DRAFT

(CONFIDENTIAL)

Use of a tracer to detect poultry offal meal in compound feeds.

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Introduction

The prohibition in 1990 on the use of mammalian meat and bone meal in compound feeds for ruminants presented a significant analytical problem. The method used - detection of bone fragments under a phase contrast microscope - does not enable the analyst to differentiate between bone particles from mammalian and avian species. Hence, if bone particles were found, one could not exclude the possibility that poultry offal meal (POM) was the source. Consequently, the use of POM was restricted to certain licensed manufacturers, primarily to prevent cross-contamination with ruminant feeds.

The objective of the trial was to determine whether tracer particles, incorporated during the rendering process, would facilitate the detection of POM in compound feeds. In addition, cross-contamination, between batches of different formulations, within feed mills is a real problem, and the usefulness of the technique in detecting such contamination was examined.

Materials and Methods

The trial was carried out at the feed mill of McCarton Bros., Shercock, Co. Cavan. The firm is licensed to manufacture compound feedingstuffs for sale. As they only produce compound feeds for poultry, they are also licensed to use POM as an ingredient in the manufacturing process. Output is in excess of 70 000 tonnes per

annum. The firm also has a chicken processing unit nearby, and the offal from this plant is rendered to produce POM which is used as an ingredient in the feed mill.

Raw offal is conveyed by floatation from the processing to the rendering plant and collected in skips. The skips are emptied manually into batch cookers and the material is cooked for about 4 hours. Cooking conditions are monitored continuously and the process must meet specified EU standards (at least 133°C for 20 minutes at 3 bar pressure). During cooking, the offal is continuously agitated by baffles within the cooker.

The tracer used the trial consistent of iron/nickel particles[1]. This product was chosen for its ability to withstand the high temperatures and pressures used in the rendering process. The manufacturers specification for Microtracer F-Ni is as follows:

- Particle size: 95% between 125 and 500 μm
- Number of particles/g: 50,000 minimum
- Range: 50,000 - 65,000/g

The trial consisted of two treatments, a high and a medium level of Microtracer in the POM (Treatment 1 = 370 g/tonne; Treatment 2 = 180 g/tonne). Assuming the sample of Microtracer supplied contained 57 500 particles per gram, the POM would contain 21 275 and 10 350 particles per gram for each treatment. The first treatment was carried out on the 18/8/97 and the second on 17/9/97.

The cooker used in the trial had a nominal output of 1.25 tonnes POM and the tracer was added on top of the raw offal in the loaded cooker. The weight of POM produced is quite variable, depending on the composition of the offal in the batch (proportion of feathers, bone, flesh, blood) and its moisture content. For Treatment 1, 312 g Microtracer was added to the cooker which yielded 845 kg POM (equivalent to 370 g/tonne). The dose rate for Treatment 2 was 180 g, yielding 1005 kg POM. An outline of the Treatments used in the trial is given in Table 1.

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[1] Microtracer F-Ni, supplied by Guinness Chemical (Ireland) Ltd.

On discharge from the cooker, the POM was conveyed by auger to a ribbon mixer, fixed on load cells. Eight samples of spiked material were drawn at fixed intervals from the stream to give a representative set for each batch and numbered subsequently. An equal amount of soya bean meal (SBM) was weighed automatically to the mixer and mixed for 15 minutes. A further set of 8 samples, representative of the POM/SBM mixture (1:1), were drawn on discharge to a trailer and numbered.

The material was transported to the feed mill and discharged into an empty bin. A three tonne batch of broiler pellets was produced under normal conditions using the POM/SBM mix as an ingredient at 14 % inclusion for both Treatments. The manufacturing process is computer controlled, giving a print out of the actual quantities of ingredients used in each batch. The actual inclusion rate of the POM/SBM was within 99 to 103 % of the target level for all batches. Four samples of the pelleted broiler feed (~ 500 g each) were drawn at fixed intervals during discharge from the cooler. To determine carry-over effects, 4 samples (~ 1000 g each) were also drawn from the next two batches of compound feed produced.

This procedure was replicated three times for each treatment.

Analysis of the samples was carried out in the State Laboratory, Abbotstown. The iron particles were extracted magnetically from the ground pellets using a rotary detector and treated with a reagent which gives a colour reaction on contact with nickel. The red spots formed on the filter paper were counted.

