling’ flow mode according to the literature [1]. This mode was described in the ‘Introduction’ section. The drum was filled with material to 1/8, 1/6 and 1/4 of the total volume, which corresponded to low product loads used in the industry for pharmaceutical materials.

In order to generate bi-dispersed particulate populations, the drum was filled half by half in volume with the two different classes of beads: two different sizes (bi-size bed) or two different densities (bi-density bed). The studied (experimentally or numerically) beds were composed of polypropylene (PP) spherical beads with 3 mm diameter (specifications according to Marteau and Lemarié, Sorbiers, France) and cellulose acetate (CA) spherical beads with 2 mm and 3 mm diameters (specifications according to CIMAP, Paris, France). Each class (size or density) of the beads had a different color. The quantity (number) of beads that were used for preparing the two types of bed and the three drum filling ratios are given in Table 1. For the purpose of experiments, the two classes of beads were loaded into two adjacent separate compartments and the vertical separating wall was removed just before the mixing started. For the purpose of simulations, the two classes were initially virtually perfectly mixed by the software.

**Table 1.** Bed compositions for the experimental and simulated trials.

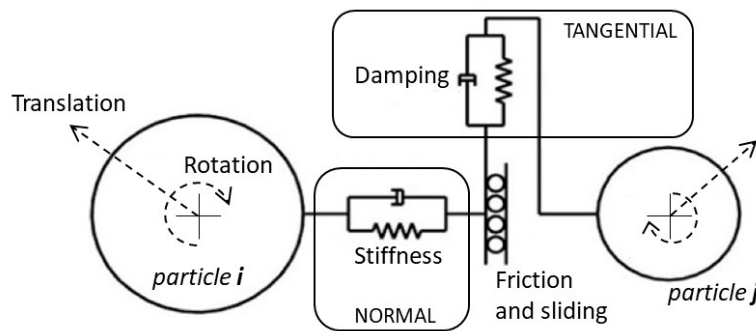
|                    | Bi-Size Bed |        |        | Bi-Density Bed |        |        |
|--------------------|-------------|--------|--------|----------------|--------|--------|
| Drum filling ratio | 1/4         | 1/6    | 1/8    | 1/4            | 1/6    | 1/8    |
| Beads CA 3 mm      | 16,388      | 10,847 | 8183   | -              | -      | -      |
| Beads CA 2 mm      | 55,884      | 37,332 | 27,905 | 55,884         | 37,332 | 27,905 |
| Beads PP 2 mm      | -           | -      | -      | 57,381         | 36,328 | 27,154 |
| Total              | 72,272      | 48,179 | 36,088 | 113,265        | 73,660 | 55,059 |

The photographs of the frontal face of the drum were taken with a digital single lens reflex camera (Nikon D500), equipped with fixed focal distance lens (Nikon 50 mm f/1.4 AF-S) well suited for high quality portrait-style photography. The shootings were made at a distance of 1 m, with additional fluorescent tube lighting from beneath, with lens aperture at f/2.5 and camera sensibility at 8000 Iso. The digital pictures were processed with Image-J software, with several processing steps: color thresholding, grey scale conversion, contour sharpening, targeted size detection, bead center points positioning. The bead coordinates were exported to MATLAB for bead counting in each mesh of the grid and calculation of segregation indexes.

### 2.2. Mechanical DEM Model

The simulations of milli-bead mixing were realized with the commercial software EDEM 2017 (DEM Solutions, Edinburgh, UK). This software is based on the discrete elements method (DEM) which is a very powerful tool used more and more nowadays to investigate and develop granular solid

processes. In the DEM framework, each particle of the granular bed is considered to be distinct and has its own trajectory and speed. The particle–particle and particle–boundary interactions are checked at each time step and the resulting individual particle positions are updated. The main assumption of DEM modeling is that the particles are rigid (non-deformable) solid bodies but have the capability of small interpenetration during their contacts. The resulting decrease in the distance between the centers of two adjoining particles is called the interpenetration depth ( $\delta_{ij}$ ) or the overlap which is shown in Figure 1.



**Figure 1.** Schematic representation of the mechanics of the contact between two beads.

The determination of the position and the speed of a given particle (identified by index  $i$ ) is the time solution of the Newton second law for translational and rotational movement of a solid body. For translation, the dynamics of a single particle is then expressed as [3,7,10]:

$$m_i \frac{d^2 \vec{x}_i}{dt^2} = \sum_j F_{ij} + m_i g \quad (1)$$

where  $m_i$  is the particle mass,  $x_i$  its position vector and the right hand side of the equation is the resulting force, cumulating interactions with all adjoining ( $j$ ) particles (and the wall of the vessel) and gravity (see Figure 1). A similar equation can be written for the rotational movement.

