IMPROVEMENT OF SEGREGATION PROBLEMS DURING POWDER HANDLING OPERATIONS

Boix, J.[(1)], Aguilella, M.[(1)], Mallol, G.[(1,2)], Llorens, D.[(1,2)], Foucard, L.[(1,2)]

[(1)] Instituto de Tecnología Cerámica (ITC). Asociación de Investigación de las Industrias Cerámicas (AICE)

[(2)] Universitat Jaume I. Castellón. Spain.

1. ABSTRACT

Segregations, understood as the partial separation of the components of a granular material, can pose serious problems in the industrial processes that use particulate materials in some of their stages. One of the main causes of the segregations that take place during powder handling in industrial practice is the difference in particle sizes in these powders.

This paper describes the basic causes of the segregations that appear most frequently in powder handling processes and the actions that can be adopted to prevent or minimise these. Given special importance of powder segregation in the ceramic industry, the study focuses on the segregation problems relating to spray-dried powder transport and storage operations.

The study is rounded off with a series of pilot experiments that demonstrate the efficiency of the actions proposed to minimise the effect of spray-dried powder segregations.

2. INTRODUCTION

At present, numerous industrial sectors use powdered materials in some of their manufacturing processes or stages, such as the pharmaceutical industry, food and agriculture sector, mining, and ceramics manufacture. Very large quantities of particulate materials are manipulated annually worldwide. By way of example, in the year 2010, the Spanish ceramic tile, glaze, frit and ceramic pigment manufacturing sector handled 25,000 tons of powdered materials daily.

Traditionally, the study of the rheological behaviour of these types of materials has drawn little attention. The processes that use particulate materials thus very frequently exhibit processing problems associated with powder handling, with negative effects, reducing plant production capacity and efficiency, as well as end product quality, with the ensuing costs.

Together with the flow breaks in silo discharges, the presence of dead zones inside the silos, failure or alteration of the silo structure, explosions in certain types of flammable materials, and uncontrolled silo discharges, segregations constitute one of the most frequently appearing problems in powder handling and storage operations [1,2]. Segregations involve the partial separation of the constituents of a particulate material or of the components of a granular mixture owing to their differences in size, shape, or density [3].

Segregation problems are common in numerous industrial sectors and processes. For example, they often appear in bulk coal operations, the handling of pharmaceutical powders in the preparation of tablets, or in storing spray-dried powders. The key to avoiding segregations is understanding the behaviour of a certain material and how it will perform on going through a facility's different transport and storage systems. Understanding such behaviour enables definition, in the design phase, of the actions that need to be adopted to prevent segregations or even, should segregations occur, to recombine separated materials in order to maintain product quality.

3. SEGREGATION MECHANISMS

Four physical segregation mechanisms are generally identified: percolation, differences in particle trajectories, fluidisation, and vibration. Each of these mechanisms is described in detail below, indicating the conditions in which they appear with some practical examples.

3.1. PERCOLATION

Percolation is the most common particulate segregation mechanism. It occurs when the finest particles in a mixture are able to move through the voids or interstices left by larger particles. Segregation by percolation in a material is encouraged when the following four conditions concur [4]:

- There is a noticeable difference between particle sizes of the mixture components.
- Average particle size is quite high because, otherwise, the greater cohesiveness of the fine fractions will reduce particle mobility.
- The material displays easy-flow behaviour, associated with low cohesiveness and avoidance of agglomerate formation that will adversely affect particle motion.
- The material is in motion and subjected to a shear rate, because if the particles in the powder bed are stationary or moving at a steady rate, these will be jammed against each other, the tendency to segregate being practically non-existent.

The image in Figure 1 illustrates the percolation phenomenon that customarily develops during storage of spray-dried powder used in ceramic tile manufacture, when this is fed in from a central point at the top of the silo. The photograph shows a cylindrical laboratory silo that has been cross-sectioned, with a view to displaying its contents through a meth-acrylate sheet fixed to the side. The spray-dried powder had been coloured before it was charged into the silo: the granule size fractions below 400 μm were white and the larger fractions were black.

