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Addition of feed additives and the danger of carry-over

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SUMMARY - The correct addition of feed additives should be controlled by the working accuracy test of the production plant for compound feed or premixes, which includes the test of the carry-over level. According to market requirements the carry-over should be less than 4% for manufacture of compound feed and less than 1% for premix plants. Considering the different causes of additive carry-over recommendations for stabilization of the mixtures and avoidance of carry-over and cross contamination are given. The addition of liquid formulated additives at the end of the production line can reduce the carry-over level. Unprofessional admixing of liquid components in the main mixer causes the formation of agglomerates and deposits as sticks and crusts which cannot be compensated by rinsing batches. Test results show the bad suitability of ribbon mixer for admixing of liquid components.

Key words: Feed additive admixing, feed additive spraying, liquid component processing, carry-over.

RESUME - "Incorporation d'additifs alimentaires et danger de persistance". L'addition correcte d'additifs alimentaires devrait être contrôlée par un test de précision des opérations à l'usine de production pour les aliments composés ou prémélanges, comprenant le test du niveau de persistance. Suivant les exigences du marché, la persistance devrait être de moins de 4% pour la fabrication d'aliment composé et de moins de 1% pour les usines de prémélange. En considérant les différentes causes des résidus d'additifs, des recommandations sont données pour la stabilisation des mélanges et éviter la persistance et la contamination croisée. L'incorporation d'additifs liquides de formulation à la fin de la chaîne de production peut réduire le niveau de persistance. Une incorporation peu professionnelle de composants liquides dans le mélangeur principal provoque la formation de grumeaux qui se déposent sous forme de bâtonnets et de croûte, ce qui ne peut pas être remédié par des opérations de rinçage. Les résultats des tests montrent que le mélangeur à ruban est peu approprié à l'addition de composants liquides.

Mots-clés : Incorporation d'additifs alimentaires, nébulisation d'additifs alimentaires, traitement de composants liquides, persistance.

Introduction

Manufacture of compound feed means at first homogeneous mixing of extremely small parts of additives (micro components) into large quantities of feed mixture components (macro components). Therefore, mixing is one of the essential technological processes for production of compound feed for animals and it has important influence on the quality of the final product.

The characteristics of the mixing technology in feed compounding are: (i) batch mixing of high capacity for solid components; (ii) use of trough-mixers with different mixing-tools; (iii) different structures of the mixture components; (iv) addition of supplementary substances in the ppm-range by mass, i.e., mg/kg; (v) partly continuous mixing of liquid components (e.g., molasses, fat) after the batch mixer; (vi) increasing admixing of liquid parts in the main batch mixer; and (vii) single-line structure of the production plant used for manufacturing all kinds of animal feed.

The explosive increase of the formulations resulting from the customer oriented production enhances the probability of carry-over and cross contamination.

For mixing the so-called macro components there are no problems for sufficient homogenization. The problems are the addition and homogeneous distribution of the micro components (additives) and of liquid parts in the mixture of the macro components, the stability of this final mixture and the avoidance of concentration losses and carry-over of the additives in the total production plant. Consequently, the criteria for the mixing quality and for the proof of the accurate production in the feedmill are: (i) the homogeneity of the additives in the mixture and in the final product; (ii) the

concentration of the additives in the final product in accordance with the declared level; and (iii) the level of the carry-over and possible contamination of following batches.

Considering technical tolerances, feed compounding with total avoidance of the carry-over is not possibly.

Evaluation of the mixing technology and requirements

According to the European and German legal requirements, it is to distinguish between the mixing process for manufacture of compound feed and mixing process for production of premixtures. The application of a number of essential additives (e.g., vitamins, provitamins, performance promoters, trace elements, carotins, xanthophylls, additives for prevention of histomoniasis or coccidiosis, additives with similar activities) has to be carried out by means of a premixture only. The minimum concentration by mass of the premixture in the compound feed has to be 0.2%.

The mixing technology will be evaluated by the so-called working accuracy of the production plant, which is different from the mixing efficiency of the mixer. This efficiency of the mixer will be expressed by the homogeneity obtained after different or usual mixing time for the used mixer and material composition (Fig. 1).

