

# A user's guide to value-added vitamins

*How do premix manufacturers make the right choice of vitamins for each feed application, whilst optimising production parameters and maintaining premix quality? A few helpful guidelines make adding value much more coherent.*

By Eric Paillard, Adisseo, France

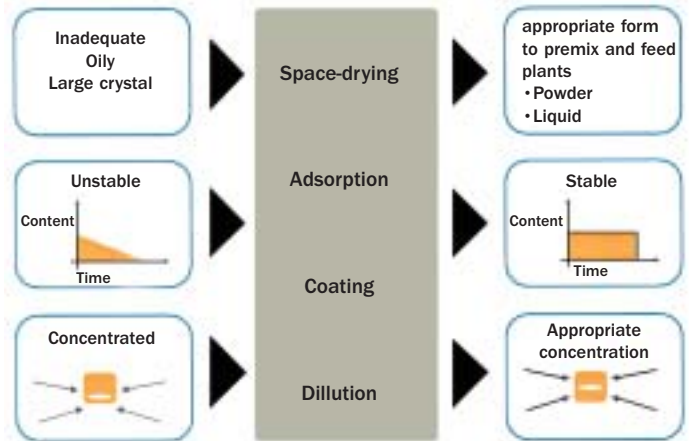
Ensuring that a feed formulation contains the correct level of the correct vitamins for a particular species at a particular level of production is only the first stage in optimising vitamin supplementation. Effective premix production and utilisation also requires detailed knowledge about how individual vitamins perform during storage, mixing, processing, and feeding, as well as how each one is digested, absorbed and metabolised. Taking these variations into account is key to adding and maintaining value in the premix.

## From molecule to product

Pure vitamins are generally not suitable for direct use in feed applications, but must first be processed to ensure their efficacy. Pure oil or large crystal vitamins are sometimes unstable or too highly concentrated and must be converted into a form that is more suitable for use in premix or feed plants. Some of the various conversion processes are shown in *Figure 1* and include atomisation (drying a sprayed liquid to produce a fine powder), adsorption (using a solid medium to convert a liquid into a powder), coating (to protect the vitamin against physical or chemical damage) and dilution (to achieve a specific concentration).

The properties of vitamins and their manufacturing processes are summarised in *Table 1*. The restrictions of use of the oil-based (fat soluble) vitamins, such as vitamins A and E occur mainly due to their physical form. This is usually overcome by coating or spray-drying, although adsorption onto a carrier or some form of chemical complexing with a carrier is also used. The water-soluble vitamins, such as the B-vitamins or vitamin C, that are synthesised as crystalline powders can cause a range of problems during premix or feed manufacture. Cohesion of the granules, electrostatic clumping or dispersion and hygroscopicity (absorption of

**Figure 1 - Supplying vitamins in an appropriate form is the first step in optimising their utilisation in feed**



**Table 1 - Problems encountered using pure vitamins in feed and formulation solutions to overcome them**

Vitamin	Physical form of	Problem with pure synthetic product	Existing solutions form of vitamins	Main types of formulation
<b>A</b>	Oil which can crystallize at room temperature	Physical form Stability Concentration	Microbeadlets Protection Dilution	Coating Spray-drying
<b>D<sub>3</sub></b>	Resin	Physical form	Powder formation Stability	Coating Spray-drying Protection Dilution Concentration
<b>E</b>	Oil	Physical form	Powder formation	Adsorption Spray-drying
<b>K<sub>3</sub></b>	Oil	Physical form Stability	Powder formation	Chemical complexing Blending with carrier
<b>B<sub>1</sub></b>	Crystalline powder	Cohesiveness	Grinding, sieving	-
<b>B<sub>2</sub></b>	Crystalline powder	Cohesiveness Electrostaticity	Physical treatment of the powder	Spray-drying Granulation
<b>B<sub>3</sub></b>	Crystalline powder	Cohesiveness Hygroscopicity	Physical treatment of the powder	Granulation Agglomeration by compaction
<b>B<sub>5</sub></b>	Crystalline powder	Hygroscopicity		
<b>B<sub>6</sub></b>	Coarse crystalline powder	Mixability	Physical treatment of the powder	Grinding
<b>B<sub>9</sub></b>	Crystalline powder	Cohesiveness	Electrostaticity Physical treatment	Atomisation of the powder
<b>H</b>	Crystalline powder	Concentration Physical form	Dilution Physical treatment of the powder	Spray-drying Grinding and blending with carrier
<b>B<sub>12</sub></b>	Crystalline powder Dried fermentation broth	Concentration Concentration homogeneity	Dilution	Blending with carrier
<b>C</b>	Crystalline powder	Hygroscopicity Stability	Physical treatment of the powder	As is coating

\*) **B<sub>3</sub>** nicotinic acid or nicotinamide

# Nutrition

moisture from the surrounding air) are common obstacles that need to be overcome and that cause problems during dosing, mixing, stability and general usage. The most common solutions involve a physical treatment of the powder that result in granular formulations, atomisation, grinding or blending with a carrier.

