i'Tec_Q3
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Measuring the angle of repose (flow) found in animal feed additives

Tecaliman carried out the first angle of repose measurement tests based on a technique described in standard NF T 73-008 (1978) that we refer to as the angle of repose (distribution). The results obtained using this method initially designed for powder and pellet surface agents demonstrated that the method was not suitable for use with animal feed additives. This angle of repose measuring technique required products to flow during their distribution even if they had very limited flowability.
In his 1974 thesis, Devise described various pile formation techniques used to measure what he referred to as "drained angles of repose". One of these techniques was based on the use of a cylinder placed on a flat surface. Removing this cylinder made it possible to form a pile with no need for the tested products to flow.
This technique gave rise to the method and apparatus described in this datasheet. The apparatus was designed jointly by TECALIMAN and INRA IN Nantes; the measurement protocol was developed by TECALIMAN.

1. Principle

The method consists in creating a cone-shaped pile of powder and measuring the angle formed at the base of the cone (**angle α**).
The sample of test product is placed in an open-ended cylinder which is itself placed on a flat circular surface. The pile is obtained by letting the powder flow by raising the cylinder vertically.

2. Apparatus

The whole apparatus was placed on a horizontal **slab** (Figure 1). The **cylinder** was raised up vertically using a winch. The movement's verticality was ensured by a rail fitted in **stand** perpendicular to the slab. The cylinder's travel speed was adjusted using the **variable speed control**. The **baseplate**, placed in the centre of a **basin**, had diameter 100 mm.

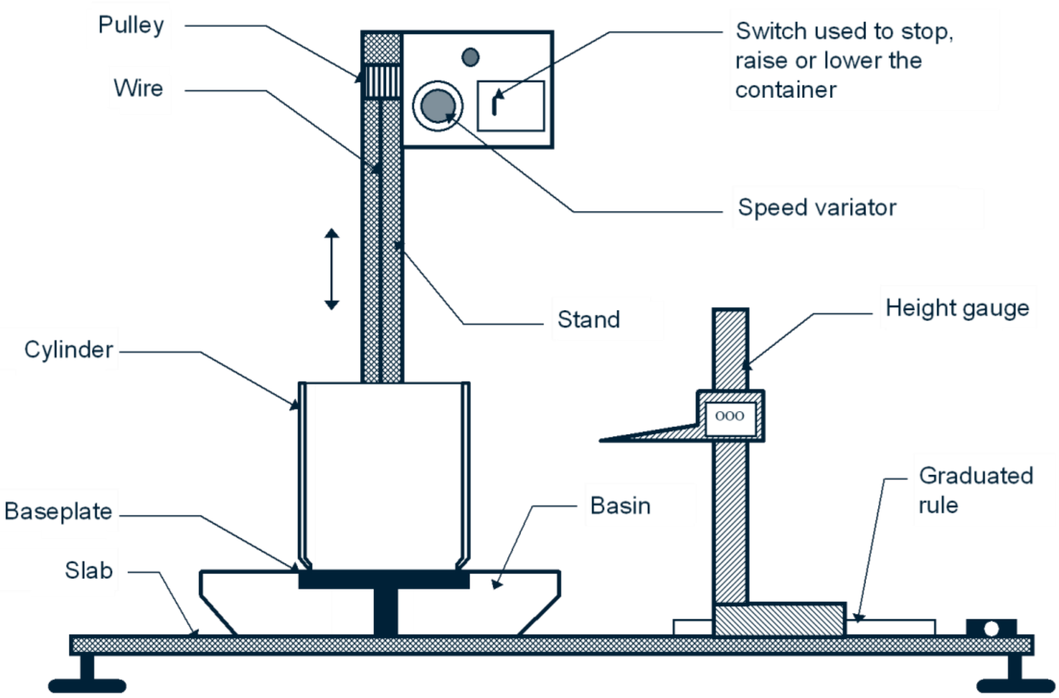

Figure 1: Diagram of the apparatus used to measure the angle of repose (flow)

Its upper surface, ribbed with concentric circles, had an internal rim of 10 mm. This system allowed the product to overflow the baseplate during formation of the pile. A mobile **height gauge** that moves horizontally along a graduated **ruler** was used to measure the pile's height property "**h**".