Results

Analysis results for the POM and POM/SBM mix are presented in Table 2. Assuming that the tracer contained 57,500 particles/g, this would give a theatrical level of 21,275 particles/kg for Treatment 1 and 10,350 particles/kg for Treatment 2; and half these levels for the POM/SBM mix. On this basis, recovery values for Treatment 1 were good (97 % and 112 % for POM and POM/SBM respectively). For Treatment 2,

recovery values from both materials were poor (20 % and 57 %). However, subsequent analysis of the compound feeds shows that a significant proportion of particles present were not detectable in these materials.

Analysis results for the compound feeds containing spiked POM/SBM are set out in Table 3. Dose rates for the Microtracer in the compound feed were 25.9 g/tonne and 12.6 g/tonne for each treatment (1490 and 725 particles/kg compound feed). The data in Table 3 shows that recovery values for both Treatments were significantly less than these estimated values. In contrast to the data in Table 2, recovery values for Treatment 2 were higher than for Treatment 1. Of greater significance, however, is the decrease observed from the first to the last replicate within each treatment.

Data on the carry-over of the Microtracer to the next two batches of compound feed (COB 1 and COB 2) are shown in Table 4. For Treatment 1, particles were found in all carry-over samples collected from the next two batches. In addition, there is a clear difference between the average levels found in COB 1 and COB 2 samples (14 and 2 particles/kg). In the case of Treatment 2, particles were found in all COB 1 samples but only in 8 of the 12 samples from COB 2. Also the difference in mean levels between COB 1 and COB 2 is not as apparent in Treatment 2 as in Treatment 1. In fact it is surprising that any particles were found in COB 2 samples for this treatment considering the levels found in the corresponding COB 1 samples. The level of tracer particles found in COB 2 samples for both treatments indicates that most of the contamination was occurring prior to mixing, possibly from the blending bin feeding mixer but more likely from the elevators feeding the blending bin. Overall the data indicates carry-over of POM in the region of 0.05 - 0.2 % for COB 1 and 0.01 - 0.06 % for COB 2.

The carry-over batches manufactured after Treatment 2 did not contain POM and hence a comparison was possible between the Microtracer method and the current method for detecting bone particles (microscopic analysis). This data is presented in Table 5 and may be summarised as follows:

	Positive samples	
	Microtracer	Bone
COB 1	6/6	3/6
COB 2	3/6	3/6

Given the very low level of contamination in these samples and expected variation at this level, it would be unrealistic to expect a high correlation between the number of particles found using both methods. Nevertheless, the results would indicate a reasonable probability of finding one or more tracer particles in samples found positive for bone at low levels of contamination.

Discussion

A number of tracer products have been marketed for use in food and feedingstuffs since the early 70's. Usually they consist of small particles of an innocuous material, (such as iron, graphite, stainless steel or salt) which are coated with an approved food colour. The colour acts as a means of identification of the particles when extracted from a mixture of ingredients. Their main use is to enable quick on site analysis for the presence or absence of an ingredient in a mixture e.g. checking that the correct premix has been added to a compound feed. In practice, they are seldom used by premix manufacturers or compounders in Ireland.

An important consideration in choosing a tracer is its stability when incorporated in a particular product or formulation. Food colourants would not withstand the processing conditions used by the rendering industry and these firms do not have mixers to incorporate a tracer in the finished product. Microtracer F-Ni was chosen to test addition to the raw offal prior to cooking.

With the exception of the POM and POM/SBM in Treatment 1, recovery values for the Microtracer were disappointing,. While the Manufacturer's literature indicates an

average recovery of 75 % from pellets, most samples were well below this level. Some loss can be expected from magnets in the mill(4) and a single sample from one magnet contained 1600 particles/g. However, the main factor effecting recovery is probably due to coating of the particles during cooking and feed manufacture. POM can contain up to 45 % oil which leaves the product very sticky and difficult to handle in a feed mill (bridging in bins). SBM was used in this mill to reduce this effect by making the material more manageable. The oil-coated tracer particles may tend to become embedded in larger particles and thus prevent magnetic recovery.

The decrease found in recovery levels from the 1st to the 3rd replicate within each Treatment could be very significant if this is a real effect and not just a chance observation. Approximately two hours elapsed between replicate samples and if this effect continued it would severely limit the potential use of the product to identify POM.

The variability of the data within replicates is much greater than that normally found and indicates lack of homogeneity in the dispersion of tracer particles throughout the mix. (Usually the tracer is added either directly to the mixer or in a free-flowing premix which helps dispersion). In addition to the possible adverse effect of the oil, the relatively small aliquots taken for analysis would have contributed to this variability (POM & POM/SBM = 2-3 g; Compound feed = 20-30 g).

