i'Tec_T12
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**Keywords :** Carry-over - elevator

# Pilot study on micro-ingredient carry-over via a bucket elevator : Method

## 1. Focus

This datasheet describes the equipment and methods developed during the POUSSALIM research project (**i'Doc_T16**) in order to assess micro-ingredient carry-over on a bucket elevator. This technical datasheet links up with **i'Tec_T13**.

## 2. Equipment and apparatus

### 2.1. Pilot testing center

The testing centre (Figure 1) comprises a bucket elevator supplied with meal via a screw conveyor. The product is introduced via the feed hopper, evacuated via the screw conveyor, and lastly, carried to its discharge point at the elevator head, where it is directed towards a manually operated by-pass. This system means the product can be directed either towards a discharge hopper, connected to the feed hopper via a mechanical hatch (closed loop operation or product held in the discharge hopper), or towards the discharge pipe where it is recovered in a bin (open loop operation).

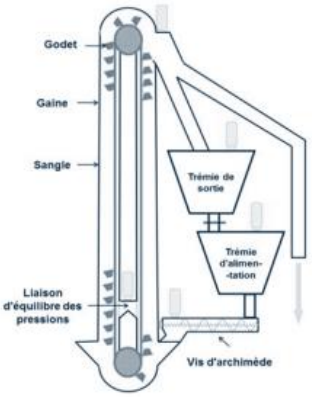


**Figure 1 : Diagram of the pilot testing center**

The head and foot of the elevator, together with its casings, are mostly made of antistatic Plexiglas that provides maximum visibility of the moving product inside the system during operation. In addition, most of the testing centre's component parts can be dismantled in order to sample deposits in every area of the elevator. This adaptability optimises the pilot centre's cleaning capacity, meaning that this configuration maximises control over the pilot centre's initial state of cleanliness for each test. Also, the buckets and belt have been chosen to maintain the system's aeraulics and ensure that the product behaves as it would in an industrial elevator. The space between the buckets and the casing (in the median position) is maintained by preserving the ratio $\frac{Bucket\_Casing\ Space}{Bucket\ widht}$. The angles of the various inclined sections (feed angles, link between strands and product discharge at the elevator head) and the elliptical shape of the head are also preserved in order to ensure an identical product flow. Lastly, the range of available belt and screw speeds are defined so as to provide 3 discharge options at the elevator head: gravity, mixed and centrifugal.

### 2.2. Reference product

#### 2.2.1. Preparation

Feed formulations change over the course of the year and from one plant to another. In order to ensure a product that would have constant physicalchemical characteristics throughout the study, a reference product is therefore developed by mixing corn cobs with varying grain size distributions. The reference product's main physical characteristics (Table 1) reflect average feedstuff characteristics (based on the Tecaliman database) in terms of particle size distribution (Gaussian distribution around 500µm), Hausner ratio and angles of repose.

| | Feedstuff | | | |
|---|---|---|---|---|
| | Standard | Pig | Rabbit | Poultry |
| $D_{50}$ (µm) | 531.2 | 592.6 | 716.6 | 510.8 |
| Hausner ratio | 1.1 | 1.1 | 1.1 | 1.2 |
| Angle of repose (°) | 53.9 | 54.3 | 62.8 | 62.4 |

**Table 1 : Main physical charecteristics of the reference product compared against those of three compound feedstuffs**

The first tests revealed that the reference product, comprised solely of corn cobs, did not behave like the feedstuff in terms of deposition mass and fines (airborne particulate matter) in the elevator following batch throughput (Figure 2). This explains why sunflower oil was added to the reference product at a ratio of 1.5%.

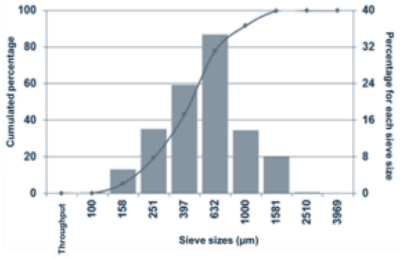


**Figure 2 : Particle size distribution by mass of the reference product-Tecaliman database**

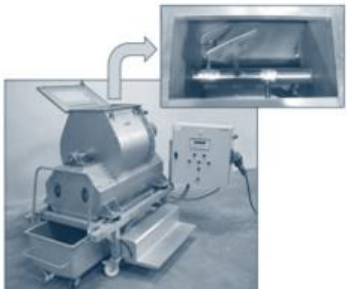

**Figure 3 : Pilot blade mixer**

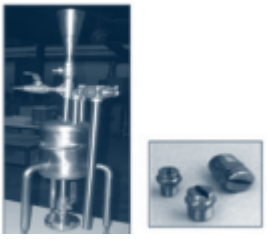

**Figure 4 : Spray tower and nozzles**

### 2.2.2. Assessing oil distribution homogeneity

The mixture is tested to assess its oil distribution homogeneity. Once mixed, the product is tipped into a tray. It is levelled to provide a relatively flat surface, and then quartered into 10 areas in order to take 10 samples from the top section of the tray. The upper fraction of the feedstuff is removed and the same operation repeated on its lower section. Only 10 of the 20 samples taken are analysed (5 from each layer) (Figure 5).