It may be observed that the finest fractions were concentrated in the central part of the silo, right under the feed point. This was because as the pile of material formed inside the silo, the coarsest particles tended to roll across the slope that formed, moving towards the sides. In their displacement, the coarse particles occasionally carried away some fine particles, giving rise to the stratification shown in the image.

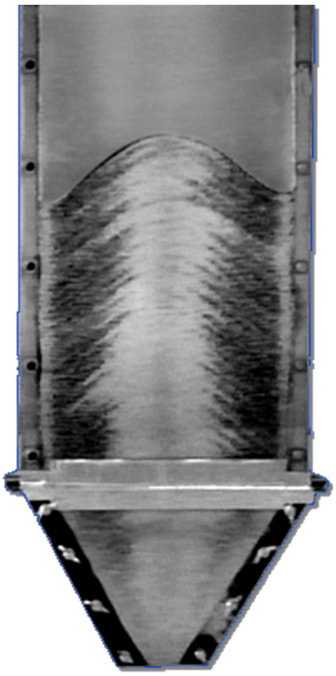

Figure 1. *Cylindrical laboratory silo with spray-dried powder segregated by percolation.*

The phenomenon described above evidences the great proneness to segregation of the spray-dried powder used in ceramic tile manufacture. Indeed, this material fully displayed the four conditions described previously: the powder exhibited a high diameter ratio (7:1); its average particle size was above 250 μm; it displayed easy-flow behaviour; and in addition it was in motion.

Sometimes the high proneness to segregation of spray-dried powder poses serious problems during processing, which materialise in an incorrect development of some process stages and lead to a reduction in end product quality. The plot in Figure 2 shows, as a function of the percentage of discharged material, the evolution of spray-dried powder moisture content recorded continuously by a moisture sensor installed right at the outlet of an industrial silo in which the material exhibited segregation like the one shown in Figure 1.

Owing to inappropriate silo design, the material was discharged in a typical funnel flow pattern. This involves initially discharge of the central part of the silo, in which a completely stable central duct sometimes forms, depending on powder cohesiveness, followed by discharge of the material located most closely to the walls. As a result, the finest spray-dried powder fractions were discharged first. On containing not very porous granules with a low water absorption capacity, in industrial practice, these powder fractions usually display moisture contents about 1 to 1.5% lower than the average moisture content of the material. In the test, the average moisture content was 6%.

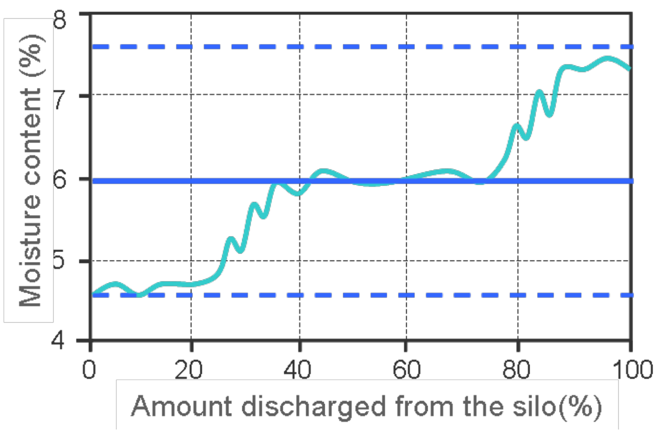

Figure 2. *Evolution of moisture content at the outlet of a silo containing spray-dried powder segregated by sizes.*

As the discharge proceeded, part of the coarse material located near the walls began to discharge. As these coarse fractions had more porous granules, with higher moisture contents than the fine granules, the recorded moisture content progressively increased in the course of the discharge. In the final part of the discharge, as only coarse material poured out, the moisture content reached values between 1 and 1.5% above the average moisture content of the powder.

