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Evaluation of Mixing Efficacy of Carriers for Supplemental Nutrient Premixes in Animal Feed

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Summary

Animal feed is commonly formulated with vitamin and mineral premixes to supply the micronutrient requirements of the animal. Premixes typically require carriers which act as a delivery system for micronutrients to aid in uniformity, dilution, and dispersibility across the entire mix. However, little information is available regarding how different carriers might influence the dispersion of nutrients throughout a premix. Therefore, the main objective was to develop a method to systematically evaluate the mixing efficacy of different carriers in a premix formulation. A currently established analysis of mixer uniformity was adapted and repurposed for the evaluation of the premix carriers, rice hulls, pea fiber, and soy hulls. These carriers were evaluated in duplicate 40-lb batch sizes using a single-shaft, double ribbon horizontal mixer. Additionally, these carriers were described based on their physical properties and flow behavior. For the mixer efficacy evaluation, pea fiber had the largest coefficient of variation (CV) (17.08%). Rice hulls, which are the industry standard, and soy hulls were similar to one another (6.02 and 5.36%, respectively) and had acceptable CV. Soy hulls had the largest mean particle size (802.46 μm ± 2.04), followed by pea fiber (458.42 μm ± 2.84), and rice hulls had the smallest mean particle size (339.35 μm ± 1.79). Soy hulls had the largest critical orifice diameter (COD), followed by pea fiber, and COD was smallest for rice hulls, (30, 28, and 20 mm, respectively). Pea fiber had the largest angle of repose at 41.00° and was followed closely by rice hulls at 40.24°. Soy hulls had shallowest angle at 35.28°. The particle size analysis, COD, and angle of repose give useful indications of an ingredient's handling behavior. However, there is no easily discernible relationship between these tests and mixing efficacy of these carriers. This supports the utility of a separate analysis to evaluate mixing efficacy for specific ingredients. Rice hulls are the industry standard and continue to rank better on numerous metrics than other carriers, including their lower cost and improved ingredient-handling capabilities. Rice hulls and soy hulls also had better mixing uniformity, indicating that these two ingredients would better facilitate the distribution of nutrients across a premix. However, some sectors of the feed/pet food industry have pushed to remove cereal grains and/or soy from their products. With these market demands, feed manufacturers may need to find alternatives to the more traditional ingredients that can be evaluated by these techniques.

Introduction

Mixing is a crucial step in the manufacturing of animal feed, necessary for the uniform distribution of nutrients or medication, and to meet the label guarantees.[1] Failure to achieve an adequate dispersal of ingredients in a ration can lead to nutrient toxicity in one portion of a product container and deficiency in another. This is especially pertinent in regard to supplemental nutrient premixes, which are often utilized to meet micronutrient requirements. Supplemental minerals and synthetic vitamins may have physical characteristics, which hinder their dispersal across a ration. Therefore, it is common to formulate premixes with a carrier to help facilitate uniform distribution during mixing.

Carriers play an important role in premix formulation as they provide a delivery system for micronutrients; carriers also help to improve uniformity, dilution, and dispersibility across the entire mix. Careful consideration should be given when selecting a carrier for use in supplemental premixes in order to sufficiently achieve these goals. To date, the most used premix carrier has been rice hulls. Recently, some companies have utilized pea fiber, and others have used soybean hulls. However, little information is available regarding how different carriers might influence the dispersion of nutrients throughout a premix. There is need to be able to systematically evaluate the mixing efficacy of different carriers. Thus, the objective here was to evaluate three carriers within a standard premix formulation, by adapting a currently established analysis of mixer uniformity. Additionally, these carriers were described based on their physical properties and flow behavior.

Materials and Methods

The test ingredients included rice hulls, pea fiber, and soybean hulls (Lortscher's Animal Nutrition Inc. Bern, KS), all of which are commonly utilized as premix carriers within feed industries. These carriers were evaluated for mixing efficacy, particle size distribution, and flow ability characterized by critical orifice diameter and angle of repose.

