

Laboratory methods for assessing the segregation of additives tested by Tecaliman

A methodology for assessing segregation may consist of a straightforward comparison of coefficients of variation before and after mechanical or pneumatic transfers (Friedrich and Jansen, 1974 – Tecaliman, 1996a - Wild, 1999). However, this must involve $CV_{\text{homogeneity}}$ values as residual variations can, as usual, hide the actual effects of stimulated factors, and in this case, transfers. Other tests, conducted in a laboratory, can be used to stimulate the effects of segregation using the angle of repose or elutriation, in line with the definitions of these phenomena (Tecaliman, 1996b).

1. Angle of repose

1.1. Existing methods

One of the first known tests for segregation was developed by Williams and Shields (1967) as part of their study of the segregation of fertilizer pellets. How their apparatus for investigating segregation functions is not clear, but their study led to the proposal of a simple segregation coefficient (C_s), which was subsequently used in another study (Williams and Khan, 1973) and was based on the comparison of concentrations in two areas (A and B) of a mix or a heap:

$$C_s = \frac{C_A - C_B}{C_A + C_B} \cdot 100$$

Schneider (1985) described the technique used by the company BASF to assess the segregation of additives in various excipients (Figure 1).

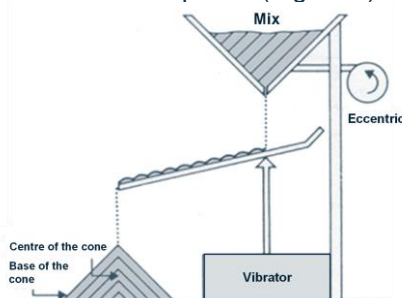


Figure 1: System for stimulating segregation using the angle of repose designed by Schneider (1985)

A segregation test using the angle of repose consists of comparing the concentrations of samples collected in the centre of the cone and at the foot of the cone with the theoretical concentration.

1.2. Tecaliman's tests

1.2.1. Method

Tecaliman used a segregation test inspired by Schneider's approach (1985). The heap is created in the same way, but samples are taken at the top (T), in the centre (T) and at the base (B) of the cone by a series of divisions (Figure 2).

The concentration of traced product is analysed in each section of the mound. An attempt to summarise the results of the three analyses in the form of a global index was made, similar to the work of Williams and Shields (1967), but without it being possible to clearly highlight the segregating behaviour, which is nevertheless visible when looking at the raw data.

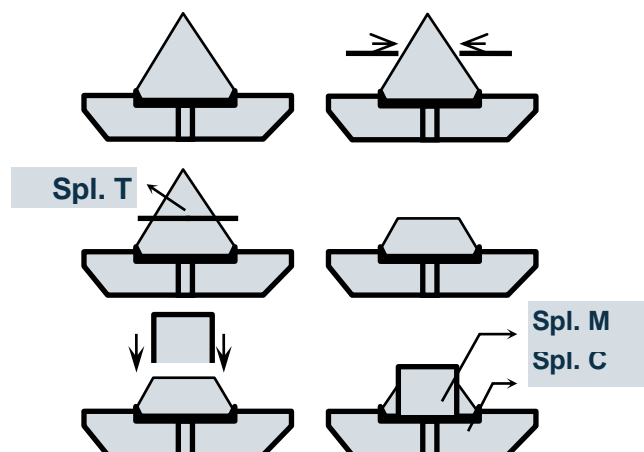


Figure 2: Sampling protocol for segregation diagram

1.2.2. Results

A test was carried out by Tecaliman, using copper sulphate (246 μm – 10000 ppm) dispersed in the laboratory in glass beads of two sizes (510 and 260 μm), exhibiting no interaction with the tracer. Three mixes were produced using each of the matrices with mixing times of 60, 120 and 240 s.

With a higher grain size, clear segregation occurred during homogenisation in the laboratory. This behaviour was also confirmed (Figure 3) by the detection of more significant segregation with the largest matrix: copper sulphate concentrating in the centre and at the top of the heap.

In another test, the behaviour of two additives (Amprolium and manganese sulphate) was tested at 3 different concentrations in 2 carbonates with different grain sizes. The results of homogenisation detected an interaction effect between the traced products and the matrices. Assessments of segregation behaviour were subsequently undertaken, using either the angle of repose or elutriation (See next chapter).

The results of segregation tests using the angle of repose (Figure 4) clearly reveal that differences in concentrations are created between the different

parts of the heap, when mixes are produced in coarse carbonate (CG), while there is no difference in fine carbonate (CF).