For the purpose of this study, the classical Hertz–Mindlin contact model was used [3,7,10], which is the default modeling option in EDEM software. In this model, the particle–particle mechanical interaction involves the normal impact force and the tangential friction force. This normal force is essentially elastic but both normal and tangential forces include damping components. The normal force is thus expressed by means of the non-linear visco-elastic behavior law:

$$F_{ij}^n = \alpha \delta_{ij}^{3/2} - \beta (v_i - v_j) \quad (2)$$

where  $\alpha$  is the elasticity or ‘stiffness’ coefficient depending theoretically on the material mechanical properties (essentially the Young’s modulus) and particles radius,  $\beta$  is the damping coefficient depending on the empirical restitution coefficient of the particles (ratio of relative velocity of two particles after collision to their relative velocity after collision),  $\delta_{ij}$  is the normal overlap between particle  $i$  and  $j$ , and  $v$  is the velocity of the particle. A similar equation can be written for the tangential force, based on a hypothetical tangential overlap. However, the tangential force has an upper physical limit due to the Coulomb’s law of friction which involves a material dependent friction coefficient:

$$F_{ijmax}^t = f F_{ij}^n \quad (3)$$

The overlap between two adjoining particles is a key variable because it determines the interaction mechanical force (Equation (2)). While performing a DEM simulation this variable must be controlled. The particle ‘stiffness’  $\alpha$  is in fact a user-defined simulation parameter. Its value is adjusted in order to

limit the interpenetration depths to a desired fraction of the particle diameter, generally not more than 0.01, and at the same time to keep the minimal calculation time step as high as possible.

### 2.3. Numerical Solver Settings and Material Properties

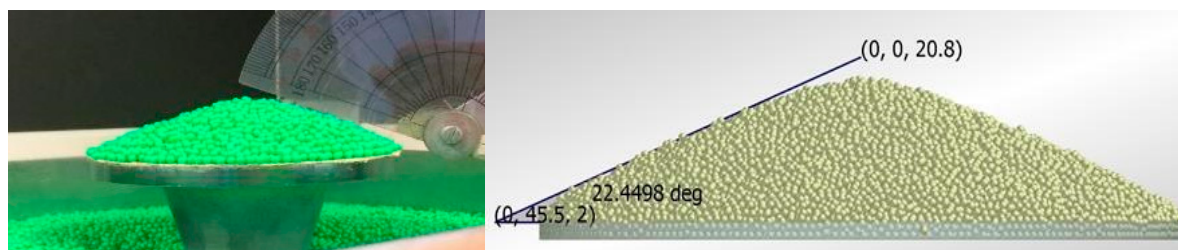
The simulations were realized on a DELL workstation (T7910) with two 10 cores processors (Intel Xeon E5-2660v3). The maximum processing time step was set at 40% of the theoretical Rayleigh time step. This is the time taken for a shear wave to propagate through the particle which depends on the particle size and on its mechanical properties. The adjoining particles numerical search distance was set at 5-fold particle radius. The standard “Hertz–Mindlin” particle–particle contact model was selected. All materials mechanical and thermophysical properties needed for EDEM simulations are given in Table 2.

**Table 2.** Materials mechanical properties.

| Beads | Property                                     | Units                | CA    | PP    |
|-------|----------------------------------------------|----------------------|-------|-------|
|       | Density <sup>a</sup>                         | (kg/m <sup>3</sup> ) | 1280  | 910   |
|       | Poisson's ratio <sup>a</sup>                 | (-)                  | 0.4   | 0.42  |
|       | Elastic modulus *                            | (MPa)                | 2.8   | 2.84  |
|       | Shear modulus *                              | (MPa)                | 1     | 1     |
|       | Coefficient of rolling friction <sup>c</sup> | (-)                  | 0.01  | 0.01  |
|       | Coefficient of restitution <sup>c</sup>      | (-)                  | 0.3   | 0.3   |
| Drum  |                                              |                      | Steel | Glass |
|       | Density <sup>b</sup>                         | (kg/m <sup>3</sup> ) | 7800  | 2500  |
|       | Poisson's ratio <sup>b</sup>                 | (-)                  | 0.3   | 0.21  |
|       | Elastic modulus <sup>b</sup>                 | (GPa)                | 182   | 94.3  |
|       | Shear modulus <sup>b</sup>                   | (GPa)                | 70    | 39    |
|       | Coefficient of rolling friction <sup>c</sup> | (-)                  | 0     | 0.01  |
|       | Coefficient of restitution <sup>c</sup>      | (-)                  | 0.3   | 0.3   |

<sup>a</sup> manufacturers data, <sup>b</sup> free access web data: <https://www.engineeringtoolbox.com/>, <sup>c</sup> data from this study, \* user defined value for speeding up the simulations.

A DEM model is extremely sensitive to static friction coefficients among the particles and between the particles and the wall [10]. In this study, the particle–particle friction coefficients were obtained by model identification on the basis of specific experiments. The natural slope experiments were performed for each kind of bead (CA and PP) by pouring the beads vertically and very gently on a horizontal flat plate. The static angle of repose was measured. Exactly the same procedure was modeled and simulated by the EDEM software and the particle–particle friction coefficient was adjusted in order to match the experimental angle. The experimental and simulated piles of beads are presented in Figure 2.



