Processing

Pro's and cons on the use formulation techniques for

Why are some additives in the form of granules and how do they differ over other dosing forms such as powders and liquids? In part one of this article the focus will be on the various techniques that can be used to formulate feed additives.

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A recent survey published in Feed Tech showed that approximately 28 companies were involved in the manufacture of feed enzymes. Of the enzymes used most of these were available in powder form or as liquid application for PPA (post pelleting application). Some of are available as granulate.

Of course, not only enzymes are used as additives in the feed industry. There is a whole range of additives varying from anti-biotics, pre-biotics, pro-biotics, vitamins, coccidiostats and medicines which are added in the feed mix. Why are some additives in the form of granules and how do they differ over other dosing forms such as powders and liquids?

In this article, the medicine, the vitamin or the coccidiostat is called the 'active material'. It is that biological or chemical compound that has a health-promoting or nutritional effect. This active material is mixed with a variety of materials (lubricants, fillers, binders and coatings) and processed by means of specialized processes into round or potato-shaped particles. These are the commercially available additives, which are subsequently used in feed concentrates, premixes or blend directly into the final feed.

For example, Diclazuril (a coccidiostat) is used in a concentration of one (1) gram per tonne of animal feed. It is not possible to use the pure active material for direct feed manufacturing of broiler feed. Such active materials are very potent, often non-free flowing, sometimes hygroscopic and may generate potential hazardous dusts for operators that work with these in the factory. Therefore, the active material has to be diluted and made available in a form, which can be better handled in large animal feed producing installations.

In case of Diclazuril, this is diluted with a specific carrier to obtain a final product with only 0.5% active material (e.g. Clinacox® 0.5%). Instead of adding one gram of pure active material per tonne, 200 grams of the product is added per tonne of feed. The technique used for the formulation of Clinacox 0.5% will ultimately determine the dosing accuracy and mixing stability of Diclazuril at 1 gram per tonne level in the final feeds.

Costly procedures

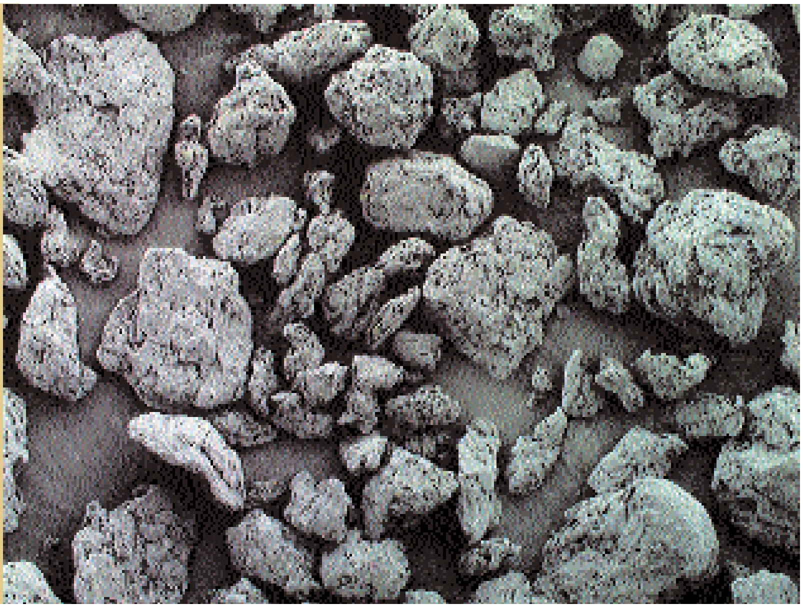
One could easily blend the active material with some carrier commonly found in the feed industry (*Table 2*) and use that as a product. Why using additional, costly, manufacturing procedures to obtain feed additives? For that, there are a number of reasons:

- Provision of definite quantity units for metering, dispensing and administration.
- Reduction of (potential) allergenic dust, reduction of handling hazards.
- Creation of non-segregating blends of particles.

Processing of different feed additives (1)

Photo 1 left - Electroscanning microscopial picture (62x) of a feed additive (generic diclazuril 0.5%) consisting of active material blend with a mineral carrier

Photo 2 right - Electroscanning microscopial picture (62x) of vacuum coated and dried Clinacox (0.5%). The carrier is polysoy™ (Photos: Janssen Animal Health)



- Improved product-appearance and flow-properties
- Control of solubility and in some instances time-released liberation of active material.
- Control of heat transfer and moisture diffusion by, e.g. control of porosity and surface-to-volume ratio.
- Control over the release of active substances in certain parts of the digestive tract by applying coatings with different solubility's.