The variability of spray-dried powder moisture content, together with segregation by percolation inside the silo, entails serious manufacturing problems, given the importance of spray-dried powder moisture content for the bulk density of newly pressed tile bodies [5]. As will be seen below, these types of problems can be minimised and even eliminated by implementing a series of measures when designing storage facilities.

3.2. DIFFERENCE IN TRAJECTORIES

The second mechanism that can lead to the appearance of segregations is the difference in particle trajectories when particles are launched at a certain speed. Indeed, if there are differences in size or shape between particles in a mixture, the finer or irregular particles will offer greater aerodynamic resistance during their displacement, resulting in a slower speed and hence segregation.

This mechanism causes size segregation in the fall of material like the one shown in the schematic illustration of Figure 3. When a mixture of different size particles is driven horizontally, it can be demonstrated that the distance travelled by each particle is proportional to the particle diameter squared. Thus, the finest particles usually pile up near the edge of the fall, while the coarse particles tend to fall farther away.

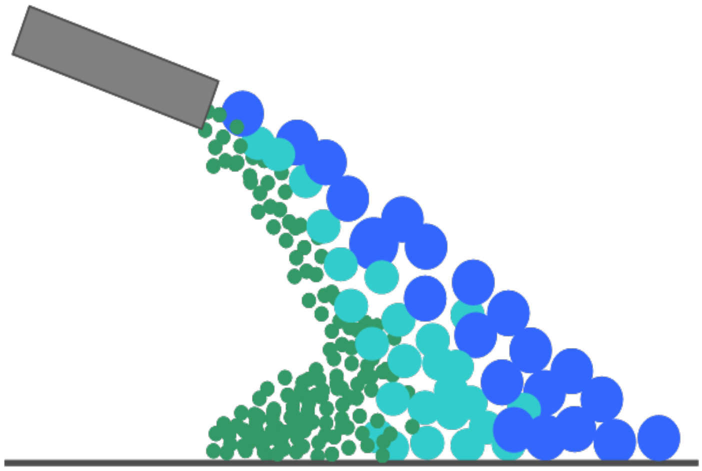

Figure 3. *Segregation by differences in particle trajectory in a powder fall.*

3.3. FLUIDISATION

The fluidisation mechanism takes place in materials or mixtures that contain very fine particles that can easily pass into the gas phase when the material is handled.

An example of segregation by fluidisation is what occurs when a pharmaceutical mixture, comprising an active principle in the form of a very fine powder and an excipient of larger particle size, is charged in a silo or a hopper (see Figure 4). During the fall and subsequent impact against the material previously fed in, much of the active principle can pass into the gas phase, while the excipient is largely deposited on the bottom of the silo. When the feed stops, the active principle is deposited generating a layer with a greater concentration in the upper part of the powder bed in the silo. During discharge, the existing segregation can then generate problems associated with the heterogeneity of the active principle content in the resulting products.

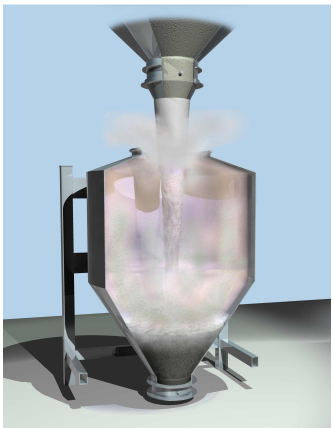

Figure 4. *Segregation by fluidisation (Source: www.jenike.com).*

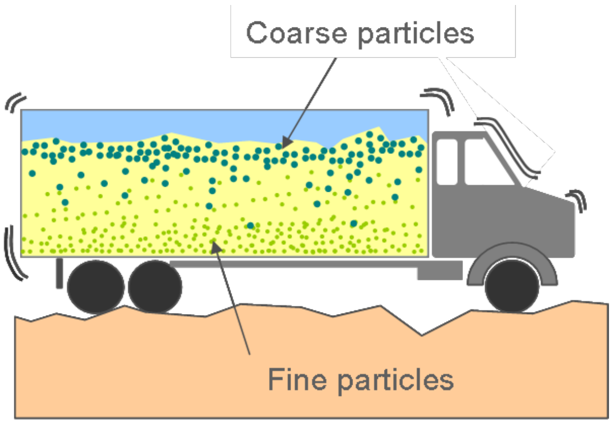

Figure 5. *Segregation by vibration during lorry transport of a particulate material.*

3.4. VIBRATION

There are external dynamic effects that can contribute to segregating components of a particulate material, such as vibration. As a result of the vibration, only a coarse particle is able to rise through a bed of finer particles, as the latter accommodate themselves under the coarse particle by the percolation mechanism.