Mixer efficacy analysis

This analysis was an adaptation of the Extension Service Bulletin *Testing Mixer Performance.*[2] Two 40-lb batches were mixed and evaluated per treatment, as shown in Table 1. Each test batch was conducted between 50–75% of rated mixer capacity. Mixer operation was performed using a 2-ft^3 single-shaft, double ribbon horizontal mixer (Hayes & Stolz model HP2SSS0106, Fort Worth, TX). During operation, all dry ingredients were added to the mixer, starting from highest to lowest inclusion and this was blended for 1 minute. After this, mineral oil was added and mixing continued for 3 more minutes. At 4 minutes, roughly half of the mixer's contents were discharged into a separate container, and immediately placed back into the mixer to help clear out any dead space between the ribbons and the side walls. The ingredients were mixed for an additional 3 minutes for a total 7 minutes of mixing time. The mix was then discharged

[1] Stark, C & Saensukjaroenphon, M. 2017. Testing Mixer Performance. MF3393. Kansas State University Agricultural Experiment Station and Cooperative Extension Service Bulletin, Manhattan, KS: Kansas State University.

[2] Herrman, T. & Behnke, K. 1994. Testing mixer performance. MF1172. Kansas State University Agricultural Experiment Station and Cooperative Extension Service Bulletin, Manhattan, KS: Kansas State University.

from the bottom slide gate and 10 samples were collected at equally spaced 10 second intervals, placed in resealable plastic bags, and stored at -4°F for later analysis.

Samples were later ground with a coffee grinder, tumble-mixed for subsampling, and analyzed for salt concentration by Quantab chloride titrator test strips (Hach Company, Loveland, CO). Briefly, 10 g of each ground sample was placed in a plastic disposable cup and mixed with 90 g of boiling distilled water. The mixture was stirred for 30 seconds, allowed to rest for 60 seconds, and stirred again for an additional 30 seconds. Filter paper was folded into the shape of a cone and placed inside the cup, allowing the liquid to pool at the bottom without any particulate. A Quantab strip was inserted into the liquid at the bottom of the filter paper to measure the percentage of salt within the sample. A coefficient of variation (CV) was computed for salt content among the 10 samples of each batch. Each treatment was evaluated in duplicate with two separately mixed batches serving as experimental units and the mean CV was reported.

Particle size distribution

Particle size analysis[3] was completed on a Ro-Tap machine (Model RX- 29, W. S. Tyler Industrial Group, Mentor, OH) using a 13-sieve stack with the inclusion of sieve agitators and flow agent, tapped for 10 min.[4] Sieves were weighed and the amount of material on each sieve was used to calculate the geometric diameter average (d_{gw}) and geometric standard deviation (S_{gw}).[3] Sieves were cleaned after each analysis with compressed air and a stiff bristle sieve cleaning brush. Each carrier was evaluated in duplicate and the mean weights retained on each sieve were used to calculate geometric mean and standard deviation.

Flowability characteristics

The flowability characteristics of test carriers were evaluated using the results of angle of repose and critical orifice diameter (COD). Angle of repose was determined by allowing a sample to flow from a vibratory conveyor above a free-standing platform until it reached maximum piling height.[5] Each carrier was evaluated in duplicate and mean angle was reported. The critical orifice diameter, defined as the smallest diameter opening through which the material will pass unaided,[6] was determined using a powder flowability test instrument (Flodex Model WG-0110, Paul N. Gardner Company, Inc., Pompano Beach, FL) to represent ingredient flow characteristics in bins. Discs were used to determine the appropriate bin hole opening for material to flow freely. Three sequential positive results were then used to determine the critical orifice diameter.

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[3] ASABE Standards. 2008. S319.2: Method of determining and expressing fineness of feed materials by sieving. St. Joseph, Mich.: ASABE.

[4] Kalivoda, J. R., Jones, C. K., & Stark, C. R. 2017. Impact of varying analytical methodologies on grain particle size determination. Journal of Animal Science, 95(1), 113–119.

[5] Appel, W. B. 1994. Physical properties of feed ingredients. 4th ed. Feed Manufacturing Technology, (pp. 151). Arlington, VA: American Feed Industry Association, Inc.

[6] Taylor, M. K., Ginsburg, J., Hickey, A. J., & Gheyas, F. 2000. Composite method to quantify powder flow as a screening method in early tablet or capsule formulation development. AAPS PharmSciTech, 1(3), 20–30.