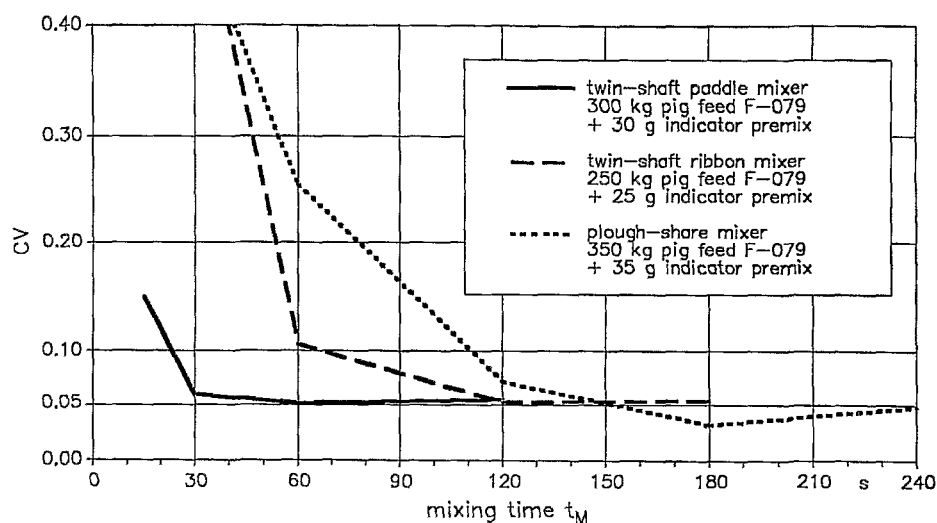


Fig. 1. Mixture quality curves for pig feed and different types of mixers.

The coefficient of variation CV is the quantitative expression of the homogeneity calculated by the standard deviation and the mean value of the concentration of the additive (mostly simulated by a marker substance because of the high analysis tolerance of real additives) in several samples taken from the mixture. The working accuracy of the production plant includes three components, which have to be determined experimentally:

- (i) The real concentration of the considered additive in the compound feed after the usual mixing time and after conveying the mixture to the final station (e.g., silo for delivery) in comparison to the nominal concentration value (Fig. 2).
- (ii) The homogeneity of the mixture after the usual mixing time and after conveying the mixture to the final station expressed by the coefficient of variation CV in each case (Fig. 2).
- (iii) The cross contamination of the rinsing batch by the considered additive, produced without this additive after two test batches (Fig. 3).