**Figure 2.** Natural slope forming: experimental set-up on the left and simulated equivalent on the right.

The particle–wall friction coefficients were also obtained by model identification but not with an additional specific experiment but using some of the mixing trials performed to study the segregation phenomenon (see Section 2.1). In fact, the trials without baffles ( $N = 0$ ) were used for parameter

identification and consequently were not used afterward for model validation. The front layer segregation index was first determined from photographs and then calculated from the EDEM simulated data for the same configuration. The friction particle–wall friction coefficients were adjusted in order to get the same front layer segregation index from experiment and simulation. Two other important but hardly known parameters, the coefficient of rolling friction and the coefficient of restitution, were obtained in the same way. It must be acknowledged that it would be more reliable to obtain these parameters independently of each other and directly. Hlosta et al. [11] proposed a methodology where each of the critical mechanical DEM model parameters was determined by a specific experiment with an original apparatus.

The front layer segregation index was first determined from photographs for a reference configuration and then calculated from the EDEM simulated data for the same configuration. The model coefficients were adjusted in order to match the experimental values. The friction coefficient values for pairs of materials obtained by experimental data fitting in this study are given in Table 3.

**Table 3.** Solid–solid static friction coefficients.

|    | Bead-Steel | Bead-Glass | Bead-Bead |     |
|----|------------|------------|-----------|-----|
|    |            |            | CA        | PP  |
| CA | 0.3        | 0.2        | 0.3       | 0.6 |
| PP | 0.3        | 0.2        | 0.6       | 0.6 |

Despite using a rather powerful work station and modulating the elastic material properties, the simulation time was prohibitive, as is often true in DEM studies. For this reason, only a limited number of simulations were performed, and the applied mixing times were limited. The simulation duration depended of course mostly on the total number of beads in the drum. For a drum filled to 1/8 of its volume half by half with 2 mm and 3 mm beads, the 2 min mixing process was simulated over 2 days. For a drum filled to 1/4 with only 2 mm beads, the 2 min mixing process was simulated over one week.

#### 2.4. Particulate Bed Characteristics

In order to characterize macroscopically the geometrical distribution (mechanical segregation) of each class of beads within the bed, a mechanical segregation index already introduced in the literature was used [3,4,7]:

$$SI = \sqrt{\frac{1}{K-1} \sum_{i=1}^k (C_i - \langle C \rangle)^2} \quad (4)$$

where  $C_i$  was the fraction by number of one class of beads (of the considered size or density) in a control volume  $i$  among  $k$  other control volumes,  $\langle C \rangle$  was the average fraction by number of this kind of bead in the entire bed divided into  $k$  equal size control volumes ( $k = 400$  in this study). For both of the bi-dispersed beds, the number fraction  $C$  was that of the 2 mm AC beads. The control volumes were defined by a  $20 \times 20$  rectangular grid superimposed over the frame containing the drum front layer.

Two kind of segregation indexes were calculated: the global one (GSI) where the control volume extended over the entire bed depth (42 mm) and the local one where the control volume covered only the thickness of one layer of beads (3 mm). This local index was evaluated for the front layer and rear layer of the bed, but the front layer segregation index (FLSI) was mainly analyzed and discussed. The minimum SI value is 0 for a perfectly mixed bed. Its maximum value depends on how the perfectly segregated bed is defined. In the case of a single layer, if there are no smaller or denser beads at all in the considered layer, the FLSI will reach the bed overall fraction of this class of beads, for instance 0.77 for the bi-size bed. In the case of the entire bed, if half of the control volumes contain only smaller (or

denser) beads and the other half contain only the bigger (or less dense) beads, the GSI will reach 0.57 for the bi-size bed and 0.5 for the bi-density bed.

Concerning specifically the front layer of the bed, the following characteristics were also determined:

- the dynamic angle of repose (DAR) which is the angle in the radial section of the bed formed by the line representing the free surface of the bed and the horizontal line,
- the number fraction of smaller or denser beads (FSB) which is simply the number of the considered beads divided by the total number in the front layer.

### 3. Results and Discussion

#### 3.1. Bed Front Layer Experimental Observations and Comparison with Simulations

##### 3.1.1. Bi-Size Bed

The experimental photograph and the simulated picture of the front panel of the rotary drum after 2 min (six revolutions) of mixing are shown in Figure 3. The distribution of the two classes of beads in the first layer of the bed can be observed thanks to the colors. There is obviously a segregation between the small (orange) and large beads (black). The small (orange) beads are grouped in the core of the bed, and the large ones are spread on the periphery.



**Figure 3.** Simulated and experimental images of the front panel of the rotary drum showing the distribution of the beads in the first layer of the bi-size bed after 2 min (6 revolutions) of mixing. The drum is filled at  $\frac{1}{4}$  of its volume and equipped with 8 baffles of 4 cm length.