Statistical analysis

Mixer data were analyzed using statistical software via the ANOVA procedure (SAS v. 9.4, SAS Institute, Inc., Cary, NC). Treatment batches were performed in duplicate and the resulting CV was used as the experimental unit in a completely randomized design. Results were considered significant at a $P \leq 0.05$ and treatment means were separated using Tukey's test. Particle size distribution was performed in duplicate and average weight retained in each sieve was used to generate a single value reported as the mean ± standard deviation. Angle of repose was also performed in duplicate and mean value was reported. For the critical orifice diameter, three sequential positive results were used to confirm a single reported value.

Results and Discussion

Several tests have been developed to evaluate the performance of mixing equipment; either to optimize the mixing process or to test when equipment maintenance is needed. The industry standard is to use salt (as the source of chloride) as an indirect marker of uniformity. After a specified mix time, samples (typically 10) are collected from either equally spaced locations within the mixer or from the discharge port at equally timed intervals. The collected samples are then analyzed for salt concentration using chloride titrator test strips, and a CV is calculated among the samples. As a general rule, the CV should be less than 10%.[7] These tests are generally used to evaluate the mixer itself and may be employed for two main reasons: 1) testing the optimal mixing time of a new mixer with commonly used ingredients (i.e., soybean meal or cereal grains) or diet formulations; or 2) evaluating the performance of a mixer with an already established optimum mixing time as a function to test when preventative maintenance is needed. In these two examples, any increase from an ideal CV would be attributed to improper mixing time or wear of internal parts, respectively. However, when an optimum mixing time is well established and the mixing equipment is maintained, this method may also be adapted to evaluate how new ingredients may be affecting the mixing process.

The main goal of the current experiment was to evaluate the efficacy of each carrier when used in a typical premix ration. A standard premix formula was emulated (Table 1) in which: calcium carbonate represented the mineral portion, corn was the vitamin portion, salt was used as a marker, and mineral oil provided dust suppression. Batches differed only by test carriers and were otherwise subjected to identical mixing conditions. Pea fiber had the largest CV (Table 2; 17.08%) and was above an ideal CV of 10% and considered "fair".[2] Rice hulls, which are the standard, and soy hulls were similar to one another and had acceptable CV between 5 and 10%. Assuming equipment wear and ingredient sequencing are not a concern and are held constant across treatments, the suggested remedy would be a 50% increase in mixing time. While this is an easy corrective action, it consumes time and energy. Alternatives, such as rice hulls and soy hulls, with lower CV would offer improved efficiency to the manufacturing process.

A number of lab-scale tests were also employed to help describe the flow properties of these ingredients; the tests may provide some insight into the ingredient handling behavior of these materials. Ground ingredients are comprised of many particles

[7] Fahrenholz, A. & Stark, C. R. 2014. Mixing feeds and mixer test procedures for batch mixers. Feed Additive Compendium, Pages 105-108. ed. T. Lundeen, Minnetonka, MN: Miller Publishing Co.

that can vary widely in size depending upon the material composition, screen used in grinding, and method of milling. Among the tested samples, rice hulls had the smallest geometric mean particle size (Table 3; 339.35 μm ± 1.79). Soy hulls had the largest mean particle size (802.46 μm ± 2.04) and pea fiber had an intermediate mean particle size (458.42 μm ± 2.84). Rice hulls also showed the most consistency in particle size, having the smallest variation among treatments. Pea fiber had the greatest variation in particle size, whereas soy hulls were intermediate. Considering particle size and geometry play a large role in the ingredient handling properties of feed ingredients, consistency in particle size is a favorable characteristic for better prediction of ingredient behavior.

As previously mentioned, the COD is defined as the smallest diameter through which a material will flow. Free flowing materials tend to flow through the smaller diameters, while more cohesive materials flow through larger diameters. Soy hulls had the largest COD, followed by pea fiber, and COD was smallest for rice hulls, (30, 28, and 20 mm, respectively). The angle of repose is one of the simplest and most common methods used to evaluate and compare flow properties. It is standard that a shallower angle indicates better flow properties. Pea fiber had the largest angle of repose at 41.00° and was followed closely by rice hulls at 40.24°. Soy hulls had shallowest angle at 35.28°.

