THE EFFECT OF TRACERS' PHYSICAL PROPERTIES ON RETENTION TIME MEASUREMENTS INSIDE THE CONDITIONER OF A PELLET MILL

by

ESAM A. SALIM

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Approved by:

Major Professor
Dr. Keith Behnke

Abstract

The effect of tracer particle size on the accuracy of measuring retention time inside the conditioner of a pellet mill was studied. Three experiments were conducted using tracers with different particle sizes. The control retention time was calculated using the hold-up capacity method, while retention times for treatments were calculated using the pulse-input method. In addition to the previously prepared tracers, crystalline salt and powder color dye were used as tracers in this study to represent small particle tracers.

In experiment one, large, medium, and small tracers were used. The treatment with large particle size was closer to the control with differences around 1.5%. Conversely, using the tracer with small particle size resulted in larger differences, approximately 18%. Two tracers were used in experiment two, resulted in 33% difference between treatment and control using a small particle size tracer, and a 10% difference using a tracer with similar particle size to the main materials. A tracer similar in particle size to the materials flowing inside the conditioner was used in the third experiment in addition to the salt and dye. Statistical analyses for this experiment indicated that particle size affects the accuracy of retention time measurements. There was a significant difference ($P<0.05$) in the comparison between salt and red color dye in treatment two, while there was no significant difference ($P>0.05$) between them in treatment one (same particle size). Moreover, in another comparison of the differences between treatments and their related controls, there was significant difference ($P<0.05$). However, the P-value for the red

dye comparison (0.0126) was higher than that of salt (0.0026), which adds density as another influential factor that affects retention time measurement.

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Dedication

For my father who planted the dream.

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For Lamees who helped make it true.

For Ameen and Hamza.

CHAPTER 1 - Literature Review

Pelleting

History

The pelleting process was introduced to the United States feed industry in the late 1920s through an imported pellet machine from Europe. Later, the first American pellet machine was built by S. Howes Company in 1930 (Schoeff, 2005) and since then the concept has been used widely in the feed industry because of the numerous benefits that pelleting offers. Although the additional cost to produce pellets vs. mash in animal feeds is averaged from $3.00 to $7.00 per ton (Harper, 1998), pelleted feed has been used due to the benefits that affect both animal performance (Hussar et al., 1962; Jahan et al., 2006) and physical handling of the feed (Robinson, 1975). Improved animal performance has been attributed to starch and protein modification, destruction of pathogenic organisms, less time and energy consumption for prehension, and improved palatability (Behnke, 1994). In addition, physical properties of pelleted feed have been recognized to decrease feed wastage and ingredient segregation, while increasing bulk density (especially for material with high fiber content), and reducing the cost of feed handling and transportation (Robinson 1975; Behnke, 1994). Robinson (1975) describes the advantages of pelleting on materials handling and storage capacity, stating that a railcar load can handle pelleted feed almost double the amount that of mash feed. Hussar et al. (1962) found that pelleting increases bulk density by 24%. Therefore, the cost of handling and transporting these materials is less.

Effect of feeding pellets on animal performance

Pelleted feed is widely used in the United States due to the physical and nutritional benefits (Behnke, 2001). There are numerous studies in this area that found the effectiveness of pelleting on animal performance, in both the poultry and the swine industries. In the poultry industry, Jahan et al. (2006) reported that body weight and body weight gain from the 4th to the 8th week were higher for broilers fed crumbles, pellets, and mash, respectively. The results also demonstrated that the feed efficiency was superior for broilers fed pellets compared to birds fed mash. These results are probably because feeding pellets or crumbles ensures that broilers get the required amount of nutrients in each mouthful, presuming that the material is mixed properly. In addition, there is also less energy consumed during feeding (Behnke, 1994). Hussar et al. (1962) found that growing chickens fed pelleted feed consumed 15% more feed and gained 25% more weight than those fed mash. Moreover, the feed conversion ratio was higher for chickens fed mash than those fed pellets or reground pellets. In this study, they assessed the apparent metabolizability, which was not affected by the physical properties of the feed. Consequently, they suggested that these results are due to the "improved nitrogen and/or energy retention, or simply due to increased feed intake." Thus, the increased feed intake is due to the palatability of the feed or the amount of material in each mouthful, which in turn reduce the movement of chickens and saves energy.

