THE EFFECT OF INGREDIENT PROPERTIES, LIQUID SYSTEM AND MIX TIME ON UNIFORMITY OF MIX AND TESTING OF UNIFORMITY OF MIX

by

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Abstract

The uniformity of a feed mixture is determined from the coefficient of variation (CV) of ten samples in a single batch of feed. The feed industry standard is a %CV of less than 10 using a single source tracer such as salt, trace minerals or iron filings. The uniformity of mix can be affected by many factors including ingredient properties, equipment design, mix time, sampling method, sample preparation and overall precision of method. The objectives of this thesis were to verify the chloride ion test by the Quantab® chloride titrator method and to determine the effect of extended mixing time, salt particle size, sample preparation, wet mix time, spray condition, application time of the liquid addition, percent liquid addition and mixer type on the uniformity of mix. The first experiment evaluated the effect of water temperature, technician, extraction time, dissolution time on the chloride ion test by the Quantab® chloride titrator method. The salt concentration was significantly affected by water temperature and dissolution time. The results of Experiment 1 demonstrated the 60ºC water temperature and the 30 s extraction time by stirring followed by immediately placing the strip into the solution after extraction should be used for the Quantab® chloride titrator method. The second experiment evaluated the effect of extended mix time, particle size of salt and sample preparation on the %CV. The extended mix time did not result in segregation ($P > 0.30$). However, particle size of the salt and sample preparation significantly affected the uniformity of mix. The results of Experiment 2 indicated that the extended mix time of up to 60 minutes did not increase the %CV of the feed; the particle size of the salt used in the uniformity of mix test can significantly change the results of the test; and grinding the sample prior to analysis improved the precision of the Quantab® chloride titrator method when coarse salt is used in the manufacturing process. The third experiment evaluated the effect of wet mix time and the timing of the liquid addition using different spray

conditions on the %CV. The wet mix time significantly changed the %CV of the mixture (P = 0.0057). The use of a spray nozzle had no effect on the %CV of the feed mixture when a 1.14% of a 23% saline solution was sprayed on to the feed (P = 0.7435). The results of Experiment 3 indicated the wet mix time had a greater influence on the uniformity of mix than the type of nozzle used to apply the liquid; and the shorter liquid application time allowed more time for the mechanical shear of the ribbons and paddles to break up the agglomerated wet particles and distribute them throughout the feed mixture when the total time of the liquid addition plus wet mix time was fixed. The fourth experiment evaluated the effect of different percent liquid addition, application time and mixer type with different wet mix times on the uniformity of mix. The %CV of feed mixed using a fixed wet mix time setting did not change when the percent liquid addition was increased, while the %CV of the feed mixed using a fixed total liquid mix time setting increased when increasing the percent liquid addition. The %CV of feed mixed with a ribbon mixer did not change when the liquid application time was decreased while the %CV of the feed mixed with a paddle mixer increased when decreasing the liquid application time. Differences were observed in the %CV based on the parameters of liquid cycle time, mixer type and mixer size. The results of Experiment 4 indicated the liquid addition time and the percent of liquid addition affected the uniformity of mix. The uniformity of liquid application should be tested with the highest percent liquid addition. Furthermore, application and wet mix times should be determined for each mixer type and size to establish the optimal batch cycle.

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Acknowledgements

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Chapter 1 - Uniformity of Mix

Introduction

The mixing process is one of the most important steps in feed manufacturing. The goal of mixing is to produce a perfect mix, "where the probability of selecting a particular type of particle is the same at all positions in the mix and is equal to the proportion of such particles in the total mix" (Aulton and Taylor, 2013). This goal will ultimately provide the correct proportion of nutrients for optimal animal growth. The feed industry has historically used the percent coefficient of variation (%CV) to evaluate the uniformity of mix. Several markers used to determine the uniformity of mix include amino acid, salt, micro minerals, drugs and colored iron fillings (Clark et al., 2007). The most common marker used in feed mills is salt, which is added to the diet between 0.3 to 0.5%. The percent of salt in a diet can be determined with a Quantab® strip that measures chloride ions in the mixture. Herrman and Behnke (1994) suggest the mixer should have a %CV below 10 (Table 1.1). Mixer evaluation is also required to comply with the FDA regulations under 21 CFR part 225.30 (b) for a licensed feed mill and under 21 CFR 225.130 for a non-licensed feed mill. Several factors can affect the uniformity of mix, such as ingredient particle size, density, shape and static, sequence of ingredient addition, amount of ingredient filled, mixer design; mix time, cleanliness of the mixer, and wear of the mixer (Herrman and Behnke, 1994).

Uniformity of mix influences animal performance

Several experiments were conducted to study the effect of uniformity of mix on animal performance using different mix times or revolutions to mix the same diet; or feeding two different diets that had different target nutrient composition levels on different daily schedules.

Paulk (2011) stated that the species and age of animals can require a different %CV to obtain optimal performance.

Poultry

McCoy et al. (1994) conducted two experiments to study the effect of mixing uniformity on broiler chick performance. The author used five different markers to determine the uniformity of mix: salt, red particles, blue particles, chromium and sodium. The first experiment evaluated the effect of mixer revolutions of 20, 40 and 80 on broiler performance, which were categorized as a poor, intermediate and adequate uniformity of mix, respectively. The %CV of the poor treatment had significantly higher %CV using chromium as a tracer (49.7, 15.3 and 16.7% for poor, intermediate and adequate mix, respectively) as compared to intermediate and adequate treatments. The intermediate and adequate treatments were similar in %CV. A quadratic response in %CV was observed as the revolutions increased ($P = 0.001$). However, the average daily gain (ADG), average daily feed intake (ADFI), gain:feed ratio (G:F ratio), bone breaking strength, bone ash, carcass crude protein (CP), carcass fat and carcass ash were not significantly different in the 24-d growth assay ($P > 0.12$). The second experiment involved diets formulated at 80% of the National Research Council (1984) nutrient requirements for crude protein, lysine, methionine and calcium while the other nutrients were formulated to meet or exceed nutrient requirements. The number of revolutions was also changed from 20, 40 and 80 revolutions to 5, 20 and 80 revolutions to create the poor, intermediate and adequate treatments. The %CVs of poor, intermediate and adequate treatments were 40.5-53.9%, 12.1-30.0% and 9.7- 30.3%, depending on the type of marker. Similar to the first experiment, the %CV quadratically decreased as the revolutions increased ($P = 0.001$) for each marker. The average daily feed intake (ADFI) and mortality were not significantly different when the broilers were fed three diet

treatments over a 28-d growth assay. The results of the 28-d growth assay indicated the average daily gain quadratically increased as the revolutions increased in the study ($P < 0.04$). The G:F ratio of the poor uniformity treatment was significantly lower as compared to the intermediate and adequate uniformity treatments. The results of these experiments indicated the uniformity of mix had a dramatic effect on the broiler performance when the broilers were fed at 80% of the requirement as compared to a diet that met or exceeded NCR requirements.

Clark (2006) studied the effect of mixing uniformity on broiler growth performance from d 0 to 41 by varying only synthetic DL-Methionine levels. The corn-soy diet was formulated to meet the NRC requirements for starter (d 0 to 16), grower (d 17 to 32) and finisher (d 33 to 41). The DL-methionine was added and mixed for 10, 20, 30, 40 and 120 s for three phases of growth. The resultant %CVs of starter phase were 145%, 52%, 25%, 17% and 3%, respectively. The ADG and F:G ratio quadratically decreased as the mix time increased ($P < 0.001$) for starter phase. The ADG, ADFI and F:G of the broilers in grower and finisher phases as well as overall were not affected by decreasing methionine CVs of the diets which were fed from 135 to 4%.

Research conducted by Johnston and Southern (2000) reported a tendency for poorer ADG and significantly decreased bone strength and bone ash in broiler chicks as the variation in added enzyme was increased by varying the amount of phytase the bird received in the diet.

Swine

Groesbeck et al. (2007) and Traylor et al. (1994) conducted experiments to study the effect of mixing time on nursery pig performance. Both studies concluded that increasing mixing time improved nursery pig performance. Groesbeck's experiments demonstrated increasing mix time from 0 to 330 s decreased CVs from 178 to 5% when the mixer was sampled and from 26 to 7% when the bag was sampled for a phase 1 diet; and from 172 to 26% when the mixer was

sampled and from 56 to 12 % when the bag was sampled for a phase 2 diet. The results of the 28-d growth assay indicated a linear decrease in F:G ratio as the %CV decreased ($P = 0.04$). Traylor's trials concluded increasing mix time had a positive response on %CV. The %CV for Cr were 107%, 28%, 16%, 12% for 0, 30, 120 and 240 s, respectively. A cubic response was observed in F:G ratio as the %CV decreased ($P = 0.02$). However, Paulk et al. (2015) and Traylor et al. (1994) concluded the finishing pig performance was not affected by the mix time. Paulk's experiments indicated increasing mix time from 0 to 360 s and decreasing %CV of Cr from 51% to 15% had no effect on ADG and F:G in the 27-d growth assay. Traylor's experiments indicated decreasing %CV from 54% to 10% also did not increase the ADG and G:F ratio when the finishing pigs were raised from 56 kg to 118 kg.

Testing mixer performance

There have been multiple procedures used in the feed industry to determine the uniformity of mix: chemical assay for drug, mineral, amino acid, chloride ion rapid test – Quantab® strip and colored iron filling – microtracer (Behnke, 1996). The tracer should come from only one source, be approved for use in feed and have a precision method of analysis in order to accurately evaluate the uniformity of mix (Fahrenolz and Stark, 2014). The uniformity of a mixture can be affected by the properties of the tracer, ingredients as well as interaction between tracer and ingredients such as particle size, particle number, density and flowability (Axe, 1995).

Representative samples should be taken in the mixer from 10 different locations or be obtained as near to as the discharge point as possible. Samples taken during discharge of the mixer should be at equally spaced time intervals. Sample preparation has a greater effect when larger particle size is present in the sample as compared to the smaller particle size (Groesbeck,

2004). Decreasing the particle size of the tracer has a positive relationship with the number of particles per gram when the volume or weight is constant (Axe, 1995). The amount of marker per unit increases, yielding a greater recovery of marker within the sample. This will ultimately result in a lower %CV.

ASABE Standard method

The American Society of Agricultural and Biological Engineers (ASABE) developed a procedure to evaluate mixer performance. The "Test Procedure for Solid-Mixing Equipment for Animal Feeds-ASABE S303." ASABE (2012) recommended the addition of 2% sodium chloride salt with a particle size of 450 $\pm$ 100 µm, or 50 g of colored iron particles per ton of feed. However, several methods have been used to analyze the Cl^- concentration in feed. Headley (1967) conducted an experiment to compare three different methods: potentiometric, sedimentation and Quantab® chloride titrator. The results indicated the Quantab® chloride titrator method had the highest %CV (4.66%, 1.30% and 2.84% for Quantab® chloride titrator, potentiometric and sedimentation, respectively). Behnke (1996) recommended the %CV for the method should be less than 5% in order to evaluate mixer performance. Therefore, the Quantab® chloride titrator can be used to determine the uniformity of mix. The Quantab® chloride titrator as described by Nielsen (2010) is a thin plastic strip that is laminated with a capillary column impregnated with silver nitrate and potassium dichromate, which together form silver dichromate. When the strip is placed in a chloride ion solution, the color of the column is changed from a reddish-brown (silver dichromate) to a white color (silver chloride) due to the reaction of the chloride ion with silver dichromate, which creates silver chloride. The reactions in the strip are shown in the equations below (Korkmaz, 2005).

$$Ag_2(Cr_2O_7)_{(s)} \leftrightarrow 2Ag^+_{(aq)} + Cr_2O^{2-}_{7\,(aq)} - Step\ 1$$

$$Ag^+_{\ (aq)} + Cl^-_{\ (aq)} \leftrightarrow AgCl_{(s)} - Step\ 2$$

$$Cr_2O^{2-}_{7\,(ag)} + \ H_2O_{(l)} \leftrightarrow 2CrO^{2-}_{4\,(aq)} + 2H^+_{(aq)} - Step\ 3$$

$$2Ag^+_{\ (aq)} + CrO^{2-}_{4\ (aq)} \leftrightarrow Ag_2(CrO_4)_{(s)} - Step\ 4$$

Another method typically found in the feed industry is colored iron fillings as a tracer. Micro-tracers were developed for testing for completeness of mix with Microtracer F by Micro-Tracers, Inc. (2013). The method determines the number of color spots which were created from the colored iron particles after being separated from the sample by a magnet.

Interpretation of results

The interpretation of mixing uniformity using chemical composition or amino acid or drugs as a tracer can be evaluated by %CV (Behnke, 1996). The %CV is calculated by dividing the standard deviation by the average of 10 samples and then multiplied by 100. Herrman and Behnke (1994) categorized mixer tests as excellent, good, fair and poor based on %CV ranges. A %CV of less than 10% was excellent, 10-15% was good, 15-20% was fair and greater than 20% was poor (Table 1.1). In contrast to %CV, colored iron filling compares the variation of the number of counted spots with the variability characteristic of a Poisson Statistical distribution. If the number of counted spots has less variables than the expected value from Poisson distribution, the mixture is complete or a perfect mix.

Factor influencing mixer performance

The mixing process can be affected by ingredient properties, equipment design and control system during process and management. The three mechanical forces that occur during the mixing process are convection, diffusion and shear (Maynard, 2007). The convection process moves the particles throughout the mixer by the directional force applied by the ribbon or paddle

on the rotating shaft. The second process occurs when the particles are diffused from high density to low density within the mixer. Finally, clusters of particles that were stuck together are broken apart by the shearing force of the ingredient particles, ribbons, paddles and shaft. These particles can then be spread throughout the mixer. Feed typically contains both dry and liquid ingredients. Dry ingredients will absorb liquid into particles then the absorbed particle will be dispersed throughout the mixture, similar to the other dry ingredients (Bunzel, 2008, Figure 1.1).

Ingredient properties

Several ingredient properties can affect the uniformity of mix such as particle size, particle shape, density, hygroscopicity, statistic charging and adhesiveness (Behnke, 1996). Axe (1995) stated "When two ingredients' particle sizes are extremely different, the ingredients can separate." The particle size also affects the number of particles per gram. Increasing particle size decreases the number of particles when the volume or weight is constant. Increasing the number of particles improves the distribution in the mixture. In addition, ingredient particles have different shapes such as a cube, tetrahedron or sphere. Extreme differences in the shape of the ingredient particles may lead to segregation as they are mixed and moved throughout the feed mill (Axe, 1995). Ingredient density also influences the batch size of the mixer. Diets are formulated by weight while a mixer is designed by volume. If the diet contains low density ingredients, the mixture level may be higher than the mixing zone. Over-filling the mixer beyond its rated capacity creates dead spots and causes improper mixing (Vogel and Laudert, 2015). Research conducted by Wicker (1991) demonstrated that when a 5-ton mixer was used to mix 6 tons of a ration, increasing the mix time did not decrease %CV. The %CV was 34.9%, 31.4% and 29.8% for 2.0, 2.5 and 3 min, respectively. However, the %CV of a 5-ton mixer was less than 10% when 5 tons of a ration was mixed for 2.5 and 3.0 min. On the other hand, if the diet

contains high density ingredients, the mixture level may be lower than the optimal mixing zone. Under-filling of the mixer decreases the mixing zone that can lead to poor mixing performance (Froetschner, 2005). Froetschner (2005) recommend the mixture level should be greater than 30% of the mixer rated volume to perform the proper mix.

Equipment properties

The mixing pattern is different for horizontal mixers, vertical mixers and drum mixers. The horizontal mixer is the most common mixer used in the commercial feed industry and vertically integrated feed mills; whereas, the vertical mixer is commonly used in on-farm mills and small feed mills. Horizontal mixer designs continue to evolve to improve the mixer capacity and the uniformity of the mix. Shaft model designs include double shaft ribbon, double shaft paddle, single shaft double ribbon and single shaft paddle. Wilcox and Unruh (1986) demonstrated that different mixer designs have different mixing zone patterns (Figures 1.2-1.5). Ingredients can be uniformly distributed with any type of mixer given the proper mix time. The revolutions per minute (rpm) of the shaft and the tip speed of the ribbons or paddles affected the mix time of the mixer and ultimately the manufacturing capacity of the feed mill. McCoy (1994) conducted an experiment suggesting that increasing the number of revolutions of the ribbons improved the uniformity of the mix. The %CV of salt decreased from 40% to 9.7% when the total number of revolutions increased from 5 to 80 revolutions.

The number and amount of liquids added into a mixer has been increased in the last ten years. Liquids are added to diets to help control dust in the animal production house as well as decrease diet and labor costs. Clark (2009) recommended the liquid addition system should use nozzles to improve the uniformity of mix and to decrease ingredient build up on agitators and side walls of the mixer. The liquid addition system with spray nozzles creates small droplets of

liquid that coat the dry particles and penetrate the particles; as well as cover the greatest number of particles in the mixture. Small droplets are produced when the potential energy of the pressure at the sprayer nozzle is greater than the surface tension of the liquid (Powder and Bulk Engineering, 2011). The factors that affect nozzle pressure include the number of nozzles, nozzle hole diameter, distance between pump and nozzle, pipe diameter and force of the pump. The effectiveness of the liquid addition system is also influenced by the time and duration of the liquid application during the mixing cycle.

