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Results of the pilot tests on particle segregation by elutriation

The method used to assess the segregation of particles by elutriation was published in 2002 (Technical Datasheet No. 41). In short, this method consists in comparing the concentration of tracer above and below a sample that has been dropped in a column. The elutriation ratio defined by this method ranges from – 200 to + 200; a null ratio indicates the stability of the traced product. In 2004, it was demonstrated that, to a certain extent, this method could be used to predict segregation at an industrial site (Technical Datasheet No. 50).
The next stage in our research into this segregation assessment method was therefore to study which parameters were likely to impact on this segregation.
The first parameter is the ratio of volumes at the time of discharge (RVD). This ratio was defined by Campbell and Bauer in 1966, who demonstrated that the ratio of particle volumes in a mix was a key factor in effectively gauging possible segregation. This ratio compares the volume of feed fraction with particle size greater than a 400-µm sieve against the volume of feed fraction with particle size less than 400 µm. The measurement involves sieving 4 to 5 kg of feedstuff through a 400-µm sieve and then measuring the weights and densities of the fractions above and below this sieving diameter. According to Bruggemann and Niesar (1962), the RVD should be interpreted as follows:
Zero risk of segregation below 0.8
Uncertainty over mix resistance between 0.8 and 1.1
Risk of segregation above 1.1
In 1968, Delort-Laval *et al.* demonstrated the usefulness of this ratio by showing that overall segregation of proteins, chlorides and D.O.T effectively increased with increasing RVD.

1. Objective

The purpose of this new study, rolled out via two campaigns, was to investigate how various parameters effect segregation:

- variation in RVD
- type of matrix used
- variation in microtracer particle size
- column height

2. Principle

For the first campaign, the tests were performed on 2 different matrices and used an 8-m column:

- One mineral matrix: Glass beads of two different diameters
- One organic matrix: Soybean cake for coarse particles and rofelys for fine particles

Varying the fractions of fine and coarse particles promotes a variation in RVD, creating 8 levels that range from 0 to ∞. Interval limits are set by the presence of fine or coarse particles alone.
For the second campaign, 4 feedstuffs were used, together with two columns of 4 and 8 m.
During these campaigns, two microtracers (RF and F) were used to study the effect of their difference in particle size.

3. Equipment and apparatus

3.1. Microtracers

Laser diffraction particle size analysis on the 2 RF and F microtracers revealed median diameters of 100 and 269 µm respectively (Figure 1).

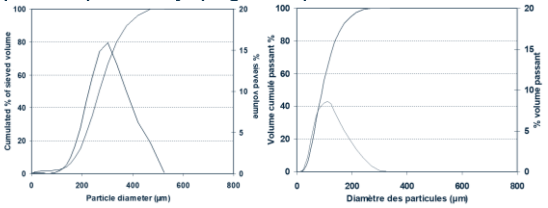


Figure 1: Laser diffraction particle size distribution of microtracers RF (left) and F (right)

3.2. RVD matrices

	Rofelys	Soybean cake
Median diameter (µm)	293.0	688.0
Bulk density (g/l)	481.5	679.5
Tap density (g/l)	671.0	719.3

	Glass beads	
Median diameter (µm)	180 to 300	400 to 600
Bulk density (g/l)	1589.6	1498.5
Tap density (g/l)	1632.8	1599.8

Table 1: Physical properties of the organic (top) and mineral (bottom) matrix bases

	RVD	0	0.5	0.75	1	1.25	1.5	2	∞
Mineral matrix	Small beads (% by weight)	100.0	65.4	55.7	48.6	43.0	38.6	32.1	0.0
	Large beads (% by weight)	0.0	34.6	44.3	51.4	57.0	61.4	67.9	100.0
Organic matrix	Rofelys (% by weight)	100.0	57.0	48.8	41.5	36.2	32.1	26.2	0.0
	Soybean cake (% by weight)	0.0	43.0	51.2	58.5	63.8	67.9	73.8	100.0

Table 2: Change in percentages by weight of the fine and coarse fractions according to RVD for the mineral and organic matrices

For the first campaign, the matrices comprised a mix consisting of some or all of the products listed in Table 1. Table 2 gives the percentages by weight for the fine and coarse fractions of both matrices.

3.3. Feedstuffs

For the second campaign, 4 feedstuffs were used (Table 3).

