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Measuring the smallest flow diameter found in animal feed additives

Measuring the smallest flow diameter is a fairly standard powder characterisation technique. It is generally performed using a "FLODEX" brand appliance. The measurement protocol designed by TECALIMAN can be used to streamline the results process.

1. Principle

The principle is to identify the smallest diameter of a circular aperture through which powder can flow. First, a cylindrical metal container is filled with powder via a funnel. The lower part of this cylinder is successively obstructed by a removable metal disc pierced by an aperture and by a valve. The operation consists in inserting a removable disc with a known aperture diameter, filling the container with powder, activating a handle to open the valve and observing whether the powder flows through the disc aperture.

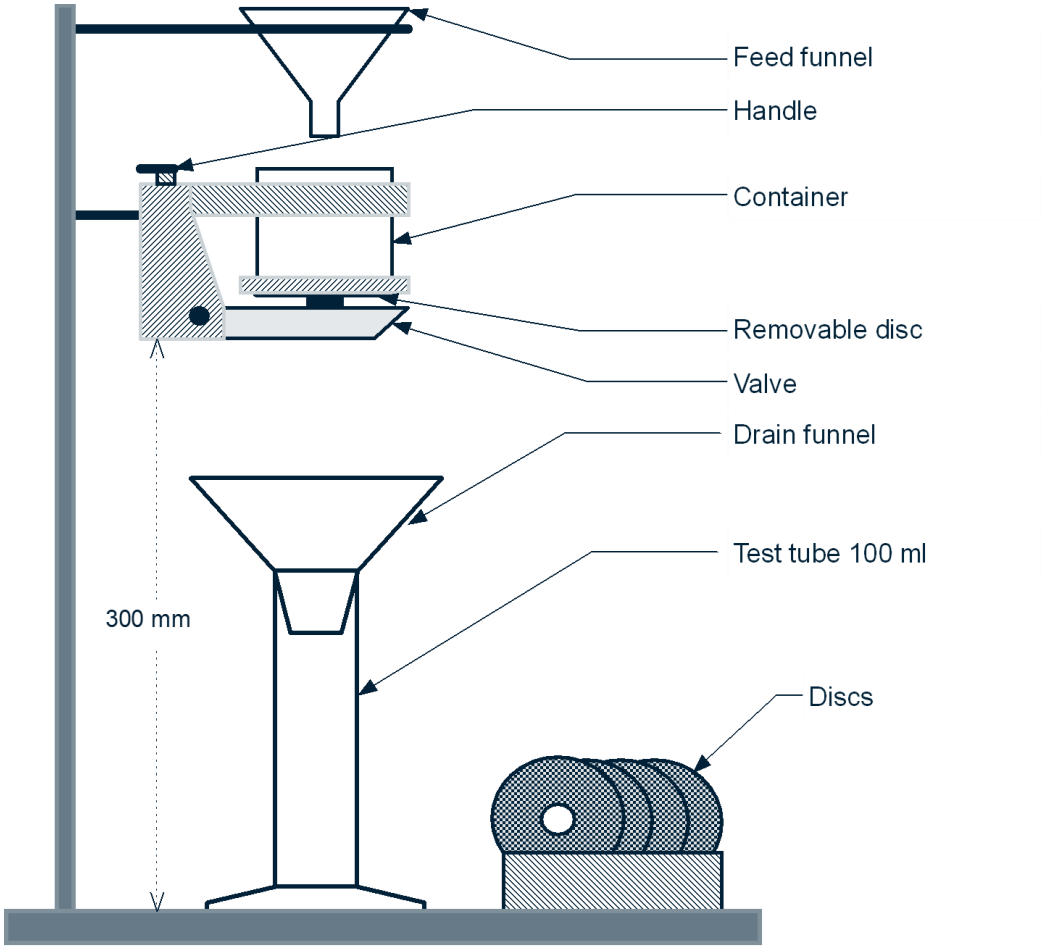

Figure 1: Diagram of the apparatus used to measure the smallest flow diameter

2. Apparatus

Figure 1 shows a diagram of the apparatus.

A stand is used to position the assembly 30 cm above the base. The feed funnel is positioned so that its bottom end is on a level with the top of the container. The disc holder at the base of the container can be removed in order to change the disc. The company that markets this equipment supplies discs with aperture diameters ranging from 4 mm to 32 mm. It recommends starting the measurements at a diameter of 16 mm. To increase the measurement spread, TECALIMAN had other discs made with aperture diameters ranging from 32 mm up to 42 mm.

To take the measurement, a 100-ml test tube and a second funnel is also needed.