Control system

The size, type and design of a mixer can dictate the manufacturing capacity of the feed mill. However, the automation of the batching process significantly increased manufacturing capacity and improved product quality in the last 20 years. The automation control system now controls the batching, mixing and liquid addition processes. Automation not only controls the batching process but also the discharge sequence of the scale, which may affect the uniformity of mix. If the micro scale or mineral scale is discharged before the major scale, the vitamin premix, mineral premix, drug or feed additive could remain in the gap between ribbon or paddle and bottom of mixer tub causing them to not be properly dispersed throughout the mixer. In addition to batching and scale discharge, the automation system controls the mix time, which is separated into two parts: dry mix and wet mix time, and the mix time can influence both feed manufacturing capacity and uniformity of mix. Several studies demonstrated that increasing the mix time improves the uniformity of mix. However, proper mix time varies based on the type of mixer. Moreover, automation systems have developed different approaches to wet mix time. The two most common set-ups found in automated control systems are associated with the wet mix time. The first approach uses a fixed total liquid mix time (liquid addition plus a wet mix) as

compared to a fixed wet mix time after liquid application. The fixed total liquid mix time starts once the liquid pump is activated to begin the liquid addition. The fixed wet mix time set-up starts after the liquid pump stops the addition of liquids, which creates a variable total mix time that influences the total batch cycle time.

Froetschner (2005) recommended that the dry and wet mix time in the automation system should be set based on mixer type (Table 1.2). In addition, the recommendation of Hayes & Stolz (2015), a manufacturer of mixers, suggested the wet mix time should be 75% of the mixer's total mix time based on mixer design. However, the FDA requires all licensed feed mills to test mixers upon installation as well as annually. Therefore, feed mills should not rely on book values but conduct tests at installation and at least annually. Stark (2012) recommended testing a mixer twice a year using different markers.

Maintenance management

Preventive maintenance is an important management tool in feed mills to control manufacturing costs and maintain quality. A worn or improperly adjusted mixer can affect the efficiency of mixing (Vogel and Laudert, 2015). Wilcox and Unruh (1986) studied the effect of ribbon wear and paddle wear on mix time. The results of the experiment demonstrated that a worn outer ribbon on a 2-ton double-ribbon horizontal mixer could not produce an acceptable %CV (<10%) when the feed was mixed from 3 to 10 min. However, after the outer ribbon was replaced, the %CV was less than 10% when the feed was mixed at 4 min. The research also demonstrated the same effect with a worn paddle mixer. Initially, the paddle mixer could not achieve the target %CV, but after replacing the paddles the %CV was achieved in 3.5 min. The cleanliness of a mixer can also be shown to influence the mixing efficiency. Ingredient build-up such as molasses on mixer ribbons and paddles decreases the efficiency of mixer (Vogel and

Laudert, 2015). Wilcox and Unruh (1986), also conducted an experiment to study the effect of build-up on paddles and the mixer body. The results indicated the build-up of molasses on the paddle and mixer body extended the mix time from 3.5 min to 5 min to reach the target %CV. These results demonstrate the need have a good preventive maintenance program at the feed mill.

Conclusion

The uniformity of mix can be affected by ingredient properties, equipment design, control system during process and management. Researchers have demonstrated uniformity of mix is most important when feeding younger chicks, diets formulated to minimum nutrient requirements and complex swine nursery diets. Although the feed manufacturers should always strive to produce an uniform feed, poor performance was not observed in older birds, diets formulated at recommend nutrient levels, and corn-SBM based nursery diets.

ASABE (2012) recommended two tracers to evaluate the uniformity of mix. The first one is sodium chloride salt with a particle size of 450 + 100 μm. The uniformity of mix is evaluated by %CV that should be less than 10% which is the percent commonly recognized by the feed industry as the cut-off for uniformity of mix analysis (Fahrenholz and Stark, 2014). Another one is colored iron particles that are evaluated by the variation of the number of iron particles in the set of samples. If the number of iron particles has less variable than the expected value from Poisson distribution, the mixture is complete or a perfect mix.

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Figures and Tables

Figure 1.1 Dispersion of ingredients in mixer (modified from Bunzel, 2008)

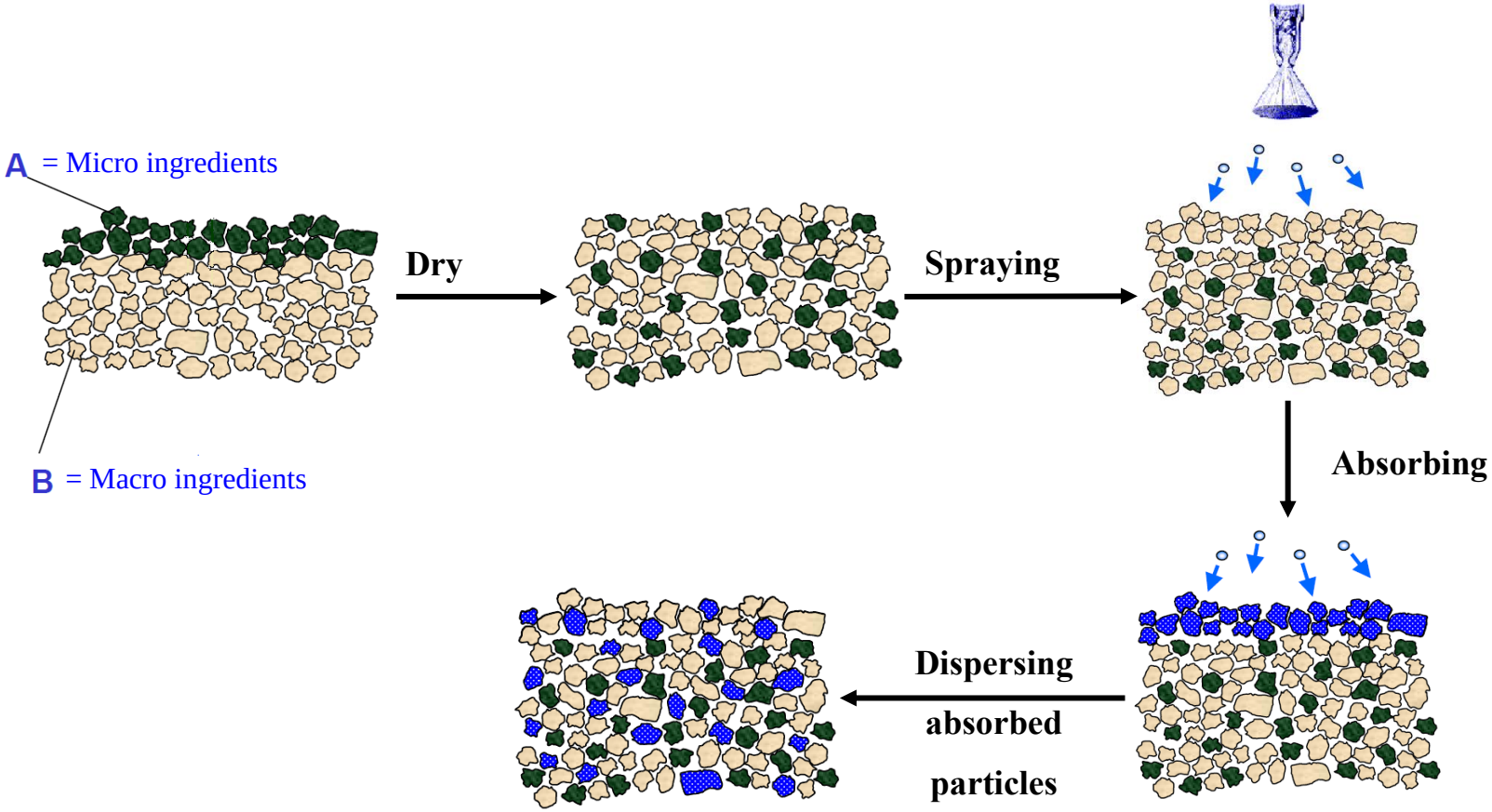

Figure 1.2 Vertical Mixer – Mixing Zone (modified from Wilcox and Unruh (1986))

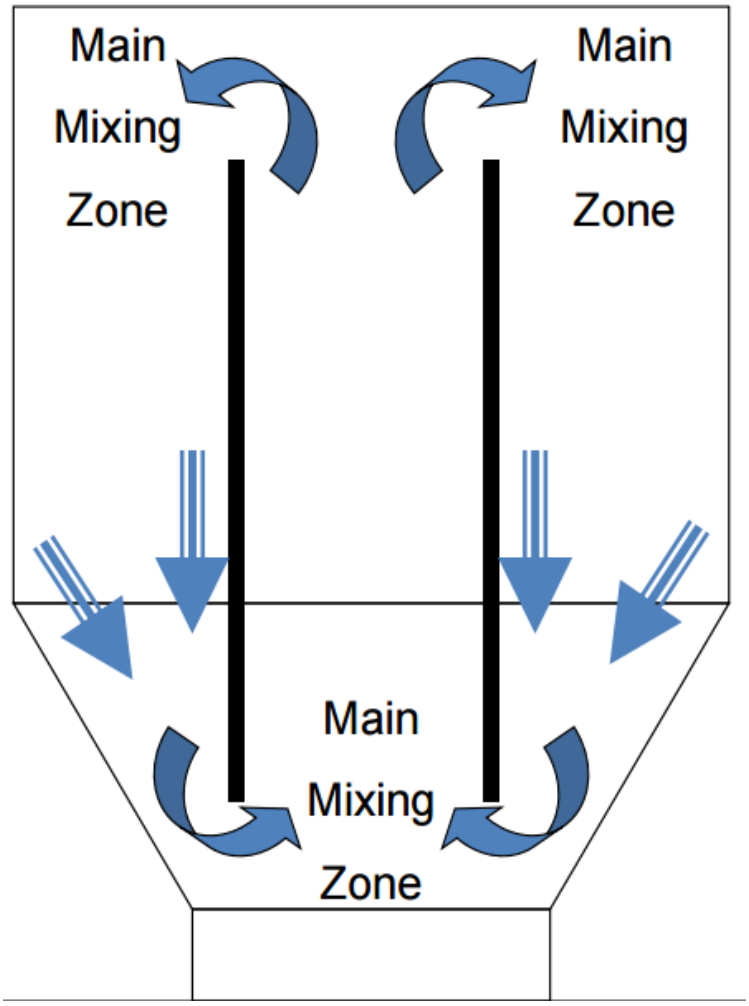

Figure 1.3 Double Ribbon Mixer – Mixing Zone (modified from Wilcox and Unruh (1986))

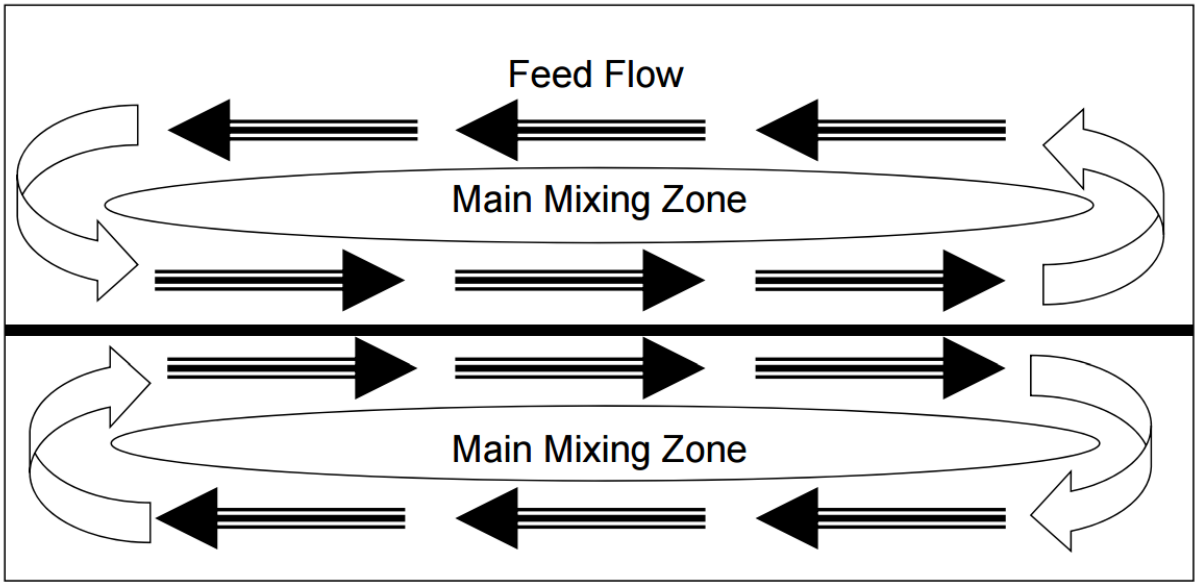

Figure 1.4 Paddle Mixer – Mixing Zones (modified from Wilcox and Unruh (1986))

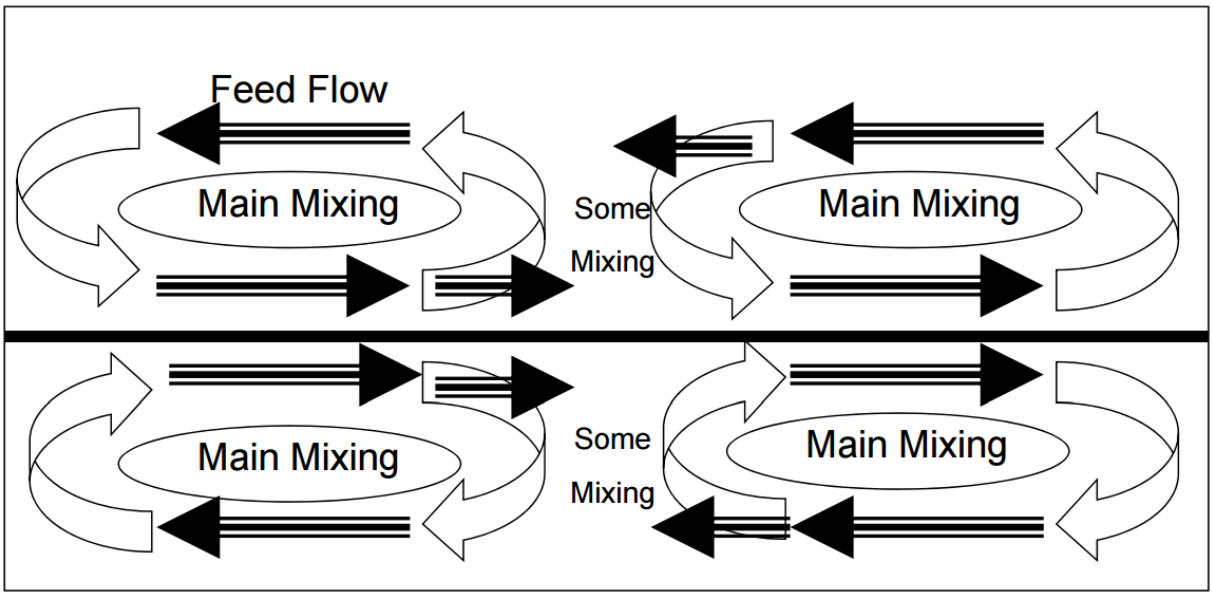

Figure 1.5 Drum Mixer – Mixing Zone (modified from Wilcox and Unruh (1986))

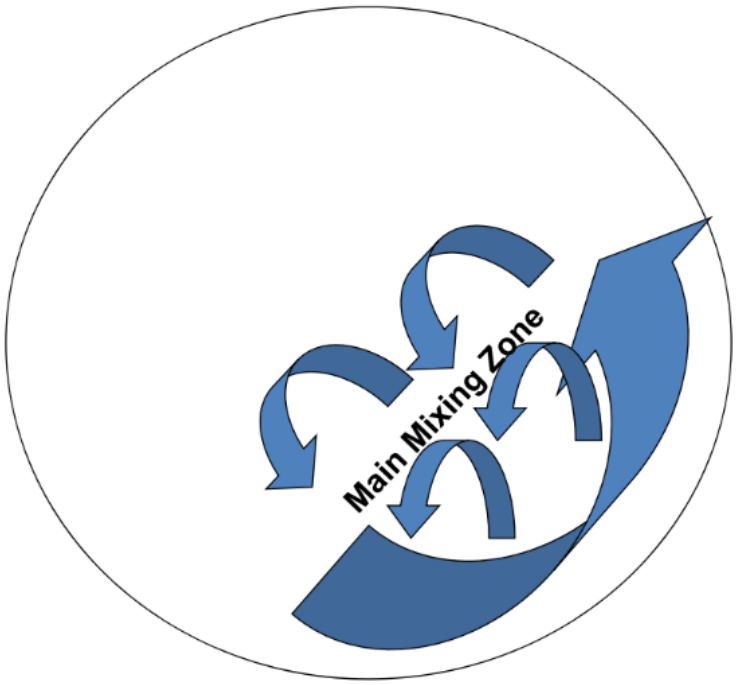

Table 1.1 Interpretation of mixer tests (Herrman and Behnke, 1994)

Percent coefficient of variation	Rating	Corrective action
<10%	Excellent	None
10-15%	Good	Increase mixing time by 25-30%
15-20%	Fair	Increase mixing time 50%, look for worn equipment, overfilling, or sequence of ingredient addition
>20%	Poor	Possible combination of all the above. Consult extension personal or feed equipment manufacturer.