Similar to the poultry industry, there are many studies in the swine industry that found a positive correlation between using pelleted feed and the improvement of animal performance. Wondra et al. (1995) found that feeding pellets to finishing pigs improved ADG by 5% and

gain/feed by 7%. In an earlier study by Baird (1973), similar results were reported. The study indicated that feeding pelleted feed improved feed gain by 4.6% and improved feed conversion by 7.8%. The explanations for these results might be due to increased digestibility, increased bulk density (Skoch et al., 1983), or improved palatability (Behnke, 2001). In addition to the factors stated above, reducing particle size in pigs' diets improves animal performance, but affects the flowability of the feed in the mash form. Pelleting the feed eliminates this outcome (Wondra et al., 1995). In general, both reduced particle size and pelleted feed are significant factors in improving pigs' performance.

While there is no proof in the literature that pelleting improves the nutritional value of the feed, there are some suggestions that starch gelatinization during the process makes the starch more susceptible to enzymes. Starch gelatinization improves the digestion of dry matter and nitrogen (Skoch et al., 1983); (Wondra et al., 1995).

Pellet Quality

Feeding pellets as compared to mash improves animal growth performance. Most of the research evaluating pelleting effects on swine and poultry would suggest that the mode of action is associated with feed form (Hussar et al., 1962; Jahan et al., 2006). Consequently, if the pellet form deteriorates the beneficial effect of pelleting is diminished. Studies on pellet quality and its effect on animal performance have shown that poor quality pellets lessen the benefit of pelleting (Zatari et al., 1990). Studies on swine and poultry have shown that high quality pellets have positive affects, while poor quality pellets resulted in reduced animal performance (Behnke, 2001). One of the reasons for this is the breakage of pellets when fed to animals. This might lead the animal to select between different particles of the feed resulting in the lack of needed nutrients. Zatari et al. (1990) reported that a group of broilers fed 75% pellets and 25% fines

have higher final body weights and cumulative feed conversions than those fed 25% pellets and 75% fines. In addition to the effect on animal performance, poor quality pellets diminish the physical benefits acquired from pelleting like increasing feed bulk density, reducing dust, and improving flowability especially with fine ground materials. Since pelleting is the most expensive feed manufacturing process, it is important to understand the factors that either add to or take away from pellet quality.

Factors affecting pellet quality

There are several factors that affect pellet quality or pellet durability index (PDI). Reimer (1992) indicated that 60% of pellet quality is influenced by factors unrelated to pelleting process. In other words, only 40% of pellet quality is dictated by the pellet mill. He categorized that 20% of the quality is attributed to the conditioning and 15% to the die specification. He also recognized that the last 5% is due to cooling and drying of the pellets.

1. Formulation

Formulation describes the different kinds of ingredients and their characteristics, like pellet ability, and their content of fiber, fat, and protein (MacBain, 1966). Some of these characteristics may improve or deteriorate pellet quality. For example, fat and protein are two important factors that affect pellet quality. Adding fat to the formula, before pelleting, hinders pellet quality, as fat insulates feed particles inhibiting effective steam penetration into the particles, which, in turn, lessens the starch gelatinization. If fat addition is required, it is recommended not to exceed 2% before pelleting and the remainder after pelleting (MacBain, 1966). Conversely, protein tends to improved pellet quality. One of the reasons identified by MacBain is that the bulk density of these materials is high. In general, dense material is easier to pellet.

Research by Briggs (1999) shows that the maximum oil limit for a diet that contains 20% protein is 5.6%; exceeding this level significantly reduced the PDI. Accordingly, Stark (1994) found that the addition of fat increased throughput and decreased electricity consumption, but the addition of more than 3% fat reduced PDI dramatically. Also, Stevens (1987) found that protein content is positively correlated with high pellet quality while fat content has negative effects.