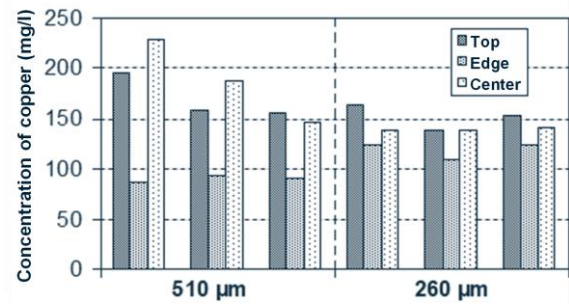


Figure 3: Results of the segregation test on copper sulphate mixes in two types of glass beads

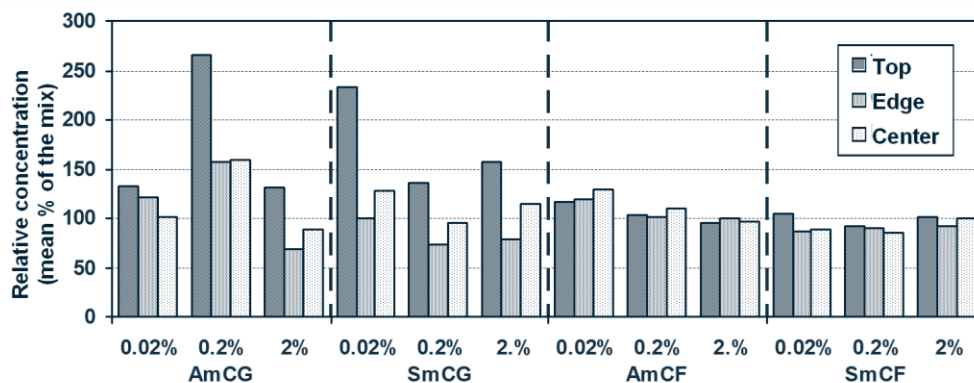


Figure 4: Results of the segregation test using the angle of repose for Amprolium and manganese sulphate mixes produced in three increasing concentrations and in two carbonates with different grain sizes

2. Elutriation

2.1. Existing methods

This kind of test was proposed by Amornthwaphat *et al* (1998) using a height of 6 m in a pipe with a diameter of 21 cm, without further details.

2.2. Tecaliman's tests

2.2.1. Method

This same type of test was developed by Tecaliman in a pilot workshop. Its very simple principle consists of using a sample of approximately 300 ml of mix, in which the distribution of tracer is assumed to be homogeneous, and subjecting it to a vertical drop in an 8 m high PVC tube (Figure 5).

After this drop, a proportion of the sample is taken from its surface (H) and another from the base (B). Segregation is expressed by an elutriation index based on a principle that is similar to that of Williams and Shields segregation index (1967): a ratio of the difference in concentration between the surface of the sample (H) and the base of the sample (B) over the average of these two concentrations:

$$IE = \frac{C_H - C_B}{\left[\frac{C_H + C_B}{2} \right]} \cdot 100$$

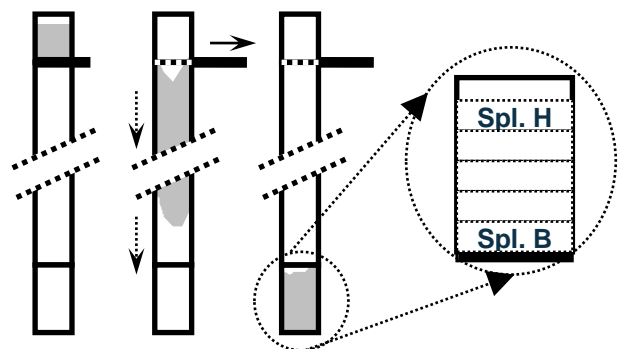


Figure 5: Sampling protocol for elutriation diagram

2.2.2. Results

The repeatability of this measurement was tested using mixes of RF-blue lake microtracers in two

cattle feeds with different grain sizes (280 and 460 µm). The test was repeated 10 times. The coefficients of variation obtained - below 10 % - were deemed to be acceptable as regards the durability of the test (Table 1).

Elutriation index	Fine feed	Coarse feed
Average	88.5	151.3
Standard deviation	8.8	12.8
CV (%)	9.9	8.5

Table 1: Results of the repeatability assessment for elutriation tests

This elutriation test was applied to Amprolium and manganese sulphate mixes in two carbonates and with different concentrations (See previous chapter). The results (Figure 6) show that in line, to a degree, with the segregation test using the angle of repose, segregation occurs with the coarse carbonate. The top of the mix samples that have been subjected to a vertical drop is still more concentrated than the base for the two additives used. This means that these additives have a greater air resistance than the carbonate forming their environment and that they land slightly later when the mix is dropped. This phenomenon appears to increase slightly when the concentration in the mix increases. However, a

new phenomenon occurs with fine carbonate. In effect, Amprolium displays significant elutriation in this carbonate. This segregation is clearly greater when the concentration of Amprolium increases and also occurs in reverse: there is more Amprolium in the base of the sample. Finally, the manganese sulphate mix in the fine carbonate demonstrates its stability with equal concentrations of manganese at the top and the base of the sample after it is dropped. This stability can be explained by the similarity in grain size of the additive and the fine carbonate.

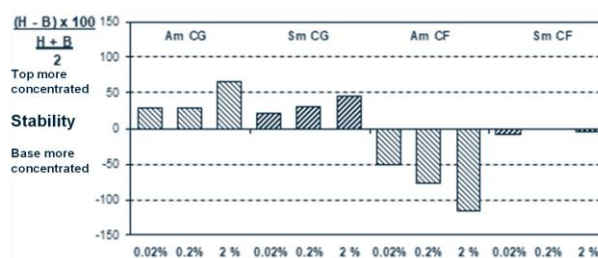


Figure 6: Results of the elutriation tests on Amprolium and manganese sulphate mixes produced in three increasing concentrations and in two carbonates with different grain sizes

Another larger pilot test was subsequently carried out with five other additives (Table 2) and one other excipient: ground flaxseed press cake with three calibrated grain sizes (Table 3).