Table 1.2 Recommended mix times by mixer type (Froetschner, 2005)

	Mix Time, min	
Mixer Type	Dry Mix	Wet Mix
Paddle	3.0	3.0
Double shaft, double paddle	0.5	1.0
Ribbon	2.0	3.0
Double ribbon	1-2	2-3
Double shaft, double ribbon	0.75-1	2.0

Chapter 2 - The Effect of Water Temperature, Technician, Extraction Time and Dissolution Time on The Salt Concentration by Using Quantab® Chloride Titrator Method

Abstract

There are several methods to test the uniformity of mix, such as chemical assay, colored iron filings and chloride ion test. The chloride ion test by Quantab® is the only test that can test the uniformity of the mix of multiple batches of feed in one day, but this method cannot be used for a mixture that contains multiple sources of chloride. The two Quantab® procedures commonly used by the food and feed industries to determine the uniformity of mix are not approved by scientific methods. The objectives of these studies were to determine the effect of 1) water temperature, 2) technician, 3) extraction time and 4) dissolution time on the concentration of salt measured with the Quantab® strip. Experiment 1 treatments were arranged as a completely randomized design to determine the effect of water temperature (20, 40 and 60ºC) on salt concentration. Experiment 2 treatments were arranged in a 3 × 3 factorial of equipment type (adjustable dispenser 100 mL, graduated cylinder and balance) with three technicians to determine the accuracy of measuring 90 g (90 mL) of distilled water at room temperature. Experiment 3 treatments were arranged in a 2 × 3 factorial of water temperature (room temperature, 20-23ºC and 60ºC) and equipment type (adjustable dispenser 100 mL, graduated cylinder and balance) to determine the accuracy of measuring 90 g (90 mL) of distilled water. Experiment 4 treatments were arranged in a 3 × 3 factorial of water temperature (20, 40 and 60ºC) and extraction time by stirring (15, 30 and 60 s) to determine the salt concentration.

Experiment 5 treatments were arranged in a 3 × 3 factorial of water temperature (20, 40 and 60ºC) and dissolution time (0, 6 and 12 min) to determine the salt concentration and determine the effect of dissolution time on %CV, respectively. There were 3 replicates per treatment and data were analyzed using the GLM and GLIMMIX procedures of SAS. The results of Experiment 1 indicated that increasing water temperature increased the concentration of salt extracted from the feed sample ($P < 0.0001$). The results of Experiment 2 indicated an interaction between the type of equipment used to measure the water and technician ($P < 0.0001$). The results of Experiment 3 indicated an interaction between the type of equipment and water temperature used to measure the water weight ($P < 0.0001$). The results of Experiment 4 indicated there was no interaction between water temperature and extraction time by stirring ($P = 0.2295$) and the stirring time did not significantly affect the salt concentration while increasing water temperature increased the salt concentration ($P < 0.0001$). The results of Experiment 5 indicated there was no interaction between water temperature and dissolution time ($P = 0.5679$). Both water temperature and dissolution time affected the salt concentration ($P = 0.0136$ and $P < 0.0001$, respectively). In addition, the %CV results demonstrated the different dissolution times had no effect on %CV ($P = 0.1864$). These experiments indicated only the scale should be used for measuring hot distilled water at 60ºC, and the 30 s extraction time by stirring followed by immediately placing the strip into the solution after extraction should be used for the Quantab® chloride titrator method.

Introduction

There are several methods to test the uniformity of mix such as chemical assay, colored iron filings and chloride ion test. The colored iron filings and chloride ion test can provide same day results. However, the colored iron filings method is time consuming and requires more resources and, therefore, limits the number of tests that can be conducted daily. On the other hand, the chloride ion test by Quantab® can test the uniformity of the mix of multiple batches of feed in one day. However, a limitation of the Quantab® is that it cannot be used for a mixture that contains multiple sources of chloride. Therefore, the Quantab® chloride titrator method is commonly used to determine the uniformity of mix in diets that contain 0.3 to 0.5% added salt.

Nielsen (2010) described the Quantab® chloride titrator as a thin plastic strip that is laminated with a capillary column impregnated with silver nitrate and potassium dichromate, which together form silver dichromate. When the strip is placed in a solution that contains chloride ions, the color of the column is changed from a reddish-brown to a white color due to the reaction of the chloride ion with silver dichromate, which creates silver chloride. The Quantab® chloride titrator method was originally used in the food and drink industries but was adapted for the feed industry in 1967 (Headley, 1967). The method was developed to analyze for chloride ions in a mixture of feed and distilled water. The salt in the feed sample was dissolved by stirring the sample with hot or boiling distilled water prior to inserting the Quantab® strip into the solution.

Both McCoy (2005) and Nielsen (2010) have described the use of Quantab Chloride titrators to evaluate mixer efficiency, but with different methods, but neither are approved scientific methods. Variation in the procedures may impact the accuracy of the result, but no data currently exists to evaluate the impact of these variables in methodology. Therefore, the

objectives of these studies were to determine the effect of 1) water temperature, 2) technician, 3) extraction time and 4) dissolution time on the concentration of salt measured with the Quantab® strip.

Material and Method

Experiment 1

Experiment 1 determined the effect of water temperature on the concentration of salt in a feed sample. A swine grower diet was manufactured for the experiment (Table 2.1). The ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX) and mixed for 5 min. A 1 kg sample was collected during the discharge process of the mixer. The sample was ground with a coffee grinder (model Krups-F20342, Groupe SEB, West Orange, NJ) for 30 s and remixed. A 10 g sample of the ground feed was mixed with 90 g of distilled water at three difference water temperatures (20, 40 and 60ºC). The mixture was stirred for 30 s, allowed to rest for 60 s and stirred for another 30 s. A folded filter paper was place into a cup and then a Quantab® strip was inserted into the liquid at the bottom of the filter paper for 20 and 40ºC treatments or three Quantab® strips from the same lot were inserted into the liquid at the bottom of the filter paper for 60ºC treatment.

Experiment 2

Treatments were arranged in a 3 × 3 factorial of equipment type (adjustable dispenser 100 mL, graduated cylinder and balance) with three technicians to determine the accuracy of measuring 90 g (90 mL) of distilled water at room temperature, 20-23ºC. The object of the experiment was for the three technicians to obtain 90 g (90 mL) of water using an adjustable 100 mL volumetric dispenser, graduated glass cylinder and top loading balance (resolution 0.1 g) into a foam cup. The amount obtained by each technician in the cup for each type of equipment was

weighed with an analytical balance (resolution 0.001g). Each technician replicated the process 10 times for each type of equipment.

Experiment 3

Treatments were arranged in a 2 × 3 factorial of water temperature (room temperature, 20-23ºC and 60ºC) and equipment type (adjustable dispenser 100 mL, graduated cylinder and balance) to determine the accuracy of measuring 90 g (90 mL) of distilled water. The object of the experiment was to obtain 90 g (90 mL) of distilled water using an adjustable 100 mL volumetric dispenser, graduated glass cylinder and top loading balance (resolution 0.1 g) into a foam cup with 2 different water temperatures (room temperature, 20-23ºC and 60ºC). The amount obtained in the cup for each type of equipment and water temperature were weighed with an analytical balance (resolution 0.001g). Each water temperature and measuring device were replicated 10 times.

Experiment 4

Treatments were arranged in a 3 × 3 factorial of water temperature (20, 40 and 60ºC) and stirring time (15, 30 and 60 s) to determine the salt concentration in feed. The test material was prepared by mixing corn (27.12 kg) and 350 µm salt (0.10 kg) for 5 min in a 0.056 m^3 double ribbon mixer (Hayes and Stolz, model HP2SSS-0106, Fort Worth, TX). A 5 kg sample was collected during the discharge process of the mixer. The sample was then ground with a coffee grinder (model Krups-F20342, Groupe SEB, West Orange, NJ) for 30 s and remixed. A 10 g sample of the ground sample was mixed with 90 g of distilled water. The mixture was stirred for the designated time (15, 30 and 60 s), allowed to rest for 60 s and stirred for another designated time equal to the initial stirring time. A folded filter paper was placed into the cup and the

Quantab® strip was inserted into the liquid at the bottom of the filter paper. The water temperatures of the experiment were adjusted to 20, 40 and 60ºC for each treatment.

Experiment 5

Treatments were arranged in a 3 × 3 factorial of water temperature (20, 40 and 60ºC) and dissolution time (0, 6 and 12 min) to determine the effect of water temperature and dissolution time on salt concentration in feed and the effect of dissolution time on %CV. The test material was prepared by mixing corn (27.12 kg) and 350 µm salt (0.10 kg) for 5 min in a 0.056 m^3 double ribbon mixer (Hayes and Stolz, model HP2SSS-0106, Fort Worth, TX). A 5 kg sample was collected during the discharge process of the mixer. The sample was then ground with a coffee grinder (model Krups-F20342, Groupe SEB, West Orange, NJ) for 30 s and remixed. A 10 g sample of the ground sample was mixed with 90 g of distilled water. The mixture was stirred for 30 s, allowed to rest for 60 s and stirred for another 30 s. A folded filter paper was placed into the cup. The dissolution time, which is the period of time after stirring prior to the addition of the Quantab® strip, was adjusted to 0, 6 and 12 min. The Quantab® strip was inserted into the liquid at the bottom of the filter paper after the dissolution time. The water temperature used to dissolve the salt was adjusted to 20, 40 and 60ºC for each treatment.

Statistical Analysis

Data were analyzed as a completely randomized design for Experiment 1 and a factorial treatment design for Experiments 2 through 5. Experiment 1 determined the effect of water temperature (20, 40 and 60ºC) on the salt concentration of the feed as measured by the Quantab®. Experiment 2 treatments were arranged in a 3 × 3 factorial of equipment type (adjustable dispenser 100 mL, graduated cylinder and balance) with three technicians to determine the accuracy of measuring 90 g (90 mL) of distilled water at room temperature, 20-

23ºC. Experiment 3 treatments were arranged in a 2 × 3 factorial of water temperature (room temperature, 20-23ºC and 60ºC) and equipment type (adjustable dispenser 100 mL, graduated cylinder and balance) on determining the accuracy of measuring 90 g (90 mL) of distilled water. Experiment 4 treatments were arranged in a 3 × 3 factorial of water temperature (20, 40 and 60ºC) and stirring time (15, 30 and 60 s) to determine the salt concentration in the feed sample. Experiment 5 treatments were arranged in a 3 × 3 factorial of water temperature (20, 40 and 60ºC) and dissolution time (0, 6 and 12 min) to determine the effect of water temperature and dissolution time on salt concentration and the effect of dissolution time on %CV. Data were analyzed using the GLM and GLIMMIX procedures of SAS. Means were separated by least squares means adjustment for Bonferroni's multiple comparisons. Result were considered significant at $P \leq 0.05$.

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Results and Discussion

The results of Experiment 1 indicated that increasing the water temperature increased the concentration of salt extracted from the feed sample. The samples extracted with 60ºC water had significantly greater salt concentrations ($P < 0.0001$; 0.53, 0.56 and 0.61% for 20, 40 and 60ºC, respectively) as compared to the 20 and 40ºC temperatures. The 20 and 40ºC were similar concentrations. There was a linear increase in salt concentration as the water temperature increased ($P < 0.0001$). The average standard deviation of the Quantab® strip was 0.014% when the same lot of three Quantab® strip were placed in the solution extracted with 60ºC water for 10 replications.

The results of Experiment 2 (Table 2.2) indicated an interaction between the type of equipment used to measure the water and technician ($P < 0.0001$). Technician 1 was the only

technician that achieved similar results with the dispenser and graduated cylinder but was not as accurate as the balance. Technician 1 was also the closest with the graduated cylinder (89.27 g) to the 90 g target as compared to technicians 2 and 3 (88.12 and 88.55 g, respectively). The main effects indicate that equipment can affect the amount of water used in the extraction of the salt ($P < 0.0356$). The technicians, who were chosen at random, did not significantly affect the accuracy of measuring 90 g (90 mL) of distilled water ($P = 0.7460$). The balance and dispenser were similar (89.92 and 89.76 g), whereas the use of the graduated cylinder resulted in the poorest measurement of water (88.64 g). Because there was interaction between the technician and the equipment used to measure the amount of water, there was a higher standard deviation due to the technician when determining the bottom of the meniscuses of the water in the graduated cylinder. Therefore, the measurement of water by a graduated cylinder was less accurate, as compared to the scale and the adjustable dispenser which were more accurate due to less interpretation by the technician as to the amount in the cylinder. While small differences in water measurement may not affect the %CV results based on the Quantab® chloride titrator method, it does support having one technician perform the uniformity of mix analysis and weighing the amount of water versus using the graduate cylinder.

The results of Experiment 3 (Table 2.3) indicated an interaction between the type of equipment and water temperature at time of weighing ($P < 0.0001$). The scale was the only equipment that achieved similar results for both room temperature water and 60ºC water (89.97 and 90.02 g, respectively). The difference in water weight between room temperature water and 60ºC water using the dispenser and graduated cylinder were 1.44 and 0.81 g, respectively. The density of water decreases as the water temperature is increased. The measurement of 60ºC water by volume using the dispenser and graduated cylinder were 88.09 and 88.46 g, respectively. Both

types of equipment resulted in less than the target of 90 g. The main effects indicate that both equipment and water temperature can affect the amount of water used in the extraction of the salt ($P < 0.0001$ and $P < 0.0001$, respectively). The dispenser and graduated cylinder were similar (88.81 and 88.86 g), whereas the use of the scale resulted in the best measurement of water (89.99 g). When water is heated, the O-H bond of water is bended and stretched after molecules obtained the kinetic energy from heat (Shapley 2012). The hydrogen bond length between water molecules increases as temperature increases (Dougherty 1998). Thus, water molecules are free to move and take more space then the volume of water increases and water density decreases. For instance, if water temperature is increased from 22° to 60°C, water volume is increased from 1 ml to 1.015 ml when the density of water at 22° and 60ºC are 0.9978 and 0.9832 g/ml, respectively (Shapley 2012). Because the density of water is decreased when the water temperature increases, the volumetric equipment did not properly measure the hot water. Therefore, the measurements of water by a dispenser and graduated cylinder were less accurate, as compared to the scale which was more accurate due to mass measurement. The data indicated a scale should be used with the Quantab® chloride titrator method for water measurement in order to decrease equipment error when performing the uniformity of mix analysis.

The results of Experiment 4 (Table 2.4) indicated that temperature had a greater effect on the amount of salt dissolved as compared to extraction time by stirring. There was no interaction between water temperature and extraction time by stirring ($P = 0.2295$). The results for the 20, 40 and 60ºC water temperatures were 0.333, 0.321 and 0.334 ($P < 0.0001$). However, the salt concentration for 15, 30 and 60 s extraction times were not significantly different ($P = 0.0506$; 0.316, 0.339 and 0.343, respectively). The data in the current experiment support the time recommendations of McCoy (2005) and Nielson (2010).

The results of Experiment 5 (Table 2.5) indicated both water temperature and dissolution time affected the salt concentration. There was no interaction between water temperature and dissolution time ($P = 0.5679$). However, the 60ºC water had a significantly higher salt concentration, as compared to 20 and 40ºC water ($P = 0.0136$). There was a linear increase in the salt concentration response as temperature increased ($P = 0.0147$). Additionally, the dissolution time for the 0 min treatment had a significantly lower salt concentration, as compared to 6 and 12 min treatment ($P < 0.0001$). There was a quadratic increase in the salt concentration response as dissolution time increased ($P = 0.0003$). The results of 10 samples within the nine treatments of combination between water temperature and dissolution time were used to compute a %CV to determine uniformity of mix. The %CV results calculated using the different dissolution times were not different ($P = 0.1864$; 6.75, 3.66 and 4.86 %CV for 0, 6 and 12 min, respectively) even though the salt concentration was different for the 0, 6 and 12 min. This experiment demonstrated that although different dissolution times affected the salt concentration, the %CV was not significantly different. The data in the current experiment suggest that the method should specify a standard dissolution time. McCoy (2005) recommends the strip should be placed in the solution immediately after extraction. However, Nielson (2010) recommends waiting for the water temperature to cool to room temperature. McCoy's method could potentially have less variation because the technician does not need to monitor the time during the analysis. The results of the current study demonstrated that when the Quantab® strips were placed in the solution at the same time, the variation in salt concentration did not affect the %CV within the same analyzed batch of feed.

Conclusions

The result of these experiments indicated only the scale should be used for measuring hot distilled water at 60ºC, and the 30 s extraction time by stirring, followed by immediately placing the strip into the solution after extraction should be used for the Quantab® chloride titrator method.