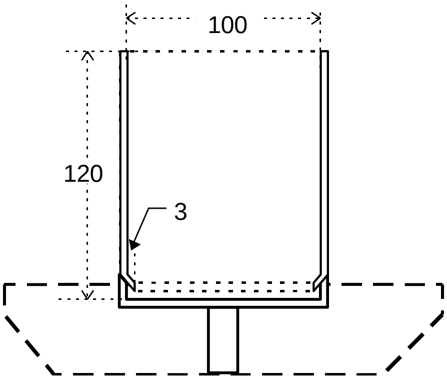

Figure 2: Cylinder cross-section

Cylinder dimensions are given in Figure 2. The 2-mm recess at its base:

- allows the cylinder to slot into the baseplate, which had an opposing recess.
- erodes the sides of the product load, in order to limit the risk of forming "blocks" that would have an angle of 90°, and would limit the measurement.

3. Operating procedure

The volume of powder required to take a measurement corresponded to filling the cylinder to the top i.e. a maximum amount of 942 ml. The filling operation was performed by carefully placing primary product loads using a metal hand. This deposit was placed on the cylinder's internal walls.
Filling stopped once the total load of powder approached the top of the cylinder. This means that the total quantity of powder poured out may have varied slightly. The product was not levelled so as to avoid any risk of compaction.
The cylinder was raised on speed setting 3 on the speed control cursor, which corresponds to 19.6 m/h.
The measured angle was the maximum angle of the base of the cone. The operator took the measurement as follows:

- the height gauge was placed at the edge of the baseplate and reset to 0.
- the height gauge was raised.
- the height gauge was moved horizontally along a distance of **10 mm** (Figure 3).
- the cone's highest point was identifed by rotating the baseplate on its base.
- the gauge was lowered till it reached the slope, without touching it.
- it was checked that this was the highest point by rotating the baseplate on its base, adjusting the height of the gauge if necessary.
- the height **h** was read on the gauge's digital display.

Where possible, this measurement should be taken under controlled ambient conditions and, especially, within an environment that is fully insulated against vibrations.

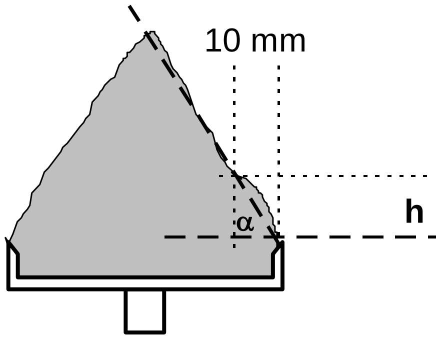

Figure 3: Measuring the angle of repose (flow)

4. Expression and interpretation of the results

The height **h** was expressed in mm, and was accurate to 0.02 mm. The angle of repose α in radians was obtained by the following trigonometric formula, derived from the Pythagorean theorem:

$$\alpha = \text{arctangent } (h/10)$$

This angle was expressed in degrees so as to make it easier to read and to compare against other angles. Mathematically, the accuracy of 0.02 mm on the height gauge gave an uncertainty of 0.11°. This uncertainty did not, however, factor in the operator's assessment error concerning height gauge/additive contact.
From an interpretative point of view, it is easy to see that product flowability decreases as the angle increases.

5. Intrinsic qualities

The method's intrinsic qualities (sensitivity, accuracy, reproducibility) were determined for 5 very different products, using measurements that were repeated 5 times at four-day intervals. The tested products were chosen as being representative of the type of additives generally used in animal feed. The results demonstrated that the method has good sensitivity (Table 1).

Products	1	2	3	4	5
Angle (°)	63.0	68.6	61.9	45.5	74.0
Group	*c*	*b*	*c*	*d*	*a*

Table 1: Test results for the method's intrinsic qualities

The method is accurate to 2.96%, enabling a reliable datum to be obtained with just **2 measurements**. The angle of repose (flow) is a method that can be reproduced over time.

A population of 30 representative additives (see i'Tec_Q2) was formed and then divided into two groups, based on their angle of repose: **α <45°** and **α > 55°** (Figure 4).