Feedstuffs	Dairy cow	Piglet	Turkey	Broiler
Median diameter (µm)	408.9	323.3	558.1	409.1
Bulk density (g/l)	639.9	704.5	592.2	596.5
Tap density (g/l)	738.0	792.0	721.0	747.7
Hausner ratio	1.10	1.20	1.27	1.25
RVD	1.07	1.17	2.00	0.99
Angle of repose (°)	51.1	60.3	64.9	67.3

Table 3: Physical properties of the feedstuffs

4. Method

The method used to measure the elutriation ratio has been described in a previous paper (Technical Datasheet No. 41).

4.1. Campaign 1

Each test was performed at least twice. The quantities transferred in the column were approx. 350 ml, i.e. 550 g of glass beads and 250 g of Soybean/Rofelys mix. The fractions collected below and above the samples were 100 g for glass beads and 50 g for the organic matrix. These fractions were then halved to make up the test portions used at an analytical level to perform analyses in duplicate.

4.2. Campaign 2

Mixes of 1.6 kg were made up for each feedstuff and each microtracer (500 ppm). These were then divided to produce samples of approx. 300 ml in order to perform two tests per column, per feedstuff and per microtracer. As described above, each fraction was analysed twice.

5. Results

5.1. Campaign 1

5.1.1. Mass balances

The loss in mass during passage through the column (% of mass introduced) was determined by weighing the products before and after this operation. While these losses were recorded with both types of matrix, they were comparatively less in the mineral matrix (-0.33%), than in the organic matrix (- 1.42%).

5.1.2. Mineral matrix

5.1.2.1 RF microtracer

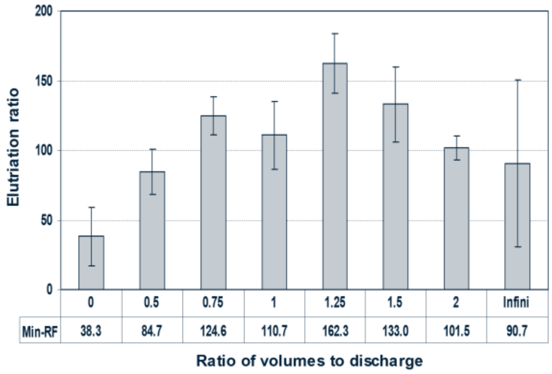


Figure 2: Change in RF-microtracer elutriation ratios according to RVD in the mineral matrix

All the ratios were positive, which suggests that the microtracer rises within the samples (Figure 2). RF-microtracer showed greater stability in the presence of fine particles; ratios increased up to an RVD of 1.25. Above this value, although the ratios appeared to fall, this could be largely explained by the microtracer particles percolating down through the large glass beads sieve each time the samples were handled. Without this effect, it is likely that the value would have remained at a threshold of about 160.

5.1.2.2 F microtracer

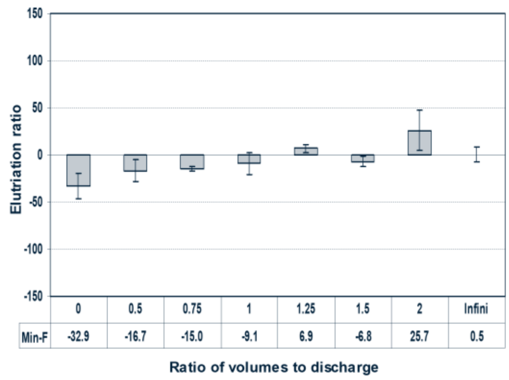


Figure 3: Change in F-microtracer elutriation ratios according to RVD in the mineral matrix

In this case, the ratios were close to zero (Figure 3), indicating the overall stability of this microtracer in these mineral matrices, whatever the RVD. However, F-microtracer tends to show a more stable distribution pattern in mixes mainly formed of coarse particles. RVD has a negligible, but nonetheless perceptible, impact on mix stability. Values were seen to rise from negative to positive up to 1.25, before appearing to reach a plateau at about 0, with a strong oscillation at RVD level 2 that is based on a single datum.