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Tables

Table 2.1 Diet compositions of swine grower diet

Ingredients	Percent
Corn	71.50
Soybean meal (SBM)	25.70
Mono-calcium phosphate 21%	0.55
Limestone	1.13
Swine vitamin premix[1]	0.15
Swine trace mineral premix[2]	0.15
L-Lysine 78.8% HCl	0.30
DL-Methionine	0.07
L-Threonine	0.08
Phytase[3]	0.02
Salt[4]	0.35
Total	100.00

[1] Composition per kilogram: 110 g Iron, 110 g Zinc, 26 g Manganese, 11 g Copper, 198 g Iodine and 198 g Selenium.
[2] Composition per kilogram: 4,409,171 IU Vitamin A, 551,146 IU Vitamin D3, 17,637 IU Vitamin E, 15 mg Vitamin B12, 1,764 mg Menadione, 3,307 mg Riboflavin, 11,023 mg d-Pantothenic Acid and 19,841 mg Niacin.
[3] Ronozyme HiPhos (GT) 2700 (DSM Nutritional Products, Parsippany, NJ) provided 476.3 phytase units (FTU)/kg with a release of 0.10% available P.
[4] Particle size was 350 µm.

Table 2.2 Effect of technician and types of equipment on water measurement (Exp. 2)

Equipment type	Technician	n	Water, g
Interaction effects			
Balance	1	30	89.97[A]
Dispenser	1	30	89.53[A,B]
Graduated Cylinder	1	30	89.27[B]
Balance	2	30	89.93[A]
Dispenser	2	30	89.96[A]
Graduated Cylinder	2	30	88.12[C]
Balance	3	30	89.86[A]
Dispenser	3	30	89.78[A,B]
Graduated Cylinder	3	30	88.55[C]
SEM			0.103
Main effect			
Balance		90	89.92[a]
Dispenser		90	89.76[a]
Graduated Cylinder		90	88.64[b]
SEM			0.060
	1	90	89.59
	2	90	89.34
	3	90	89.39
	SEM		0.060
			P-value
Source of variation			
Equipment type × Technician (random effect)			<0.0001
Equipment type			0.0356
Technician (random effect)			0.7460

[a,b] Means with different superscripts are significantly different ($P \leq 0.05$).
[A-C] Means with different superscripts are significantly different ($P \leq 0.01$).

Table 2.3 Effect of water temperature and types of equipment on water measurement (Exp. 3)

Equipment type	Water temperature	n	Water, g
Interaction effects			
Balance	Room (20-23ºC)	30	89.97[A]
Dispenser	Room (20-23ºC)	30	89.53[B]
Graduated Cylinder	Room (20-23ºC)	30	89.27[B]
Balance	60ºC	30	90.02[A]
Dispenser	60ºC	30	88.09[D]
Graduated Cylinder	60ºC	30	88.46[C]
SEM			0.054
Main effect			
Balance		90	89.99[A]
Dispenser		90	88.81[B]
Graduated Cylinder		90	88.86[B]
SEM			0.039
	Room (20-23ºC)	90	89.59[A]
	60ºC	90	88.86[B]
	SEM		0.031
			P-value
Source of variation			
Equipment type × Water temperature			<0.0001
Equipment type			<0.0001
Water temperature			<0.0001

[A-D] Means with different superscripts are significantly different ($P \leq 0.01$).

Table 2.4 Effect of water temperature and extraction time on the percent of salt in a corn-salt mixture (Exp. 4)

Water temperature, ^{o}C	Extraction times, s	N	Salt, %
Interaction effects			
20	15	30	0.338
40	15	30	0.317
60	15	30	0.339
20	30	30	0.333
40	30	30	0.326
60	30	30	0.339
20	60	30	0.329
40	60	30	0.320
60	60	30	0.325
SEM			0.0042
Main effect			
20		90	0.333 ^{A}
40		90	0.321 ^{B}
60		90	0.334 ^{A}
SEM			0.0025
	15	90	0.331
	30	90	0.333
	60	90	0.325
	SEM		0.0025
			P-value
Source of variation			
Water temperature × Extraction time			0.2295
Water temperature			<0.0001
Extraction time			0.0506

^{A,B} Means with different superscripts are significantly different ($P \leq 0.01$).

Table 2.5 Effect of water temperature and dissolution time on the percent of salt in a corn-salt mixture (Exp. 5)

Water temperature, ^{o}C	Dissolution time, min	n	Salt, %
Interaction effects			
20	0	30	0.313
40	0	30	0.314
60	0	30	0.325
20	6	30	0.333
40	6	30	0.340
60	6	30	0.346
20	12	30	0.344
40	12	30	0.333
60	12	30	0.354
SEM			0.0056
Main effect			
20		90	0.330[a]
40		90	0.329[a]
60		90	0.341[b]
SEM			0.0033
	0	90	0.317[A]
	6	90	0.340[B]
	12	90	0.344[B]
	SEM		0.0033
			P-value
Source of variation			
Water temperature × Dissolution time			0.5679
Water temperature			0.0136
Linear			0.0147
Dissolution time			<0.0001
Linear			<0.0001
Quadratic			0.0003

[a,b] Means with different superscripts are significantly different ($P \leq 0.05$).
[A,B] Means with different superscripts are significantly different ($P \leq 0.01$).

Chapter 3 - The Effect of Extended Mix Times and Sample Preparation with Different Salt Particle Sizes on The Uniformity of Mix of a Corn-Soy Swine Diet

Abstract

The uniformity of a feed mixture is determined from the coefficient of variation (CV) of ten samples in a single batch of feed. The feed industry standard is a CV of less than 10% using a single source tracer such as salt, trace minerals, or iron filings. The uniformity of mix can be affected by many factors, including mixer design, particle size of the ingredients, sample preparation and mix time. Previous research has determined the minimum mix time to maximize the mixing efficiency, but some hypothesize that over-mixing may lead to ingredient segregation. However, there is limited data regarding the effects of extended mixing, appropriate particle size of the analytical marker and the analytical sample preparation for maximum precision of the assay. The objectives of these experiments were to determine 1) the effects of extended mix time, 2) particle size of the marker and 3) sample preparation of the CV in a corn-soy swine diet. Experiment 1 treatments were arranged in a 3 × 7 factorial with 3 salt particle sizes (fine-350 µm, medium-464 µm and coarse-728 µm) and 7 mix times (2, 3, 5, 15, 30, 45 and 60 min). Experiment 2 treatments were arranged in 2 × 3 × 3 factorial with 2 sample preparations (unground and ground), 3 salt particle sizes (fine-350 µm, medium-464 µm and coarse-728 µm) and 3 mix times (3, 30 and 60 min). There were 3 replicates per treatment and 10 samples per replicate. Salt concentrations were determined using a Quantab® chloride titrator. The results of Experiment 1 indicated no interaction between mix time and salt particle size ($P = 0.4366$). The extended mix time did not result in segregation ($P = 0.3073$; 11.5, 13.8, 12.9, 13.1, 13.9, 11.6 and 11.3% CV for 2, 3, 5, 15, 30, 45 and 60 min, respectively). Particle size of the salt significantly affected the uniformity of mix ($P < 0.0001$; 21.2, 8.6 and 7.9% CV for the coarse,

medium and fine salt, respectively). The results of Experiment 2 indicated no interaction of sample preparation, salt particle size and mix time ($P = 0.3823$). However, there was interaction between sample preparation and salt particle size ($P = 0.0002$). The difference in the %CV between unground and ground samples was significantly greater for the mixture with coarse salt (8.89%) than the mixtures with medium salt (2.59%) and fine (1.35%). The ground treatment had a significantly lower %CV than the unground treatment ($P < 0.0001$; 8.7 and 13.0% for ground and unground samples, respectively). The fine and medium salt treatments had significantly lower %CV as compared to the coarse salt treatment. ($P < 0.0001$; 7.4, 7.7 and 17.4% for fine, medium and coarse, respectively). These results indicated that feed did not segregate after mixing for one hour, and that the greater number of particles per gram of the marker increased the precision of the analysis. This is likely due to an increased probability that the marker was present in greater proportionate quantities in the sample tested. However, when coarse salt is used in the manufacturing process, the samples should be ground prior to analysis.

Introduction

The composition of a diet directly impacts growth rate and feed conversion of animals. Nutritionists formulate diets based on the assumption that the animal will receive all the nutrients needed for maintenance and growth each time they go to the feeder. Researchers have demonstrated that poorly mixed feed can negatively affect the feed conversion ratio (F/G ratio) of nursery pigs (Traylor et al., 1994). The results of the study demonstrated poor F/G ratio of nursery pigs (1.81 and 2.24) as the percent coefficient of variation (%CV) of the diet increased (12.3% and 106.5%, respectively). In addition to meeting the nutrient requirements of the animal, variation within a mixture of feed can lead to toxicity or deficiency of minerals and vitamins. For instance, poultry feed has been recalled because the salt level was over the maximum dietary level. A salt toxicity in birds can result in trouble breathing, fluid discharges from the beak, wet droppings, weakness or paralysis of the legs and even death (FDA, 2015). Therefore, FDA has regulations that require licensed feed mills to test their mixers annually [21 CFR part 225.30 (b)].

The uniformity of a batch of mixed feed is described by the variation of ingredients or nutrients relative to their position in the mixer. The uniformity of mix is expressed as the %CV. The CV should be less than 10%, which is the percent commonly recognized by the feed industry as the cut-off for uniformity of mix analysis (Fahrenholz and Stark, 2014). However, Aulton and Taylor (2013) defined uniformity of mix as "a mix where the probability of selecting a particular type of particle is the same at all positions in the mix and is equal to the proportion of such particles in the total mix." A discrete probability distribution for the number of counts (markers) that occur randomly in different locations is explain by the Poisson distribution (Marchini, 2008). Therefore, the complete mix can be explained by the Poisson distribution.

Several factors can affect the uniformity of mix, such as mixer design, particle size of the ingredients and mixing time. The objective of the feed mixing process is to create a uniform mixture of feed in the minimum amount of mix time in order to maximize the efficiency of the mixing process. In addition to determining the minimum mixing time, there is a concern that extended mixing may lead to ingredient segregation.

The selection of an appropriate tracer is important when determine the uniformity of the mix. Pfost (1976) compared the uniformity of mix using rolled corn and salt, which had extremely different particle sizes in a two-ton vertical mixer. The results indicated the two ingredients were not mixed properly (%CV > 10) after mixing time for 32 min. Groesbeck et al. (2004) conducted two experiments to determine the effect of various salt particle sizes and different sample preparation on %CV. The results of Experiment indicated that the %CV decreased when the particle size of the salt decreased from 3,000 to 400 µm. Moreover, grinding the sample of feed to less than 400 µm prior to analysis decreased the %CV of the mixture as compared to the unground sample (700 µm) when using larger than 440 µm salt as a marker.

Previous research has determined the minimum mix time to maximize the mixing efficiency, but there are concerns that over-mixing and extremely different particle size between ingredients may lead to ingredient segregation. In addition, grinding the feed sample before analysis should improve uniformity of mix. However, there is limited data regarding the effects of extended mixing, appropriate particle size of the analytical marker and the analytical sample preparation for maximum precision of the assay. The objectives of the current study were to determine the effect of extended mixing time, salt particle size and sample preparation on the uniformity of mix.

Material and Method

Experiment 1

Treatments were arranged in a 3 × 7 factorial of salt particle sizes (fine, medium and coarse) and mix time (2, 3, 5, 15, 30, 45 and 60 min) to determine the effect on uniformity of mix. A swine grower diet was used for the experiment (Table 3.1). The ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed was mixed for 2, 3, 5, 15, 30, 45 and 60 min. A 30 g per sample was obtained at 10 points in the mixer (Figure 3.1). The salt concentration of the samples was analyzed by the Quantab® chloride titrator method.

Experiment 2

Treatments were arranged in 2 × 3 × 3 factorial of sample preparation (unground and ground), salt particle size (fine, medium and coarse) and mix time (3, 30 and 60 min) to determine the effect on uniformity of mix. A swine grower diet was used for the experiment (Table 3.1). The ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed was mixed for 3, 30 and 60 min. A 30 g per sample was obtained at 10 points in the mixer (Figure 3.1). The samples were ground with a coffee grinder (model Krups-F20342, Groupe SEB, West Orange, NJ) for 30 s. The salt concentration of unground and ground samples was analyzed by the Quantab® chloride titrator method.

Data Collection

The salt concentration of the samples was determined with the Quantab® chloride titrator method (McCoy, 2005). A 10 g sample was weighed into a cup and 90 g of hot distilled water (60ºC) was added to the cup. The mixture was stirred for 30 s, allowed to rest for 60 s and stirred

for another 30 s. A folded filter paper was placed into the cup and the Quantab® strip was inserted into the liquid at the bottom of the filter paper. The results of the 10 samples per batch were used to compute a CV to determine mixing uniformity. The CV for each batch was calculated by dividing the standard deviation by the average value multiplied by 100. The particle size of the three types of salt (fine, medium and coarse) was determined with a Ro-Tap model RX-29 (W.S. Tyler Industrial Group, Mentor, Ohio) using the method of determining and expressing fineness of feed materials by sieving (ANSI/ASABE S319.4) without a flow agent for 15 min.

Statistical Analysis

Data were analyzed as a factorial treatment design for all experiments. Experiment 1 treatments were arranged in a 3 × 7 factorial of salt particle sizes (fine, medium and coarse) and mix time (2, 3, 5, 15, 30, 45 and 60 min) to determine the effect on uniformity of mix. Experiment 2 treatments were arranged in 2 × 3 × 3 factorial of sample preparation (unground and ground), salt particle size (fine, medium and coarse) and mix time (3, 30 and 60 min) to determine the effect on uniformity of mix. There were 3 replicates per treatment. Data were analyzed using the GLIMMIX procedure of SAS. Means were separated by least squares means adjustment for Bonferroni's multiple comparisons. Results were considered significant at $P \leq 0.05$.

Results and Discussion

The results of Experiment 1 (Table 3.2) indicated no interaction between mixing time and particle size of the salt ($P = 0.4366$). The results indicated no significant difference in mix times ($P = 0.3073$; 11.5, 13.8, 12.9, 13.1, 13.9, 11.6 and 11.3% for 2, 3, 5, 15, 30, 45 and 60 min, respectively). The results indicated that salt particles did not separate during the extended mixing

process. These results disagree with Hasting's (1961) statement "there is a constant tendency for particles which have been mixed to become separated". However, there may be separation of the particles as the feed moves through the feed mill; especially, when there is a wide variation of the particle size of the ingredient (Wilcox and Balding, 1976). The results of the current study indicated that separation of particles did not occur after mixing for 60 min. However, the %CV between salt particle sizes was significantly different ($P < 0.0001$: 7.9, 8.6 and 21.2% for fine, medium and coarse, respectively). The mixture that contained the coarse salt was different from the fine and medium salt treatments. The %CV decreased as the number of marker particles per gram increased. This result is explained by Poisson distribution in that the CV of Poisson is calculated by $1/\sqrt{mean}$ so the %CV of Poisson is decreased, as the average number of counts (marker) increases (Bolker, 2007). The gradual decrease in the %CV of the coarse salt particle size treatment after 2 min of mixing may be a result of particle reduction of salt due to the friction between particles during the extended mix time.

The results of Experiment 2 (Table 3.3) indicated no interaction of sample preparation, salt particle size and mix time ($P = 0.3823$). However, there was an interaction between sample preparation and salt particle size ($P = 0.0002$). The %CV of the ground samples was significantly different on %CV as compared to unground samples for the coarse salt treatment while there was no significant difference between ground and unground samples for medium and fine salt treatments. The difference in the %CV between unground and ground samples was greater for the mixture with coarse salt (8.89%) than the mixtures with medium salt (2.59%) and fine (1.35%). These results are in agreement with the findings of other research. Groesbeck et al. (2004) reported a lower %CV after the particle size of the sample that contained coarse salt ($\geq$ 730 µm) was reduced from 700 to 400 µm. The ground treatment had a significantly lower %CV

than the unground treatment ($P < 0.0001$; 8.7 and 13.0% for ground and unground samples, respectively). The effect of salt particle size was similar to Experiment 1, the mixtures that contained fine and medium salt had significantly lower %CV as compared to the coarse salt treatments ($P < 0.0001$; 7.4, 7.7 and 17.4% for fine, medium and coarse, respectively).

Conclusions

These results of Experiment indicated that an extended mix time up to 60 minutes did not increase the %CV of the feed, and the particle size of the salt used in the uniformity of mix test can significantly change the results of the test. Furthermore, the data suggests that if coarse salt is used in the manufacturing process, the samples should be ground prior to analysis.

