i'Tec_L1
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Results of pilot tests on spraying liquid additives on pellets by load

1. Focus

The aim was to investigate the distribution profile of an active solution incorporated into a mixer according to various methods onto animal feed in pellet format. The stimulated factors were:

- The quantity of incorporated solution
- The concentration of the incorporated solution
- Pellet durability
- Incorporation flow rate

2. Principle

In order to simulate the behaviour of liquid additives dispersed in an aqueous medium, a coloured tracer solution was sprayed over separate batches of pelleted poultry feed under a range of different conditions. This operation was performed by fitting a spraying device with adjustable flow nozzles onto a pilot blade mixer.
The stimulated conditions were as follows:

- Quantity of incorporated solution from 5 to 25 ml, i.e. from 100 to 500 ppm
- Concentration of incorporated solution from 10 to 30 g/l
- Three pellet durability grades
- Incorporation flow rate by adjusting the sprayer nozzle from 0.50 to 2.00 gal/h

3. Equipment and apparatus

3.1. Feedstuff

The test used poultry feed in pellet format at 50 kg per load.
Following preliminary testing, poultry feed pellets were produced with three different durability grades.

3.2. Colorant

Green S or **Brilliant Green BS** or **Lissamine Green B** is a food colouring agent, with molecular formula $C_{27}H_{25}N_2NaO_7S_2$ marketed in the form of small green crystals. It is cited in the positive list of additives permitted in animal feeds (E 142), as its presence is tolerated. Depending on the pH, its colour varies from Yellow (acidic pH) to green and then to blue-violet (alkaline pH). It is soluble in water and ethanol.
Its maximum concentration in an aqueous solution is 30 g/l.

3.3. Test apparatus

This comprises (Figure 1):

- One 100-litre mixer: horizontal static water drop mixer with internal axial bladed shaft
- One single-point compressed air injection system, fitted with a pressure sensor
- Nozzles of various sizes

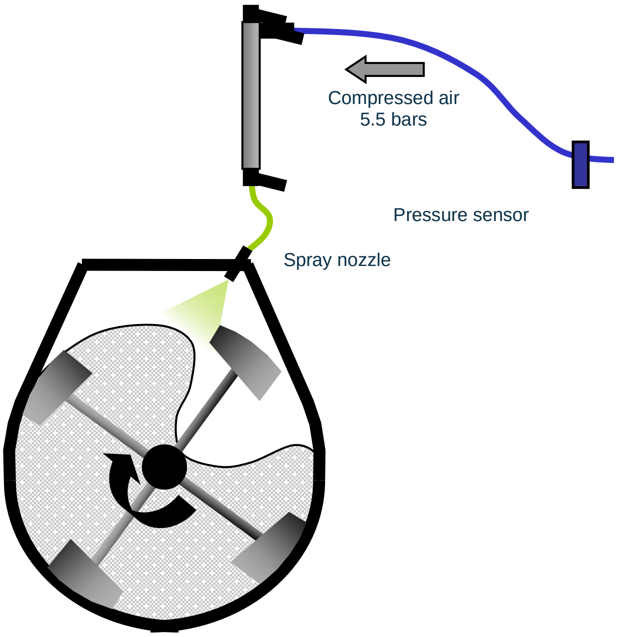

Figure 1: Diagram of the spraying system

4. Method

4.1. Preparation of the Green S solution

The tracer solutions were incorporated under the same conditions as the liquid additives at 100, 300 or 500 ppm in a 50-kg mix, i.e. 5, 15 or 25 g of tracer solution.

Solubilized tracer concentrations therefore had to be calculated so as to ensure that the quantity of tracer in 25 g of solution was sufficient to enable their analysis in samples of approx. 20 g.

Green S solutions at 10, 20 and 30 g/l were prepared by dissolving 0.5, 1 or 15 g of Green S in graduated flasks of 50 or 500 ml filled with distilled water. The tracer was weighed directly in the flask. The flask was made up to the mark, and then stirred gently with a magnetic stirrer.