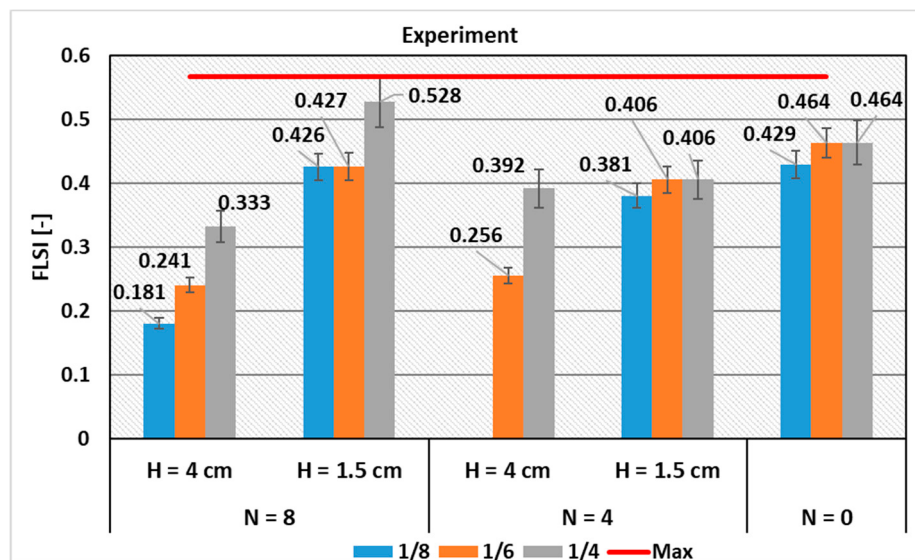
Qualitatively, the simulated picture corresponds well to the experimental one. Quantitative bed characteristics, the dynamic angle of repose (DAR) and the number fraction of the small beads (FSB) in the front layer are given in Table 4. The experimental and simulated values agree rather well as concerns DAR but agree rather poorly concerning FSB for the considered mixing conditions. The same discrepancies were found for other filling ratios and baffles configurations (results not shown).

**Table 4.** Characteristics of the front layer of the bed after 2 min (6 revolutions) of mixing (drum filled at  $\frac{1}{4}$ , 4SB: 4 baffles of 1.5 cm, 8LB: 8 baffles of 4 cm).

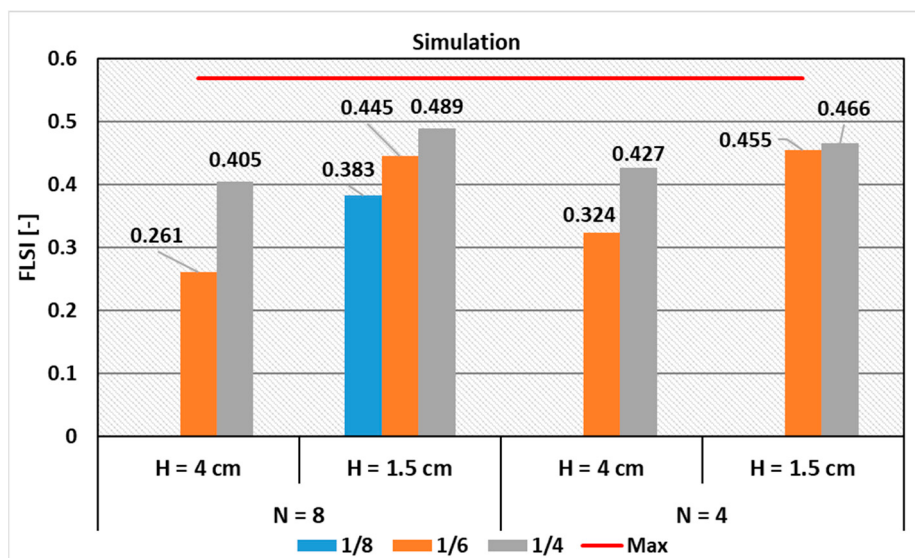
|            | FSB (%) |     | DAR (deg) |      |
|------------|---------|-----|-----------|------|
|            | 4SB     | 8LB | 4SB       | 8LB  |
| Experiment | 65      | 63  | 29.8      | 24.7 |
| Simulation | 78      | 80  | 26.8      | 26.4 |

Mixing experiments and simulations were performed for two bed types and five baffles configurations and the frontal layer mechanical segregation index (FLSI) was determined for each

trial. The experimental results for the bi-size bed are presented in Figure 4 and the simulated ones in Figure 5.



**Figure 4.** The front layer segregation index (FLSI) from experiments for the bi-size bed and for different drum filling ratios and different baffles heights (H) and numbers (N).