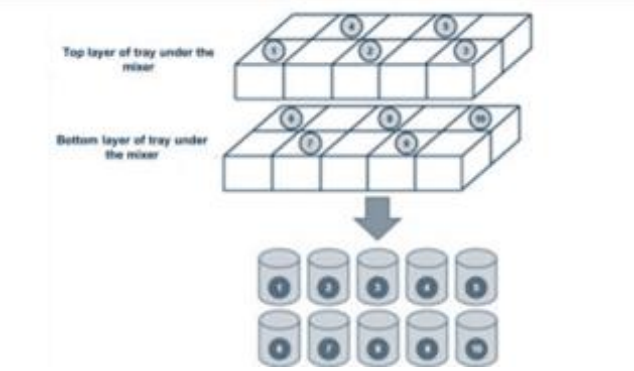


**Figure 5 : Diagram of the quartering method used to check oil distribution homogeneity in the reference product**

Figure 6 shows the oil content in each of the analysed samples. It demonstrates that the oil content in each sample is very close to the desired value (1.5%) with a CV of 4.6%.

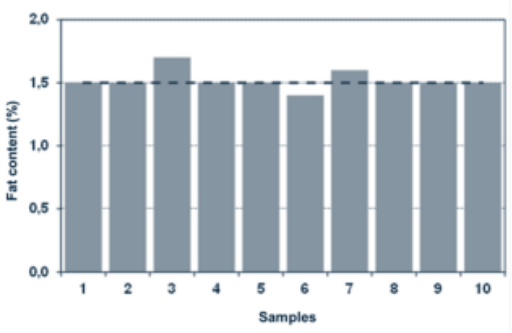


**Figure 6 : Oil distribution homogeneity in the reference product**

## 2.3. Tracer used

RF-blue lake microtracer is used as an external tracer (not used in animal feed production formulations) in order to assess carry-over rates. This microtracer comprises iron particles that have been coated with blue food colorant (Brilliant blue) using gum arabic. Table 2 lists the microtracer's physical characteristics.

| Physical characteristics | |
|---|---|
| $\rho_{bulk}$ | 2754.2 $g.L^{-1}$ |
| $\rho_{tap}$ | 3131.9 $g.L^{-1}$ |
| Hausner ratio | 1.1 |
| ATE* 20 mm (angle of repose [flow]) | 38.1° |
| $D_{50}$ (laser diffraction particle size analysis) | 92 µm |

**Table 2 : RF-blue lake microtracer-physical characteristics**

The tracer's median diameter of approx. 100 µm places it within the fine-particled fraction of the reference product (Figure 2). Despite having a density significantly higher than the additives generally used in the animal nutrition sector, this tracer is largely sufficient for qualifying handling Datasheet No. 96 - June 2018 - Page 3/4 procedures as this characteristic has little, or even no, effect on the mechanisms that drive airborne particle flows. Lastly, it has the advantage of providing easy and cost-effective treatment and analysis of a large range of samples.

# 3. Experimental methods

The standard method used to assess micro-ingredient carry-over at an industrial site requires the system to operate as an open loop in order to take the samples. This modifies the air flow (and, therefore, the flow of airborne particles). A non-intrusive pilot protocol has therefore been developed to avoid this problem. i.e. that does not disturb the system during operation. The carry-over process can be broken down into two phases:

- Product deposition during throughput of batch n,
- Collection, by batch n+1, of part of the product deposited by batch n.

To determine the characteristics of these two phases, a test is broken down into two subtests: "Tracer (Tr)" and "Tracer + Collector (Tr+C)".

## 3.1. « Tracer » subset

This subtest is designed to evaluate the product's tendency to deposit residues during its transit through the bucket elevator. A 50-kg batch of reference product, containing 250 ppm of RF-blue lake microtracer, is added to the feed hopper. The pilot testing centre is systematically cleaned prior to starting the test in order to control initial test conditions. In order to recreate plant conditions as closely as possible, the tracer batch is run through the test set-up three times before being discharged to a bin via the discharge pipe.

## 3.2. « Tracer + Collector » subtest

This subtest is used to reveal the product's capacity to collect deposits already present on the walls and dead spots of the bucket elevator. Two 50-kg batches of reference product are produced. The Tracer batch contains 250 ppm of microtracer. This is then added to the feed hopper and run through the system three times prior to discharge (identical to the "Tracer" subtest). The Collector batch (minus the microtracer) is run through the elevator once only, and then recovered. Figure 7 shows the sequencing of the main steps in the experimental protocol.

