



Experimental evaluation of the effect of particle properties on the segregation of ternary powder mixtures

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ABSTRACT

Segregation of components, especially the low content level of a highly active substance, has serious deleterious effects on powder formulation. This study investigates the effect of particle properties, particularly size, shape, density and cohesion, on the segregation of laundry detergent powders. Tetraacetylenediamine (TAED) particles, spray-dried synthetic detergent powder referred to as Blown Powder (BP) and Enzyme Placebo Granules (EP granules) are used as a model formulation. The segregation of components is evaluated using image processing of the photographic records taken from the front face of a two-dimensional heap of powders. Coefficient of variation concept is considered as segregation index. It is shown that EP granules, the component used as low-level ingredient (< 2 wt%) in the ternary mixture, are prone to extensive segregation due to their higher density as compared to BP and TAED particles. Desired properties for segregation minimisation of EP granules have been further investigated. It is found that the segregation of EP granules can be reduced noticeably by applying a thin layer of a sticky liquid on them before mixing with the rest of powders with full particle size distribution of the components. Polyethylene glycol has been used for this purpose. Addition of 2.5 wt% gives an optimum level to reduce the segregation of EP granules without compromising the flowability of the mixture as well as EP granules themselves.

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1. Introduction

In many industrial sectors involved in manufacturing and handling of granular materials, such as pharmaceutical, food, fertiliser, mining and agriculture, segregation phenomenon is encountered particularly during handling, transportation and storage of the product. For example, variation of active pharmaceutical ingredient content (API) from tablet to tablet in pharmaceutical industry or taste variation in a drink powder mixture could have adverse impact on the product quality as well as costs of the production. Therefore, most powder formulations aim to achieve the most possible homogeneous products [1,2].

Material properties are important factors affecting the segregation of granular materials [3–5]. Among different material properties, the size and density of particles have been widely investigated [6–12]. Liao et al. [13] studied the granule segregation in a rotating drum. They found that increasing the particle density led to an increase in mixing time as dense particles gathered more into the centre of drum. Liu et al. [14] investigated the segregation of granular samples of microcrystalline cellulose and starch during blending in a cylindrical container. They concluded that larger starch granules had more tendencies to move to the top of the mixture. On the other hand, the smaller microcrystalline cellulose granules moved to the bottom of the mixture. In

another research carried out by Cho et al. [15], segregation of coloured glass beads was investigated in a double cone blender. They reported a better mixing of fines as compared to free-flowing large particles because of their cohesive nature arising from van der Waals forces.

Effects of shape and/or surface roughness of particles were also explored by other researchers [16,17]. Segregation of binary mixtures containing salt and other food seasoning powders was examined by Shenoy et al. [18] in a paddle mixer. They concluded that particle size, shape and density could influence the segregation of binary powder mixtures. They showed that differences in bulk density and shape had a greater effect on particle segregation than differences in particle size. Remy et al. [19] examined the effect of particle roughness of cohesionless glass beads on their kinematics in a bladed mixer by both experimental and numerical simulation methods, using particle image velocimetry and Discrete Element Method (DEM). Particles of various roughness with different friction coefficients were examined. They found that the amplitude of the velocity fluctuation of components increased as particle surface roughness was increased which led to the formation of less uniform flows inside of the mixer.

Important segregation mechanisms are trajectory, percolation, fluidisation, agglomeration and push-away effect [4,20]. In inclined chute flow, larger particles could travel further than smaller particles by the trajectory segregation. Based on the percolation mechanism, fines could find their way through the voids of coarse particles during handling, provided the void space between particles is large enough to

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admit the fine particles to pass. In fact in this mechanism, large particles could act as a screen letting the smaller particles to penetrate. When discharging from a height, dense particles could sink to the bottom while fine particles become airborne by fluidisation segregation. Agglomeration segregation mainly occurs when particles are cohesive (e.g. in the presence of moisture). Based on this mechanism, fine particles could form large clusters and move towards the periphery of the bulk of mixture. Dense particles behave as if they are small and could build up more in the centre of powder mixtures by a mechanism known as push-away effect.

By manipulating particle properties such as changing the particle size distribution and/or optimising the bulk cohesion, it is possible to reduce segregation [4,21,22]. Jain et al. [23] showed that the segregation of both binary and ternary mixtures of glass beads with different sizes could be reduced in vertically vibrated cylinders by slightly increasing the size distribution of each species or by increasing the mass fraction of the intermediate size specie in the ternary mixture. In another work done by Chou and Hsiau [24], the effect of wet granular material on the segregation reduction of particles in a rotating drum was investigated. They reported that at a higher liquid content, the segregation index could be reduced, presumably due to the formation of liquid bridge clumping the small particles forming larger particles. The formation of bigger particles could decrease the size ratio in the mixture and therefore could reduce the extent of particle segregation. Optimising the geometrical design of equipment could be another approach for segregation reduction [25,26]. The effect of geometry design of a mixing process on particle segregation was investigated by Windows-Yule and Parker [27]. In this study, they found that density-driven segregation could be controlled by manipulating the aspect ratio of the equipment without the need of changing particle material properties. In a work done by Vanarase and Muzzio [28], an optimum impeller configuration of a continuous mixer was reported to be when blades could push the powders backward (back mixing) to keep a relatively mixed state of the powders. Nevertheless, optimising the equipment design for the segregation reduction of particles is less favourable in many industries due to the high level of capital investment.