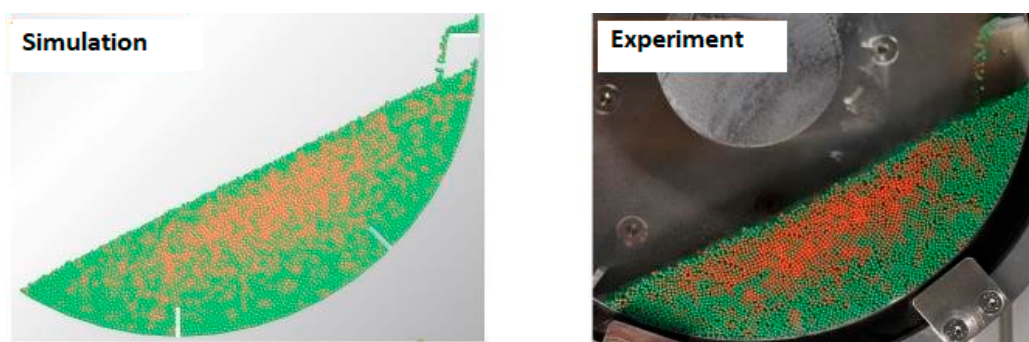
**Figure 5.** The front layer segregation index (FLSI) from simulations for bi-size bed and different baffles configurations.

The simulated FLSI are generally slightly higher than the experimental ones. However, the difference between experiment and computation always lies within the uncertainty interval of the experimental results. Thus, the DEM simulation correctly represents the empirical data. It must be emphasized that this acceptable agreement was obtained after identification of friction coefficients in the DEM model as described in the ‘Methods’ section. According to the Figures, generally the FLSI decreases when increasing the number and the height of the baffles and when decreasing drum filling ratio. However, a noticeable reduction of segregation was obtained only for the highest baffles. The height of the baffles seems to be more influential than their numbers. These trends confirm those from the literature [4,7].

Experiments were also realized on a bigger (600 mm internal diameter) drum. It was equipped with 0 or 6 or 12 straight equidistant baffles with a height of 25 or 50 mm. The revolution speed was 3 rpm. The drum was filled with material to 1/6, 1/8 and 1/10 of the total volume. The front layer segregation index was evaluated for every tested baffles configuration and filling ratio. In case of the bi-size bed, the same general trends were obtained as for a smaller (300 mm internal diameter) drum. The segregation was considerably refrained only for the highest baffles, practically independently of their number.

### 3.1.2. Bi-Density Bed

The real life photograph and the simulated picture of the front panel of the rotary drum after 2 min (six revolutions) of mixing are shown in Figure 6. There is obviously a segregation between the heavy (orange) and light beads (green). The heavy (denser) beads are grouped in the core of the bed, and the light ones are evacuated to the periphery. However, in comparison with the bi-size bed, the orange core is more diffused, and the segregation seems less pronounced. As with bi-size bed, the simulated picture corresponds qualitatively well to the experimental one.

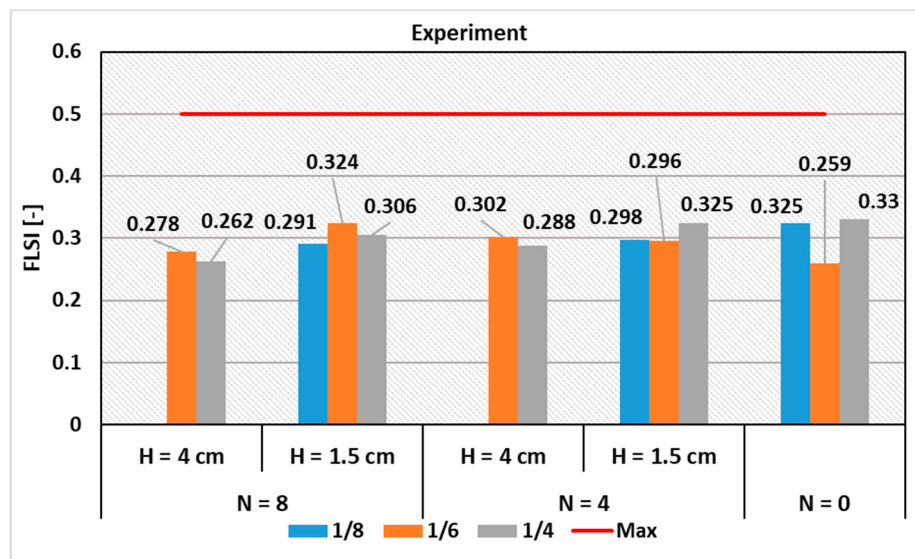


**Figure 6.** Simulated and experimental images of the front panel of the rotary drum showing the distribution of the beads in the first layer of the bi-density bed after 2 min (6 revolutions) of mixing. The drum is filled at 1/6 of its volume and equipped with 8 baffles of 1.5 cm height.

Mixing experiments were performed for two bed types and five baffles configurations and the frontal layer mechanical segregation index (FLSI) was determined for each trial. The experimental results for the bi-size bed are presented in Figure 7.

The simulations were performed for only one baffles' configuration: eight baffles 1.5 cm high. For all drum filling ratios, the FLSI was practically the same, equal to 0.3, which corresponded very well with the experimental results. According to Figure 7, the filling ratio, the number and height of the baffles have all a minor influence on the frontal segregation for the bi-density bed, having much less influence than for the bi-size bed. It is not possible to draw any trend from these experimental data.