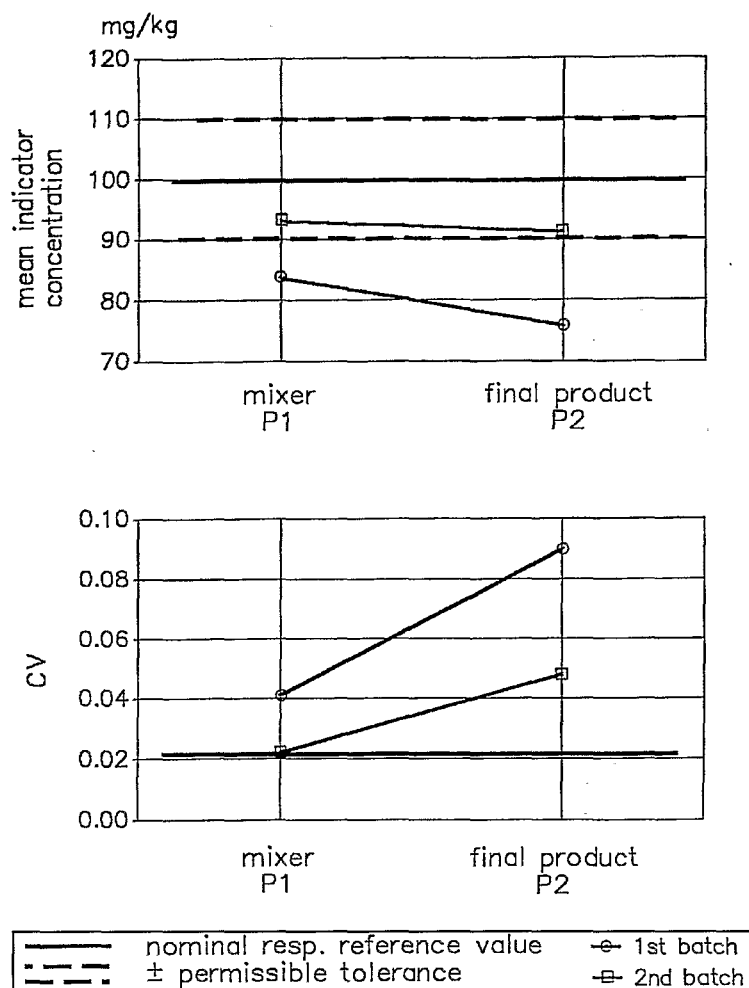


Fig. 2. Comparison of concentration and homogeneity.

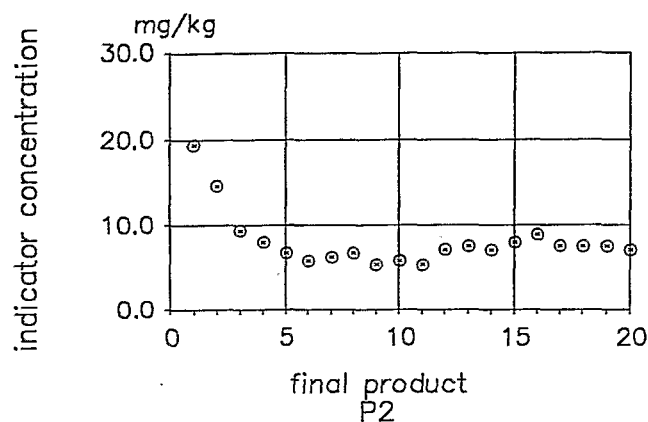


Fig. 3. Contamination of the rinsing batch.

The required working accuracy of the production plant manufacturing premixtures is 1:100,000 and of the plant producing compound feed 1:10,000. These values are related to the concentration of the considered additive.

The known legal requirements do not contain any quantitative values for the allowed carry-over or contamination of the following batches. The EC-guideline 95/69/EC and the German Feed Decree require technical and organizational measures in order to avoid cross contaminations and errors as far as possible, that means in accordance with the up-to-date technical level.

The market demands become more rigorous, especially regarding the so-called critical additives of the categories A, D and J as well as selected trace elements (e.g., copper, selenium). Some contracts exclude not only the use of performance promoters as example, they do not allow any content in the compound feed. Because of the real probability of the carry-over and contamination of following batches such absolute requirement can be fulfilled by separate and specialized production lines only. The marketing association (productshop) for animal feed of the Netherlands has the responsibility for the GMP standard which contains limits for permitted carry-over. These limits for selected additives are deduced from the maximum residue level (MRL-values) for meat. The lowest permitted carry-over is expressed for nicarbazine with 0.1 mg/kg, i.e., less than 0.5% of the used concentration. In case of relative high level of carry-over of the production plant the number of required rinsing batches can be calculated in order to meet the limits for cross contamination.

Investigations of the working accuracy carried out by the IFF Research Institute Feed Technology at more than 350 feed mills show contamination rates for the following batch between 4 and 10% for compound-feed production plants and between 1 and 5% for premix plants.

Although no quantitative criteria for the acceptable working accuracy of the production plant for compound feed are given by legislation limits are necessary for evaluation of the test results. Taking into account the permitted tolerances for additive concentration the experimentally determined concentrations should be within the range of $\pm 10\%$ of the nominal concentration. With regard to the technical level the homogeneity of the mixture evaluated by the coefficient of variation CV should be less than 0.07 in case of mainly organic components and less than 0.10 for mineral mixtures. Carry-over and contamination of the following batch should be minimized as far as possible, i.e., lower than 4% for manufacture of compound feed and lower than 1% for premix plants.