A typical case of segregation by vibration occurs during lorry transport of certain aggregates or even of bulk foods (see Figure 5). Over sufficiently long distances, an initially homogeneous mixture can segregate the coarsest elements towards the upper part of the bed in the lorry as a result of the vibrations during transport.

4. MATERIALS AND EXPERIMENTAL PROCEDURE

There are different measures that enable the segregations that arise on handling spray-dried powder to be eliminated. With a view to illustrating the efficiency of these measures, a series of pilot experiments were conducted using the laboratory silo shown in section 3.1.

All the trials were conducted with a spray-dried powder customarily used in porcelain tile manufacture, whose granule size distribution (GSD), obtained by dry sieving, is shown in Figure 6.

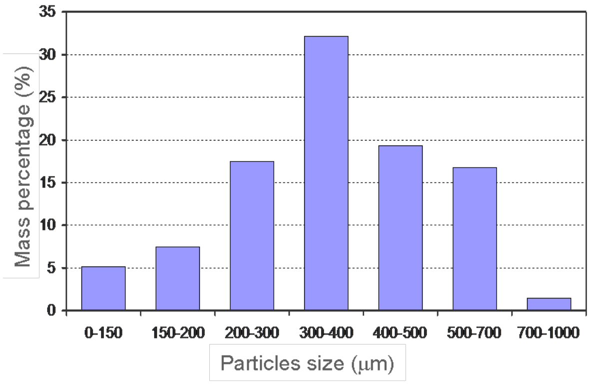

Figure 6. *Granule size distribution of the spray-dried powder used in the trials.*

To better view the inside of the silo during the trials, the work was conducted, on the one hand, with a white and a black powder, both having the GSD shown in Figure 6, and on the other, with a spray-dried powder, prepared from the previous powders, in which the granule fraction larger than 400 microns was black and the smaller fraction was white. The experimental procedure used to evaluate each of the studied improvement actions is described below.

4.1. DETERMINATION OF THE DEGREE OF SEGREGATION OF THE DISCHARGED SPRAY-DRIED POWDER

Before performing the pilot trials, it was necessary to fine-tune a method for evaluating the degree of segregation of the spray-dried powder discharged from the silo. The method ultimately used consisted of determining the percentage of granules larger than 400 µm in powder samples collected at the silo outlet as the material discharge took place. Subsequently, to evaluate the occurrence of segregation, the evolution was plotted of the granule fraction larger than 400 µm versus the time elapsing from discharge start. Thus, the greater the degree of segregation in the material discharge, the greater would be the variation in the coarse granule fraction with time.

4.2. DESIGN OF A MASS DISCHARGE SILO

The first possible measure for minimising the occurrence of spray-dried powder segregations in the manufacturing process is to design and construct storage silos with a discharge zone angle that assures emptying according to a mass flow pattern. This type of flow, as opposed to the funnel flow described above, assures that the material moves steadily during discharge so that, even though segregation occurs during filling, its effect is minimised during emptying when fresh mixing of all the material takes place at the discharge outlet.

According to Jenike's theory [6], there is a discharge zone angle below which material emptying takes place according to a mass flow pattern. It can be demonstrated that this critical discharge angle (θ_c) depends exclusively on silo shape, the cohesiveness of the material contained, and the coefficient of friction between the material and the silo walls.