Concerning the experimental results for the bigger (600 mm) drum in the case of bi-density bed, the influence of operating conditions on the segregation intensity was very weak as with the smaller drum. The unbaffled configuration presented however a lower segregation index among all. That leads to the conclusion that segregation due to a bi-density population is more difficult to tackle. It is also much less described in the literature.



**Figure 7.** The front layer segregation index (FLSI) from experiments for the bi-density bed and for different drum filling ratios and baffles configurations.

### 3.2. Full Bed Depth Analysis by Simulations

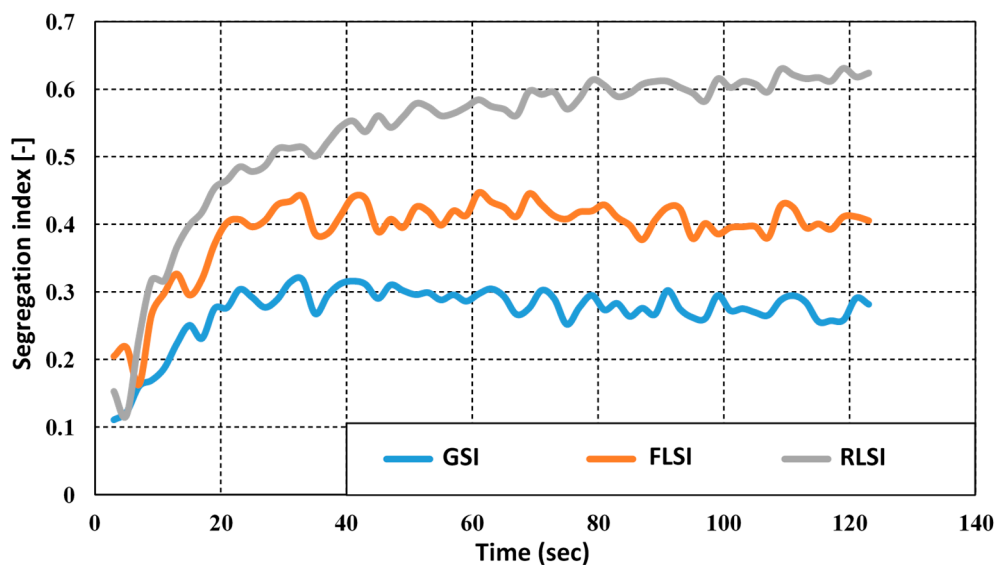
The experiments provided only the beads distributions in the front layer of the bed, at the front panel of the drum made of glass. In order to know what happens behind, only simulations can help. The simulated distributions of the beads in a bi-size bed in the front, center and rear layer of the bed are presented in Figure 8.



**Figure 8.** The simulated beads distributions after 2 min (6 revolutions) of mixing for the bi-size bed (drum filled at  $\frac{1}{4}$ , 4 baffles of 1.5 cm), from the left to right: front layer, center layer, rear layer.

In the case of bi-size bed, the bigger (black) beads accumulate at both drum ends but preferentially on the rear panel. At the center of the drum, the smaller (orange) ones are largely predominant. A kind of axial bead segregation developed due to friction effects at drum ends. In order to quantify this friction effects, the single layer segregation index was calculated at the rear (steel) panel and the front (glass) panel and compared to the global segregation index (GSI) in Figure 9.

According to Figure 9 and corroborating the qualitative results of Figure 8, the segregation index is the highest at the rear (steel) panel, which has a higher friction coefficient than the front (glass) one (see also Table 5). Moreover, both frontal and rear layer indexes are higher than the global one.



**Figure 9.** The simulated segregation indexes: global (GSI), front layer (FLSI) and rear layer (RLSI) versus mixing time for the bi-size bed (drum filled at  $\frac{1}{4}$ , 8 baffles of 4 cm).

**Table 5.** Global and front layer segregation indexes after 2 min (6 revolutions) of mixing (drum filled at  $\frac{1}{8}$ , 0B: no baffles, 4SB: 4 baffles of 1.5 cm, 8LB: 8 baffles of 4 cm).

|            | GSI     |            | FLSI    |            |
|------------|---------|------------|---------|------------|
|            | Bi-Size | Bi-Density | Bi-Size | Bi-Density |
| <b>0B</b>  | 0.181   | 0.122      | 0.429   | 0.325      |
| <b>4SB</b> | 0.214   | 0.151      | 0.381   | 0.298      |
| <b>8LB</b> | 0.166   | 0.146      | 0.181   | -          |

The global segregation index (GSI) was calculated for the two bi-dispersed beds and for different baffles configurations. These GSI values are given in Table 5, together with the values of the front layer index (FLSI). As already shown in Figure 7, the GSI is significantly lower than the FLSI as it integrates the bead distribution variation over the entire bed depth. Moreover, the GSI variation with baffles number and height are quite different to that of the FLSI, in a slightly surprising way. In the case of the bi-size bed, the configuration with eight long baffles made the mixing of the two bead classes better, with less segregation, but the configuration with four short baffles made the mixing worse, with more segregation than without any baffles. For the bi-density bed, the results are even stranger, because for all baffles configurations, the segregation is stronger than without baffles.