2. Particle size

It is acknowledged that a small particle size results in good quality pellets. Consequently, the concept is that small particles let the steam penetrate particles more than in the case of large and coarse particles, which leads to the increase of water absorption during conditioning and improves gelatinization. Stark (1994) found improvement in pellet durability when he reduced particle size from 561µ to 222µ. Conversely, Young (1960) did not find significant differences between different particle sizes (coarse, medium, and fine). This was also confirmed by Stevens (1987) who found that there was no effect on PDI between fine and medium particles in both corn and wheat meals. However, reducing particle size may lead to the reduction of throughput of the pellet mill, because with large particle size there is less work for the pellet mill (MacBain, 1966).

3. Pellet mill die

The main purpose of the die is to shape the pellet and provide resistive force to the mash. This is done by forcing the conditioned mash through the die holes with approximately (25,000 psi) pressure applied by the rolls (Behnke, 2007). To produce relatively good quality pellets, residence time of the material inside the die holes need to be increased, and that can be achieved by increasing the length of the die hole for a certain diameter. Thus, as the L/D ratio is increased, the quality of pellets is improved.

4. Conditioning

MacBain (1966) states that, “Steam conditioning is one of the most important factors in pelleting” as it accounts for 20% of the pellet quality. During conditioning, steam penetrates particles and breaks up the starch bonds and improves gelatinization, which positively affects pellet quality. Generally, long conditioning times improve pellet quality because it gives adequate time for the steam to penetrate particles. Skoch et al. (1981) studied the effect of steam conditioning on production rate, pellet durability, and energy consumption. They found that steam conditioning increased flow rate by 250% (65°F) and 275 %(80°F) compared to dry pelleting. They also found that steam conditioning reduced electrical energy consumption by almost 50%. Increased production rate and lower energy consumption have been attributed to the act of steam as a lubricant, as indicated by MacBain (1966), which reduces the friction inside the pellet mill die holes. In the same study, there were also improvements in pellet durability from an average of approximately 75% in dry pelleting to an average of approximately 93% for pellets that were steam conditioned.

Pathogens and microorganisms can also be confined by conditioning because of the high temperature inside the conditioner, which can be affected by retention time and temperature. A study by Himathongkham (1996) has shown that a heat treatment of 93°C for 90 seconds can reduce viable *salmonella* 10000 folds in feed with 15% moisture content. Aflatoxin, which is a naturally occurring toxin that can be produced in feed materials, may also be controlled through the high temperature during conditioning. A study by Rao & Deyoe (1977) on four types of Aflatoxin, found that pelleting in general reduced Aflatoxin by 25%; however, half of that reduction found after conditioning and before pelleting.

Conditioning is also important for cooling the pellets, the evaporation of water cools the pellets while it migrates from the core to the outside surface (Fairfield, 2003). Increasing retention time during conditioning improves pellet quality and reduces the amount of fines after pelleting, which in turn creates good air flow through the cooling and drying trays.

Since conditioning has a major affect on the pelleting process, care must be taken to acquire the required products and achieve an overall efficient process. Special attention is needed for the factors that affect conditioning, which can be summarized as:

- Steam addition is considered one of the important factors in the conditioning process. Maier and Gardecki (1993) indicated that steam addition to the mash depends on the type of ingredients and their initial moisture. MacBain (1966) classified the feed formulas into five categories: “High Grain, Heat Sensitive, High Natural Protein, Complete Dairy, and High Urea-Molasses Feed.” Each one of these categories has its own need for moisture addition. Steam quality is another important term related to improving gelatinization of starch inside the conditioner. Since steam is a source for both heat and moisture, it is essential to have high quality steam, otherwise the large amount of moisture may exceed the ability of the pellet die and plug it (Maier and Gardecki, 1993). Gilpin (2001) stated that there is a correlation between steam quality and initial mash moisture. With low initial mash moisture around 12%, steam with 70% quality might be needed to compensate the lack of the required moisture (Gilpin, 2001). However, high temperature cannot be achieved with wet steam because it has less energy than dry steam, therefore care must be taken if high temperature is needed with materials that have low moisture content. Steam generation is a costly process, therefore understanding steam properties and their effects on the pelleting process is

very important. Steam pressure has been studied by Stevens (1987) and Briggs et al. (1999), who found that there was no significant difference on pellet quality or production rate by using two different steam pressures.

