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# Segregation of Formulated Bulk Powders due to Electrostatic Effects

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## Abstract

Particle electrification is a common phenomenon that occurs in many powder handling industries such as pharmaceuticals and detergents etc. The unit operations involved in the manufacture of pharmaceutical formulations frequently subject particles to frictional sliding and impact against processing surfaces, giving rise to tribo-electric charging. Pharmaceutical materials, in particular, are susceptible to electrostatic charging due to their low bulk density, small particle size and often irregular shape and electrically insulating nature. The electrostatic charge may cause particles to adhere to container surfaces leading to a loss of powder through deposition; however the problem often extends further and affects the end products' quality. Segregation can also occur when powders have been subjected to tribo-electrification. In this paper we investigate whether such adhesion resulting from the electrostatic charging could give rise to the segregation of components within a binary mixture. Binary mixtures comprising of  $\alpha$ -lactose monohydrate ( $\alpha$ -LM) and hydroxypropyl cellulose (HPC) were firstly tribo-charged and then the wall adhered particles were separated by a selective dissolution of one component and the filtration of the non-dissolving component, followed by a gravimetric analysis. The findings reveal that a considerable level of segregation can take place on the wall-adhered particles.

## 1. Introduction

In this study, a range of  $\alpha$ -lactose monohydrate ( $\alpha$ -LM) and hydroxy propyl cellulose (HPC) mass ratios (charging positively against a PTFE container as a result of tribo-electrification) were analysed for the level of material that had adhered to the wall. The particles were tribo-charged using the method described by Supuk *et al.*, 2009, i.e. a shaking device and container made from PTFE. There were several reasons for selecting the shaking device to study segregation due to electrostatic effects. Firstly, the use of shaking containers removes any sampling errors, as the whole of the sample is taken into account. Secondly, the powder adhering to the walls due to a high level of tribo-electric charging can be easily removed and the powder can then be dispersed/dissolved in solvent and filtered to determine the level of segregation. Furthermore the shaking machine provides further mixing of the components ensuring that each component is impacted against the shaking container walls. The mixture composition post shaking was analysed by dissolution and filtration to determine the proportion of  $\alpha$ -LM in comparison to the proportion of HPC. An attempt was made to link the tribo-electric charging tendencies of the selected excipients with the segregation tendencies by comparing the composition of particles adhered to the walls with that of the original formulations.

## 2. Methodology

Binary mixtures were tribo-charged using the shaking device and the charge measured by the Faraday cup and an electrometer. The powders were charged inside a PTFE container at 20 Hz. The shaking time was selected based on time required to reach the saturated charge level for each binary system as shown in Table 1. The length of time that each binary mixture was tribo-charged prior to analysing segregation study depended on the composition ratio of a mixture. A binary mixture was charged at time increments until no further increase in the net charge was measured, i.e. three consistent readings were obtained. At this point, it was anticipated that the charge had reached a saturation level.

The wall adhered material was then recovered using an electrostatic gun together with a gentle tapping on the outer wall of the shaking container. Scraping of the material from the sides was avoided to ensure that no particles were lost or broken. Following tribo-electric charging, the sample from the wall was collected, dispersed in the solvent and filtered. A known amount of wall-adhered mass of  $\alpha$ -LM and HPC was dispersed in 50 ml of propan-2-ol. The HPC particles dissolved, leaving only  $\alpha$ -LM particles dispersed in the liquid. After filtering and drying in the oven at 35 °C for one hour  $\alpha$ -LM particles were weighed. Table 1 below shows the binary mixture compositions by weight of  $\alpha$ -LM and HPC particles. Systems 1 and 7 are omitted as they were used for calibration purposes and are not presented here. Approximately 1 g of pure  $\alpha$ -LM was placed in 50 ml of propan-2-ol. The dispersion was agitated and left for a period of 72 hours to allow enough time to test the extent of dissolution of the  $\alpha$ -LM particles in propan-2-ol. After this time, the sample was filtered and dried and the mass of  $\alpha$ -LM was measured again. The procedure was repeated three times. The same procedure, as above, was carried out for the HPC sample, including repeats. The HPC sample dissolved completely. The amount of  $\alpha$ -LM recovered is slightly less than originally used. This is less likely to

have happened due to  $\alpha$ -LM particles dissolving in propan-2-ol, but rather due to some fine particles being lost as they adhere to walls following drying stage.