Considering the market requirements and the existing experience especially from more than 350 tested production plants for premixes and compound feed it is difficult to meet these requirements. That is the reason for installation of separate production lines especially for premixes, i.e., separate line for premixes containing critical additives, separate line for so-called clean premixes and separate line for medicated premixes. Another way consists in the use of rinsing batches and/or restrictions for the sequence of the formulations in case of only one available production line, but not every carry-over effect can be compensated by rinsing batches.

Causes of additive carry-over and its avoidance

Different causes of additive carry-over are known, e.g.:

(i) Segregation because of differences in densities and/or particle sizes between additives, carriers and macro components of the compound feed and insufficient bond of the micro components to the macro components.

(ii) Segregation because of the mixture stress at the processing steps (mainly conveying processes, filling and emptying processes for bins and silos).

(iii) Formation of residues in parts of the equipment caused by insufficient emptying of the mixer, bins or silo cells as well as by overcrowding of elevators.

(iv) Formation of residues in parts of the equipment as sticks or crusts of parts of the mixture as well as of demixing products as consequence of incorrect admixing of liquids.

The mentioned effects depends on the used materials as well as on the elements and structure of the production plant. Mainly relative fine particles having the ability for aerosol formation tend to segregation and carry-over. Therefore the dusting behaviour, existing streaming forces, adhesion and electrostatic effects support the carry-over of additives. The first three mentioned effects causing

carry-over can be compensated by rinsing batches because the so-called dilution model can be used for explanation of the effect of rinsing batches. The last mentioned effect causes carry-over stochastically and any compensation by rinsing batches is not plausible.

Each mixture of disperse solids as used in feed compounding is quasi instable, i.e., each handling of the mixture has to take into consideration this relative instability and the possibility of segregation. Long conveying ways, vertical transports by elevator, conveying of the premixes through the screen before the hammer mill and through the hammer mill itself increase the probability of segregation and carry-over. Unprofessional admixing of liquids in order to reduce dusting behaviour can cause the formation of residues as sticks or crusts. Carry-over and cross contamination are often caused by handling errors and or defective working diagram of the production plant.

Stabilisations of the homogeneity of solid feedstuff mixtures are possible by:

- (i) Adaptation of the structure and densities of the mixing components.
- (ii) Homogeneous addition of liquid components (e.g., molasses, fat) for supporting the agglomeration of the fine parts.
- (iii) High intensity conditioning including mechanical shearing stress for formation of a matrix structure which envelops the fines.
- (iv) Pelleting or expanding the mixture.

An elementary requirement for a successful mixing process is the correct concentration of the feed additive in the final mixture, beginning with the accurate dosing of the additives.

For manufacturing stable premixtures the selection of the well-structured carrier is very important (Heidenreich and Löwe, 1995). The main point is the irregular shape of the carrier particles which enables the binding of the fine additive particles with the carrier ones by shape structure. Fig. 4 shows the mixing and segregation properties of different carrier substances obtained by experimental investigations at the IFF Research Institute of Feed Technology. Because of the reduction of the particle shape irregularities by comminution a change for the worse of these properties can be expected.