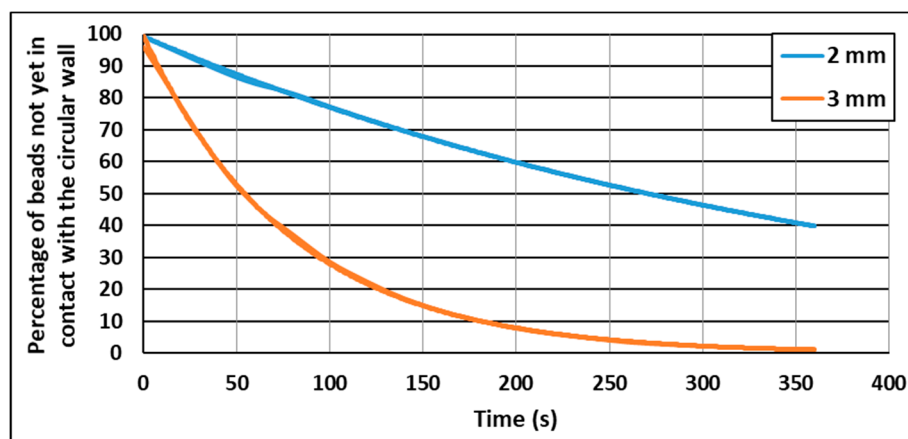
Our findings are nevertheless supported by published results. Bhattacharya [6] claimed that a drum with peripheral (attached to the drum wall) baffles may induce more segregation than an unbaffled one. Vargas [4] argued that the peripheral baffles could be effective for segregation reduction only if their length allows them to interfere with the active zone of the bed. Both of these publications recommend a single baffle placed centrally at the level of the active zone of the bed. Jiang [12] studied only centrally (axially) placed baffles of three different geometrical (single, cross and star) configurations and demonstrated by DEM simulations that baffles with multiple arms are more efficient than a single baffle and that an optimal arm length exists.

Furthermore, the influence of the bead friction at the frontal (glass) and rear (steel) drum panels appears paramount. In the case of a long drum, the global segregation index would be much less sensitive to the friction effects at the drum end panels and the observed trends could be different, as reported by Maione [7]. In case of a very short ‘slice’ type drum, the segregation index at the front layer does not seem to sufficiently express the segregation intensity in the entire bed.

### 3.3. Introductory Analysis of Contact Thermal Processing of Bi-Dispersed Beds

In rotary contact heating or cooling equipment, it is not the mixing quality i.e., compositional homogeneity of the bed, that essentially matters but the frequency and duration of contacts of the particles with the peripheral wall. Especially, in order to obtain the same thermal treatment for each particle, every particle should at least spend a similar cumulated time on the wall during the process. In bi-dispersed beds, due to mechanical segregation phenomena, one class of particles (size or density class) could transit preferentially on the wall compared to the second class. That would induce preferential and thus quicker heating or cooling of this class, and should be avoided.

In order to have a first insight into this complicated issue, the frequency of bead contacts with the peripheral circular drum wall was analyzed. The time evolution of the number of beads which have not yet been in contact with the wall from the beginning of the process is presented in Figure 10. According to this figure, the proportion of the smaller beads that have not been in contact with the wall decreased slowly and linearly with time, while the portion of bigger beads decreased quickly and exponentially. After quite a long time (6 min, 36 revolutions), there was still a considerable amount of small beads that had not made contact with the wall, while all the bigger beads had already made contact.



**Figure 10.** The simulated percentage of beads that had not yet been in contact with the peripheral wall versus mixing time for the bi-size bed (drum filled at 1/3, 4 baffles of 4 cm, rotation 6 rpm).

These results are consistent with those concerning the segregation phenomenon. In the case of the bi-size bed, the small beads accumulate in the core of the bed, while the big ones move to the periphery. The big beads are predominantly in the active zone, are carried by the main stream and are returned again and again to the wall, while the small one are trapped into the bed core which rotates by itself. If the peripheral circular wall is heated, small beads will remain cold longer and the bigger ones will become hot faster. The influence of mechanical segregation on the thermal dispersion of beads during rotary contact heating was analyzed thoroughly in a previous paper by the same authors [9].

## 4. Conclusions and Prospects

The rotary mixing of bi-dispersed (binary composition) beds was considered and the impact of the baffles' configuration and drum filling ratio on segregation intensity was investigated. The distribution of particles in the front layer was analyzed both numerically and experimentally. A local segregation index, defined for the frontal layer of the bed, was extracted from the data. Good agreement between the simulated and experimental results was achieved by adjusting the friction coefficients at the front and rear walls of the drum. In the case of the bi-size bed, the segregation intensity was considerably diminished only for the highest baffles, practically independently of their number. For the bi-density bed, the number and height of the baffles had little influence on the frontal segregation index.