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Figures and Tables

Figure 3.1 The sampling points of the mixer surface

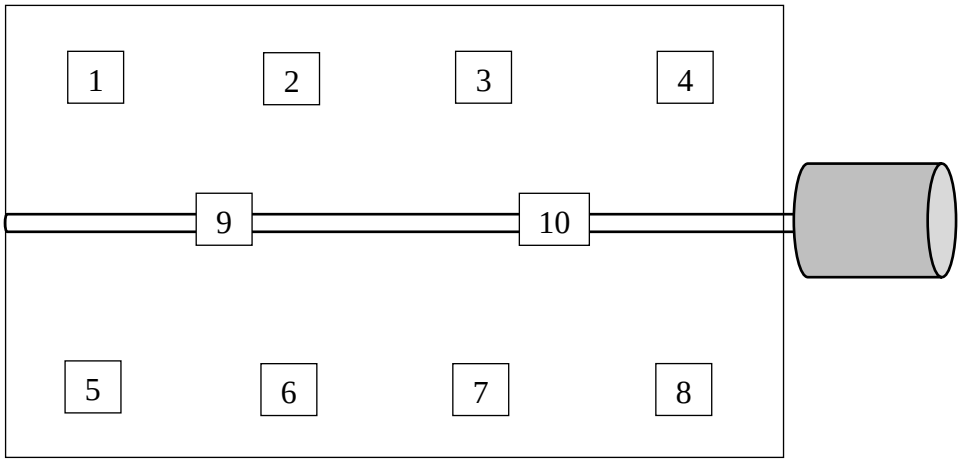

Table 3.1 Diet compositions of swine grower diet

	Salt[1]		
Ingredients	Fine	Medium	Coarse
Corn	71.50	71.50	71.50
Soybean meal (SBM)	25.70	25.70	25.70
Mono-calcium phosphate 21%	0.55	0.55	0.55
Limestone	1.13	1.13	1.13
Swine vitamin premix[2]	0.15	0.15	0.15
Swine trace mineral premix[3]	0.15	0.15	0.15
L-Lysine 78.8% HCl	0.30	0.30	0.30
DL-Methionine	0.07	0.07	0.07
L-Threonine	0.08	0.08	0.08
Phytase[4]	0.02	0.02	0.02
Salt[1]	0.35	0.35	0.35
Total	100.00	100.00	100.00

[1] Particle sizes were 350, 464 and 728 µm for fine, medium and coarse salt, respectively.
[2] Composition per kilogram: 110 g Iron, 110 g Zinc, 26 g Manganese, 11 g Copper, 198 g Iodine and 198 g Selenium.
[3] Composition per kilogram: 4,409,171 IU Vitamin A, 551,146 IU Vitamin D3, 17,637 IU Vitamin E, 15 mg Vitamin B12, 1,764 mg Menadione, 3,307 mg Riboflavin, 11,023 mg d-Pantothenic Acid and 19,841 mg Niacin.
[4] Ronozyme HiPhos (GT) 2700 (DSM Nutritional Products, Parsippany, NJ) provided 476.3 phytase units (FTU)/kg with a release of 0.10% available P.

Table 3.2 Percent coefficient of variation (%CV) of feed mixed at different mix times and salt particle sizes (Exp. 1)

Mix Time, min	Salt particle size	n	Coefficient of Variation(CV), %
Interaction effects			
2	Fine	3	7.39
2	Medium	3	6.04
2	Coarse	3	21.13
3	Fine	3	8.20
3	Medium	3	8.05
3	Coarse	3	25.04
5	Fine	3	9.21
5	Medium	3	9.77
5	Coarse	3	19.63
15	Fine	3	7.10
15	Medium	3	8.49
15	Coarse	3	23.66
30	Fine	3	8.82
30	Medium	3	10.59
30	Coarse	3	22.23
45	Fine	3	7.53
45	Medium	3	8.95
45	Coarse	3	18.46
60	Fine	3	7.24
60	Medium	3	8.36
60	Coarse	3	18.24
SEM			1.696
Main effect			
2		9	11.52
3		9	13.77
5		9	12.87
15		9	13.08
30		9	13.88
45		9	11.65
60		9	11.28
SEM			0.979
	Fine	21	7.93^{A}
	Medium	21	8.61^{A}
	Coarse	21	21.20^{B}
	SEM		0.641
			P-value
Source of variation			
Mix time × Particle size			0.4366
Mix time			0.3073
Particle size			<0.0001

^{A,B} Means with different superscripts are significantly different ($P \leq 0.01$).

Table 3.3 Percent coefficient of variation (%CV) of feed mixed with different salt particle sizes between unground and ground samples (Exp. 2)

Salt particle size	Sample preparation	Mix time, min	n	Coefficient of Variation(CV), %
Interaction effects				
Fine	Unground		9	8.09[A]
Medium	Unground		9	9.00[A]
Coarse	Unground		9	21.84[C]
Fine	Ground		9	6.74[A]
Medium	Ground		9	6.41[A]
Coarse	Ground		9	12.95[B]
SEM				0.880
Main effect				
Fine			18	7.41[A]
Medium			18	7.71[A]
Coarse			18	17.39[B]
SEM				0.622
	Unground		27	12.97[A]
	Ground		27	8.70[B]
	SEM			0.508
		3	18	11.49
		30	18	11.04
		60	18	9.99
		SEM		0.622
				P-value
Source of variation				
Salt particle size × Preparation × Mix time				0.3823
Salt particle size × Preparation				0.0002
Salt particle size				<0.0001
Preparation				<0.0001
Mix time				0.2320

[A,B] Means with different superscripts are significantly different ($P \leq 0.01$).

Chapter 4 - The Effect of Wet Mix Time and Application Time When Applied with Different Spray Conditions on the %CV in a Corn-Soy swine grower diet

Abstract

The number of liquids added into the mixer has increased over the last 10 years. These ingredients are typically added simultaneously to the feed mixture using several spray nozzles located across the top or on the side walls of the mixer. Since the addition of liquids may cause agglomeration between small dry particles and liquid drops, the feed should be mixed longer to break up the agglomerations and ensure uniform dispersal of liquid in the mixture. The effectiveness of the liquid addition system is also influenced by the time and duration of the liquid application during the mixing cycle. The objectives of the current experiments were to determine the effect of wet mix time and the timing of the liquid addition using different spray conditions on the %CV in a corn-soy swine grower diet. Experiment 1 treatments were arranged as a complete randomized design to determine the effect of a fixed wet mix time (15, 30, 45 and 60 s) on uniformity of mix of a liquid ingredient. Experiment 2 treatments were a 3 × 2 factorial arrangement of fixed wet mix time (15, 30 and 45 s) and sprayer nozzle (with and without nozzle) to determine the effect of liquid addition on uniformity of mix. Experiment 3 treatments were arranged as a complete randomized design to determine the effect of a liquid application time (15, 30, 60 and 75 s) on uniformity of mix of a liquid ingredient. Experiment 4 treatments were a 4 × 2 factorial arrangement of liquid application time (15, 30, 60 and 75 s) and fixed total liquid mix times (75 and 90 s) to determine the effect of liquid addition on uniformity of mix. The results of Experiment 1 indicated increased fixed wet mix time had significantly decreased %CV when the feed was mixed from 15 to 60 s after the addition of the saline solution (P =

0.0057). The results of Experiment 2 indicated there was no interaction between fixed wet mix time and use of a spray nozzle ($P = 0.8938$), and the use of a spray nozzle had no effect on the %CV of the feed mixture when a 1.14% of a 23% saline solution was sprayed on the feed mixture ($P = 0.7435$). The results of Experiment 3 indicated that increased liquid application time did not affect the %CV when the feed was mixed for 60 and 75 s after the addition of the liquid ($P = 0.3708$). Finally, Experiment 4 results indicated both liquid application time and fixed total liquid mix time had an effect on the %CV of the feed mixture ($P = 0.0046$ and $P = 0.0001$, respectively), but there was no interaction between liquid application time and fixed total liquid mix time when adding 1.14% of a 23% saline solution ($P = 0.0510$). The results of these experiments indicated the wet mix time had a greater influence on the uniformity of mix than the type of nozzle used to apply the liquid. Furthermore, the shorter liquid application time allowed more time for the mechanical shear of the ribbons and paddles to break up the agglomerated wet particles and distribute them throughout the feed mixture when the total time of the liquid addition plus wet mix time was fixed.

Introduction

The number of liquids added into the mixer has increased over the last 10 years. In the past feed mills added either animal fat or molasses. However, the introduction of new liquid feed ingredients such as amino acids, betaine, choline chloride, propionic acid and formaldehyde has resulted in a more wide-spread use of liquids in animal feed. These ingredients are typically added simultaneously to the feed mixture using several spray nozzles located across the top or on the side walls of the mixer.

Using liquid ingredients reduces the amount of labor required to handle, receive and store the product, reduces fugitive dust during processing, decreases dry ingredient batching time, decreases friction in the pellet die, reduces product loss and eliminates the disposal of bags (Steen, 2013). Additional benefits include a lower price for liquids compared to dry products and additional space in the micro-system (Steen, 2013).

Several studies have identified limitations on the use of liquids in the mixing process. Fahrenholz and Behnke (1984) recommended less than 3% molasses and 10% liquid fat be added to feed in a drop-bottom mixer. Since the addition of liquids may cause agglomerations between small dry particles and liquid drops, the feed should be mixed longer to break up the agglomerations and ensure uniformity of liquid in the mixture. The amount and type of liquid addition will affect the frequency of cleaning. Products such as molasses, liquid lysine and glycerol tend to build up on the ribbons, paddles and side walls, which requires more frequent cleaning.

Bunzel (2008) described the process of mixing dry ingredients with liquid in two steps, starting with coating the dry particles and then allowing the liquid to absorb into the particle. The second step is the dispersing of the particles throughout the mixture. Although most of the wet

clumps that are generated during the liquid addition process are usually reduced by the shear forces in the mixer, other factors can influence uniformity of mix. These other factors include droplet size of the liquid, amount of liquid, viscosity of liquid, mixer type and mix time after all liquids are added. The uniformity of liquid ingredient application is often measured as the number of coated or absorbed single particles in the dry mixture.

The liquid addition system should be designed with spray nozzles that create small droplets of liquid that coat the dry particles and penetrate the particles; as well as cover the greatest number of particles in the mixture. Small droplets are produced when the potential energy of the pressure at the spray nozzle is greater than the surface tension of the liquid (Powder and Bulk Engineering, 2011). The factors that affect nozzle pressure include the number of nozzles, nozzle hole diameter, distance between pump and nozzle, pipe diameter and force of the pump. The effectiveness of the liquid addition system is also influenced by the time and duration of the liquid application during the mixing cycle.

Several studies have been conducted to determine the appropriate dry mix time. However, there is limited data in the literature related to determining the appropriate wet mix time. This is evident by the fact that batch automation controls have developed different approaches to liquid addition and wet mix time. The two most common settings in automated control systems used for wet mix time are: 1) fixed total liquid mix time (liquid addition plus a wet mix) and 2) fixed wet mix time. The fixed total liquid mix time starts when the liquid pump is activated to add the liquid, whereas the fixed wet mix time setting starts after the liquid pump stops, which creates a variable total mix time. Limited data is available in the literature that supports the uniformity of mix for either wet mix settings. Therefore, the objectives of the

current experiments were to determine the effect of wet mix time and the timing of the liquid addition using different spray conditions on the %CV in a corn-soy swine grower diet.

Material and Method

Experiment 1

Experiment 1 treatments were arranged in a 3 × 2 factorial of fixed wet mix time (15, 30 and 45 s) and sprayer nozzle (with and without nozzle) to determine the effect of liquid addition on uniformity of mix. A swine grower diet was used for the experiment (Table 4.1). The dry ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed ingredients were dry mixed for 15 s followed by the addition of 1.14% of a 23% saline solution (272 mL). The 23% saline solution was applied with a spray nozzle (model TP11006, Teejet Technologies, Springfield, IL) or without a nozzle using a pressurized hand held sprayer (model 26329, Orscheln Farm & Home LLC, Moberly, MO) to the dry feed in the mixer. A total of 10 samples was obtained from 10 different locations (Figure 4.1) after the feed was mixed for 15, 30 and 45 s wet mix time after the liquid was applied with and without the nozzle, respectively.

Experiment 2

Experiment 2 treatments were arranged as a completely randomized design to determine the effect of a fixed wet mix time (15, 30, 45 and 60 s) on uniformity of mix of a liquid ingredient. A swine grower diet was used for the experiment (Table 4.1). The dry ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed ingredients were dry mixed for 15 s followed by the addition of 1.14% of a 23% saline solution (272 mL). The 23% saline solution was applied with a spray nozzle (model TP11006, Teejet Technologies, Springfield, IL) or without a nozzle using a pressurized hand

held sprayer (model 26329, Orscheln Farm & Home LLC, Moberly, MO) to the dry feed in the mixer. A total of 10 samples was obtained from 10 different locations (Figure 4.1) after the feed was mixed for 15, 30, 45 and 60 s wet mix times after liquid addition.

Experiment 3

Experiment 3 treatments were arranged as a complete randomized design to determine the effect of liquid application time (15, 30, 60 and 75 s) on uniformity of mix of a liquid ingredient followed by a 60 s fixed wet mix time. A swine grower diet was used for the experiment (Table 4.1). The dry ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed ingredients were mixed for 15 s follow by the addition of 1.14% of a 23% saline solution (272 mL). The 23% saline solution was applied with four different liquid application times (15, 30, 60 and 75 s) using a pressurized hand held sprayer (model 26329, Orscheln Farm & Home LLC, Moberly, MO) without a nozzle and with three different nozzle models (TP11004, TP8002 and TP80015, Teejet Technologies, Springfield, IL) to the dry feed in the mixer. A total of 10 samples was obtained from 10 different locations (Figure 4.1) after the feed was mixed for 60 s wet mix time after the liquid was applied without and with three different nozzle models (Figure 4.2).

Experiment 4

Experiment 4 treatments were arranged in a 4 × 2 factorial of liquid application time (15, 30, 60 and 75 s) and fixed total liquid mix times (75 and 90 s) to determine the effect of liquid addition on uniformity of mix. A swine grower diet was used for the experiment (Table 4.1). The dry ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed ingredients were mixed for 15 s follow by the addition of 1.14% of a 23% saline solution (272 mL). The 23% saline solution was applied with four

different liquid application times (15, 30, 60 and 75 s) using a pressurized hand held sprayer (model 26329, Orscheln Farm & Home LLC, Moberly, MO) without a nozzle and with three different nozzle models (TP11004, TP8002 and TP80015, Teejet Technologies, Springfield, IL) to the dry feed in the mixer. A total of 10 samples was obtained from 10 different locations (Figure 4.1) after the feed was mixed for the total time of 90 or 105 s (Figure4.2).

Data Collection

The salt concentration in the collected samples was determined with the Quantab® chloride titrator method (McCoy, 2005). A 10 g sample was weighed into a cup and 90 g of hot distilled water (60ºC) was added to the cup. The mixture was stirred for 30 s, allowed to rest for 60 s and stirred for another 30 s. A folded filter paper was placed into the cup and the Quantab® strip was inserted into the liquid at the bottom of the filter paper. The coefficient of variation was calculated for each batch of feed.

Statistical Analysis

Data were analyzed as a factorial treatment design for Experiments 1 and 4; and a completely randomized design for Experiments 2 and 3. Experiment 1 treatments were arranged in a 3 × 2 factorial of fixed wet mix time (15, 30 and 45 s) and sprayer nozzle (with and without nozzle) to determine the effect of liquid addition on uniformity of mix. Experiment 2 determined the effect of fixed wet mix time (15, 30, 45 and 60 s) on uniformity of mix of a liquid ingredient. Experiment 3 determined the effect of liquid application time (15, 30, 60 and 75 s) on uniformity of mix of a liquid ingredient. Experiment 4 treatments were arranged in a 4 × 2 factorial of liquid application time (15, 30, 60 and 75 s) and fixed total liquid mix times (75 and 90 s) to determine the effect of liquid addition on uniformity of mix. There were 3 replicates per treatment. Data were analyzed using the GLIMMIX procedure of SAS. Means were separated by least squares

means adjustment for Bonferroni's multiple comparisons. Results were considered significant at $P \leq 0.05$.

Results and Discussion

Experiment 1 results (Table 4.2) indicated that increased fixed wet mix time had a greater effect on %CV than the use of a spray nozzle. The average actual liquid application time for the sprayer with and without nozzle was 16.7 and 9.7 s, respectively. There was no interaction between fixed wet mix time and use of a spray nozzle ($P = 0.8938$). The 45 s fixed wet mix time had the lowest %CV as compared to 15 and 30 s fixed wet mix time ($P = 0.0055$). There was a linear decrease in %CV response as the fixed wet mix time increased ($P = 0.0016$). The use of a spray nozzle had no effect on the %CV of the feed mixture when a 1.14% of a 23% saline solution was sprayed on to the feed ($P = 0.7435$). The results of this experiment contrast those of Clark (2009) where a liquid added with a single hose to the mixer did not produce a uniform mixture. The viscosity of liquid could explain the difference between Clark's statement and the result of Experiment 1. The viscosity of water, 25% saline solution and soybean oil at 20ºC are 1.0, 2.4 and 75.0 centistokes, respectively (Engineers Edge, 2016). Based on the current study, the use of a nozzle to generate smaller droplets was less important when the viscosity of saline solution was low and similar to water. Since the bridge strength of a low viscosity liquid between coated particles is less than the weight of the particle, cohesion between coated particles is low. When low viscosity liquid is added into mixer using a liquid application system without a nozzle, large drops of liquid can generate lots of clumps and some big clumps. However, liquid clumps can be broke up easily by the shear force of a mixer during mixing. Therefore, feed with the added saline solution may have less clumps and the coated particles dispersed more efficiently. However, the use of spray nozzles for high viscosity liquids such as soy oil may further reduce

the liquid and feed agglomeration in the mixture as well as more efficiently disperse the coated particles (Powder and Bulk Engineering, 2011). Furthermore, the increasing of viscosity tends to prevent fluid from breaking up into a droplet (Graco Inc., 2014).