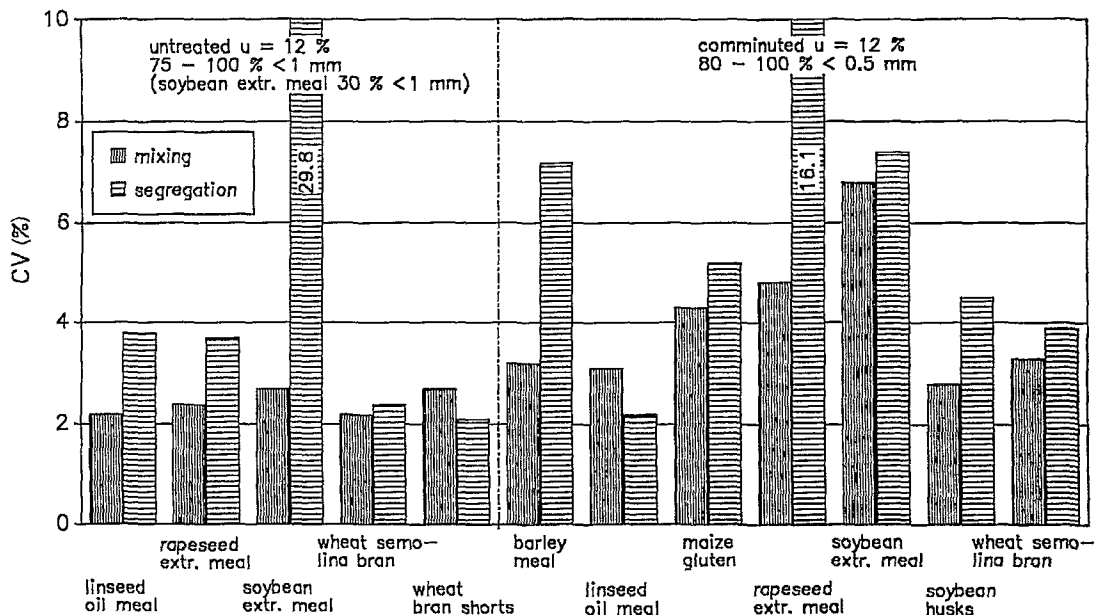


Fig. 4. Coefficients of variation for indicator/carrier mixtures after mixing and pneumatic conveyance.

For selection of the mixer different aspects should be considered. Because of the possible difficulties for axial mixing the length-diameter ratio of the mixer should be in the level of 1:1. Mixtures of disperse solids have a flowing behaviour in the mixer which is similar to liquids of high viscosity. Therefore the experience shows that mixers with low number of revolution and mixing tools covering the total volume are more effective than mixers with high number of revolution and few mixing tools only. Using up-to-date mixers the required homogeneity can be obtained after a mixing time of approximately 1 minute. For avoidance of residues large opening for assurance the total emptying and possibilities for cleaning respectively self-cleaning system are essential aspects.

Segregation caused by dusting behaviour, streaming forces, adhesion or electrostatic effects delivers lower concentrations than have in view. It can be an essential source for undesired contamination of the following batch. In order to avoid these effects, a so-called new generation of additives are in development and partly on the market which have a reduced dusting ability and/or antistatic properties. These additive preparations (granulated products) have coarser particle sizes than conventional ones (Heidenreich *et al.*, 1992). In cases of very small quantities of the addition of these micro components with relatively coarse particle sizes, inhomogeneities of the mixture can be expected because of insufficient numbers of the particles. The distributibility of coarse disperse additives is limited.

For reduction of the probability of cross contamination additional measures have to take into consideration:

- (i) Careful cleaning of the delivery point for raw material.
- (ii) Complete emptying of the dosing cells and buffer boxes in case of product changes.
- (iii) Careful feeding of liquids in order to avoid the formation of sticks or crusts as well as periodical removal of deposits (e.g., in screw conveyers too).
- (iv) Control of the emptying of the mixer, pre bins and post bins as well as the state of wear of the conveying equipment.
- (v) Realization of short conveying ways for the mixtures without intensive mechanical stress.
- (vi) Avoidance of oversized conveying elements which have usual a store effect.
- (vii) Direct addition of the additives or premixture into the mixer.
- (viii) Minimum hindrances and small-radius bends in tubes.
- (ix) Correct back-feeding of aspiration and filter dusts.
- (x) Keeping the restrictions for the sequence of different formulations.
- (xi) Cleaning of the production plant after manufacture of medicated feed by rinsing batches.

The most of the mentioned activities for cleaning, maintenance and control are structured as elements of the quality-management system, e.g., according to ISO 9000 at seq. or HACCP or GMP in the meantime. The required proof of the inspections, cleanings and other measures into the system of internal audits supports the effectiveness of the activities for avoidance of carry-over and cross contamination.

Addition of liquid components

Because of the limited stability of sensitive additives as vitamins, enzymes or probiotics their availability as liquid formulation is increasing which enables the spraying of these additives after high-intensive treatment processes on the agglomerates (e.g., pellets, expandates, extrudates). The application of liquid formulated additives and the realization of the spraying process at the end of the production line can be a contribution for the avoidance of carry-over and cross contamination. Before

each spraying process the fine particles (abrasion of the agglomerates) has to be separated because of their relative high surface area and carry-over potential. In order to ensure the sufficient distribution of the liquid formulated additives the well-designed spraying process for single liquid additives has to be used. For liquid application of selected additives, different technical principles and equipments are in discussion or on the market.

