

Measuring the hardness of pellets using a "SCHLEUNIGER" device

1. Context and aim of the study

Hardness is a measurement of the compressive strength of pellets when they are subjected to a compressive force. This measurement enables the cohesion of the pellet (compressive strength) and the force applied by the animal when chewing to be assessed.

In France, a number of methods are used to measure this feature of pellets: the Kahl, Sodexim and INRA methods (Tecaliman 1994). In Switzerland and Holland, the animal feed sector has been using a method for measuring the hardness of pharmaceutical tablets (the Schleuniger 6 D method) for several years, however no results have been published on the application of this method for compound feed pellets.

The aim of the study undertaken by Tecaliman and the Coopérative de Broons (22) is to determine the performance of the Schleuniger 6 D method when measuring the hardness of compound feed pellets.

2. Equipment and methods

2.1. Principle

The method used to assess the performance of the Schleuniger 6D 400N (S6D) device is similar to that proposed in AFNOR standard V03 - 110 - Assessment protocol for an alternative quantitative analysis method in comparison with a reference method. Consequently, four actions are undertaken:

- Calibration and assessment of the linearity of the "S6D" device
- Assessment of the repeatability of measurements obtained using the "S6D" device
- Examination of the correlations between the values obtained using a reference method and those obtained using the "S6D" device.
- Examination of the correlations between two ways of using the "S6D" device.

2.2. Expressing hardness results

Hardness is generally expressed as a kilogram-

force (kgf) or in Newtons.

To compare the various measurement methods and the different batches of pellets, it is necessary to use a common mode of expression (Melcion 1981), i.e. to take account of the length of the pellet on which measurement is being undertaken and the diameter of the pellet. This results in the megapascal (Mpa) being used as the unit for expressing the hardness of pellets.

$$\text{Hardness en Mpa} = \frac{\text{hardness measured} \times 2K}{L \times 3.14159 \times D \times 1000}$$

or

$$K = 1, \quad \text{if the hardness measured is in decanewtons}$$

$$K = 0.98665, \quad \text{if the hardness is expressed as a kilogram-force}$$

$$L \text{ (cm)} = \text{Length of the pellet or pellets subjected to the compressive force}$$

$$D \text{ (m)} = \text{diameter of the pellet.}$$

2.3. Schleuniger method

A pellet is placed horizontally on a tray, between two vertical plates. The first plate or plunger is mobile and is equipped with a force sensor.

By moving, it compresses the pellet against the fixed plate or jaw. The compressive force is analysed during the course of this operation, which enables the pellet's breaking strength to be determined. This value is recorded and displayed (it is expressed in newtons). The device can be connected to a printer and the data processed using statistical software.

Several types of jaws can be used: a pointed jaw (fig. 1), a flat jaw (fig. 2) or a square jaw (fig.3).

The flat jaw is longer than the pellet, as a result, in order to determine the hardness of pellets in Mpa, it is necessary to measure the length of pellets (L) using slide calipers.

The square jaw, produced for the purpose of the study, is the same length as the plunger on Kahl pliers (0.64 cm). As this length is generally less than the length of pellets, the length L on which the measurement is taken is, therefore, constant and equal to 0.64 cm.