The so-called Jenike abacuses allow the critical angle of discharge to be determined for a particular silo geometry, knowing the internal friction angle of the stored powder (δ) (directly related to powder cohesiveness) and the friction angle between the silo walls and the powder (ϕ) (directly related to the coefficient of friction). The graph in Figure 7 represents the Jenike abacus for a conical silo like the one used in the pilot trials conducted.

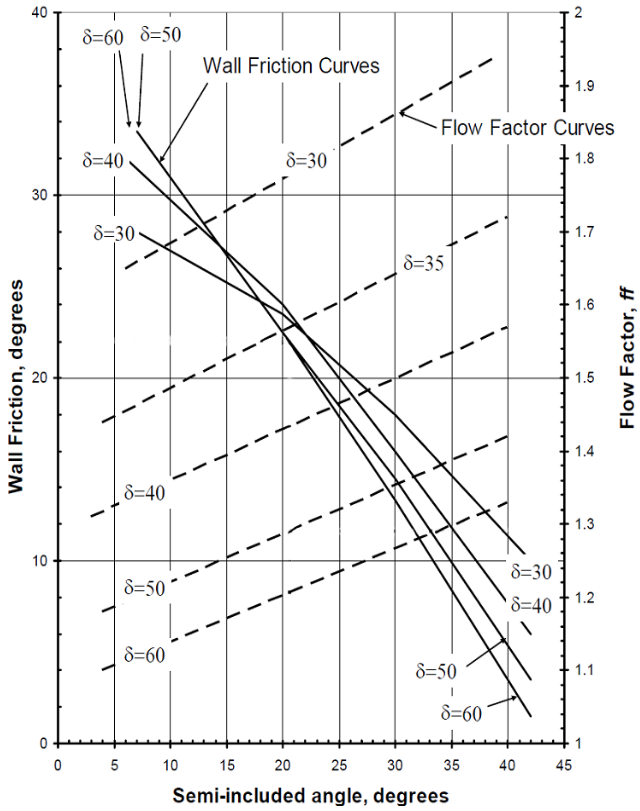

Figure 7. *Jenike abacus for a conical silo.*

The determination of the powder internal friction angle and of the friction angle with the silo walls was performed using a shear cell. Details of the experimental procedure that allows these angles to be calculated are reported elsewhere [7].

The pilot trial consisted of evaluating the degree of segregation of the powder discharged from the silo, according to the method described in section 4.1, for two different silo configurations: a first configuration, in which the discharge angle was the same as the critical angle calculated according to the above procedure, and a second one, in which the discharge zone was modified to give it an angle that was 3° larger than the calculated one.

4.3. INCORPORATION OF LININGS WITH A LOW COEFFICIENT OF FRICTION

As it is often unfeasible to modify the shape of an already constructed silo, the effect of spray-dried powder segregations can be minimised by adopting other measures. One of the most effective measures is modification of the internal surface of the silo discharge zone, with a view to reducing the coefficient of friction between the material and the silo walls. Indeed, according to the abacus in Figure 7, reducing the friction angle with the walls allows larger discharge zone angles to be used without producing funnel discharge. Thus, silos already constructed with an angle that produces funnel discharge can become silos with mass flow at the same angle of discharge if they are internally lined with a material having a low coefficient of friction.

The materials customarily used for this purpose are hydrophobic polymer materials, with high wear resistance and a low coefficient of friction. In particular, this study evaluated in a practical form the effect of a Teflon®-based lining on the segregation of the material discharged from the silo. To do so, the degree of segregation of the discharged powder unloaded when operating with a discharge hopper made of steel was compared with the degree of segregation resulting on using the same hopper lined with a thin sheet of Teflon® adhered to its surface.

4.4. MODIFICATIONS IN THE CHARGE MODE

The previously described actions minimise the effect of the segregations generated during silo charge owing to mixing in the discharge outlet as a result of mass material flow. However, segregation of the material can also be avoided during silo charge by modifying the material feed mode.