Production of feed additives

There are many processes available by which feed additives can be made. In *Table 1* an overview is given of these methods (of which not all apply to the manufacture of feed additives) and some of the general properties of the produced particles that can be observed with these methods. These are guideline values but provide a good overview on the performance of the various processes relative to each other. We will have a closer look at four different methods to manufacture feed additives. Although many more processes exist to make feed additives these four methods are for the largest part used in the manufacture feed additives. These are:

- simple mixing
- vacuum coating ➢
- fluid bed processes
- mixer granulation

Table 1 - Comparison of various granulation methods (After Ennis 1997)

Methods	Product size [mm]	Product density	Capacity
Tumbling granulators			
- drum	0.5-20	Moderate	0.5-800 tonne/hr.
- disc			
Mixer granulators			
- continuous high shear	0.1-2	low - high	< 50 tonne/hr.
- batch high shear	0.1-2	high	
Fluidised granulators			
- fluidised bed	0.1-2		100-1500 kg/batch
- spouted bed		low - moderate	50 tonne/hr cont.
- Wurster coater			
Spray methods			
- spray drying	0.05-0.5	low	<10 tonne/hr.
- prilling	0.7-2	Moderate	< 5 tonne/hr.
Pressure compaction			
- extrusion	>0.5		< 5 tonne/hr.
- roll press	>1	high - very high	< 50 tonne/hr.
- tabletting	10		< 1 tonne/hr.
Thermal processes			
- sintering	2 - 50	high- very high	<100 tonne/hr.

SIMPLE MIXING

Making feed additives the simple way involves blending of the active material with a filler/diluting material, called the carrier-material. Many of these carrier-materials are used in animal feed manufacturing and *Table 2* gives an overview of some of these materials.

There is a wide range of physical properties associated with these carrier materials, for instance; bulk-density may range from as low as 294 kg/m^3 (dried wheat-middlings) to 1,020 kg/m^3 for calcium carbonate. The particle size ranges from 50 microns to over 1 mm. The amount of dust that can be generated from the carrier materials ranges from non-detectable (e.g. rapeseed, wheat feed flour) to 1.47-1.48 gr/m^3 for bleaching earth and extracted soybeans respectively.

It is dependent on the properties and size of the carrier and active material how the mix behaves during feed manufacturing. When active material particles are small (<50 micron) Vanderwaals forces may glue these particles on the larger carrier particles. Surface properties and hygroscopic activity may affect the tightness of such bonds.

In all cases the active material is loosely present between and on the carrier-particles and when the material is subjected to handling, active material can become air-borne with subsequent hazards for operators. It may segregate from the mix and remain in screws, elevators or other transport and handling equipment thereby increasing the amount of carry-over in the next batch. *Picture 1* is an example of Diclazuril simply mixed with a mineral carrier. Such products

Table 2 - Physical properties of assorted carrier materials (Heidenreich, 1995)

Carrier material	Moisture-content	Bulk density	Tap density	True density	Angle of repose	Heubacher[1] test	Specific surface	Particle size
	%	[kg/m^3]	[kg/m^3]	[kg/m^3]	[°]	[gr/m^3]	cm^2/gr]	[d50:micron
Bentonite	12.3	853	1010	2150	43	1.06	575	50
Calcium carbonate	0.5	1020	1190	2180	39	2	438	70
Bleaching earth	6.8	314	357	1580	43	1.47	675	60
Barley 1 (milled)	11.3	508	529	1550	48	0.62	319	330
Barley 2 (milled)*	12.5	484	520	1550	40	0.3	176	560
Linseed (extr.)	12.2	592	658	1453	39	0.08	154	480
milled	11.7	583	613		40	0.11	368	150
dried	7.3	597	649		43	0.03	154	440
milled and dried	7.2	588	625		43	0.05	319	160
Maizegluten feed								
milled	10.3	649	676	1519	40	0.38	311	200
milled and dried	5.6	659	699		44	0.37	281	190
Rape seed extr. 1	11.7	501	538	1280	38	-	117	560
Rape seed extr. 2	10	542	602	1470	39	-	131	470
milled	9.6	590	633		47	0.05	180	380
dried	6.6	527	592		40	-	122	470
milled and dried	4.6	602	641		48	0.05	171	450
Soya hulls								
milled	11	539	625	1545	37	0.95	192	290
milled and dried	4.3	546	595		40	0.47	226	270
Soya beans extr.	11.8	607	641	1468	26	0.29	48	1360
milled	11.4	671	714		42	0.95	427	170
dried	7.2	638	704		35	0.3	56	1150
milled and dried	6.3	672	735		43	1.48	347	170
Wheat middlings	13.3	287	333	1462	41	0.08	118	620
milled	13	372	410		42	0.16	215	310
dried	6.2	294	329		43	0.06	109	590
milled and dried	4.2	354	394		47	0.02	199	310
Wheat feedflour	12.5	505	538	1457	41	-	289	220
(dried)	5.9	492	526		46	-	214	250