Experiment 2 results (Table 4.3) indicated that increased fixed wet mix time improved the coefficient of variation of the mixture when 1.14% of a 23% saline solution was added with a spray nozzle. The feed mixed for 15 s after the addition of the saline solution had a significantly higher %CV, as compared to the feed mixed for 45 and 60 s (23.5, 13.6, 7.4 and 6.6 % for 15, 30, 45 and 60 s, respectively; $P = 0.0057$). There was a linear decrease in the %CV response as the fixed wet mix time increased ($P = 0.0011$). The liquid agglomerates created during the liquid addition time were eliminated by the shearing forces of the mixer during the wet mix time period allowing the coated or absorbed particle to be distributed throughout the mixture. However, the elimination of the liquid agglomerates need a minimum period of time for the mechanical shear of the ribbons to break up the agglomerates and to distribute coated particles throughout the feed mixture.

Experiment 3 results (Table 4.4) indicated that increased liquid application time did not affect the %CV when the feed was mixed for a fixed 60 s after the addition of the liquid (7.6, 7.8, 6.4 and 5.6 %CV for 15, 30, 60 and 75 s; $P = 0.3708$). Furthermore, all results were less than the 10% CV which is the percent commonly recognized by the feed industry as the cut-off for uniformity of mix analysis (Fahrenholz and Stark, 2014). The average actual liquid application times for 15, 30, 60 and 75 s were 12, 31, 62 and 72 s, respectively. The ratio of liquid application time period in total mix cycle time were 17, 29, 44 and 50% for 15, 30, 60 and 75 s, respectively, liquid application time when the total mix cycle times were 90, 105, 135 and 150 s, respectively (dry mix 15 s and fixed wet mix 60 s). As liquid application time is reduced, the

number of liquid agglomerates may increase. For instance, the 15 s liquid application time may have a higher number of liquid agglomerates than the 75 s liquid application time. However, all agglomerates of both 15 and 75 s liquid application time treatments were eliminated during the 60 s fixed wet mix time by the mechanical shear of the ribbons. The results of Experiment 1 indicated the agglomerates should be eliminated after the mixture was mixed for 45 and 60 s. In the current study the ratio of fixed wet mix time (60 s) to total mix ranged without liquid addition time (75 s) was 80% (15 s dry mix time and 60 s fixed wet mix time). The ratio of wet mix time was similar to Hayes & Stolz's (2015) recommendation that the wet mix time should be 75% of the mixer's design mix time. Hayes & Stolz did not include the liquid addition time in their suggested mix time. The results of this experiment were in contrast to Bunzel's suggestion that the liquid application time should be one-third of the mix cycle time.

The results of Experiment 4 (Table 4.5) indicated both liquid application time and fixed total liquid mix time affected the %CV of the feed mixture. The percent of liquid application time, which are the ratio between timing of liquid addition and mix cycle time, were 17, 33, 67 and 83% for 15, 30, 60 and 75 s, respectively, liquid application time when the mix cycle time was 90 s, respectively (dry mix 15 s and fixed total liquid mix 75 s); and were 14, 29, 57 and 71% for 15, 30, 60 and 75 s liquid application time when the mix cycle time was 105 s, respectively (dry mix 15 s and fixed total liquid mix 90 s). There was no interaction between liquid application time and fixed total liquid mix when adding 1.14% of a 23% saline solution ($P = 0.0510$). The 75 s application time had a significantly higher %CV as compared to the 15 and 30 s treatments (6.0, 5.8, 7.6 and 8.5 %CV for 15, 30, 60 and 75 s, respectively; $P = 0.0046$). However, all treatments were below the 10% CV. There was a linear increase in the %CV response as the liquid application time increased from 15 to 75 s ($P = 0.0008$). The 90 s fixed

total liquid mix time had a lower %CV than the 75 s fixed total liquid mix time (P = 0.0001). The results demonstrated that 90 s fixed total liquid mix time improved the uniformity of mix, as compared to the 75 s fixed total liquid mix time. However, both results were less than the 10% CV which is the percent commonly recognized by the feed industry as the cut-off for the uniformity of mix analysis (Fahrenholz and Stark, 2014). The longer liquid application time should create less liquid agglomerates than the shorter liquid application time because the mechanical shear of the ribbons eliminates some liquid clumps during liquid addition. Moreover, the mixture still required the additional mix time after the liquid was added to break up the remaining clumps and distribute the coated particles. The results of Experiment 1 and 3 supported the results of Experiment 4 that the increased fixed wet mix time had a positive effect on %CV more than the increased liquid application time. This study demonstrated that the shorter addition time resulted in a significantly lower %CV, in contrast to Bunzel's recommendation (2008) that the dry mix, liquid application time and wet mix time should be set equally.

Conclusions

The results of these experiments indicated the wet mix time had a greater influence on the uniformity of mix than the type of nozzle used to apply the liquid. Furthermore, the shorter liquid application time allowed more time for the mechanical shear of the ribbons and paddles to break up the agglomerated wet particles and distribute them throughout the feed mixture when the total time of the liquid addition plus wet mix time was fixed.

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Figures and Tables

Figure 4.1 The sampling points of the mixer surface

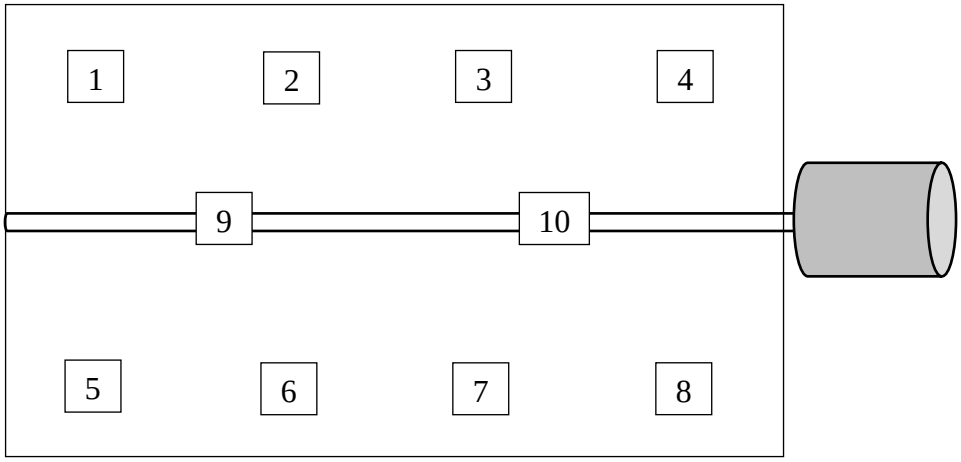


Figure 4.2 Illustration of the mix time of Experiments 3 and 4 for each treatment

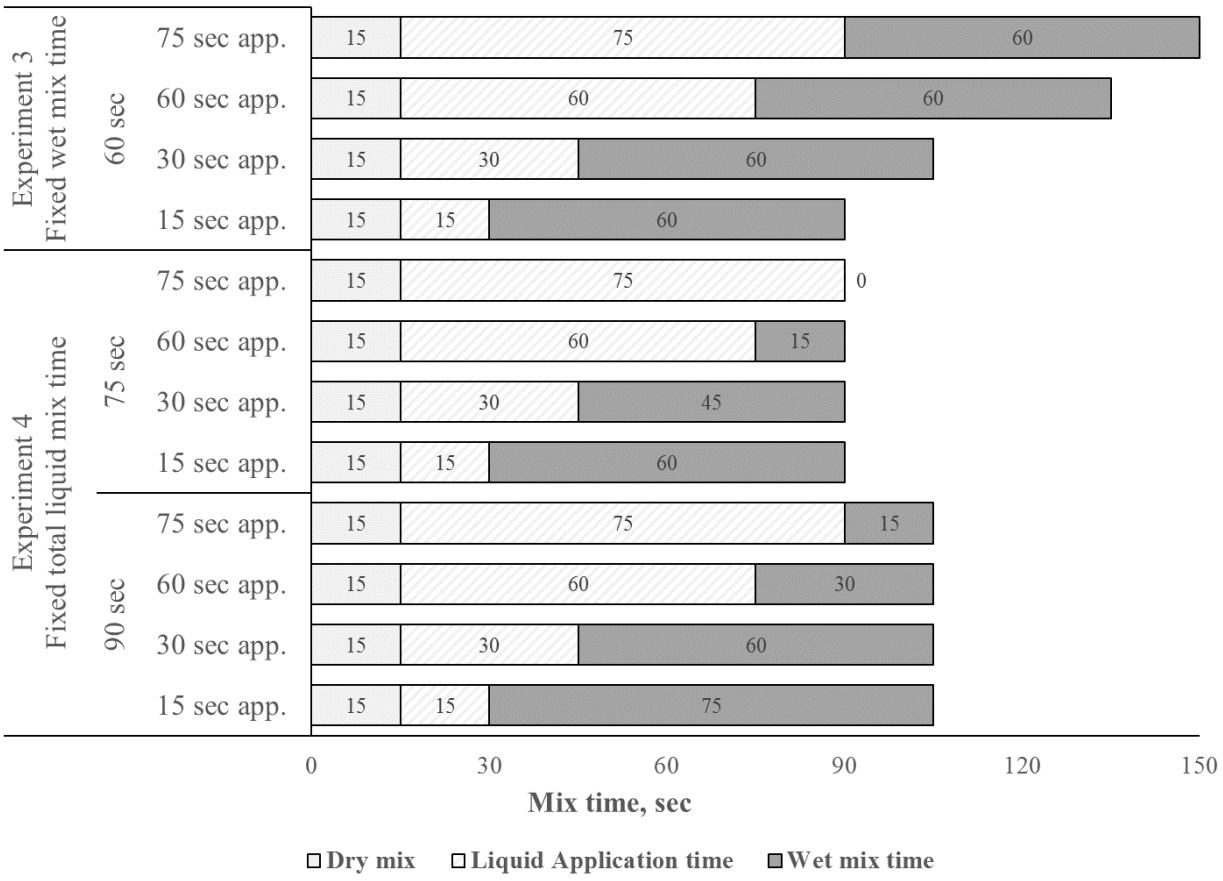


Table 4.1 Diet compositions of swine grower diet

Ingredients	Percent
Corn	71.57
Soybean meal (SBM)	25.72
Mono-calcium phosphate 21%	0.55
Limestone	1.13
Swine vitamin premix[1]	0.15
Swine trace mineral premix[2]	0.15
L-Lysine 78.8% HCl	0.30
DL-Methionine	0.07
L-Threonine	0.08
Phytase[3]	0.02
Salt[4]	0.26
Total	100.00

[1] Composition per kilogram: 110 g Iron, 110 g Zinc, 26 g Manganese, 11 g Copper, 198 g Iodine and 198 g Selenium.
[2] Composition per kilogram: 4,409,171 IU Vitamin A, 551,146 IU Vitamin D3, 17,637 IU Vitamin E, 15 mg Vitamin B12, 1,764 mg Menadione, 3,307 mg Riboflavin, 11,023 mg d-Pantothenic Acid and 19,841 mg Niacin.
[3] Ronozyme HiPhos (GT) 2700 (DSM Nutritional Products, Parsippany, NJ) provided 476.3 phytase units (FTU)/kg with a release of 0.10% available P.
[4] The salt was dissolved in distilled water.

Table 4.2 Effect of nozzle and fixed wet mix time on the coefficient of variation (%CV) of feed mixed and sprayed with 1.14% of a 23% saline solution. (Exp. 2)

Fixed wet mix times, s	Nozzle	n	Coefficient of Variation(CV), %
Interaction effects			
15	Yes	3	23.52
15	No	3	20.54
30	Yes	3	13.60
30	No	3	13.73
45	Yes	3	7.37
45	No	3	7.25
SEM			3.62
Main effect			
15		6	22.03^{A}
30		6	$13.66^{A,B}$
45		6	7.31^{B}
SEM			2.56
	Yes	9	14.83
	No	9	13.84
	SEM		2.09
			P-value
Source of variation			
Fixed wet mix time × Nozzle			0.8938
Fixed wet mix time			0.0055
Linear			0.0016
Quadratic			0.7535
Nozzle			0.7435

^{A,B} Means with different superscripts are significantly different ($P \leq 0.01$).

Table 4.3 Effect of fixed wet mix time on the coefficient of variation (%CV) of feed mixed and sprayed with a 1.14% of a 23% saline solution (Exp. 1)

Fixed wet mix times, s	N	Coefficient of Variation(CV), %
15	3	23.52^{A}
30	3	$13.60^{A,B}$
45	3	7.37^{B}
60	3	6.61^{B}
SEM		2.62
		P-value
Source of variation		
Fixed wet mix time		0.0057
Linear		0.0011
Quadratic		0.1395

^{A,B} Means with different superscripts are significantly different *(*$P \leq 0.01$).

Table 4.4 Effect of fixed liquid application time on the coefficient of variation (%CV) of feed mixed and sprayed with 1.14% of a 23% saline solution and a 60 s fixed wet mix time. (Exp. 3)

Application time[1]s, s	N	Coefficient of Variation(CV), %
15	3	7.64
30	3	7.80
60	3	6.37
75	3	5.56
SEM		0.98
		P-value
Source of variation		
Application time		0.3708

[1]Liquid addition time plus 60 s fixed wet mix time.

Table 4.5 Effect of liquid application time and fixed total liquid mix time on the coefficient of variation (%CV) of feed mixed and sprayed with 1.14% of a 23% saline solution (Exp. 4)

Application time[1]s, s	Fixed total liquid mix times, s	n	Coefficient of Variation(CV), %
Interaction effects			
15	75	3	8.07
15	90	3	3.90
30	75	3	5.99
30	90	3	5.69
60	75	3	8.53
60	90	3	6.67
75	75	3	10.45
75	90	3	6.56
SEM			0.72
Main effect			
15		6	5.99^{A}
30		6	5.84^{A}
60		6	7.60^{A,B}
75		6	8.51^{B}
SEM			0.51
	75	12	8.26^{A}
	90	12	5.71^{B}
	SEM		0.36
			P-value
Source of variation			
Application time × Fixed total liquid mix time			0.0510
Application time			0.0046
Linear			0.0008
Quadratic			0.1533
Fixed total liquid mix time			0.0001

^{A,B} Means with different superscripts are significantly different ($P \leq 0.01$).

[1]Liquid addition time plus wet mix time to reach the fixed total liquid mix time setting

Chapter 5 - The Effect of Different Percent Liquid Addition, Application Time and Mixer Type with Different Wet Mix Times on Uniformity of Mix

Abstract

The liquid addition system is often designed to add liquid ingredients with the shortest application time in order to increase the batching capacity of the mixing process. The amount of liquid that is added into the mixer will affect the batch cycle time, especially, when there is a set wet mix time. The shorter application time could produce a bigger droplet size. When dry particles come into contact with a big droplet size, they tend to stick together and become a liquid clump. The clumps are reduced by the shear force generated by the turning shaft of the mixer. The mixer type and mix time affect the amount of shear force that occurs during the mixing cycle, thereby affecting the size and number of clumps as well as the uniformity of the liquid within the mixture. The objectives of the experiments were to determine the effect of percent liquid addition, application time, mixer type and wet mix time on the uniformity of mix. Treatments were arranged in a 2 × 3 factorial: Experiment 1 treatments were fixed wet mix time (45 and 60 s) and percent liquid addition (1.14%, 2.27% and 3.41%); Experiment 2 treatments were fixed total liquid mix time (45 and 60 s) after liquid addition and percent liquid addition (1.14%, 2.27% and 3.41%); and Experiment 3 treatments were liquid application time (20 and 30 s) and fixed wet mix time (15, 30 and 45 s) to determine the effect of liquid addition on uniformity of mix using a double ribbon mixer. Experiment 4 treatments were arranged in a 2 × 3 factorial of liquid application time (15 and 30 s) and fixed wet mix time (10, 20 and 30 s) to determine the effect of liquid addition on uniformity of mix using a paddle mixer. The results of Experiment 1 indicated the 45 s fixed wet mix time had a significantly higher %CV as compared to the 60 s fixed wet mix time ($P = 0.0467$) but there was no interaction between fixed wet mix

time and percent addition of saline ($P = 0.5114$). The results of Experiment 2 indicated an interaction between the percent addition of saline and the fixed total liquid mix time ($P = 0.0221$) and both percent addition of saline and fixed total liquid mix time affected the %CV treatment ($P < 0.0001$ and $P = 0.0002$, respectively). The results of Experiment 3 indicated that fixed wet mix time had a greater effect on %CV than application time ($P < 0.0001$ and $P = 0.6530$, respectively) when 2.27% of a 23% saline solution was added in a double ribbon mixer and there was no interaction between application time and fixed wet mix time ($P = 0.6380$). The results of Experiment 4 indicated that both fixed wet mix time and application time affected the %CV ($P = 0.0009$ and $P = 0.0296$, respectively) when 2.27% of a 23% saline solution was added in a paddle mixer and there was no interaction between application time and fixed wet mix time ($P = 0.2896$). The results of these experiments demonstrated that the liquid addition time and the percent of liquid addition affected the uniformity of mix. The uniformity of liquid application should be tested with the highest percent liquid addition. Furthermore, application and wet mix times should be determined for each mixer type and size to establish the optimal batch cycle.