4.2. Test plan

The test plan described in Table 1 stimulated the following parameters:

- The quantity of incorporated solution: 5, 15 and 25 ml, i.e.100, 300 and 500 ppm with maximum pellet durability and a nozzle at 1.25 gal/h
- The concentration of the incorporated solution: 10, 20 and 30 g/l with maximum pellet durability and a nozzle at 1.25 gal/h
- Pellet durability: 3 grades (A, B and C) - Nozzle at 1.25, solution at 30 g/l incorporated at 500 g/t, i.e. 25 ml/50 kg
- Incorporation flow rate: nozzle at 0.50, 1.25 and 2.00 gal/h - Solution at 30 g/l incorporated at 500 g/t, i.e. 25 ml/50 kg over maximum durability pellets (C)

The key test (500 ppm of solution at 30g/l sprayed with a nozzle at 1.25 gal/h over maximum durability pellets) was repeated twice, as was the test using a nozzle at 2 gal/h.

Nozzle 1.25 Max. durability		**Solution incorporation rate**		
		100 ppm	300 ppm	500 ppm
Concentration of the initial solution	10 g/l			X
	20 g/l			X
	30 g/l	X	X	X X

Solution 30 g/l at 500 ppm		**Durability**		
		A (74.0*)	B (82.7)	C (90.1)
Nozzles	0.50 gal/h			X
	1.25 gal/h	X	X	X X
	2.00 gal/h			X X

Table 1: Test plan (* durability - Eurotest)

4.3. Spraying conditions

The solutions were sprayed onto the pellets via a compressed air system. A pressure sensor was fitted on the compressed air circuit downstream of a control valve and within one metre of the spraying system intake in order to optimise flow uniformity throughout the spraying operation.

During the pre-test phase, the system was calibrated with water in order to determine spraying times with the three selected nozzles.

4.4. Production of mixes

The overall mixing time was set at 2.5 min., regardless of the spraying time. This meant that mixing times after full incorporation of the liquid varied depending on which nozzle was used.

The initial mixer load was 50 kg of pellets.

After zeroing the system with a pot containing the intended solution, the required test quantity was weighed by negative measurement after sampling with a syringe (the density of the sprayed solution was considered as being equal to that of water).

The solution was injected into the spraying system with the syringe. After closing all the valves, the whole assembly was weighed.

The system was placed on the mixer and connected to the compressed air supply. The solution was sprayed through a slit in the mixer cover, through which the sprinkler head of the spraying system was inserted at an angle.

After checking the compressed air pressure value, the mixing cycle was started for 2.5 min. at 30 rpm. Spraying began ten seconds after the start of the mixing cycle. Spraying time was measured by recording pressure oscillations.

The compressed air system continued to operate up to the end of the mixing cycle, meaning a significant amount of time after the end of the injection, thus ensuring maximal spraying of the solution.

At the end of the cycle, the feedstuff was emptied out of the machine. The injection system was flushed with compressed air, and then weighed to obtain with reasonable accuracy the weight of liquid actually sprayed into the mixer chamber, thus giving the expected concentration.

4.5. Sampling

Due to the possible presence of fines in the pellet mix post-test, each batch was transferred in two bags. This initial total batch was successively halved in order to obtain 16 samples of approx. 100 g. Each sample (containing pellets and fines) was then ground in a roll mill and homogenised before being halved again twice to obtain two sub-test portions of approx. 25 g.

4.6. Analyses

Colorant dosage was based on the following method:

- Weigh the test portion
- Add 50 ml of ethanol/distilled water solution (70/30) using a graduated applicator
- Wait for 10 minutes (stir horizontally 4 to 5 times while waiting)

- Stir briskly at the end of the waiting period
- Sample one millilitre of supernatant and filter
- Read the optical density at 635 nm against a white control made using a blank feed.

The analyses were performed in duplicate on a total of 16 samples per batch. Standard ranges were made up from the 3 stock solutions sprayed onto the feed.

4.7. Data processing

The leading coefficients for standard curves such as illustrated in Figure 2 were used to calculate colorant concentrations in the samples.