1 The amount of air-borne dust generated by 4 moving steel balls generated from 20 ml of feed material in upstream current of 0.2 m/s (Beekman, 2000).

All milling over 1 mm sieve, except * milled over 2 mm may give rise to dust-formation and loss of the active material during manufacturing thereby inducing cross-contamination.

VACUUM COATING AND DRYING

In case of vacuum coating and drying, the active material in the form of a suspension or solution is thoroughly mixed with the carrier particles under vacuum. Subsequently, the vacuum is released and the solute is pushed in the cores of the particle. Then, by using vacuum evaporation, the solvent is boiled off at low temperature, thus preventing thermal damage to the active material. The manufacturer according to specification selectively chooses the used carrier particles.

Important properties with respect to these carrier particles are: a high porosity, strength against attrition, and a specific particle size and particle size distribution. The origin of these particles may either be mineral (e.g. sodium sulphate) or from organic origine (modified starches or fibers). The active material, together with the other components (e.g. stabilizers or colorants) is coated on the external and internal surface of the particles. Applications involve medicines, detergents and feed enzymes. A well-known feed additive manufactured with this method is Clinacox 0.5%. Picture 2 gives an example of such a product.

FLUID BED PROCESSING

In a fluidized bed, air passes through a porous bed support (plenum) on which the product to be treated rests. At a certain air flow and pressure difference over the bed the packing between the particles is broken and the air/particle mass is ‘fluidized’ e.g. behaves as a liquid. Mixing of particles with or withoud fluids is rapid and, depending on the properties of the incoming air (heated) combinations of drying, agglomeration, coating etc. may occur.

Spray nozzles are inserted to introduce liquid binder(s). Agglomerators may work either in batch or (semi-) continuous modes. Agglomerates have a tendency to have a rather ‘open’ structure. Particle size may be wide or narrow depending on the materials to be agglomerated and the processing conditions. Typical particle sizes are between 100-2000 micron. Applications involve detergents, animal feed enzyme’s (e.g. phytase granulates).

MIXER GRANULATION

In principle all mixers can be set up as agglomerators. The collision of particles may result in agglomeration if enough binder material is available or particle sizes are small enough for moleculair interactions to develop. Mixer agglomerators can be diveded in two groups according toe Müller and Kraft:

- free-fall or tumbling mixers (double cone, twin shell V-mixer), where particles slide over each other during mixing and
- mixing by mechanical force (ribbon mixers, pug mills, Lödige mixer).

Any one of these mixers can be used, with or without modification, as agglomerator.

A wide range of particle characteristics may appear due to the different modifications and operating regimes under which these machines are driven. High intensity shear mixers mostly consist of modifications of mixers with high speed cutter heads. These are capable of introducing high amounts of energy, to the extent that thermoplastic materials in the products partially melt and form very strong bonds after resolidification. Applications involve detergents and feed enzymes.

Conclusion / take home-points

It can be concluded that simple blends of active material(s) results in a poor quality additive. Because of the fine particles involved, an increased risk of loss of active material in feed manufacturing and transportation equipment is present. Several formulation technologies for additives have been discussed in this article, which provide for a safer and easier means of handling and dosing active materials (enzymes, medicines and coccidiostats). With all of these formulation technologies good quality additives can be produced. The specific raw materials, binders, carriers and operating conditions determine the physical properties the feed additive. As a rule of thumb:

- Fluid bed processes have a porous structure and are most often spherical,
- Mixer granulated particles are generally denser and, therefore, less porous,
- Vacuum coated feed additives have physical properties largely depending on the type of carrier particle used.

There is a wide range of binders, fillers and coatings available which can be applied in all of these three processes to tailor-make particles with properties suitable for its intended application. ●

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