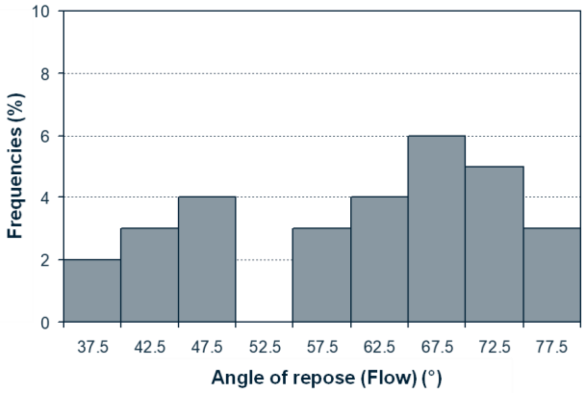

Figure 4: Frequency histogram

This distribution explained the slight difference between the mean and median values for the 30 additives (Table 2). It is not possible to state with certainty that this difference represents the boundary between favourable and unfavourable flow properties.

Mean	60.1°
Standard deviation	12.6°
Minimum	36.3°
Maximum	76.9°
Min./max. difference	40.6°
Median	62.6°

Table 2: Statistical reporting on the range of 30 representative products

The measurement range had a spread of over 40° and the angles were often greater than those observed with other methods.

7. Discriminating power and redundancy

In terms of additive differentiation, the angle of repose (flow) has a discriminating power comparable to that of the smallest flow diameter but less than that of laser diffraction particle size analysis or bulk density.
Conversely, this angle has the best discriminating power out of all the flow behaviour measurement methods trialled by TECALIMAN (Hausner ratio, angle of slope, angle of spatula, etc.). While this method is redundant with that of the smallest flow diameter in the lower part of their respective scales (Figure 5), there are significant differences in the upper part of the scales.

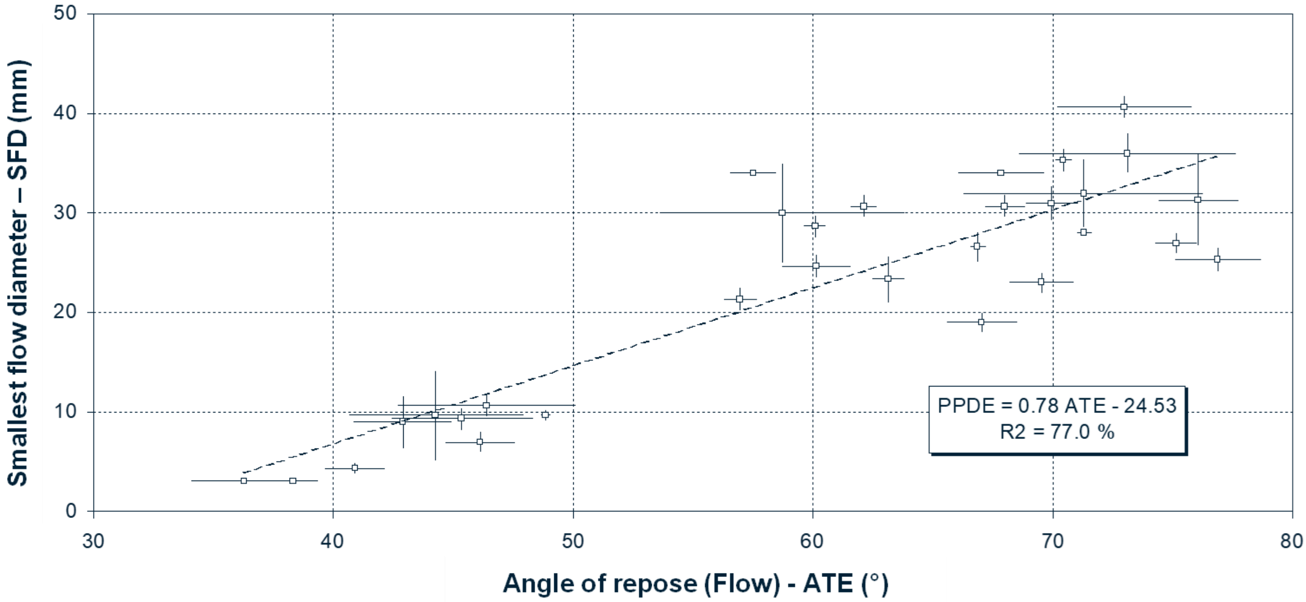

Figure 5: Relationship between the angle of repose (flow) and the smallest flow diameter for the range of thirty representative additives.

There is also a fairly close relationship between this angle of repose and the angle of spatula measured using a HOSOKAWA appliance (Figure 6). However, the angle of repose (flow) method has the advantage of superior accuracy and discriminating power.