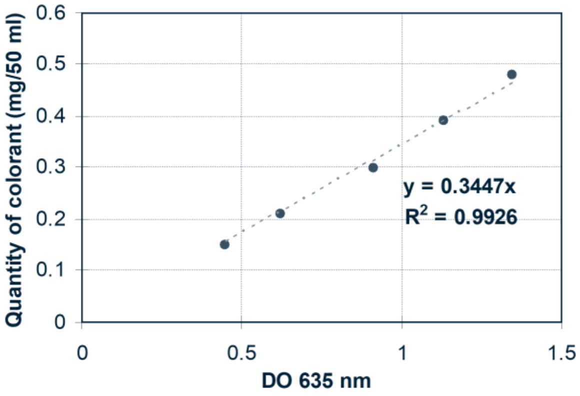

Figure 2: Standard curve describing the relationship between the weight of Green S in 50 ml of 70/30 solvent and the optical density at 635 nm

The recovery rates for each tracer were calculated based on the quantities actually incorporated. Having duplicated the analyses using separate test portions from each sample, it was possible to perform randomised model variance analyses in order to obtain the homogeneity variance and evaluate the residual variance.

5. Results

Nozzle 1.25 Max. durability		Solution incorporation rate		
		100 ppm	300 ppm	500 ppm
Concentration of the initial solution	10 g/l			95.2
	20 g/l			87.7
	30 g/l	75.1	85.2	88.4 / 88.4

Solution 30 g/l at 500 ppm		Durability		
		A (74.0*)	A (74.0*)	A (74.0*)
Nozzles	0.50 gal/h			89.7
	1.25 gal/h	90.7	84.6	88.4 / 88.4
	2.00 gal/h			73.4 / 96.8

Table 2: Recovery rate (%)(* durability - Eurotest)

With the exception of one value above 110%, the recovery rates were found to be acceptable when compared against conventional homogeneity tests (Table 2).

The impact of the 4 factors was investigated using homogeneity coefficients of variation that were calculated based on the variance analyses derived from the duplicate-analyses on each sample (Table 3).

These test results suggest that the variation in durability has no impact on colorant distribution or homogeneity (Figure 3). Note that this is true insofar as, when making up the samples, the pellet/fines mix was divided up to make the final samples. It remains highly likely that the fines in the pellets contained higher quantities of colorant. Therefore, regardless of a pellet durability of 74 to 90% (Eurotest method), spraying 500 ppm of solution with a colorant content of 30 g/l onto 4-mm diameter pellets, using a conical nozzle injecting at 1.25 gal/h, gives coefficients of variation of around 10% in this blade mixer.

Nozzle 1.25 Max. durability		Solution incorporation rate		
		100 ppm	300 ppm	500 ppm
Concentration of the initial solution	10 g/l			6.3
	20 g/l			8.0
	30 g/l	10.5	7.2	11.2 / 9.2

Solution 30 g/l at 500 ppm		Durability		
		A (74.0*)	B (82.7)	C (90.1)
Nozzles	0.50 gal/h			5.1
	1.25 gal/h	10.1	9.2	11.2 / 9.2
	2.00 gal/h			10.2 / 12.1

Table 3: Homogeneity coefficient of variation (%)(* durability - Eurotest)

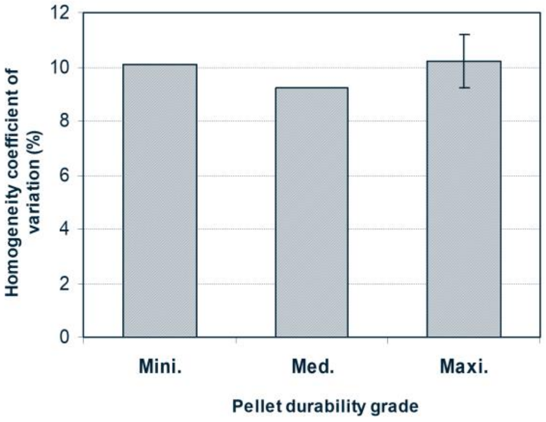

Figure 3: Levels of homogeneity coefficients of variation according to initial pellet durability grades

The other factors would appear to have a significant impact on the homogeneity of sprayed liquid distribution. Fluctuations in the quantity of sprayed solution involve an optimum at 300 ppm (Figure 4) with a value of 7.2%. Given the closeness of the results and the importance of this factor, the tests will have to be repeated in order to verify this possible effect. Spraying a lesser (100 ppm) or a higher (500 ppm) quantity would result in coefficients of variation of around 10%. A decrease in variation as the quantity increases would be more logical as, in this case, there would be a greater number of pellets exposed to the liquid solution. Increasing the number of colorant-carrying pellets would reduce variations in distribution in 100 g.