3. Operating procedure

Each measurement requires a minimum quantity of 100 ml of powder. The operating procedure consists in:

- activating the handle to release the valve and remove the disc holder.
- placing the 16-mm disc in the disc holder and then positioning the disc holder.
- filling the 100-ml test tube with the powdered product.
- slowly pouring the 100 ml of product into the container through the feed funnel.
- placing a funnel and the 100-ml test tube under the valve.
- activating the handle to open the valve.
- observing the volume of powder collected in the test tube without compacting.

When opening the valve, care should be taken to prevent generating any vibration in the assembled structure. The measurement should also be taken in a vibration-proof room.

Every measurement taken on a product will include 2 phases:

- the selection of the base diameter
- the taking of additional measurements

The base diameter is that which allows the collection of a powder volume of between 45 ml and 55 ml or of a slightly larger volume if the poured volume crosses the 45/55 ml interval in one go.

If the collected volume lies outside this range, the operating procedure should be restarted with a correspondingly smaller or larger diameter.

Once the base diameter has been selected, additional measurements can be taken with this diameter. Where necessary, this may lead to collected volumes that are greater than 55 ml or less than 45 ml. In this case, the result will be determined according to the instructions given in the next chapter.

4. Expression and interpretation of the results

The disc aperture diameter and the volume of collected powder are used to identify the smallest flow diameter or "SFD" (Table 1). While the end result is always an aperture diameter expressed in millimetres, the value may be that of a disc that is not included in the series.
It is easy to see that product flowability decreases with increasing diameter.

Volumes	Decisions
35 ml →	
45 ml →	Diameter immediately above the base diameter
55 ml →	Mean value between the base diameter and the diameter immediately above
65 ml →	Base diameter
	Mean value between the base diameter and the diameter immediately below
	Diameter immediately below the base diameter

Table 1: SFD decision table

5. Intrinsic qualities

The qualities intrinsic to the method (sensitivity, reproducibility, accuracy) were determined for 5 representative animal feed additive products. Five series of tests were performed at four-day intervals; the measurements were repeated 5 times on each product during each test campaign. The results demonstrate that this is a sensitive method (Table 2).

Products	1	2	3	4	5
SFD (mm)	27.4	32.6	21.5	6.3	26.8
Group	*b*	*a*	*d*	*e*	*c*

Table 2: Results of the test on the method's intrinsic qualities

The method is highly accurate (1.65%), meaning that a single measurement may provide a reliable

result for the smallest flow diameter. The method does, however, have reproducibility issues that may be linked to weather conditions. This defect does not, however, cause any significant variation in product ranking.

6. Range of animal feed additives

Based on the 30 representative additives (i'Doc_Q2), this method (along with the chosen protocol) provides a large measurement range with nearly 38 levels between the minimum maximum values. Fifty percent of products lie within an interval spanning 21 levels.
The distance between the mean and median values should also be noted; this can be explained by the non-Gaussian distribution of the frequency histogram (Figure 2). A study of how the products were distributed over the measurement range revealed a binary distribution lying on either side of a diameter of approx. 16 mm. This would seem to suggest that there are two additive populations.

	mm
Mean	22.6
Standard deviation	11.2
Minimum	3.0
Maximum	40.7
Min./max. difference	37.7
Median	26.0

Table 3: Statistical report on the range of 30 representative products

The products under the 16-mm diameter value are:

- 4 coccidiostatics,
- 4 vitamins
- 1 oligo-element.

However, even if a low diameter indicates good flow and that the 16-mm diameter is recommended as the starting point when determining the base diameter, it should not be inferred that this diameter demarcates the limit between superior and inferior-flowability. It should also be noted that as the median lies principally around a diameter of 26 mm, it may make more sense to take this last value into account.

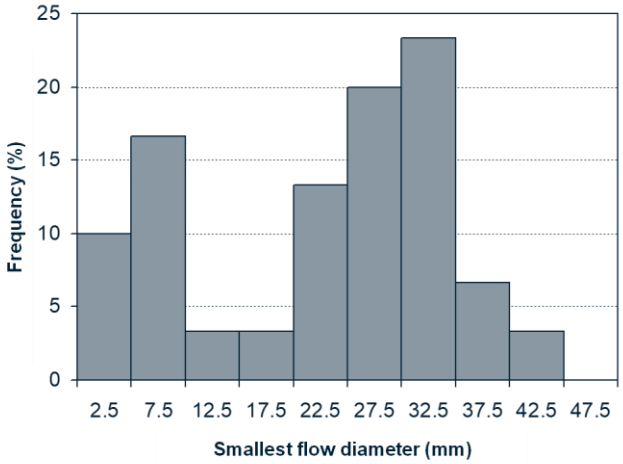

Figure 2: Frequency histogram

7. Discriminating power and redundancy

This method has a power similar to the angle of repose (flow), but lower than laser diffraction particle size analysis or bulk density or tap density.
Out of all the flow behaviour measurement methods tested by Tecaliman (Hausner ratio, angle of slope, angle of spatula, etc.), this method, with its angle of repose (flow), demonstrates the best discriminating power.
The relationship between these two methods is similar for products at the lower end of the scales.
Values for the upper part of the scales are scattered over a wider range; neither method can be used to predict the result of the other (Figure 3).
It is also interesting to note that it is often the same products that show strong fluctuations in results faced with both methods (horizontal or vertical bars).