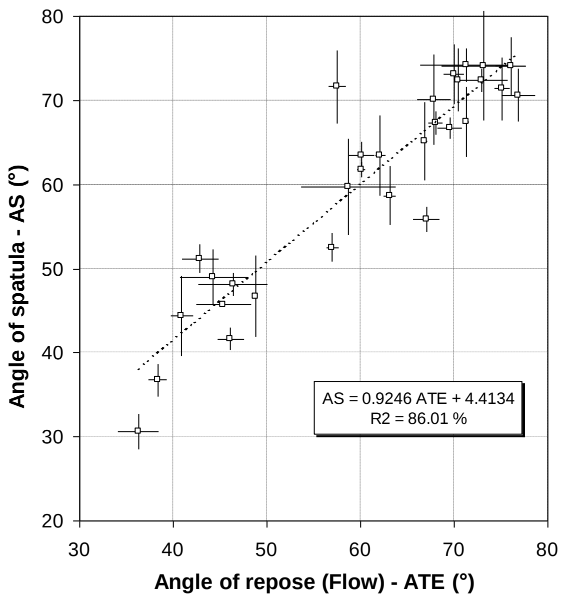

Figure 6: Relationship between the angle of repose (flow) and the angle of spatula for the range of thirty representative additives

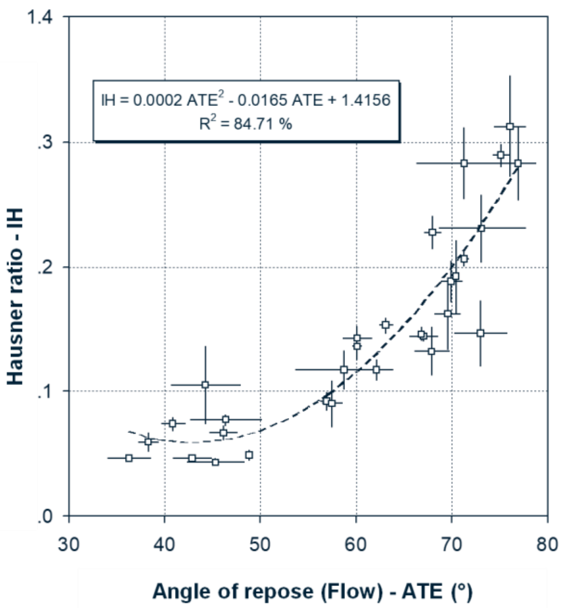

Figure 7: Relationship between the angle of repose (flow) and the Hausner ratio for the range of thirty representative additives

The results revealed a curved line relationship with the Hausner ratio (Figure 7). A quadratic equation linking the two parameters was assigned a coefficient of determination of 84.7%.
This relationship can be interpreted based on the existence of a threshold of approximately 60° for the angle α above which the Hausner ratio starts to increase above 1.1. Therefore, in the lower part of the scale where the Hausner ratio is always equal to 1.1, the angle α increases by approx. 25° between 35 and 60°. In this section, the angle of repose (flow) is therefore markedly more discriminating than the Hausner ratio.
In the upper part of the comparative measurement ranges, all the points are distributed within scatter plots. None of the methods classified the products in a similar and uniform manner.

8. Conclusions

It is very easy to measure the angle of repose (flow) While the difficulty in forming a pile that is sufficiently conical, as seen with the protocol for angle of repose (distribution), appears to have been largely resolved, certain behaviours such as the formation of clumps may hinder correct measurement.
The average time needed to take a basic measurement is short, i.e. approx. 15 minutes. The main difficulty lies in getting hold of the appropriate apparatus as there is none actually on the market. This new method for measuring angles of repose provides a means of taking measurements that are accurate, sensitive and repeatable. In practical terms, the objective of forming correctly centred piles with each product was achieved. This is a simple, low-cost technique that would be a good candidate for a method used in plant product acceptance tests.
The continued formation of powder piles of varying shapes means it is still necessary to measure the maximum height on the pile slopes. The size of the angles on some piles requires the height h (used to calculate the angle) to be measured at a distance of 10 mm from the edge, compared to 20 mm for the angle of repose (distribution). This means that an incorrect height measurement has a greater impact on the angle assessment error. However, despite this disadvantage, this method has nevertheless been proved effective.

9. Bibliography

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