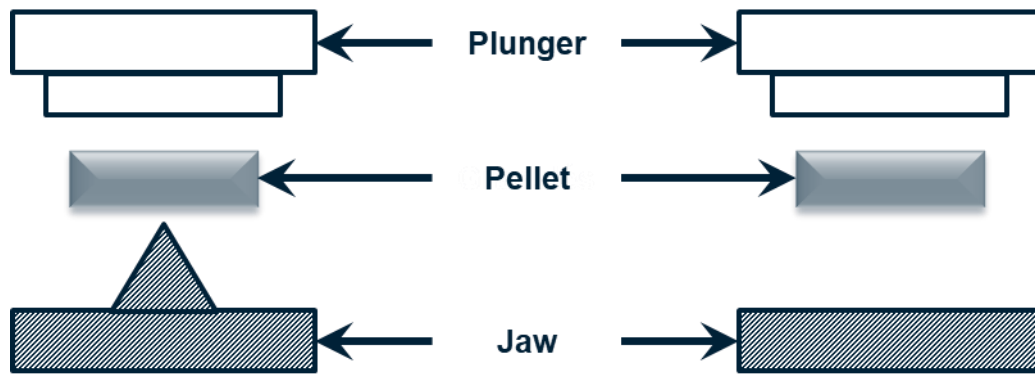


Figure 1: pointed jaw Figure 2: flat jaw

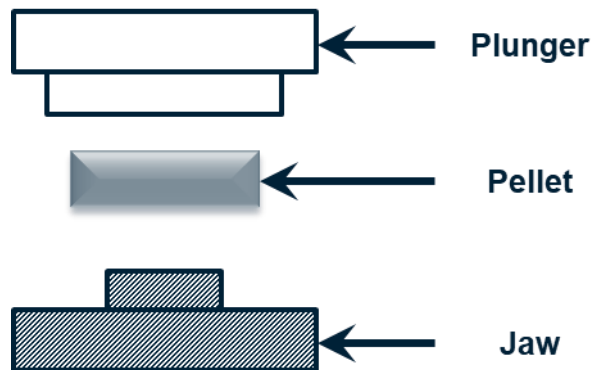


Figure 3: square jaw

The number of pellets to be analysed per sample is determined as analysis progresses, resulting in accuracy of 5% for the average hardness of the sample.

Depending on the variability of pellet hardness within a sample, the number of pellets characterised is between 40 and 70.

2.4. Calibrating the Schleuniger device

Calibration is a necessary operation for preventing the hardnesses measured during a routine operation being marred by errors, caused by drift affecting the sensor or the device's electronics. Calibrating the "S6D" device is a quick and easy operation. It is carried out by positioning the device vertically and placing calibrated weights on the force sensor (plunger) with the aid of a suitable tool. Subsequently, during measurement, the date of the last calibration appears on each document printed.

2.5. Assessing the linearity of measurements produced by the Schleuniger device

The aim of this part of the study is to assess the device's ability to provide results that are commensurate with the hardness of the sample, over its entire measuring range.

The method consists of placing the device in the calibration position and using calibrated weights to

establish a link between the weights measured and the values read. The statistical processing of results, the correlation coefficient, the regression line and the gradient of the regression line enable the linearity of the measurement method to be judged.

2.6. Assessing the repeatability of the Schleuniger method

Repeatability is defined as the closeness of measurement results obtained in the same laboratory, using the same method, on an identical material (pellet) subject to measurement by the same operator, using the same equipment over a short period.

The protocol needed to determine the repeatability value (r) consists of analysing 50 samples in duplicate, of calculating the standard deviation of repeatability (Sr) based on the differences in absolute values between duplicates and of calculating r using the following formula:

$$r = 2.8Sr.$$

The repeatability value r is the value below which it can be considered that the absolute difference between two measurements on the same sample (pellet) has a probability of 95%. In practice, this means that two individual hardness results, obtained for the same sample, must be regarded as suspect if they differ by more than r and that a

repeatable method is a method for which 95% of the absolute variances between 2 values in a pair are less than or equal to r .

Application of this method requires measurements to be taken in duplicate for 50 samples or pellets. Given that:

- hardness measurement for pellets is destructive and that it is, therefore, impossible to conduct the same measurement twice on the same pellet
- in a batch of pellets from the same production run, the variability of pellet hardness is significant, the repeatability study is carried out using calibrated weights on the device in its calibration position, to this end, measurements are taken in duplicate for 50 masses of between 3 and 50 kg.

2.7. Examination of the correlations between the values obtained using a reference method and those obtained using the Schleuniger device

2.7.1. Reference method

There are two reference methods, the Kahl method and the INRA method (Melcion 1981). For reasons of speed and in order to control the sources of operator errors, the INRA method is chosen as the reference method (Tecaliman 1994).