- Retention time of the mash in the conditioner also contributes to how well mash is conditioned. Retention time is simply the time that feed mash spends inside the conditioner, exposed to water and heat applied by the steam. During this period, steam penetrates particles and works on starch gelatinization, which, in turn, is important for pellet quality. In general, long retention times produce better quality pellets than short retention times (Briggs et al., 1999). However, some enzymes are heat susceptible and should not be exposed to high temperature for long period of time. On the other hand, some microorganisms and toxins need to be exposed to high temperature for certain amount of time in order to diminish their activity (Rao & Deyoe, 1977); (Himathongkham, 1996). In order to lengthen the retention time and keep the production rate the same, the amount of materials inside the conditioner need to be increased for a certain time, (McDonald, 2000). This is accomplished by adjusting the paddles on the conditioner shaft. Research by Briggs et al. (1999) showed two different settings for the paddles. The first setting was a 45° forward angle and they estimated the retention time about 5 sec. In the second setting, all of the mixing paddles were set parallel to the mixing shaft, except the first and last set which remained the same as the first setting. They estimated the retention time about 15 sec. However, changing these angles is not an easy method and is time consuming, especially in the industry. With the invention of the variable speed drive, it became

very easy to adjust the speed of the conditioner to achieve the required retention time and keep the same production rate.

Retention time

Retention time has been defined by McDonald (2000), "as the amount of material in the conditioner at any given time divided by the total flow rate through the conditioner." In addition to improving pellet quality, controlling microorganisms, increasing throughput, and lengthening the die working life, McDonald also suggests that measuring retention time provides an indication about the material uniformity inside the conditioner and can facilitate making adjustments for the paddles inside the conditioner or making some improvements to the design of the equipment. Although measuring retention time would be very useful tool to feed manufacturers. There is no direct formula or method that provides a precise measurement for retention time. Most of the studies that dealt with the evaluation of retention times relied on estimates. Gilpin (2001) measured retention time by injecting chromic oxide and salt as a tracer at the inlet of the conditioner. Samples of the discharged material were collected in time intervals, and analyzed by Quantab® Chloride Titrators and color meter. After plotting the concentration with time, Gilpin then considered the peaks of the curves as the average retention time.

Measuring retention time is of special interest in the extrusion field and there are studies about that in plastic and food production (Wolf et al., 1976); (Yeh et al., 1998); (Kumar et al., 2006). Most of these studies referred to the importance of measuring retention time, because reactions such as starch gelatinization, protein denaturation, enzyme activation, and browning are important in food production, which is affected by retention time (Yeh et al., 1998); (Kumar et al., 2006).

Methods for measuring retention time:

1. The "Hold-up capacity" method is based on the holding capacity of the conditioner for a certain time. This follows McDonald's (2000) definition for retention time, "as the amount of material in the conditioner at any given time divided by the total flow rate through the conditioner." This concept has been described as the measurement for the mean residence time by using the physical conditions. Levenspiel (1972) and Wolf et al. (1976), both referred to the formula:

$$\bar{t} = \frac{\text{Volume}}{\text{Flow Rate}}.$$

Measuring the amount of materials in the conditioner for a certain time has been accomplished in previous experiments which were conducted in the Department of Grain Science Feed Processing Center (2006). The results gave reasonable results for the mean retention time. This method included the following steps:

- The conditioner covers should be removed and the conditioner completely cleaned of any previous residues that might affect the flow of material.
- The screw feeder and the conditioner shaft need to be run to reach steady state conditions.
- To determine the exact mass flow rate, the discharged material should be collected for a certain time in a previously scaled container. This step should be repeated to get an average flow rate.
- The screw feeder should be stopped, while the conditioner shaft continues to operate at the same speed. The discharged material is then collected in a previously scaled container until there is no material coming out. The

collected amount of material represents the holding capacity of the conditioner.