**Table 1:** The average charge and adhesion of binary mixtures

| System | $\alpha$ -LM:HPC | Shaking<br>Time<br>[min] | Q/m<br>[nC/g] | $M_i$<br>[g] | $M_a$<br>[g] | $M_{\alpha\text{-LM}}$<br>[g] | $M_{\text{HPC}}$<br>[g] | % $\alpha$ -LM<br>measured |
|--------|------------------|--------------------------|---------------|--------------|--------------|-------------------------------|-------------------------|----------------------------|
| 2      | 80:20            | 30                       | 16.67         | 1.0024       | 0.0802       | 0.0764                        | 0.0039                  | 95:5                       |
| 3      | 60:40            | 30                       | 14.21         | 1.0007       | 0.0757       | 0.0546                        | 0.0210                  | 72:28                      |
| 4*     | 50:50            | 20                       | 11.89         | 1.0021       | 0.0695       | 0.0454                        | 0.0241                  | 65:35                      |
| 5      | 40:60            | 20                       | 10.49         | 1.0014       | 0.0673       | 0.0332                        | 0.0342                  | 49:51                      |
| 6      | 20:80            | 15                       | 5.01          | 1.0034       | 0.0552       | 0.0144                        | 0.0408                  | 26:74                      |

$M_a$ : wall adhesion,  $M_{\alpha\text{-LM}}$ : amount of  $\alpha$ -LM in  $M_a$ ,  $M_{\text{HPC}}$ : amount of HPC in  $M_a$ ,  $M_i$ : initial

### 3. Results

#### 3.1 Tribo-Electrification of Binary Mixtures

Table 1 above shows the saturated charge-to-mass ratio (Q/m) for each binary mixture following tribo-electrification inside a PTFE container. The amount of charge decreases when there is a decrease in the amount of  $\alpha$ -LM present in a binary mixture. This is expected as the saturated charge measured on pure  $\alpha$ -LM particles (24.7 nC/g) following tribo-electrification (inside a PTFE container at 20Hz) and the specific saturation time is significantly higher in comparison to that measured of HPC (3.2 nC/g) in similar test conditions. The introduction of more  $\alpha$ -LM particles into the system increases the chance of  $\alpha$ -LM particles impacting on the walls of the shaking container.

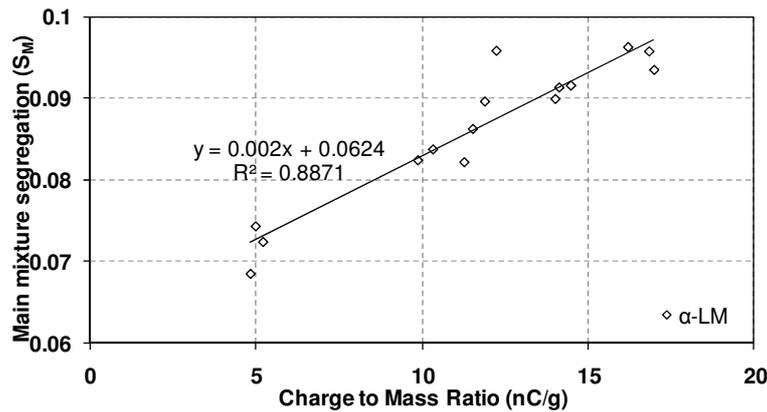
In Table 1, the mass percentage of  $\alpha$ -LM for an ideal mixture, measured by the filtration method are shown for each binary mixture system. The amount of particles adhered to walls increases as the  $\alpha$ -LM in the initial system is increased. The 80:20 system has the largest difference between the ideal and measured amounts of  $\alpha$ -LM and it is expected that this system will cause the largest amount of segregation in the main mixture.

#### 3.2 Segregation of the Main Mixture

The level of segregation of the binary mixture in the bulk of the sample and in the powder mixture adhered to the walls of the container was analysed. In this section the term "the main mixture" is used to refer to the mass recovered from the shaking container following tribo-electrification and it excludes the amount of powder adhered to the walls. In contrast, the term "wall-adhered mixture" is used to refer to wall-adhered material only. The change in  $\alpha$ -LM concentration in the system can be used as an indicator of the extent of segregation of  $\alpha$ -LM in the main mixture ( $S_M$ ). The segregation extent for the main mixture was determined by calculating the ratio between the amount of  $\alpha$ -LM recovered at the end of tribo-electrification and the amount of  $\alpha$ -LM initially used, for each sample, as shown by:

$$S_M = 1 - [(\text{Conc. of } \alpha\text{-LM after}) \div (\text{Conc. of } \alpha\text{-LM before})]$$

An extent of segregation that is equal to one indicates a fully segregated system, whilst a value of zero indicates a system where no segregation has taken place. Therefore, an increase from zero indicates that more  $\alpha$ -LM particles have adhered to the container walls and this indicates that the binary mixture has undergone segregation. In Fig. 1, the extent of segregation of  $\alpha$ -LM in the main mixture is shown as a function of charge-to-mass ratio. Despite the data scatter, a clear trend is observed. With higher charge-to-mass ratios, the extent of segregation is increased, which implies that the tendency of a binary mixture to segregate is adversely affected by tribo-electric charging of the mixture. However, the extent of segregation is relatively small (below 0.1). This is expected as the difference in the ideal and the measured values of  $\alpha$ -LM on the walls (Table 1) is small when looking at  $\alpha$ -LM variation in the whole mixture.