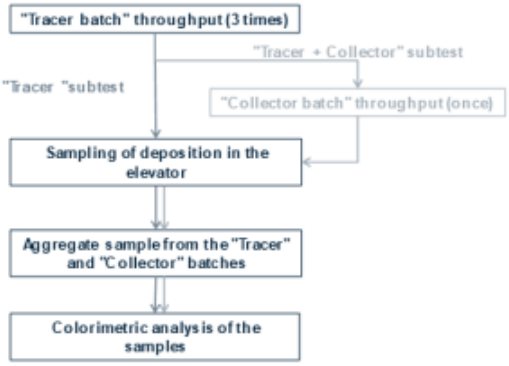


**Figure 7 : Diagram of the experimental protocol**

## 3.3. Output data

Once all the batches have been through the system, several samples are taken for each test. This output data consists of:

- Either, aggregate quantities for post-test batches,
- Or spot quantities, taken from certain, specific, areas in the elevator.

### 3.3.1. “ Spot” data

Several areas of deposition can be identified (Table 3 and Figure 8). The samples are taken directly using a brush or a vacuum bag. These bags allow the passage of particles of under 30 µm in diameter.

However, the reference product does not contain any such particles. 98% of the microtracer particles lie above this value, while the 2% of particles with diameters below 30 µm may agglomerate with others due to incorporation of [1]the oil during the mixing process. This strongly limits the amount of product loss[1] .

| Areas | | Sampling means |
|---|---|---|
| P1 | Foot | Direct |
| Li | Link between the two strands | Suction |
| P2 | Foot rest (feed angles) | |
| GM | Casing and upwards buckets | |
| GD | Casing and downwards buckets | |
| T | Head | |
| R | Rest of the elevator | |
| Tr+V | Hopper + screw | |

**Table 3 : Sampling means according to area**

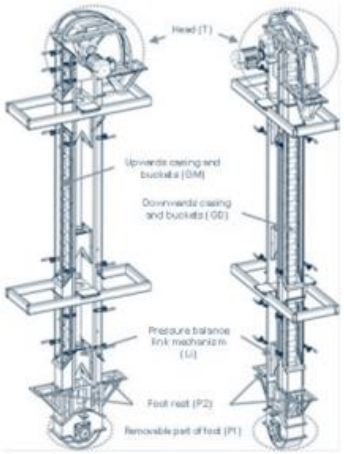

**Figure 8 : Deposition sampling areas**

### 3.3.2. « Aggregate » data

These output data concern the batches as a whole. After each test, the remaining batches are weighed and then halved. This operation is repeated several times using a quarter splitter until obtaining a sample of approx. 700g (Figure 9).

**Figure 9 : Splitter**

[1] Comment: tests were run to quantify product and microtracer losses generated by hoovering and handling of the bags. These tests revealed that product and microtracer variances were not significant.

The resulting samples are referred to as aggregate samples:

- For the tracer batch: Aggregate T,
- For the collector batch: Aggregate C.

### 3.4. Sample treatment and analysis

The vacuumed deposits are recovered by cutting the bag into strips (using scissors) and then removing the product contained within using a brush. The product is then weighed before being split using a riffle splitter (Figure 10).

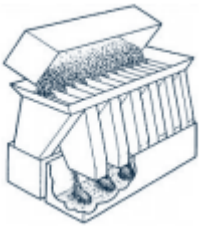

**Figure 10 : Riffle splitter**

### 3.5. Microtracer analysis

The method used to analyse a sample's microtracer content breaks down into three steps. First, the microtracer is extracted magnetically under wet conditions. It is then recovered on a 0.8-µm cellulose nitrate filter. This filter is placed in solution (45% distilled water, 50% pure denatured ethanol (E96) and 5% soda) and then stirred by hand for 30 seconds to dissolve the gum arabic coating on the iron particles. Lastly, the optical density of the coloured solution is measured using a UV-vis spectrophotometer (Shimadzu) calibrated at 629 nm. The sample's microtracer content is then established by calibration curve, which is developed using five samples of virgin standard product to which is added a known quantity of microtracer.
**i'Tec_H17**. describes the microtracer analysis method in full.

## 4. Conclusion

The fact that the testing system can be dismantled and reconfigured means that the experimental testing centre can be used to perform a wide range of carry-over studies. A reference product (elaboration protocol), an experimental method for the "Tracer" and "Tracer + Collector" tests and sampling, treatment and sample analysis protocols specific to this bucket elevator have been developed to assess the amount to which micro-ingredients are capable of generating carry-over.

## 5. Bibliography

**i'Doc_T16**, 2013 - Programme POUSSALIM Étude expérimentale du comportement des aérosols et de leurs dépôts dans un élévateur à godets : Impact sur les transferts inter-lots en alimentation animale
**i'Tec_H17**, 2014 - Méthode d'analyse du microtraceur Rf Bleu Lake après extraction en milieu aqueux