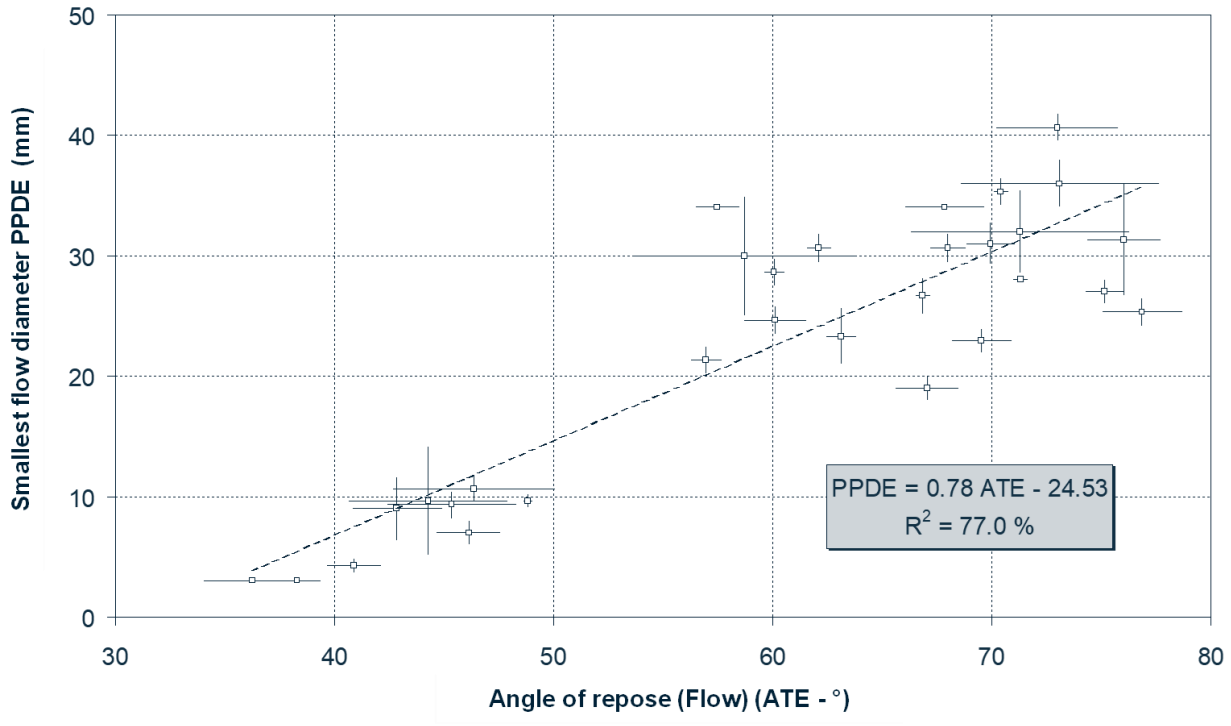

Figure 3: Relationship between the angle of repose (flow) and the smallest flow diameter for the range of thirty representative additives

8. Conclusions

While there is an appliance (BIOBLOCK [1]-Flodex) for measuring smallest flow diameter on the market, additional discs are nevertheless required.
This measurement may be considered simple to implement, even if the protocol initially appears difficult to apply. The main disadvantage concerns the base diameter identification phase. However, in cases where only a single measurement is taken, it is the measurement itself that identifies this diameter. Fifteen minutes could therefore be considered a fairly average time for obtaining a result.

The smallest flow diameter method can be defined as sensitive and accurate, but also as difficult to reproduce. The method is a little complicated, but can be adapted to fit industrial conditions. One straightforward way of assessing the quality of a product at plant input is to refer to the diameter previously assigned to the same accepted incoming product. A fluctuation margin of one, or even two diameters above could therefore be allowed for product acceptance.

Nevertheless, this measurement could be improved by:

- using discs with apertures that go down to 1 mm.
- designing a larger piece of apparatus that can include diameters greater than 44 mm.
- having aperture diameters every millimetre.
- designing stronger discs that have a longer life-time.

9. Bibliography

Tecaliman report No. 9, 1998. Assessment of the internal quality of laboratory methods used to characterise the additives used in animal feeds - Phase 2a.

i'Doc_Q5, 1998. Summary of the programme for predicting the technological behaviour of additives in industrial environments

[1] Bioblock, Parc d'innovation - Strasbourg - Illkirch - BP11, 67 403 Illkirch cedex