Segregation of minor components has a significant impact on industries dealing with powders. For example, the segregation of low content level enzyme granules in detergent industry should be closely monitored as it adversely impacts the quality of the final product. Despite considerable reported research on particle segregation, there is a lack of in-depth work on the evaluation of segregation reduction of low content level ingredients (<2 wt%), particularly in multicomponent powder mixtures. In this paper, the main segregation mechanisms

in a multicomponent mixture of laundry detergent powders are first evaluated by investigating the effect of particle properties on the segregation of powders. Furthermore, segregation mechanism of low content level enzyme granules is evaluated and the effect of particle surface stickiness on their segregation reduction is studied, where a thin layer of liquid coating is applied to the surfaces of particles. The optimum coating level of the minor ingredient (without compromising the flowability of the powders) to reduce their segregation is then reported.

2. Materials and experimental procedure

As model material systems, binary and ternary washing powder mixtures, comprising blue Tetra Acetyl Ethylene Diamine (TAED, used as bleach agent in detergent formulation), white spray-dried synthetic detergent powder referred to as Blown Powder (BP, used as active cleaning agent in detergent formulation), and red Enzyme Placebo Granules (EP granules) are used. The results of particle size distribution of all powders, obtained using British Standard sieves, are provided in Fig. S1, supplementary document, as well as in previous study [29]. To avoid enzyme exposure risk, enzyme placebo granules (EP granules) representing the actual enzyme granules were used. BP and TAED were obtained from Procter and Gamble (P&G), Newcastle Innovation Centre. EP granules were provided by DuPont, USA.

To investigate the effect of particle properties on the segregation mechanisms, components have been characterised separately. Malvern Morphologi G3, Scanning Electron Microscopy (SEM, Hitachi TM3030 Bench Top SEM system) and Nanotom X-ray computed tomography were used for particle shape analysis. Using Malvern Morphologi G3, the average circularity of the projected area of a number of particles was analysed. Also, 3D visualization of particles was obtained by X-ray microtomography (XRT) using the Nanotom X-ray computed tomography instrument (GE Phoenix, Wunstorf, Germany) to measure the sphericity of particles. For the evaluation of the effect of particle stickiness on the segregation of EP granules, Polyethylene glycol (PEG, Plurion E 600) was used as a coating agent. PEG, a family of long chain polymers, is used in many applications, such as surfactants, ointments, foods, pharmaceuticals and cosmetics. In this study, PEG is used as a coating material due to its compatibility and applicability in detergent formulation. PEG is used for many applications in detergent industry, including as a lubricant and surfactant. In some detergent formulations, PEG is used as anti-redeposition agents. It should be noted that the use of PEG with higher molecular weights should be applied with caution as it may not be fully soluble in water [30,31]. Therefore, a sticky PEG with

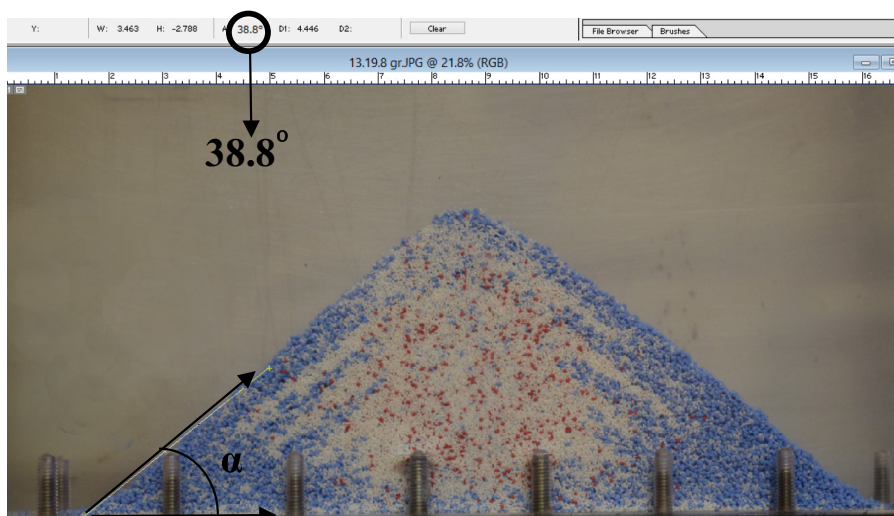


Fig. 1. Angle of repose measurement using image processing tool.

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