		A	B	C	D	E
Angle of repose	(°)	57.6	43.8	73.0	70.3	49.7
Apparent (bulk) density	(g/cm ³)	0.64	0.48	0.39	0.83	0.97
Particle density	(g/cm ³)	1.32	1.48	1.35	5.52	2.94
Grain size using laser diffraction	(µm)	167.2	749.4	702.4	8.7	79.6

Table 2: Physical characteristics of the 5 additives

Flaxseed press cake	D50 using laser diffraction	Apparent (bulk) density
Fine	73.0 µm	341.0 g/l
Medium	188.6 µm	429.3 g/l
Coarse	596.0 µm	436.5 g/l

Table 3: Physical characteristics of the excipients

Following mixing for the pilot test, the CVhomogeneity values are fairly stable depending on the grain size of the excipients for four of the five additives (Figure 7). Product B has a clear tendency towards heterogeneity in the 2 finest flaxseed press cakes. This tendency is confirmed by the results of elutriation tests carried out on the same mixes (Figure 8). In this case, an interesting comparison can be made between products B and C, which have similar grain sizes, but only one of which is stable in the mix in the three excipients (product C). However, the only difference in physical characteristics between these two products is the angle of repose. Consequently, the

assumption that is made is that the easier flow of product B assists its segregation.

In this instance, this segregation by flow would have occurred more evidently in the coarsest medium. The excipient's fineness gives the mix a cohesion that limits dispersion of the product and, therefore, results in heterogeneous distribution, but enables segregation to be limited when the mix is transferred in pipes.

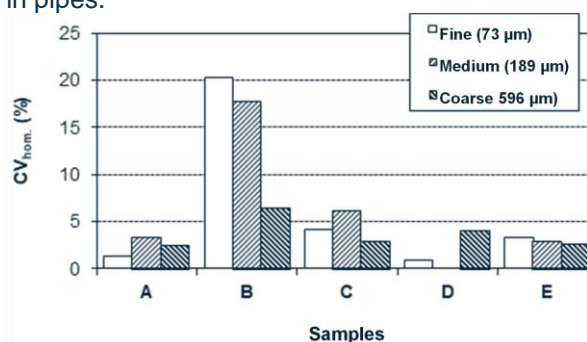


Figure 7: CVhomogeneity values depending on the additive and flaxseed press cake

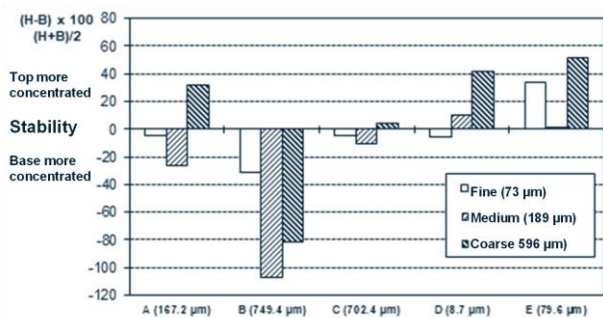


Figure 8: Result of the elutriation test on mixes of five additives in flaxseed press cakes

Other mixes of these five products were produced in an industrial setting using a carbonate base (188 µm). These mixes were also subjected to an elutriation test (Figure 9) and the result confirmed product B's tendency to segregate in this new excipient. However, product A emerged as more unstable in the carbonate than it was in the flaxseed press cake with the same median grain size. This behaviour was attributed to a difference in density between the carbonate and product A.

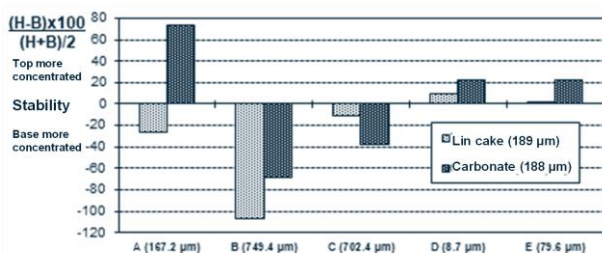


Figure 9: Comparison of the results of elutriation tests carried out on mixes containing 0.2 % of the five additives in a carbonate or a flaxseed press cake with similar median diameters

3. Conclusion

These two tests demonstrated that they provided coherent information regarding the relative stability of their distribution in mixes.

Trials of segregation testing using the angle of repose were not continued beyond the results appearing in this sheet, because it is impossible to easily express the results in the form of a summary index. Furthermore, by means of tests conducted on Amprolium and manganese sulphate, it appears that the elutriation test appeared more forceful in the stimulation of segregation possibilities. Its relatively good repeatability, its ease of implementation and its ability to reveal instabilities in mixes results in it being favoured.

4. Bibliographic references

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