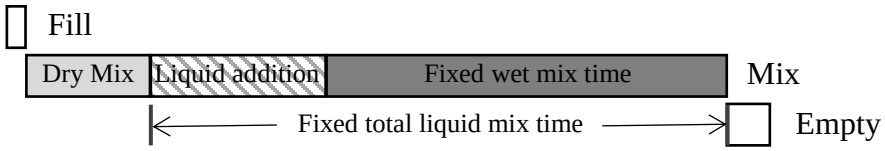
Introduction

The number of liquid ingredients and amount added to the mixer has increased over the last 10 years. Similar to dry ingredient classifications, liquid additives are separated into two groups: macro ingredients, which are more than 1 kg/ton (0.1%) of the diet, and micro ingredients, which are less than 1 kg/ton of the diet (Steen, 2013). The amount of liquids added to a mixer could affect the batch cycle time when the batch controller has a set wet mix time. The two most common settings in automated control systems are fixed total liquid mix time (liquid addition plus a wet mix time) and fixed wet mix time. The fixed total liquid mix time starts when a liquid pump is activated to add the liquid, whereas the fixed wet mix time setting starts after the liquid pumps stop. The liquid addition time in the fixed wet mix time setting creates a variable batch cycle time. Therefore, liquid addition systems are designed to add liquid ingredients in the shortest time possible in order to increase the batching capacity of the mixing process. Steen (2013) suggested that in order to achieve an uniform mix, the liquid addition time should be one-half of the wet mix time while Bunzel (2008) recommended the liquid addition time should be one-third of the total mix cycle time. However, there is limited data available to verify these recommendations for liquid addition time. The amount of liquid that is added into the mixer could increase the total cycle time required. Steen (2013) reported liquid ingredient inclusion levels in North America, Europe and Asia ranged from 0.15-0.30% for methionine, 0.05-0.50% for choline chloride, 0.15-0.30% for lysine, 0-3% for fat or oil and 0.01% for enzymes. Each liquid may be added in separated line or in a single line from a common liquid scale. The liquid addition times vary based on the percent of liquid applied and type of application system. Liquid application systems are designed to balance droplet size and liquid addition time. Rioux (2003) suggested that the number of clumps in the mixture was correlated

with the amount of fluid penetration into particles. Additionally, the fluid penetration is dependent on substrate characteristics, fluid properties and the interaction between the fluid and substrate. For instance, a wheat middlings based premix can absorb more water than a limestone based premix. A corn-soy diet can absorb water better than an oil-based fluid. Gane et al. (1999) described the interaction of fluid on the surface of the substrate, finding that spreading and penetration are competing when a liquid droplet contacts a porous surface. When particles with saturated liquid surfaces contact each other, they tend to stick together and become a liquid clump. The clumps are reduced by the shear force generated by the shaft turning in the mixer. The mixer type and mix time affect the amount of shear force that occurs during the mixing process, thereby affecting the size and number of clumps as well as the uniformity of the liquid within the mixture. Tekchandaney (2013) concluded that a relationship exists between the shear force and the mixer type, namely that double-paddle mixers have a slightly higher shear force, single-paddle and ribbon mixers have an average shear force and the tumbling mixers have a low shear force. The objective of the experiments was to determine the effect of percent liquid addition, application time, mixer type and wet mix time on the uniformity of mix.

Material and Method

Illustration of the mixing cycle



Experiment 1

Treatments were arranged in a 2 × 3 factorial of fixed wet mix time (45 and 60 s) after liquid addition and percent liquid addition (1.14%, 2.27% and 3.41%) to determine the effect of liquid addition on uniformity of mix. A swine grower diet was used for the experiment (Table

5.1). The dry ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed ingredients were dry mixed for 15 s followed by the addition of 1.14%, 2.27% and 3.41% of a 23% saline solution. The 23% saline solutions were applied to dry feed in the mixer by using a hand-held sprayer (model 26329, Orscheln Farm & Home LLC, Moberly, MO) with a nozzle (model TP11006, Teejet Technologies, Springfield, IL). A total of 10 samples was obtained from 10 different locations (Figure 5.1) after the feed was mixed for 45 and 60 s fixed wet mix time (Figure 5.2). Samples were analyzed for salt concentration with Quantab® strips.

Experiment 2

Treatments were arranged in a 2 × 3 factorial of fixed total liquid mix time (45 and 60 s) and percent liquid addition (1.14%, 2.27% and 3.41%) to determine the effect of liquid addition on uniformity of mix. A swine grower diet was used for the experiment (Table 5.1). The dry ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed ingredients were dry mixed for 15 s followed by the addition of 1.14%, 2.27% and 3.41% of a 23% saline solution. The 23% saline solutions were applied to dry feed in the mixer by using a hand-held sprayer (model 26329, Orscheln Farm & Home LLC, Moberly, MO) with a nozzle (model TP11006, Teejet Technologies, Springfield, IL). A total of 10 samples was obtained from 10 different locations (Figure 5.1) after the feed was mixed for the total time of 60 and 75 s (Figure 5.2). Samples were analyzed for salt concentration with Quantab® strips.

Experiment 3

Treatments were arranged in a 2 × 3 factorial of liquid application time (20 and 30 s) and fixed wet mix time (15, 30 and 45 s) to determine the effect of liquid addition on uniformity of

mix. A swine grower diet was used for the experiment (Table 5.1). The dry ingredients were added to a 0.056 m^3 double ribbon mixer (Hayes and Stolz model HP2SSS-0106, Fort Worth, TX). The feed ingredients were mixed for 15 s followed by the addition of 2.27% of a 23% saline solution (544 mL) to the dry feed in the mixer by using the hand-held sprayer (model 26329, Orscheln Farm & Home LLC, Moberly, MO) with 2 different application times by using different nozzles (TP11015 and TP11006, Teejet Technologies, Springfield, IL). A total of 10 samples was obtained from 10 different locations (Figure 5.1) after the feed was mixed for 15, 30 and 45 s fixed wet mix time. Samples were analyzed for salt concentration with Quantab® strips.

Experiment 4

Treatments were arranged in a 2 × 3 factorial of liquid application time (15 and 30 s) and fixed wet mix time (10, 20 and 30 s) to determine the effect of liquid addition on uniformity of mix. A sample of 90.40 kg of corn was added to a 0.170 m^3 paddle mixer (Davis model 2014197-SS-S1, Bonner Springs, KS). The corn was mixed for 15 s follow by the addition of 2.27% of a 23% saline solution (1813 mL) to the dry feed in the mixer with two different application times (15 and 30 s) by using 2 or 4 hand-held sprayers (model 26329, Orscheln Farm & Home LLC, Moberly, MO) with a nozzle (model TP11020, Teejet Technologies, Springfield, IL), respectively. A total of 10 samples was obtained from 10 different locations (Figure 5.1) after the feed was mixed for 10, 20 and 30 s fixed wet mix time. Samples were analyzed for salt concentration with Quantab® strips.

Data Collection

The salt concentration in the collected samples was determine with the Quantab® chloride titrator method (McCoy, 2005). A 10 g sample was weighed into a cup and 90 g of hot distilled water (60ºC) was added to the cup. The mixture was stirred for 30 s, allowed to rest for

60 s and stirred for another 30 s. A folded filter paper was placed into the cup and the Quantab® strip was inserted into the liquid at the bottom of the filter paper. The coefficient of variation (CV) was calculated for each batch of feed.

Statistical Analysis

Data were analyzed as a factorial treatment design for all experiments. Experiment 1 treatments were arranged in a 2 × 3 factorial of fixed wet mix time (45 s, FW45 and 60 s, FW60) and percent liquid addition (1.14%, 2.27% and 3.41%) to determine the effect of liquid addition on uniformity of mix. Experiment 2 treatments were arranged in a 2 × 3 factorial of fixed total liquid mix time (45 s, FTL45 and 60 s, FTL60) and percent liquid addition (1.14%, 2.27% and 3.41%) to determine the effect of liquid addition on uniformity of mix. The fixed wet mix time and fixed total liquid mix time factors from Experiments 1 and 2, respectively, were combined as a liquid mix cycle time setting factor. The combination of Experiments 1 and 2 treatments were arranged in a 4 × 3 factorial of liquid mix cycle time setting (FTL45, FTL60, FW45 and FW60) and percent liquid addition (1.14%, 2.27% and 3.41%) to determine the effect of liquid addition on uniformity of mix. Experiment 3 treatments were arranged in a 2 × 3 factorial of liquid application time (20 and 30 s) and fixed wet mix time (15, 30 and 45 s) to determine the effect of liquid addition on uniformity of mix using a double ribbon mixer. Experiment 4 treatments were arranged in a 2 × 3 factorial of liquid application time (15 and 30 s) and fixed wet mix time (10, 20 and 30 s) to determine the effect of liquid addition on uniformity of mix using a paddle mixer. There were 3 replicates per treatment. Data were analyzed using the GLIMMIX procedure of SAS. Means were separated by least squares means adjustment for Bonferroni's multiple comparisons. Results were considered significant at $P \leq 0.05$.

Results and Discussion

The results of Experiment 1 (Table 5.2) indicated that increasing fixed wet mix time improved the %CV. There was no interaction between fixed wet mix time and percent addition of saline ($P = 0.5114$). The average actual application times for 1.14, 2.27 and 3.41% liquid additions were 16.7, 31.0 and 43.3 s, respectively. The percent addition of saline was not significantly different on %CV ($P = 0.2372$). However, the 45 s fixed wet mix time had a significantly higher %CV as compared to the 60 s fixed wet mix time ($P = 0.0467$), but the CV was less than 10%.

The results of Experiment 2 (Table 5.3) indicated an interaction between the percent addition of saline and the fixed total liquid mix time ($P = 0.0221$). The 1.14% of saline addition combined with the 60 s fixed total liquid mix time resulted in the lowest %CV (6.26%) as a result of the longest wet mix time (43.3 s). In contrast, the 3.41% of saline addition with 45 s fixed total liquid mix time had the highest effect on %CV (42.25%) due to the shortest wet mix time (1.7 s). The main effects indicated that both percent addition of saline and fixed total liquid mix time affected the %CV. The 45 s fixed total liquid mix time had significantly higher %CV than 60 s ($P = 0.0002$). There was a quadratic increase in %CV as the percentages of added saline solution increased ($P = 0.0270$). The highest %CV occurred when the 3.41% of the 23% saline solution was applied to the feed ($P < 0.0001$).

The results of combined data for Experiments 1 and 2 (Table 5.4) indicated an interaction between percent saline addition and liquid mix cycle time setting ($P < 0.0001$). The 3.41% saline solution addition with a fixed wet mix (FW45 and FW60) treatments were less than 10% CV (9.21 and 6.01%, respectively). However, for fixed total liquid mix times of 45 s and 60 s (FTL45 and FTL60) the %CV were above the target (42.25 and 21.47%, respectively). The

results of the 2.27% saline solution addition with 45 s and 60 s fixed wet mix time (FW45 and FW60) were similar to the 3.41%. The FW45 and FW60 treatments were less than 10% CV, whereas FTL45 and FTL60 treatments were greater than 10% CV (17.73 and 13.20%, respectively). The results of the 1.14% saline solution addition with 45 s and 60 s fixed wet mix time (FW45 and FW60) were similar to the 2.27 and 3.41% addition results. However, there was a difference in the FTL45 and FTL60 treatments. The data suggested that there was sufficient wet mix time after the 1.14% solution was applied for the FTL60 treatment but not enough wet mix time for the FTL45 treatment. The FTL60 treatment actually had a 43.3 s wet mix time due to the short application time of 1.14% liquid addition. The %CV were not different as the percent of the saline solution was increased from 1.14% to 2.27 % and the %CV increased as the percent of saline solution was increased from 1.14 and 2.27 % to 3.41% (quadratic; $P = 0.0076$). The liquid mix cycle time setting significantly affected %CV ($P < 0.0001$). The short liquid mix cycle time setting had a greater effect on %CV than the longer cycle time.

The two experiments had different results due to how the wet time was controlled. In Experiment 1, the liquid addition time was not counted in the wet mix time, whereas in Experiment 2 the liquid addition time was counted in the total liquid mix time. For instance, assume a mixing cycle of a batching system consists of 45 s dry ingredient fill time, 15 s dry mix, 20 s liquid addition time, 45 s wet mix time and 15 s empty time. If the wet mix time setting of the batching control is set as fixed wet mix time (Exp. 1), then the mixing cycle is 140 s/batch and the mixer efficiency is 25 batches/hr. The number of batches per hour is decreased as the liquid addition time is increased. In contrast, when the wet mix time setting of the batching control is set as fixed total liquid mix time (Exp. 2), the mixing cycle is 120 s/batch and the mixer efficiency is 30 batches/hr. The number of batches per hour is stable when the liquid

addition time is changed. However, a mixing uniformity test is required for the fixed wet mix time setting if the % liquid addition increases and for the fixed total liquid mix time setting if the % liquid addition or liquid addition time increases based on the previous test.

The results of Experiment 3 (Table 5.5) indicated that fixed wet mix time had a greater effect on %CV than application time when a 2.27% of a 23% saline solution was added to a 0.056 m^3 double ribbon mixer. There was no interaction between application time and fixed wet mix time ($P = 0.6380$). The average actual application time for 20 and 30 s were 19.0 and 30.9 s, respectively. The %CV for the 20 s and 30 s application times were not significantly different ($P = 0.6530$; 22.66 and 21.76%, respectively). However, there was a quadratic decrease in %CV as the fixed wet mix time increased ($P = 0.0241$). The lowest %CV occurred after the feed was mixed for a 45 s fixed wet mix time ($P < 0.0001$).

The results of Experiment 4 (Table 5.6) indicated that both fixed wet mix time and application time affected the %CV when 2.27% of a 23% saline solution was added to a 0.170 m^3 paddle mixer. There was no interaction between application time and fixed wet mix time ($P = 0.2896$). The average actual application time for 15 and 30 s were 13.0 and 27.3 s, respectively. The 15 s application time had a greater effect on %CV than the 30 s application time ($P = 0.0296$). As the fixed wet mix time was increased from 10 to 30 s, the %CV decreased from 17.0 to 8.23%, respectively (linear; $P = 0.0004$).

The results of Experiments 3 and 4 indicated the importance of testing mixers at the time of installation as required for the CGMPs [FDA, 21 CFR part 225.30 (a)]. The %CV of feed mixed with a ribbon mixer did not change when the liquid application time was decreased while the %CV of the feed mixed with a paddle mixer increased when decreasing the liquid application time. The results demonstrated that mixer types and sizes affected the %CV. The results of the

experiments also demonstrated that dry mix, liquid addition time and wet mix time cannot be generically applied to mixers based on size and type.

Conclusions

The results of Experiments 1 and 2 demonstrated that both liquid cycle time and the percent of liquid addition affected the uniformity of mix. The uniformity of liquid application should be tested with the highest percent of liquid addition and different wet mix times in order to establish the wet mix time. The efficiency of the mixing process will be increased if the liquid application time is decreased. The fixed total liquid mix time computer set-up has a constant mixing cycle time, whereas the fixed wet mix time is variable based on the time required to apply liquids to the formula. Moreover, the results of Experiment 3 and 4 demonstrated that application time and fixed wet mix time must be determined for each mixer's type and size. The data from the experiments suggest that while extended liquid application times are beneficial, there should be a minimum fixed wet mix time after all of the liquids have been added to the mixer.