5.1.3. Organic matrix

5.1.3.1 RF microtracer

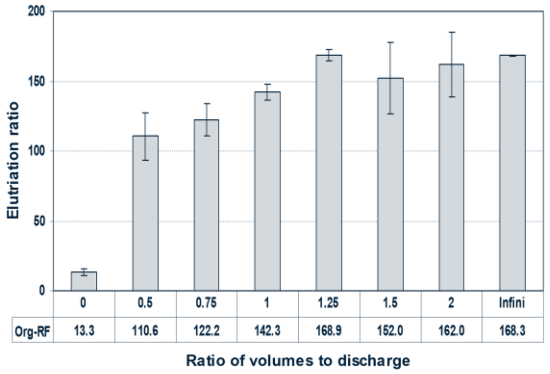


Figure 4: Change in RF-microtracer elutriation ratios according to RVD in the organic matrix

As with the mineral matrix, once again, all the ratios for this microtracer were positive (Figure 4).
The cohesive effect of fines on the stability of microtracer distribution was strongly apparent in the deviation between RVDs of 0 and 0.5. The RVD then increased gradually up to 1.25, before stalling again at a plateau of approx. 160.
Therefore, while a volume mostly made up of fines reduces the risk of segregation, this segregation can occur rapidly as soon as the ratio of coarse particles reaches 33% (by volume) (RVD = 0.5).

5.1.3.2 F microtracer

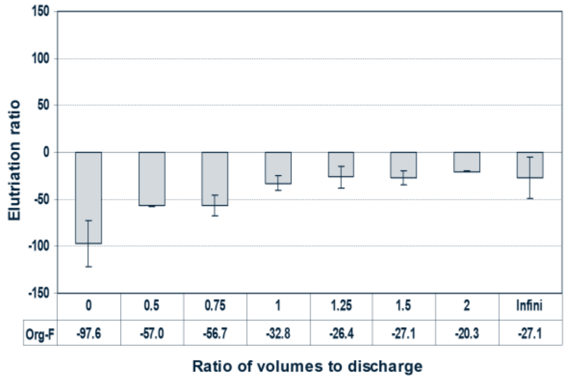


Figure 5: Change in F-microtracer elutriation ratios according to RVD in the organic matrix

For the first time, all the ratios were negative (Figure 5). Even more clearly than in the mineral matrix, the ratios were seen to increase up to an RVD of 1.25, after which the plateau appeared, but this time at a value of - 25. As with the mineral matrix, this microtracer showed greater distribution stability in mixes containing a majority of coarse particles; in this case, however, segregation persisted whatever the RVD. This type of matrix would appear to discharge F-microtracer downwards.

Campaign 2

5.1.4. Mass balances

A systematic loss of mass was observed during passage in the columns (Table 4). Losses were greater in the 8-m column for dairy cow feed and piglet feed.

Column	Dairy cow	Piglet	Turkey	Broiler
8 m	-2.8	-5.6	-0.5	-0.8
4 m	-2.5	-3.9	-1.3	-1.6

Table 4: Loss of mass of the samples during tests in both columns

Taking all columns together, losses were also larger for Turkey and broiler feeds. In all likelihood, it is the greater cohesiveness and fat content of these two feeds that caused the comparative difference with the Dairy cow and Piglet feeds which are more fluid and powdery.

5.1.5. Column effect

5.1.5.1 RF-microtracer

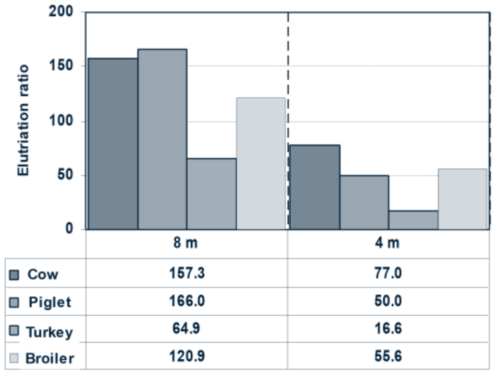


Figure 6: Elutriation ratios for RF-microtracer in the 4 feedstuffs according to column

While the ratios are all positive (Figure 6), they are systematically inferior in the 4-m column. For each feedstuff, the variation in ratios is not proportional to column height.
The range in variation between feedstuffs is greater in the 8-m column (101.1 against 60.3). The latter therefore provides a better resolution, slightly modifying feedstuff rankings.