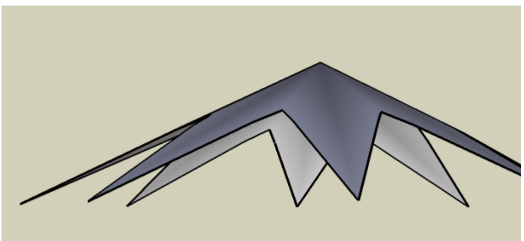

Figure 8. *Scheme of a baffle plate in the shape of a truncated cone-shaped star used for segregations during silo charge.*

In the last evaluated action, the effect of a silo charge centred at one point was compared with a charge process in which the powder was homogeneously distributed across the surface of the deposited material. For this, baffle plates can be installed like the one shown in the scheme in Figure 8, which deflect the material flow, preventing the material from falling at a single point and thus avoiding segregation by percolation.

5. RESULTS AND DISCUSSION

5.1. EFFECT OF THE TYPE OF DISCHARGE FLOW

Following the procedure described in section 4.2, based on the rheological properties of the spray-dried powder, the critical angle of mass flow discharge corresponding to the conical silo used was determined. The shear cell was used to obtain an internal friction angle of the spray-dried powder of 30° and a friction angle with the steel walls of 24°. Introducing these data in the abacus in Figure 7 yielded a critical angle of mass flow of 18°.

To validate the data obtained, the effect was evaluated of working with a steel hopper with the same angle as the calculated one and with an angle 8º larger than this, associated with funnel flow discharge. Figure 9 shows the evolution of the mass fraction percentage with a size larger than 400 μm of the samples collected at the silo outlet for the two evaluated discharge angles. In both cases, a similar starting powder distribution to that shown in Figure 1 was used.

As may be observed, the occurrence of the segregations induced during charging of the material, as a result of the percolation associated with charging from a central point, was very pronounced when the silo emptied according to a funnel flow pattern. Initially, the evaluated samples exhibited a high proportion of fine granules due to the discharge of the material in the centre of the silo. As the discharge advanced, the coarse fraction became more significant until in the last stages, coinciding with the discharge of the material next to the walls, the coarse fraction values reached 60%.

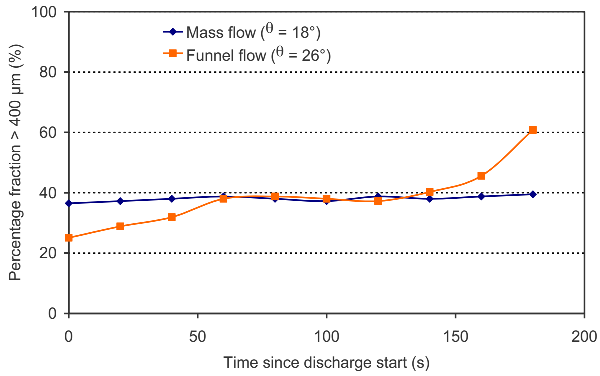

Figure 10. *Effect of the type of material flow on the degree of spray-dried powder segregation at the silo outlet.*

In contrast, when mass flow discharge took place, the discharged coarse fraction stayed practically constant throughout the time at a value of 38%. As has been shown in previous studies, the homogeneous displacement of the material when silo discharge took place by mass flow caused sufficiently good mixing to occur at the silo discharge outlet for the coarse granule fraction to remain practically constant throughout the entire monitoring step.

5.2. EFFECT OF INTERNAL LINING OF THE SILO WALLS

Following the procedures set out in section 4.2, the friction angle between the spray-dried powder and a Teflon®-based lining adhered to a steel plate was determined. This analysis yielded a friction angle between the powder and the lining of 11º, which, according to the abacus in Figure 7, would allow angles of discharge up to 38º, for a spray-dried powder with an internal friction angle of 30º to be used.