The equipment used comprises a Lhomargy CM12 compression tester connected to a plotter. This set-up enables the hardness of pellets in a test sample comprising 20 cm of pellets (i.e. approximately 20 pellets) to be measured. Measurement is repeated 6 times for each sample, i.e. a total of approximately 120 pellets for each sample to be analysed.

2.7.2. Pellets

The pellet samples originate from batches of feed produced at the plant operated by the Coopérative de Broons. Each sample, with a mass of

approximately 1.5 kg, is taken in a single operation, at the cooler outlet, when the pelleting line is running in a steady state.

The pellet diameter is 5 mm and the majority of them originate from a pig feed batch.

3. Results

3.1. Linearity of the S6D device

The Schleuniger 6D 400N device's response is linear. In effect, the results obtained reveal:

- that there is a linear correlation ($y = ax + b$) between the hardness of samples (calibration weight) and the value measured by the S6D.
- that the gradient of the line is close to 1 ($a = 1.001$),
- that this line virtually passes through the origin ($b = -0.003$)
- that the determination coefficient R^2 is equal to 0.9999.

3.2. Repeatability of the S6D device

The repeatability value (r) is equal to 0.39. The number of variances between pairs, below r , is 48 - i.e. 96%. As this percentage is above 95%, the method can be deemed repeatable.

3.3. Examination of links between hardness measurement methods

Table 1 presents a summary of existing correlations between the different hardness measurement methods. Links between the reference method and the S6D method are weak (R^2 equal to 0.59 and 0.81) while those between the two ways of using the S6D device are strong ($R^2 = 0.95$).

It appears that measuring the length of the pellet, which is necessary to carry out hardness measurement in the case of INRA and S6D-flat jaw methods, is the source of errors, which adversely affect the quality of the correlation between these two methods.

Comparison	Number of samples analysed	R^2	Estimating equation
S6D pointed jaw/INRA	33	0.59	$Y = 2.763 X + 8.434$
S6D flat jaw/INRA	33	0.81	$Y = 1.499 X - 0.385$
S6D square jaw/ S6D pointed jaw	22	0.95	$Y = 0.527 + 0.271$

Table 1: Correlation between the different measurement methods

4. Conclusion

The Schleuniger method satisfies the mandatory criteria for an analytical method (good repeatability and linearity, quick and easy calibration), therefore

it can be used to measure the hardness of pellets in animal feed.

Of the three types of jaws used (pointed, flat, square), the square or pointed jaws appear the best

suited, as they enable measurements to be taken without needing to measure the length of pellets, which reduces the sources of errors and the time needed for measurement.

In comparison with the pointed jaw, the square jaw has the advantage of functioning in conditions like the INRA method and, to the best of our knowledge, it can be viewed as superior to the latter, as it is faster, less expensive and more accurate (Table 2).

To confirm these results, it would be interesting to conduct an additional study by:

- comparing the Schleuniger 6D method with the Kahl method
- using pellets with low hardness, as these are products that are hard to characterise using the usual methods (e.g. Turkey growth feed)
- assessing the possibility of using the Schleuniger 6D method as the reference method for animal feed.

Methods	Total number of pellets to be characterised for each sample with an accuracy of 5 to 6%	Time needed to measure the hardness of a sample of pellets	Cost of equipment in Francs (excluding VAT)
INRA	120	24 minutes	
S6D pointed or square jaw	40 to 100	7 to 17 minutes	30,500
S6D flat jaw	40 to 100	14 to 34 minutes	30,500
Kahl (manual)	55 to 100	30 to 54 minutes	
Kahl (motorised)	55 to 100	23 to 42 minutes	

Table 2: Measuring the hardness of pellets - Cost calculation aspects

5. Bibliography

J.P. Melcion 1981, Prévion de la valeur nutritive des aliments des ruminants, INRA Publication, 307-323.

Tecaliman 1994, Report no. 38 - Comparaison de différents appareils de mesure de la dureté des granulés.