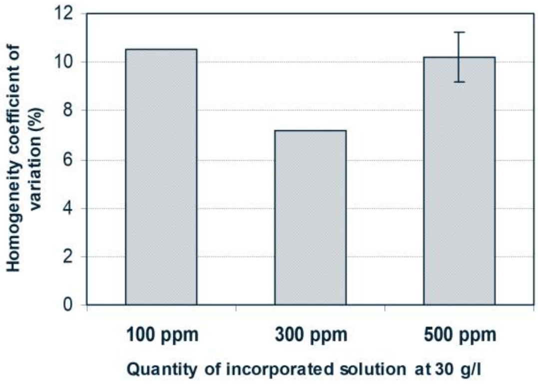

Figure 4: Levels of homogeneity coefficients of variation according to incorporation rates for the solution at 30g/l

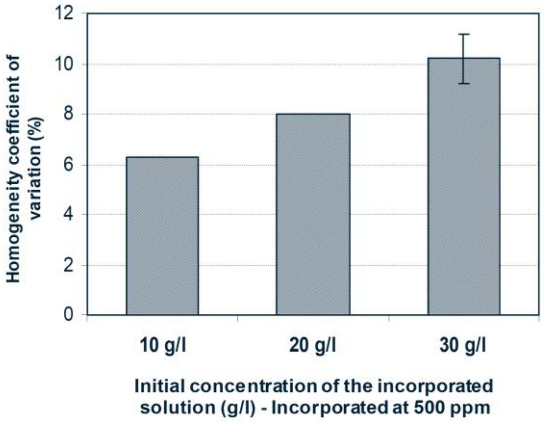

Figure 5: Levels of homogeneity coefficients of variation according to initial concentrations of the solution incorporated at 500 ppm

Diluting the solution sprayed at the same rate gave final concentrations with decreasing colorant contents. This showed that reducing the concentration may increase analysis variability. However, the residual variances revealed that this was not the case while the homogeneity coefficients of variation demonstrated that, even at lower concentrations, inter-sample variability also decreases in a regular manner (Figure 5).

This effect differs from that observed when varying nozzle flow rates (Figure 6) as, in this case, the variation could lie in the number of pellets that has received the solution.

This suggests that the lower the flow rate, the greater the number of pellets that are likely to receive and then carry the colorant.

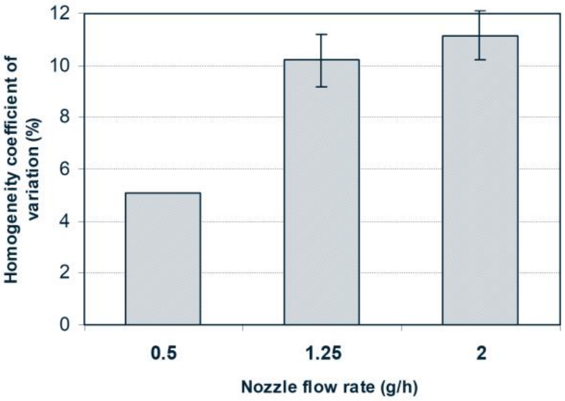

Figure 6: Levels of homogeneity coefficients of variation according to nozzle flow rate

6. Conclusion

Generally speaking, a diluted solution should be sprayed slowly in order to ensure uniform distribution of the target active ingredient. Therefore, to ensure uniform distribution, it would be advisable for the feed manufacturer, who controls their spraying facility and its operation, to choose incorporation flow rates that decrease as the concentration of the sprayed solution increases. If the plant uses a high incorporation rate, the active ingredient solutions manufacturer should choose weak solutions or suitable dilutions.

Pellet durability will have no effect on homogeneity in a solution ranging from 100 to 500 ppm provided that pellets and fines are evenly distributed on arrival in the animal's feeding trough. In other cases, which is likely, it would be advisable to use pellets with low friability as, while this would not prevent the fixation of liquid solutions, it would limit risks relating to the presence of fines.

Lastly, it should be noted that, under these conditions, the resulting homogeneity coefficients of variation that fluctuated between 5.1% and 12.1% with 4-mm diameter pellets could be considered optimal. This suggests that it would be difficult to improve on these coefficients of variation when using a continuous mixer.

This does not, however, exclude choosing smaller pellets that would provide a larger surface area, thereby improving distribution, although this remains to be confirmed.