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Experimental and computational investigation of segregation during tumblers unloading

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ABSTRACT

Dry mixing is a common operation in many industrial processes. The unloading of the mixer is an essential aspect of the process which actively contributes to the final quality of the mixture. Its effect however is often underestimate or even ignored. In the present work the consequences of the unloading process on mixture quality were investigated in three different types of tumbling mixers: a symmetric double cone, an asymmetric double cone and a conic mixer were considered. The symmetric double cone mixer is a standard geometry in the pharmaceutical industry, while the other two geometries are unconventional. The effects of mixer geometry on the mixture quality were studied and related to differences in the unloading flow patterns. A segregating polydispersed mixture was used during the experiments in order to emphasize the different performances of the three tumblers. Additional quasi-2D experiments and FEM simulation of the unloading process clearly showed the ability of the unconventional geometries to mitigate the effect of segregation improving the mixture quality evaluated at the outlet of the mixer.

1. Introduction

The handling of granular mixtures composed of particles with different physical properties such as size, density or shape is known to cause segregation, i.e. the loss of mixture homogeneity. Segregation is a complex phenomenon that can occur even in industrial devices designed to increase the level of homogeneity between two or more components. Incorrect design of mixers or careless operating procedures may indeed promote ‘demixing’ of granular mixtures instead of improving their level of homogeneity [1].

Several equipments are available for mixing solids but it is possible to recognize two main classes: tumbling (or diffusive) mixers and agitated (or convective) mixers. Tumblers are constituted by a rotating hollow shell of various shapes (cylinder, double cone, cube, v-shaped) and partially filled with the granular material. The material moves subjected to centrifugal and gravitational forces and its homogenization is obtained through shear and dispersion (diffusion-like) mixing mechanisms [2]. These mixers are operated bacth-wise. In the agitated mixers, instead, the shell is stationary and the powder motion is caused by the action of one or more rotating devices (ribbons, screws, paddles, ploughshares). In these blenders the main mixing mechanisms are convection and shear [2]. They can be operated batch- or continuous-wise.

In some important industrial field, such as the pharmaceutical one, even if regulatory bodies are promoting modernization of manufacturing process to continuous pharmaceutical manufacturing, batch-wise powder mixing remains the rule in the industrial practice [3].

Among the above two mixers categories, tumblers are often plagued by segregation when they are used to process free-flowing powders. In the literature spectacular segregation patterns are reported for rotating drums (horizontal cylinders) and several mitigating strategies suggested [4,5,6]. When a binary mixture of particles differing only by size is tumbled in the rotating drum, finer particles tends to collect around the center of the granular bed and after few revolutions can form a segregated core (radial segregation) [7,8,9]. For sufficiently long tumblers mixture can also segregate forming several bands randomly distributed along the axis (axial segregation) [10,5].

In these mixers low axial mixing is given because of the geometry. For this reason other tumblers, able to provide higher level of axial mixing, are used in the industry: V-blenders, double cone and cubic mixers. In particular, among these, double cone mixer is one of the most common blenders used in pharmaceutical batch mixing operations [11].

The assessment of the quality of a mixture is usually performed by the analysis of a discrete quantity of material removed from the bulk mixture after blending and the composition of the samples is analyzed using statistical methods. A thief probe is the most common tool used to withdraw samples from the bulk mixture [12]. It has been demonstrated that sampling performed with a probe can generate sampling

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errors, for example because the internal structure of the mixture is destroyed by probe insertion or because of uneven flow of particles inside the sampler, mostly when the powder mixture is free-flowing [13,14]. Furthermore, the samples are withdrawn from a limited number of different locations in a static powder mixtures (typically at the end of the mixing process). This way of sampling however disagree with the "golden rules of powder sampling": (i) a powder should always be sampled when in motion; (ii) the whole of the stream of powder should be taken for many short increments of time in preference to part of the stream being taken for the whole of the time [15].