## Stability vital for efficacy

A stable vitamin product is one that does not deteriorate during storage or when used in the manufacturing of premix and feed. Vitamins are complex organic molecules. Even very small structural changes may sometimes considerably reduce their effectiveness.

Oxidation-reduction reactions are the main causes of degradation of vitamins in animal feed. The sensitivity to oxidation or reduction of a number of vitamins is shown in *Figure 2*. These reactions are made possible, in premixes and feeds, by the simultaneous presence of free water from raw materials and such redox compounds as organic acids, reducing sugars, trace minerals

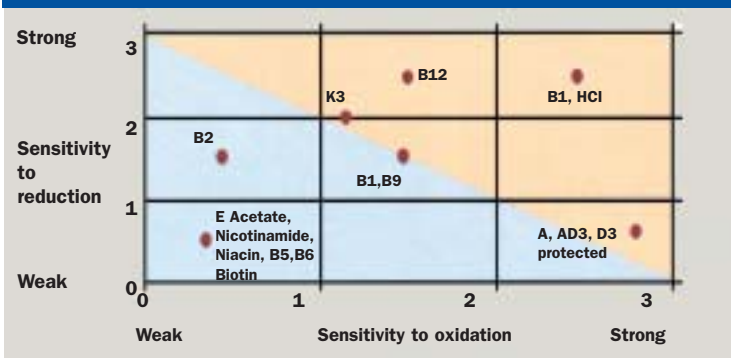
- Lack of dust emissions makes plants safer by reducing the risk of explosion, protects workers who may suffer from allergies or respiratory problems, eliminates cross-contamination of products and reduces waste and loss.
- Good flowability allows products to flow smoothly through the production process. It reduces product loss and residue in storage and conveyance equipment and enables more precise dosing.
- Good mixability enables uniform distribution of vitamins in the finished product and thus higher quality premix and feed. Uniform dispersion of active ingredients ensures that the feed contains an animal's entire daily vitamin requirement.

Assessment of practical mixability of a vitamin product is determined by three factors: theoretical mixability, efficiency of the mixer and analytical performances. The theoretical mixability, determined for each vitamin product, indicates whether the number of active particles in the product is (or is not) sufficient to yield an acceptable distribution in the intended mix.

The concept of theoretical mixability is used to:

- determine the minimum size of feed or premix samples to obtain a significant result of analysis (i.e. containing sufficient active particles).
- determine a valid tracer and the size of sample in the case of testing the efficiency of a mixer. assess the validity of a tracer and the adequate sample size to use in a de-mixing test.
- ease of use depends on various physical characteristics, such as particle size distribution, angle of repose and density.

**Figure 2 - Sensitivity of vitamins to oxidation and reduction in the feed**



and oxidised fat. Such reactions are accelerated, during manufacturing of premixes and feeds, by mixture with air during pneumatic transfers, by addition of steam, heat and mechanical actions and continue when finished products are stored. Finally, many other factors promote redox reactions: pH, light, temperature, contact surface area and mechanical constraints.

In the developmental process for new vitamin products (example: vitamin A), different formulations are methodically tested in order to determine those best suited for a specific manufacturing and storage environment.

## Ease of use increases efficiency

A product that is easy to use is one that premix and feed manufacturers can easily handle, convey and dose in their plants. The main three requirements are lack of dust, flowability and mixability— each of which delivers a number of advantages to during manufacture:

## Choice of components

The manufacture of a quality premix requires careful choice of its constituents: vitamins, trace minerals, minerals and carrier. Incorrect choice may explain difficulties in manufacture and/or aberrant analytical results. The vitamins used in the premix must satisfy a set of criteria such as those outlined in *Figure 3*.

## Good manufacturing process

The choice of components is not only essential to ensure the appropriate for various production processes but must also pay particular attention to the definition of premix plant equipment: storage, danger of dust, metering and mixing.