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Figures and Tables

Figure 5.1 The sampling points of the mixer surface

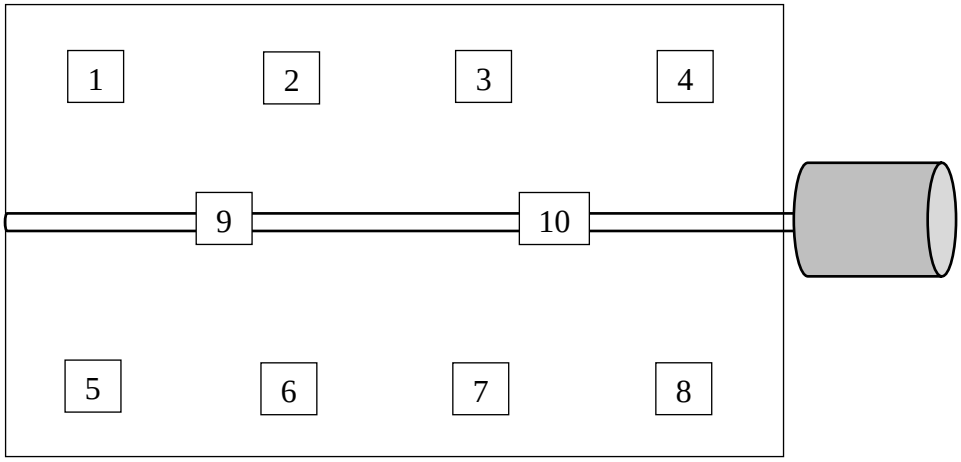

Figure 5.2 Illustration of the mix time of Experiments 1 and 2 for each treatment

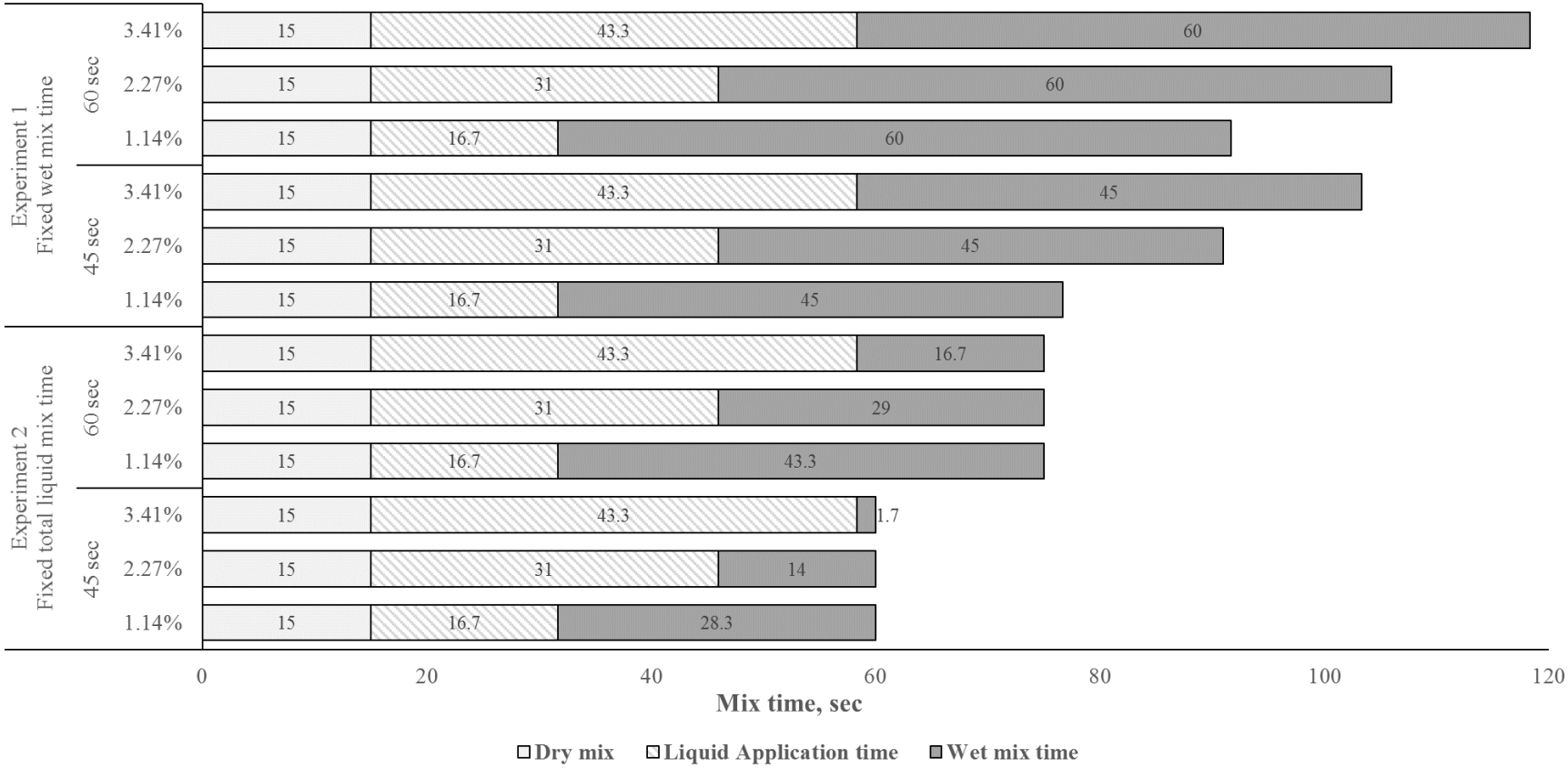

Table 5.1 Diet compositions of swine grower diet

	% added saline solution		
Ingredients	1.14	2.27	3.41
Corn	71.57	71.37	71.18
Soybean meal (SBM)	25.72	25.66	25.59
Mono-calcium phosphate 21%	0.55	0.55	0.55
Limestone	1.13	1.13	1.13
Swine vitamin premix[1]	0.15	0.15	0.15
Swine trace mineral premix[2]	0.15	0.15	0.15
L-Lysine 78.8% HCl	0.30	0.30	0.30
DL-Methionine	0.07	0.07	0.07
L-Threonine	0.08	0.08	0.08
Phytase[3]	0.02	0.02	0.02
Salt[4]	0.26	0.52	0.78
Total	100.00	100.00	100.00

[1] Composition per kilogram: 110 g Iron, 110 g Zinc, 26 g Manganese, 11 g Copper, 198 g Iodine and 198 g Selenium.
[2] Composition per kilogram: 4,409,171 IU Vitamin A, 551,146 IU Vitamin D3, 17,637 IU Vitamin E, 15 mg Vitamin B12, 1,764 mg Menadione, 3,307 mg Riboflavin, 11,023 mg d-Pantothenic Acid and 19,841 mg Niacin.
[3] Ronozyme HiPhos (GT) 2700 (DSM Nutritional Products, Parsippany, NJ) provided 476.3 phytase units (FTU)/kg with a release of 0.10% available P.
[4] The salt was dissolved in 272, 544 and 816 mL distilled water for 1.14%, 2.27% and 3.41%, respectively.

Table 5.2 Effect of fixed wet mix time on the coefficient of variation (%CV) of feed mixed and sprayed with different percent saline solution additions (Exp. 1)

Liquid addition, %	Fixed wet mix times, s	n	Coefficient of Variation(CV), %
Interaction effects			
1.14	45	3	7.37
1.14	60	3	6.06
2.27	45	3	6.28
2.27	60	3	5.30
3.41	45	3	9.21
3.41	60	3	6.01
SEM			1.01
Main effect			
1.14		6	6.72
2.27		6	5.79
3.41		6	7.61
SEM			0.71
	45	9	7.62[a]
	60	9	5.79[b]
	SEM		0.58
			P-value
Source of variation			
Percent liquid × Fixed wet mix time			0.5144
Percent liquid			0.2372
Fixed wet mix time			0.0467

[a,b] Means with different superscripts are significantly different ($P \leq 0.05$).

Table 5.3 Effect of fixed total liquid mix time on the coefficient of variation (%CV) of feed mixed and sprayed with different percent saline solution additions (Exp. 2)

Liquid addition, %	Fixed total liquid mix times, s	n	Coefficient of Variation (CV), %
Interaction effects			
1.14	45	3	14.58[a,b]
1.14	60	3	6.26[a]
2.27	45	3	17.73[a,b]
2.27	60	3	13.20[a,b]
3.41	45	3	42.25[c]
3.41	60	3	21.47[b]
SEM			2.60
Main effect			
1.14		6	10.42[A]
2.27		6	15.46[A]
3.41		6	21.86[B]
SEM			1.84
	45	9	24.85[A]
	60	9	13.64[B]
	SEM		1.50
			P-value
Source of variation			
Percent liquid ×Fixed total liquid mix time			0.0221
Percent liquid			<0.0001
Linear			<0.0001
Quadratic			0.0270
Fixed total liquid mix time			0.0002

[a-c] Means with different superscripts are significantly different ($P \leq 0.05$).
[A,B] Means with different superscripts are significantly different ($P \leq 0.01$).

Table 5.4 Effect of liquid mix cycle time setting on the coefficient of variation (%CV) of feed mixed and sprayed with different percent saline solution additions (Exp. 1 and 2)

Liquid addition, %	Time[1]	n	Coefficient of Variation(CV), %
Interaction effects			
1.14	FTL45	3	14.58^{A,B,C}
1.14	FTL60	3	6.26^{A}
1.14	FW45	3	7.37^{A,B}
1.14	FW60	3	6.06^{A}
2.27	FTL45	3	17.73^{B,C}
2.27	FTL60	3	13.20^{A,B,C}
2.27	FW45	3	6.28^{A}
2.27	FW60	3	5.30^{A}
3.41	FTL45	3	42.25^{D}
3.41	FTL60	3	21.47^{C}
3.41	FW45	3	9.21^{A,B}
3.41	FW60	3	6.01^{A}
SEM			1.98
Main effect			
1.14		12	8.57^{A}
2.27		12	10.62^{A}
3.41		12	19.73^{B}
SEM			0.99
	FTL45	9	24.85^{A}
	FTL60	9	13.64^{B}
	FW45	9	7.62^{C}
	FW60	9	5.79^{C}
	SEM		1.14
			P-value
Source of variation			
Percent liquid × Application			<0.0001
Percent liquid			<0.0001
Linear			<0.0001
Quadratic			0.0076
Application			<0.0001

$^{A-D}$ Means with different superscripts are significantly different ($P \leq 0.01$).

[1]FTL = fixed total liquid mix time, FW = fixed wet mix time, 45 = 45 s and 60 = 60 s.

Table 5.5 Effect of liquid application time and fixed wet mix time on the coefficient of variation (%CV) of feed mixed and sprayed with 2.27% of a 23% saline solution in a double ribbon mixer (Exp. 3)

Application times, s	Fixed wet mix times, s	n	Coefficient of Variation(CV), %
Interaction effects			
20	15	3	37.87
20	30	3	20.12
20	45	3	9.99
30	15	3	36.55
30	30	3	17.13
30	45	3	11.59
SEM			2.41
Main effect			
20		9	22.66
30		9	21.76
SEM			1.39
	15	6	37.21^{A}
	30	6	18.63^{B}
	45	6	10.79^{C}
	SEM		1.70
			P-value
Source of variation			
Application time × Fixed wet mix time			0.6380
Application time			0.6530
Fixed wet mix time			<0.0001
Linear			<0.0001
Quadratic			0.0241

[A-C] Means with different superscripts are significantly different ($P \leq 0.01$).

Table 5.6 Effect of liquid application time and fixed wet mix time on the coefficient of variation (%CV) of feed mixed and sprayed with 2.27% of a 23% saline solution in a paddle mixer (Exp. 4)

Application times, s	Fixed wet mix times, s	n	Coefficient of Variation(CV), %
Interaction effects			
15	10	3	20.23
15	20	3	11.83
15	30	3	8.46
30	10	3	13.76
30	20	3	7.79
30	30	3	7.99
SEM			1.82
Main effect			
15		9	13.51[a]
30		9	9.84[b]
SEM			1.05
	10	6	17.00[A]
	20	6	9.81[B]
	30	6	8.23[B]
	SEM		1.29
			P-value
Source of variation			
Application time × Fixed wet mix time			0.2896
Application time			0.0296
Fixed wet mix time			0.0009
Linear			0.0004
Quadratic			0.1004

[a,b] Means with different superscripts are significantly different ($P \leq 0.05$).
[A,B] Means with different superscripts are significantly different ($P \leq 0.01$).

Chapter 6 - Summary of Findings

The results of these experiments determined the effect of methodology on the salt concentration of a feed sample when using the Quantab® chloride titrator Method, on uniformity of dry mixture and on uniformity of liquid on a dry mixture. The first experiment evaluated the effect of water temperature, extraction time and dissolution time on the chloride ion test by the Quantab® chloride titrator method. The samples extracted with 60ºC water had significantly higher salt concentrations as compared to the 20ºC and 40ºC temperatures ($P < 0.0001$). There was an interaction between the type of equipment (adjustable dispenser 100 mL, graduated cylinder and balance) used to measure the water weight and water temperature (20, 40 and 60ºC), ($P < 0.0001$). The measurements of 60°C water by a dispenser and graduated cylinder were less accurate as compared to the scale which was more accurate due to mass measurement. An increased stirring time from15 to 60 s did not significantly affect the salt concentration ($P = 0.2295$) while increasing the water temperature increased the salt concentration ($P < 0.0001$). There was no interaction between water temperature (20, 40 and 60ºC) and dissolution time (0, 6 and 12 min) ($P = 0.5679$). The increasing of water temperature or dissolution time increased the salt concentration ($P = 0.0136$ and $P < 0.0001$, respectively). These results indicated only the scale should be used for measuring hot distilled water at 60ºC, and the 30 s extraction time by stirring followed by immediately placing the strip into the solution after extraction should be used for the Quantab® chloride titrator method.

The second experiment evaluated the effect of extended mix time, particle size of the marker and sample preparation on the %CV. There was no interaction between mix time (2, 3, 5, 15, 30, 45 and 60 min) and salt particle size (fine-350 µm, medium-464 µm and coarse-728 µm) ($P = 0.4366$). The extended mix time did not increase the %CV of the feed ($P = 0.3073$). The

mixture that contained the coarse salt had a significantly different %CV than the fine and medium salt treatments ($P < 0.0001$). There was an interaction between sample preparation (unground and ground) and salt particle size (fine, medium and coarse) ($P = 0.0002$). The difference in the %CV between unground and ground samples was significantly greater for the mixture with coarse salt (21.84 and 12.95%, respectively) than the mixtures with medium salt (9.00 and 6.41%, respectively) and fine salt (8.09 and 6.74%, respectively). These results indicated that an extended mix time up to 60 minutes did not increase the %CV of the feed, and the particle size of the salt used in the uniformity of mix test can significantly change the results of the test. Furthermore, the data suggests that fine salt should be used as a marker for the uniformity of mix test.

The third and fourth experiments evaluated the effect of wet mix time, spray condition, application time, % liquid addition and mixer type on the %CV. The increased fixed wet mix time significantly decreased %CV when the mix time was increased from 15 to 60 s after the addition of the saline solution ($P = 0.0057$). There was no interaction between fixed wet mix time (15, 30 and 45 s) and use of a spray nozzle (with and without nozzle) ($P = 0.8938$). The use of a spray nozzle had no effect on the %CV of the feed mixture when 1.14% of a 23% saline solution was sprayed on to the feed ($P = 0.7435$). There was an interaction between percent saline addition (1.14%, 2.27% and 3.41%) and liquid mix cycle time setting (45 s fixed total liquid mix time, 60 s fixed total liquid mix time, 45 s fixed wet mix time and 60 s fixed wet mix time) ($P < 0.0001$). The %CV of the 45 s fixed wet mix time and 60 s fixed wet mix time treatments were similar when the 1.14%, 2.27% and 3.41% was added. On the other hand, the %CV of the 45 s fixed total liquid mix time and 60 s fixed total liquid mix time were also similar when the 1.14% and 2.27% were added, but not the 3.41% treatment. There was no interaction

between application time and fixed wet mix time when 2.27% of a 23% saline solution was added in the double ribbon and paddle mixer (P = 0.6380 and 0.2896, respectively). Increasing fixed wet mix time (15, 30 and 45 sec-ribbon mixer and 10, 20 and 30 sec-paddle mixer) decreased the %CV for the double ribbon and paddle mixer ($P < 0.0001$ and $P = 0.0009$, respectively). Increasing application time (15 and 30 s) decreased the %CV for the paddle mixer ($P = 0.0296$). However, the %CV of 20 and 30 s application time treatments were not significantly different for the double ribbon mixer ($P = 0.6530$). These results demonstrated that the wet mix time had a greater influence on the uniformity of mix than the type of nozzle used to apply the liquid. The shorter liquid application time allowed more time for the mechanical shear of the ribbons and paddles to break up the agglomerated wet particles and distribute them throughout the feed mixture when the total time of the liquid addition plus wet mix time was fixed. The liquid addition time and the percent of liquid addition affected the uniformity of mix. The uniformity of liquid application should be tested with the highest percent liquid addition. Furthermore, application and wet mix times should be determined for each mixer type and size to establish the optimal batch cycle.

The results of these experiments indicated that the Quantab® chloride titrator method should use a scale for measuring hot water, and a 30 s extraction time by stirring the mixture followed by immediately placing the strip into the solution after extraction. Feed did not segregate after mixing for one hour. The greater number of particles per gram of the marker increased the precision of the analysis. Thus, fine salt should be used as a marker for evaluation of the mixing process. The wet mix time had a greater influence on the uniformity of mix than the type of nozzle used to apply the liquid. The data suggested that while extended liquid application times are beneficial, there should be a minimum fixed wet mix time after all of the

liquids have been applied to the mixer. Furthermore, the uniformity of the liquid application system should be tested with the highest percent liquid addition and amino acid liquid should be an appropriate marker for evaluation of liquid addition in the feed industry.