The previously described tests are related to the flow of dry materials through bins, hoppers, and feeders. However, their ability to predict the mixability of an ingredient is not obvious. Additionally, individual flow tests usually only challenge a single aspect of the phenomenon known as flow. This allows the possibility for different tests to produce conflicting results for the same test ingredient, presenting challenges in the evaluation and comparison of different samples. For example, the COD method is supposed to be a direct measure of powder cohesiveness and arch strength;[8] whereas the angle of repose is an indirect method which also characterizes the cohesive properties of a material, associated with particle size, particle shape, and moisture content.[9] Soy hulls had the worst performance for the COD test and the best performance for the angle of repose. Soy hulls also had the best mixing performance and the largest particle size. Rice hulls had the best COD performance, smallest particle size, and was very close in the mixing performance to that of soy hulls. Pea fiber had an angle of repose and particle size that was much closer to that of rice hulls than soy hulls, but had far worse mixing performance than the other two samples. It appears that there is no easily discernible relationship between either of these flow tests or particle size distribution and the mixing efficacy of these carriers; demonstrating the utility of a separate analysis.

Rice hulls continue to rank better on numerous metrics than other carriers with their apparent low cost, improved ingredient handling capabilities, and low impact on formula palatability. It has been previously reported that rice hulls have low density, low variation in particle size, and low moisture absorption.[10] It was observed here that rice hulls and soy hulls both had better mix uniformity based on salt concentration

[8] Horn, E. 2008. Development of a composite index for pharmaceutical powders. Ph.D. dissertation, Potchefstroom, South Africa: North-west University, School for Pharmacy.
[9] Guo, Y., Wassgren, C., Ketterhagen, W., Hancock, B., & Curtis, J. 2018. Discrete element simulation studies of angles of repose and shear flow of wet, flexible fibers. Soft Matter, 14(15), 2923–2937.
[10] Ahna, G. C., Jang, S. S., Kwak, H. J., Lee, S. R., Oh, Y. K., & Park, K. K. 2016. Characteristics of rice hulls, sawdust, wood shavings and mixture of sawdust and wood shavings, and their usefulness according to the pen location for hanwoo cattle. Asian-Australasian Journal of Animal Sciences, 29(4), 599–605.

variability, indicating that these two ingredients would better facilitate the distribution of nutrients across a premix. It is therefore reasonable to assume that greater uniformity across a premix would also result in superior dispersion of nutrients when included into an entire dietary ration. While rice hulls and soy hulls outperformed pea fiber in these tests, it is likely that other ingredient-sourcing constraints could present different results. Compositional differences are common among agricultural by-products, such as fiber sources, and can be different between ingredient suppliers as well as from lot to lot for the same product. This procedure merely provides a basis for which new ingredients or characteristics can be deliberately and systematically tested in the laboratory to provide insight into their use as carriers. Further, some more "premium" sectors of the feed/pet food industry may push for removal of any traces of cereals and/or soy from their formulas. With these market demands, feed manufacturers may need to find possible alternatives to the more traditional ingredients that can be evaluated by these techniques.

Table 1. Experimental premix formulation (as-is basis)

Ingredients	Inclusion, %
Carrier[1]	48.0
Calcium carbonate	44.7
Corn	6.0
Salt	0.3
Mineral oil	1.0

[1] Carrier sources: rice hulls, pea fiber, and soy hulls.

Table 2. Mixer efficacy analysis of each test carrier

Treatment ration[1]	Mixing efficacy CV, %[2]
Rice hulls	6.01[a]
Soy hulls	5.42[a]
Pea fiber	17.08[b]

[1] Ration based on test carrier (48%), calcium carbonate (44.7%), ground corn (6.0%), salt (0.3%), and mineral oil (1.0%).
[2] Coefficient of variation: (standard deviation/mean) × 100.
[ab] Means with unlike superscripts differ ($P = 0.0073$).

Table 3. Particle size, critical orifice diameter, and angle of repose of each test carrier

Sample	Particle size[1]	Critical orifice diameter, mm	Angle of repose
Rice hulls	339.35 ± 1.79	20	40.24°
Soy hulls	802.46 ± 2.04	30	35.28°
Pea fiber	458.42 ± 2.84	28	41.00°

[1] The $d_{gw} \pm S_{gw}$ (geometric mean ± standard deviation).