5.1.5.2 F-microtracer

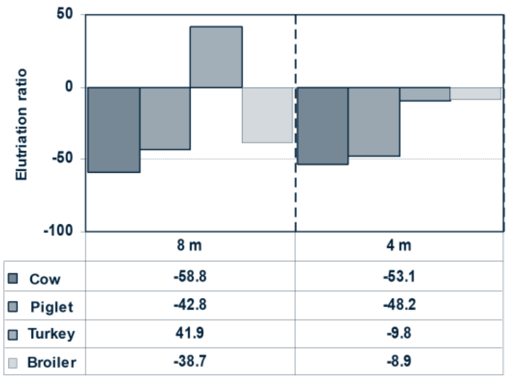
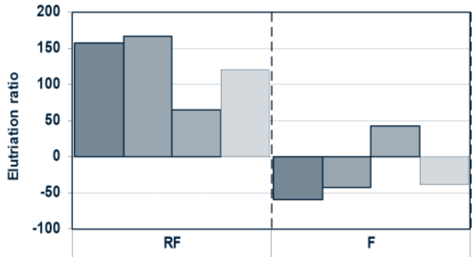


Figure 7: Elutriation ratios for F-microtracer in the 4 feedstuffs according to column

With the exception of one Turkey feed value, the ratios obtained with this microtracer were generally negative (Figure 7). As with the RF-microtracer, the ratios were generally higher for the 8-m column and the range of variation greater (100.7 against 44.2). However, the difference is not quite as clear as with the RF-microtracer. Once again, the values obtained are not proportional to column height.

5.1.6. Comparison of microtracers



	RF	F
Cow	157.3	-58.8
Piglet	166.0	-42.8
Turkey	64.9	41.9
Broiler	120.9	-38.7

Figure 8: Elutriation ratios for both microtracers in the 8-m column for all 4 feedstuffs

The two microtracers clearly behaved in different ways (Figure 8). The ratios for the Dairy cow and Piglet feeds closely matched those obtained with the organic matrix at comparable RVDs.
Conversely, for Broiler feed, the RF ratio was below the value expected based on the RVD, and for Turkey feed, the ratios for both tracers showed no link with RVD, despite this being clearly maximum (Table 3). These two results suggest that the ratio of RVD to segregation is significantly modified when feed cohesiveness increases (for instance, due to an increase in fat content).

6. Conclusions

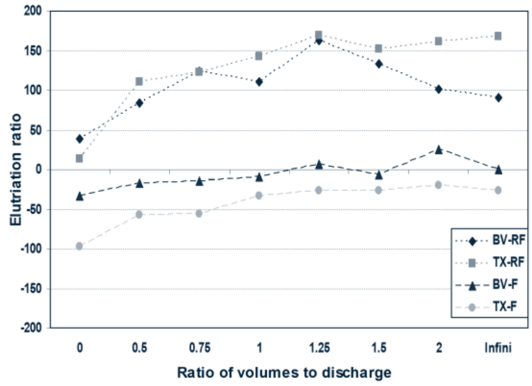


Figure 9: Findings on the change in elutriation ratios according to RVD for both matrices and both microtracers

Figure 9 summarises the results of campaign 1. It clearly demonstrates that segregation varies according to the volumetric ratios between fine and coarse particles.
The finest-grained microtracer (RF) migrated significantly towards the top of the samples while the coarsest particles tended to migrate towards the bottom.
The fine-grained particles of the RF-microtracer showed a more stable distribution in the finest matrices, while the coarsest particles of the F-microtracer was more stable in the coarsest matrices. Tracer particle size and its relationship with the matrix therefore has a major effect.
The range of variation was smaller, approaching zero with the F-microtracer. This suggests that a product with a particle size of 250/300 µm is less likely to segregate in a cattle feed than would a finer-grained product, even given the large difference in density.
Acquired segregation appears to stabilise above an RVD of 1.25; useful insight could be gained by testing for segregation above this value up to an RVD of 1.5 as, in this zone, segregation is more stable and certainly more repeatable.
It should also be noted that the organic matrix appears to amplify these phenomena (especially for the F-microtracer), while limiting percolation effects that could interfere with certain assessments.
The second test campaign revealed that increasing the drop height would increase segregation phenomena in a non-linear fashion. This might be caused by the higher drop speed that relates mass to speed squared.
This second round of tests also confirmed the effect of microtracer particle size.
The demonstration of the limiting effect of cohesiveness on segregation and the impact of RVD suggests that other tests need to be carried out to test the effect of liquid bridges and cohesion on segregation.

7. Bibliography

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