Acknowledgement

The assistance provided by McCarton Bros., and in particular Jimmy Courtney and Tom Horan, in carrying out this trial is very much appreciated

TABLE 1

Levels of Microtracer F-Ni used in Trial

Feedingstuff	Treatment 1		Treatment 2	
	(g/tonne)	(particles/kg)[1]	(g/tonne)	(particles/kg)
POM	370	21 275	180	10 350
POM/SBM (1:1)	185	10 640	90	5 175
Compound feed (14 % POM/SBM)	25.9	1490	12.6	725

[1] Assuming 57 500 particles/g in the Microtracer F-Ni used

TABLE 2

Microtracer F-Ni in POM and POM/SBM mixture
(Number of particles/kg material)

Sample	Treatment 1		Treatment 2	
	POM[1]	POM/SBM[2]	POM[3]	POM/SBM[4]
1	15 250	6 750	2 167	2 000
2	14 000	15 750	1 167	3 167
3	8 250	7 000	1 667	3 000
4	16 750	8 750	2 833	2 667
5	27 500	16 000	1 333	3 000
6	18 500	14 500	1 833	3 667
7	34 250	11 500	2 167	3 333
8	28 750	14 000	3 000	2 833
Mean	20 406	11 781	2 021	2 958
Recovery %	96	111	20	57

[1] Estimated level = 21 275 particles/kg
[2] Estimated level = 10 640 particles/kg
[3] Estimated level = 10 350 particles/kg
[4] Estimated level = 5 175 particles/kg

TABLE 3

Level of Microtracer in compound feeds containing POM/SBM (Number of particles/kg feed)

Sample	Treatment 1[1]			Treatment 2[2]		
	Rep. 1	Rep. 2	Rep. 3	Rep. 1	Rep. 2	Rep. 3
1	1 350	488	300	467	350	83
2	633	913	538	583	500	367
3	763	738	638	717	567	367
4	575	513	563	300	433	367
Mean	830	663	509	517	463	296
Recovery %	56	44	34	71	64	41

[1] Estimated level = 1 490 particles/kg
[2] Estimated level = 725 particles/kg

Table 4

Carry-over of Microtracer to the next 2 batches of compound feed
(Number of particles/kg feed)

	Treatment 1						Treatment 2					
	Rep. 1		Rep. 2		Rep. 3		Rep. 1		Rep. 2		Rep. 3	
Sample	COB 1	COB 2	COB 1	COB 2	COB 1	COB 2	COB 1	COB 2	COB 1	COB 2	COB 1	COB 2
1	6	2	26	2	24	1	3	0	1	3	4	4
2	15	1	2	3	26	1	5	0	5	1	2	2
3	15	5	7	3	2	1	3	3	8	9	4	2
4	11	3	20	1	10	1	3	0	1	1		0
Mean	12	3	14	2	16	1	4	1	4	4	3	2

TABLE 5

Comparison of carry over of Microtracer and bone particles in Treatment 2

Rep.	COB	Sample	Microtracer No./kg	Bone No./20 g
1	1	1	3	2
1	1	3	3	0
1	2	2	0	1
1	2	4	0	1
2	1	1	1	2
2	1	3	8	0
2	2	2	1	0
2	2	4	1	0
3	1	1	4	2
3	1	3	4	0
3	2	2	2	0
3	2	4	0	1

Treatment 1

Number of particles in Broiler Starter feed containing 7% POM (312g MT/850 kg) and subsequent batches

(Raw Data)

Treatment/ Replication	Feed	Aliquot tested (g)	Sample 1	Sample 2	Sample 3	Sample 4
1/1	Spiked Broiler Starter		22[1]	8[1]	22[2]	18[2]
			21[1]	10[1]	8[2]	12[2]
			18[1]	13[1]	14[2]	6[2]
			20[1]	7[1]	17[2]	10[2]
	Carry Over Batch 1	Total sample in brackets	6 (960)	13 (890)	14 (910)	10 (920)
	Carry Over Batch 2	Total sample in brackets	2 (1010)	1 (910)	4 (830)	3 (930)
1/2	Spiked Broiler Starter	20	6	24	14	10
		20	9	16	16	9
		20	13	14	14	10
		20	11	19	15	12
	Carry Over Batch 1	Total sample in brackets	24 (920)	2 (930)	6 (910)	18 (920)
	Carry Over Batch 2	Total sample in brackets	2 (920)	3 (910)	2 (770)	1 (880)
1/3	Spiked Broiler Starter	20	5	11	9	12
		20	7	11	12	11
		20	6	12	16	10
		20	6	9	14	12
	Carry Over Batch 1	Total sample in brackets	22 (930)	24 (910)	2 (900)	9 (900)
	Carry Over Batch 2	Total sample in brackets	1 (900)	1 (910)	1 (910)	1 (920)