The global (defined over the entire bed) segregation index was also calculated from simulation data for all considered cases. The first layer values were compared to the global ones and dissimilar

trends were observed. In case of bi-size bed, the global segregation intensity for the configuration with four short baffles was stronger than without any baffles. In the case of the bi-density bed, the global segregation index was higher for all baffles' configurations than for the one without baffles. These results were consistent with already published ones. Our study confirmed that short straight peripheral baffles were ineffective in rotary drum mixing. As a minimum, their length has to be correlated to the filling ratio in order to be long enough to cross the entire bed and reach the active zone.

For our geometrical configuration based on a 'slice' type drum, the friction at both drum ends was shown to strongly influence the segregation intensity profile along the drum. A serious doubt has arisen whether the frontal segregation index, which is the most easily accessible for direct observation, may represent the global bed behavior. In further experimental investigation, the depth of the drum should be substantially increased or the end panels should be made frictionless. Numerically, periodic boundary conditions creating a virtual infinite (periodic) cylinder could also be used in the axial direction of the drum in order to get rid of the wall-friction issue.

In our work, the size and density induced segregation was analyzed separately. A study of a poly-dispersed particulate system in which particle size and density dispersion coexist could be a valuable continuation since this kind of system is much closer to industrial practice.

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## References

1. Boateng, A.A.; Barr, P.V. Modelling of particle mixing and segregation in the transverse plane of a rotary kiln. *Chem. Eng. Sci.* **1996**, *51*, 4167–4181. [\[CrossRef\]](#)
2. Félix, G.; Falk, V.; D'Ortona, U. Segregation of dry granular material in rotating drum: Experimental study of the flowing zone thickness. *Powder Technol.* **2002**, *128*, 314–319. [\[CrossRef\]](#)
3. Xiao, X.; Tan, Y.; Zhang, H.; Deng, R.; Jiang, S. Experimental and DEM studies on the particle mixing performance in rotating drums: Effect of area ratio. *Powder Technol.* **2017**, *314*, 182–194. [\[CrossRef\]](#)
4. Vargas, W.L.; Hajra, S.K.; Shi, D.; McCarthy, J.J. Suppressing the Segregation of Granular Mixtures in Rotating Tumblers. *AIChE J.* **2008**, *54*, 3124–3132. [\[CrossRef\]](#)
5. Liao, C.C.; Hsiau, S.S.; Nien, H.C. Effects of density ratio, rotation speed, and fill level on density-induced granular streak segregation in a rotating drum. *Powder Technol.* **2015**, *284*, 514–520. [\[CrossRef\]](#)
6. Bhattacharya, T.; Hajra, S.K.; McCarthy, J.J. A design heuristic for optimizing segregation avoidance practices in horizontal drum mixers. *Powder Technol.* **2014**, *253*, 107–115. [\[CrossRef\]](#)
7. Maione, R.; De Richter, S.K.; Mauviel, G.; Wild, G. DEM investigation of granular flow and binary mixture segregation in a rotating tumbler: Influence of particle shape and internal baffles. *Powder Technol.* **2015**, *286*, 732–739. [\[CrossRef\]](#)
8. Zhang, Z.; Liu, Y.; Zhao, X.; Xiao, Y.; Lei, X. Mixing and Heat Transfer of Granular Materials in an Externally Heated Rotary Kiln. *Chem. Eng. Technol.* **2019**, *42*, 987–995. [\[CrossRef\]](#)
9. Mesnier, A.; Rouabah, M.; Cogné, C.; Peczkalski, R.; Vessot-Crastes, S.; Vacus, P.; Andrieu, J. Contact heating of bi-dispersed milli-beads in a rotary drum. Mechanical segregation impact on temperature distribution and on heating kinetic analyzed by DEM simulation. *Powder Technol.* **2019**, *354*, 240–246. [\[CrossRef\]](#)
10. Yan, Z.; Wilkinson, S.K.; Stitt, E.H.; Marigo, M. Discrete element modelling (DEM) input parameters: Understanding their impact on model predictions using statistical analysis. *Comp. Part. Mech.* **2015**, *2*, 283–289. [\[CrossRef\]](#)

11. Hlosta, J.; Jezerská, L.; Rozbroj, J.; Žurovec, D.; Nečas, J.; Zegzulka, J. DEM Investigation of the Influence of Particulate Properties and Operating Conditions on the Mixing Process in Rotary Drums: Part 1—Determination of the DEM Parameters and Calibration Process. *Processes* **2020**, *8*, 222. [[CrossRef](#)]
12. Jiang, M.; Zhao, Y.; Liu, G.; Zheng, J. Enhancing mixing of particles by baffles in a rotating drum mixer. *Particuology* **2011**, *9*, 270–278. [[CrossRef](#)]



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