**The dangers of dust.** Any unit which handles, stores or processes products in powder form must respect strict design and manufacturing standards so to protect against the emission of dust, and in particular against dust explosion. The potential risk of explosion for each powder product can be measured according to several criteria (minimum ignition energy; maximum explo-

sion pressure and the maximum rate of pressure rise). In Europe, the new European Directives ATEX (Explosive Atmosphere) are applicable as from July 1<sup>st</sup>, 2003. The first directive (94/9/EC-ATEX 95) provides health and safety requirements for electrical and non-electrical equipment intended for use in potentially explosive atmospheres. The second directive (99/92/EC-ATEX 137) provides minimum requirements for improving the safety and health protection of workers potentially at risk from explosive atmospheres.

**Mixing.** Although the theoretical mixability depends on particle size distribution and true specific gravity of the additive and its inclusion rate in the premix of feed formulation, in practice, the characteristics of the mixer and the analytical method used to assess mixing also play a part.

A mixer intended for preparation of vitamin premix products must be able to provide homogeneous mixtures of physically diverse particles incorporated at various inclusion levels in the mix. The specifications for a vitamin mixer are as follows:

- Affords good homogeneity with the component included at the lowest possible content
- Affords good homogeneity with components of different particle size
- Short mixing duration  
Variable degree of filling, with no loss of mixing efficiency
- Complete emptying
- Easy cleaning
- Possibility of adding liquids
- Ability to disintegrate clumps
- Absence of heating during mixing
- Low consumption of energy when starting and during mixing
- Low maintenance costs
- Reasonable purchase price
- Among these criteria, a certain number will have to be prioritised or compromised in order to achieve the best possible balance of performance versus price.

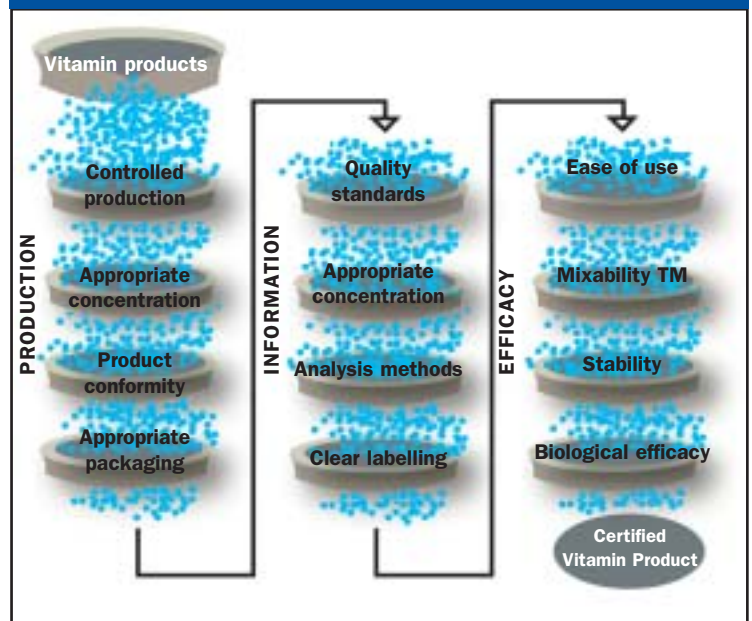
### Production quality control program

The premix or feed production quality control program is a procedure, which makes it possible to verify at any time:

- The conformity of the premix or feed with the product specification
- Good operation of the manufacturing unit at given time
- The average performance of the plant

This varies with the structure of the plant and the production process. However, general guidelines exist which can be applied.

**Figure 3 - Vitamin certification system**  
(Source: Adisseo, France)



According to the objective and nature of the quality control program, the number of samples to be analysed is variable. Thus only one sample could be sent to the laboratory for a conformity analysis, while it would be necessary to send at least seven samples for an analysis of homogeneity.

Quality control assessment is based on the choice of components to be analysed, sampling, and the analytical procedure.

**Sampling procedures.** It is essential to have a representative sample of a batch or a production, in order to have representative results. Samples dispatched to the laboratory are generally called primary samples. Primary samples are gathered from elementary samples.

European Directive 76/371/EC describes the number of elementary samples necessary to make up a primary sample according to the type of packaging. It also specifies the minimum and maximum numbers of elementary samples to be made according to the manufacturing batch size. The size of elementary samples depends on the characteristics of the component to be analysed and the probability that it contains a sufficient number of particles for good analytical precision. The preparation of the samples is the most significant part of the entire analytical procedure for obtaining good results. The primary sample is sent to the laboratory accompanied by its analysis request form.

**Analytical procedures.** In the laboratory, the preparation of the assay portion involves a certain number of operations such as division, mixing and grinding and the necessity to choose the right analytical technique. ●