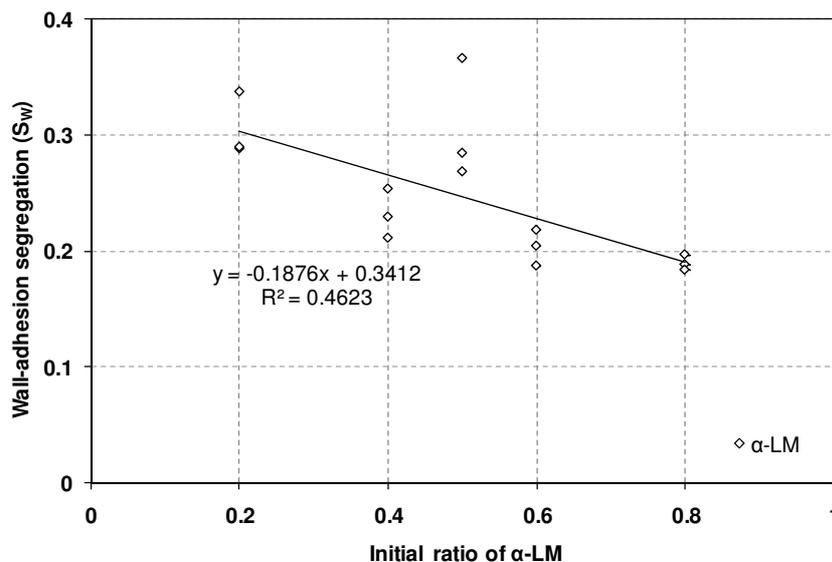
**Figure 1:** The extent of Segregation of  $\alpha$ -LM in the main mixture as a function of the initial ratio of  $\alpha$ -LM

### 3.3 Segregation of the Wall-Adhered Mixtures

In order to determine the extent of segregation of  $\alpha$ -LM on the wall ( $S_W$ ), the following equation was used:

$$S_W = 1 - [(\text{Amount of } \alpha\text{-LM on walls}) \div (\text{Ideal amount of } \alpha\text{-LM on walls})]$$

Figure 2 shows the extent of segregation of the-wall adhered mixture as a function of the amount of  $\alpha$ -LM in a binary mixture. The results show that the extent of segregation of  $\alpha$ -LM decreases with an increase in the amount of  $\alpha$ -LM present in a binary mixture. This is expected as an increase in the  $\alpha$ -LM concentration in the mixture causes segregation to approach zero asymptotically. The extent of segregation in the wall-adhered powder mixture is remarkable and, in some instances, this value is over 0.3. However, even though the extent of segregation is high on the wall-adhered material, when considering the whole of the mixture, extent of segregation is less than 0.1. Nevertheless, in an industrial context such as the pharmaceutical industry, the end products of high purity and good content uniformity are essential. The variations of components within a binary mixture adhered to walls play a significant role in the content uniformity. The mixture which is built up on the walls of the process equipment due to tribo-electric charging may fall off under its own weight due to gravity and upset the formulation. This phenomenon can take place further down the processing line, after the mixing and blending stages leading to the formation of aggregates on the walls. These aggregates may be segregated and if falling into the main mixture at the packaging stage, they will cause a transient change in the mixture composition, thus having an immense and adverse impact on the content uniformity.



**Figure 2:** Extent of Segregation of  $\alpha$ -LM in the wall-adhered mixture as a function of the initial ratio of  $\alpha$ -LM

## Conclusions

All the binary mixtures charged positively against the PTFE container at 20 Hz. The magnitude of charge increased with an increase in the amount of  $\alpha$ -LM particles in a binary mixture.

The results of the segregation analysis show that the extent of segregation in the main mixture increases linearly with an increase in the amount of  $\alpha$ -LM and consequently with the net charge on binary mixtures. The extent of segregation is highest in the 80:20 and lowest in the 20:80 ratio binary mixtures. However, the extent of segregation does not exceed 0.1 when the main mixture is considered. The extent of segregation measured for the wall-adhered mixtures shows an opposite trend to that of the main mixture, i.e. decreasing linearly with the amount of  $\alpha$ -LM increasing in the binary mixture. In this case, the extent of segregation is considerably higher than the main mixture with the highest value being 0.31 for the 20:80 ratio mixtures and the lowest of 0.19 for the 80:20 ratio mixtures.

## References

Supuk, E., Seiler, C., and Ghadiri, M. (2009), Analysis of a Simple Test Device for Tribo-Electric Charging of Bulk Powders, *Part. Part. Syst. Charact.*, **26**, 7-16