Figure 5 shows the principle of the thin layer apparatus. The feed agglomerates passes a cone as a static element and the thin layer will be arise for offering the maximum of the surface area in order to realize an intensive contact between the agglomerates and the droplets of the sprayed additives. By the free falling motion of the agglomerates an additional temperature drop takes place and a low mechanical stress appears only. Using the rotation symmetrical design several low throughput nozzles should be applied for good distribution of the droplets on the feed agglomerates.

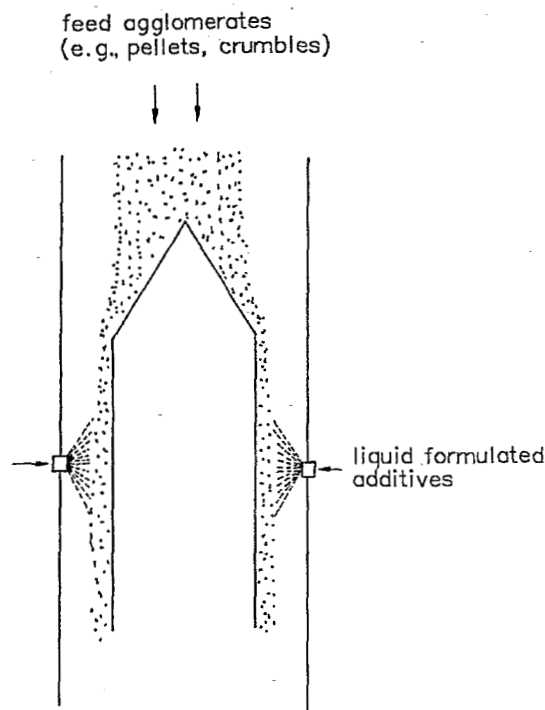


Fig. 5. Thin layer apparatus.

The essential element of the spraying system in Fig. 6 is the spraying chamber with rotary sprayer and solid distributor (Peisker, 1993). The distribution of the feed agglomerates for offering their surface area takes place firstly by the static cone element and secondly by the slowly rotating distributor. The 4 disks rotary sprayer ensures the desired droplet sizes independently on the liquid throughput. It is suitable for different liquids without the danger of disturbances and pollution (Schubert *et al.*, 1990). This spraying systems gravimetrically meters the feed agglomerate flow in the range up to 20 t/h and it doses the liquid formulated additives at amounts higher than 50 ml/t. Experimental investigations carried out by the IFF Research Institute Feed Technology have shown that homogeneities with $CV < 0.1$ can be achieved if correct dosing of the components is carried out.

Nearly independently on the design of the spraying system the realization of the optimal droplet size distribution needs attention. For homogeneous distribution of the additives in the feed a high number of small droplets seems to be the best. But, the mass of fine droplets is very low so that they follow more the stream forces than the mass forces. The consequence is the transport of the fine droplets with the air. Therefore, the optimization of the spray structure in dependence of the quantity of the liquid formulated additives should not be neglected. The aim should be a narrow logarithmic distribution of the droplet sizes.