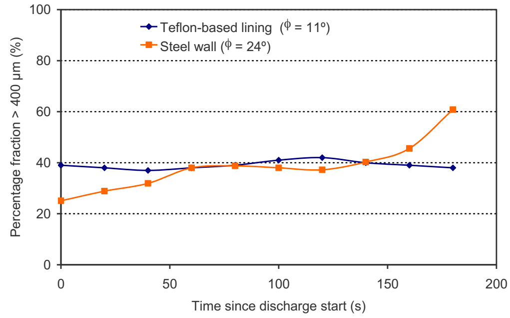

Figure 9. *Effect of the friction angle with the walls on segregation.*

According to these calculations, if the silo evaluated in the previous section with a 26º angle of discharge had the discharge zone inner walls lined with the studied Teflon®, it should produce a mass-type discharge. To verify this, the previously used hopper was lined and the degree of segregation in the spray-dried powder was evaluated during silo emptying, after charging from only one centre point.

In the graph in Figure 10, the evolution of the coarse fraction throughout the discharge for the silo with a 26º angle of discharge and steel inner wall is compared with that of the same silo lined internally with Teflon®. As may be observed, the effect of the lining on the degree of powder segregation was very similar to that produced by the change in the discharge angle set out previously. On working with the internal lining, the coarse material fraction remained constant with time, the advantage being that it was not necessary to decrease the discharge angle so that silo capability was not impaired.

5.3. EFFECT OF THE FEED MODE

The last improvement measure evaluated in the pilot trials was the switch from a fixed feed point, like the one used in the trials described thus far, to a mobile feed point.

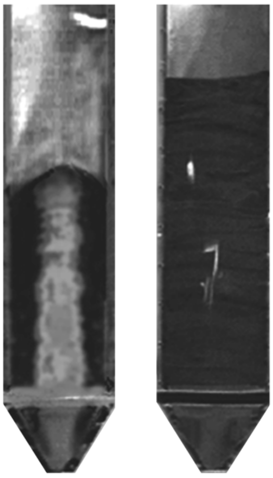

Figure 12. *Spray-dried powder distribution as a function of type of charge (right: fixed point; left: variable point).*

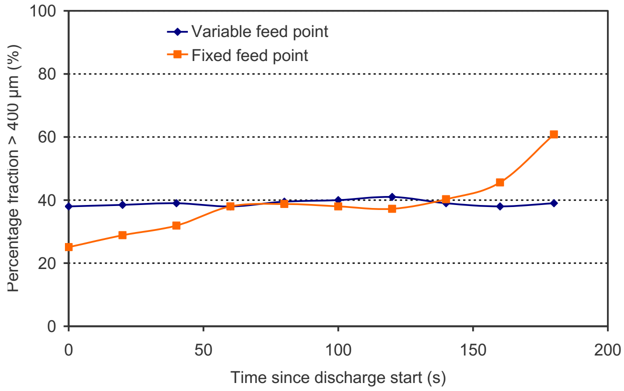

Figure 13. *Effect of silo feed mode on the degree of segregation.*

The images in Figure 11 show, for the two types of charge evaluated, the distribution of the powder inside the steel silo with a 26º angle of discharge when this was charged with the spray-dried powder containing the black-coloured fractions larger than 400 microns. As may be observed, charging from a fixed point, as had previously been confirmed, generated a powder distribution that was completely segregated by sizes, whereas the feed from a variable point gave rise to a more homogeneous powder distribution.

To evaluate the degree of segregation at the silo outlet, the discharge was sampled in the same way as in the previous trials, resulting in the comparative graph in Figure 12. It shows that the discharge coming from the silo fed from a variable point, even using a discharge angle that produced funnel flow, exhibited no segregation.

6. CONCLUSIONS

The following conclusions may be drawn from the present study:

- The segregation of the spray-dried powder by percolation during silo charging can entail serious problems during the ceramic tile manufacturing process if the necessary precautions to minimise its occurrence are not taken.
- It was demonstrated, by means of a series of pilot experiments, that appropriate design of the discharge angle, an internal lining of the silo walls with a low coefficient of friction, or the use of a charging point with a variable position completely eliminated the occurrence of segregations in spray-dried powder storage.
- From a technical and economic viewpoint, the most satisfactory solution for avoiding problems associated with the segregation of the material handled was appropriate design of the facilities, taking into account the rheological properties of the material to be processed.

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