After the mixing process, powders undergo several further processing steps which almost surely include the discharge from the mixing bin through an orifice. This simple operation can affect the homogeneity and the final texture of the mixture [13]. From an industrial point of view, it is far more important what comes out of a bin, rather than how the contents are dispersed inside it [16]. The discharge operation of a container can be a prime source of segregation. Adams and Baker [17] studied the blending of dry materials in different equipments (double cone, ribbon, shell and rotating cube blenders) withdrawing samples from the outlet of the blenders. Sampling at the outlet can give information about the granular pattern achieved inside the bulk after the blending and give information about the composition of the mixture that enters in the next equipment of the process. In addition, the sampling of the material during unloading guarantees the respect of the above mentioned golden rules of Allen [15].

In this work the segregation associated to the unloading of a binary mixture from tumblers was studied. In particular we studied the changes of composition occurring at the outlet of three different types of mixers: a conventional symmetrical double cone (SDC) mixer, typically used in the pharmaceutical industry, and two modified mixers: a single cone (SC) mixer and an asymmetric double cone (ADC) mixer, both able to promote mass flow regime during the discharge. Information on the segregation patterns inside the mixers were inferred from the composition discharge profiles and from additional quasi-2D experiments and computer simulations. The flow of the powder mixture during tumblers unloading was conjugated to powder mechanics concepts in order to suggest improvements on the design of tumbler geometries able to mitigate segregation. Both experiments and simulations confirmed that an improvement of homogeneity was obtained by modifying the discharge flow patterns in the alternative tumbler geometries. In particular the asymmetric double cone geometry provided the best results in terms of mixture composition at the outlet of the mixer.

2. Materials and methods

2.1. Granular mixtures

The material used in the experiments was tetraacetylethilenediamine (TAED) powder; it is used in detergent industry as bleaching activator and is commercially available in different colors. In the present work, white and blue powders were used (Fig. 1), but red and green also exist. The TAED powder has a bulk density of 550 kg/m^3 and is commercially available with a broad particle size distribution (PSD), typically in the range between 100 and 2000 μm. This allowed to grade the material by sieving, in several size ranges and to recombine them with the desired proportions. The raw material was first subdivided in 5 classes: A = 400–500, B = 500–600, C = 600–700, D = 700–800 and E = 800–1000 μm, and then recombined according to the following proportions: A = 10.0%, B = 24.4% C = 33.4%, D = 14.2% and E = 18.0% by weight. In order to perform the segregation experiments, fraction A of withe TAED was replaced by the corresponding blue fraction so that the amount of blue TAED particles always represented the 10% of the mixture by weight. The final PSD obtained by recombination of white and blue TAED is shown in Fig. 2. This procedure was dictated by the need of working with a controlled PSD, simplified but representative of a real powder mixture. A powder flowability test, performed with a shear cell (PFT Powder Flow Tester, AMETEK Brookfield, USA), showed that the flow factor of the mixture, *ff*, was well above the value of 10 also at low consolidation stresses (Fig. 3), confirming that powder mixture was very free flowing and therefore with a strong propensity to segregate.

2.2. Mixers geometries

Three different tumbling mixers were used to perform the experiments: a symmetrical double cone (SDC) mixer, scaled-down from a real industrial mixer, a single cone (SC) and an asymmetrical double cone (ADC) mixer. The mixers were realized with sheets of transparent PVC. A picture of the three basic geometries is reported in Fig. 4 while mixers dimensions are detailed in Table 1. The SDC mixer was made of two identical truncated cones joined by a short cylinder. The SC mixer was a single truncated cone with steeper walls than the SDC; the choice of wall slope will be detailed in a following section. The ADC was a combination of the previous two geometries since the discharge cone had the same wall slope of the SC mixer while the upper cone wall slope was comparable to that of the SDC mixer. The discharge opening was the same for the three tumblers and equal to 25 mm.

Fig. 1. Blue and white TAED particles used in the experiments (size fraction 400-500 microns).