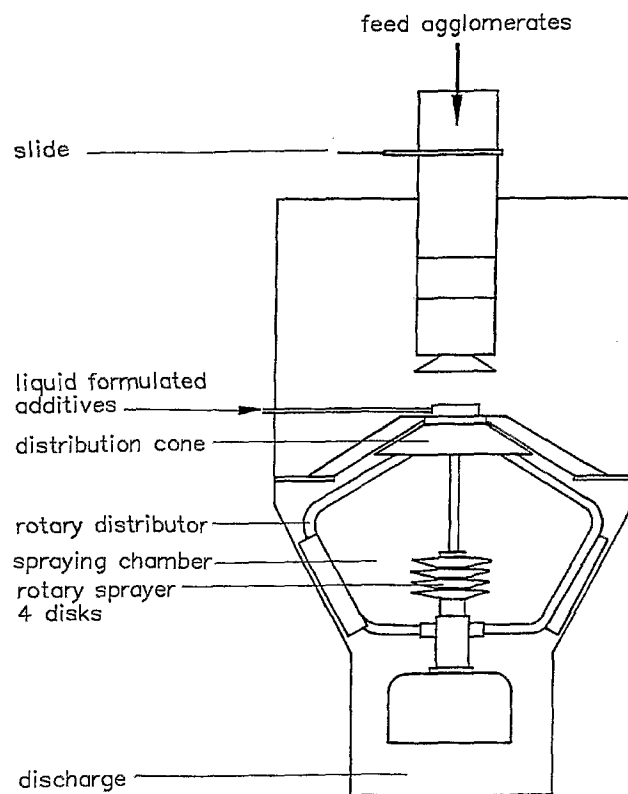


Fig. 6. Spraying chamber with rotary elements.

Another tendency is the addition of non-sensitive liquid components with quantities in the percentage level (e.g., molasses, fat, amino acids) into the main mixer. For assuring the required homogeneity of the mixture and avoidance of carry-over as sticks and crusts in the mixer some aspects has to be taken into consideration:

(i) Admixing of liquid components after sufficient mixing of the solid components (depending on the type of the mixer, less than 1 minute possible).

(ii) Paddle mixers with a low length-diameter ration ($\approx 1:1$) are better suitable for admixing of liquid components than ribbon mixers with high length-diameter ratio ($\approx 3:1$).

(iii) The liquid component should be careful distributed over the mixer volume avoiding direct contact with the mixing tools and walls.

(iv) Feed structures with high portion of fines (e.g., broiler feed) tend more to formation of agglomerates than coarse feed structures (e.g., layer meal).

These aspects can be demonstrated by results of admixing tests for crystalline methionine (DLM) and liquid formulated methionine hydroxanalogon (MHA) carried out by the IFF Research Institute Feed Technology. Both formulations were admixed into broiler feed and layer meal using a twin-shaft paddle mixer (PM) and a twin-shaft ribbon mixer (RM) as usual in feed mills respectively.

Figure 7 shows the independence of the achieved mean concentration and good homogeneity ($CV < 5\%$) from the structure of the feed and type of tested mixer for DLM. The addition of the liquid formulated MHA was realized: (i) by hand; (ii) by multiple run in (at ambient pressure); and (iii) by multiple spraying using nozzles. The results (Fig. 8) demonstrate the advantages of spray technology and the disadvantages of the ribbon mixer for admixing of the investigated liquid component. There was a high portion of agglomerates which cannot be destroyed in the ribbon mixer. Therefore, the

insufficient distribution of the liquid component in the mixture was obtained. Any prolongation of the mixing time did not improve the homogeneity into the range CV<10%.