[1] Aliquot tested = 15 g
[2] Aliquot tested = 20 g

Number of particles in Poultry Offal Meal and Soya Bean Meal mix (1/1)

(Raw data)

Treatment	Feed	Aliquot tested (g)	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8
1	(POM @312 g MT/850 kg)	1	6, 19, 12, 24[1]	9, 15, 14, 18	20, 3, 6, 4	8, 20, 21, 18	22, 29, 41, 18	23, 21, 16, 14	35, 48, 33, 21	59, 56[2]
1	POM/SBM (50:50 mix)	2	15, 12	31, 32	8, 20	23, 12	43, 21	24, 34	23, 23	25, 31
2	(POM @180g MT/1005 kg)	3	7, 6	5, 2	5, 5	12, 5	4, 4	8, 3	9, 4	8, 10
2	POM/SBM (50:50 mix)	3	4, 8	11, 8	13, 5	7, 9	11, 7	11, 11	12, 8	10, 7

[1] Four aliquots per sample
[2] Two aliquots of 2 g each

Treatment 2

Number of particles in Broiler Grower feed containing 7% POM (180g MT/1005 kg) and subsequent batches

(Raw Data)

Treatment/ Replication	Feed	Aliquot tested (g)	Sample 1	Sample 2	Sample 3	Sample 4
2/1	Spiked Broiler Grower	30	16	16	21	9
		30	12	19	22	9
	Carry Over Batch 1	Total sample[3]	3	5	3	3
	Carry Over Batch 2	Total sample[3]	0	0	3	0
2/2	Spiked Broiler Grower	30	10	12	6	16
		30	11	5	14	10
		30		13	5	
		30			9	
	Carry Over Batch 1	Total sample[3]	1	5	8	1
	Carry Over Batch 2	Total sample[3]	3	1	9	1
2/3	Spiked Broiler Grower	30	1	11	12	15
		30	4	11	10	7
	Carry Over Batch 1	Total sample[3]	4	2	4	
	Carry Over Batch 2	Total sample[3]	4	2	2	0

[3] Total sample varied between 90[illegible]nd 1000 g

Comments

1. **Incorporation of Microtracer**
The microtracer was added directly on top of the raw material in the cooker. Baffles within the cooker mixed the material continuously during cooking, which took approximately four hours. While the oil content of the POM produced for this trial was not determined, product produced at this plant normally contains 35 - 40 % oil.

2. **Replication**
The spiked POM was incorporated in three separate batches of compound feed which were sampled and analysed for each treatment. The raw data should help to clarify the sequence.

3. **Decline in Recovery within Treatments**
Only about three hours elapsed between the samples drawn for the first spiked batch of compound feed and the third within each treatment. The samples were analysed about two weeks after the first treatment and about four weeks after the second.

I can't explain why the recovery levels dropped between replicates within treatments. My main concern would be that this is a real effect which might be progressive over time and if so would seriously limit the usefulness of the product. If you have any explanation for this I would appreciate your assistance. In the meantime, we intend to examine this aspect further in a limited stability study.

4 It is not possible to estimate the amount of tracer taken up by the magnets. There were three magnets within the whole milk complex and only a small sample was taken from one.

5. **Carry Over Feeds**
The carry over feeds were pelleted and the formulation was the same as that for the spiked feed with the exception of the micro tracer. COB1 was the first batch manufactured after the spiked batch and COB2 the second for each replication.

6. **Sampling**
The POM was sampled during discharge from the cooker and the eight samples were taken at fixed intervals so as to be representative of the lot. The sample size was about 100 g.

The POM/SBM mix was sampled as it was conveyed from the ribbon mixer to a trailer. Sample sizes of approximately 500 grammes were taken.

The compound feed was sampled at the point of discharge from the cooler (after conditioning and cooling). Approximately 500 g samples were drawn in each case.

The carry over batches were sampled at the same point and, in this case, the plastic bags used for sampling were filled (approx. 950 g each).

7. **Dose Level**

The amount of micro tracer added to the cooker for Treatment 2 was180 g (I added it myself). However, it is possible that the micro tracer was not homogeneously distributed in the POM.

8. **Method of Analysis**

The method of analysis used was the one supplied by Kevin Harte. A phase contrast microscope was not used to count the particles. They were counted with the naked eye. Perhaps this is not the best method?

In my statement that a significant number of particles were not detectable in these materials (mainly Treatment 2 POM), I was assuming that the tracer was distributed uniformly in the material but that the particles in some way did not show up on analysis - possibly coated with oil. Is this likely?