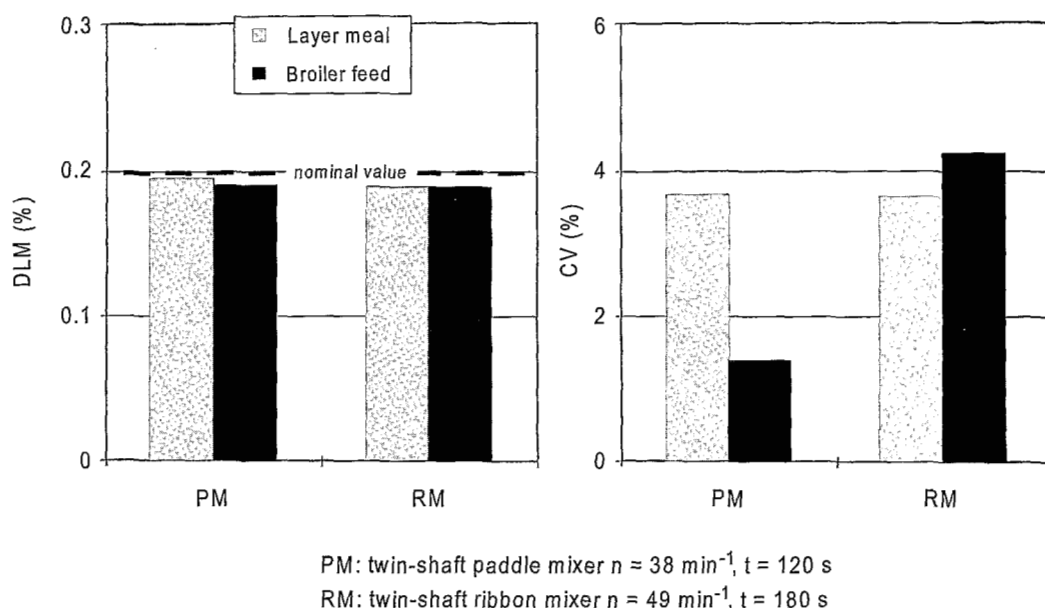


Fig. 7. Concentration balance and homogeneity for DLM, different mixers and feed structures.

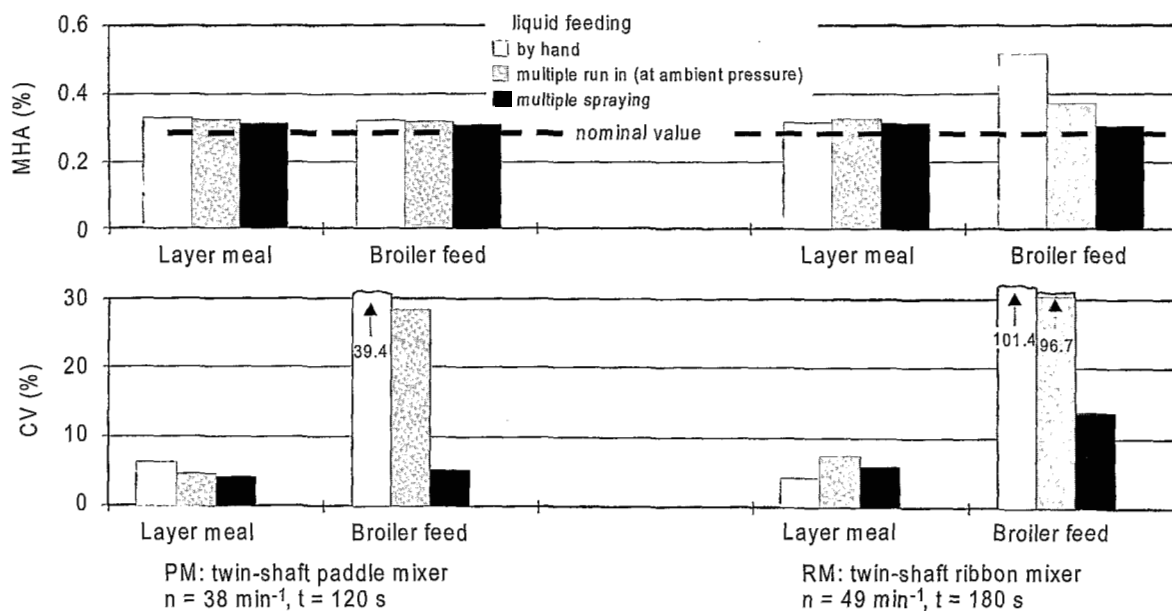


Fig. 8. Concentration balance and homogeneity for MHA, different mixers, feed structures and liquid feeding procedures.

Conclusions

The level of carry-over is an essential part of the working accuracy of the production plant for compound feed or premixes which has to be determined experimentally for the proof of the quality

ability. In order to meet especially the market requirements different measures are necessary, mainly in cases of one-line production plants manufacturing all types of animal feed or premixes. The measures needed in a company should take in mind the shown causes of carry-over and the recommendations for the avoidance.

The addition of liquid formulated additives on feed agglomerates at the end of the production line can reduce the carry-over level if the fine particles are separated before and a tested well operating spraying equipment is selected. Unprofessional admixing of liquid components in the main mixer can cause insufficient homogeneity by formation of agglomerates and deposits on the mixing tools and walls. These deposits as sticks and crusts cause carry-over and cross contamination stochastically which cannot be compensated by rinsing batches. Mainly in cases realizing the mixing process by ribbon mixer solid disperse components have a better suitability than liquids.

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