- Applying the following formula gives an average retention time for the materials inside the conditioner.

$$\bar{t} = \frac{\text{Amount of Materials inside the Conditioner (Kg)}}{\text{Mass Flow Rate (Kg/hr)}} \times 3600\ {}^{\text{sec}}\!/_{\text{hr}} = \text{Retetion Time (seconds)}$$

A disadvantage of this method is that it is not easily applied in a commercial setting due to the large size conditioners, since it requires collecting large amounts of material and also stopping the production many times.

2. "The Pulse Input" method is based on adding tracer for a short time at the inlet of the conditioner and start collecting samples at time zero at the conditioner discharge. The concentration of the tracer increases for a certain time and then decreases until there is no tracer coming out. These concentrations of the tracers are then plotted against the time spent for this material to come out (Fogler, 1999). The equations required for measuring the average residence time have been derived by Levenspiel (1972) and Fogler (1999). These equations have been used for research in the extrusion field and are as follows:

$$E(t) = \frac{C}{\sum_0^\infty C.\Delta t}$$

$$F(t) = \sum_0^t E(t).\Delta t = \frac{\sum_0^t C.\Delta t}{\sum_0^\infty C.\Delta t}$$

$$\bar{t} = \frac{\sum_0^\infty t.C.\Delta t}{\sum_0^\infty C.\Delta t}$$

Where E(t): residence time distribution function.

F(t): cumulative residence time distribution function.

C: concentration distribution.

t : average sampling time.

$\bar{t}$: Average retention time.

Source: Wolf et al., 1976.

As indicated previously, Gilpin (2001) has used the pulse input method in an experiment concerning the effect of different retention times on pellet quality. However, the retention time in that study was calculated based on the peaks of the curves, which were obtained from plotting the concentrations against sampling time, without using the equations above. Smallman (1996) also indicated that retention time can be considered as the peak point of the curve.

This method is considered an acceptable method to give an approximate mean residence time. However, because the general tracer used is either NaCl (salt) or powder color dye, each having a relatively small particle size, there is concern that these tracers might flow in different patterns compared to the feed material that flows inside the conditioner. Creger (1957) concluded that NaCl can be used as a tracer for mixing performance because it tends to segregate when it is mixed with particles that are larger in size. Pfost et al. (1966) mentioned that salt is difficult to be mixed with other feed ingredients. If this characteristic for salt is true, that means it will not be mixed with the material in the conditioner especially when the conditioning time is short. For the latter reason, flow characteristics for salt and fine dye may differ from those for the rest of the material. In general tracers should have the same physical properties of the flowing materials (Fogler, 1999).

Purpose of the study

The main reason of measuring retention time is to know the time that the conditioned material spends during conditioning, especially when dealing with heat susceptible material, or to control microorganisms and to improve pellet quality and reduce energy consumption. It is also important to understand the flow pattern of material inside the conditioner, which may help in recognizing any defect in the equipment or improving the design of the conditioner. Study on the effects of particle size on the accuracy of measuring retention time is required to investigate if there are differences in using particle size different from the conditioned material.

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CHAPTER 2 - Pre-Research Experiments

Introduction

Average retention time describes the amount of time materials spend inside the conditioner exposed to moisture and high temperatures. It is an important term that is usually related to the pelleting process because of its numerous affects on the process. The affects of retention time on the pelleting process can be summarized as follows:

- Pellet quality
- Microorganisms' elimination
- Enzyme activity
- Power consumption
- Throughput

The enhancement of pellet quality has been attributed to long retention times. This is due to the fact that the steam applied to the materials inside the conditioner has enough time to penetrate feed particles. Steam penetration causes starch gelatinization, which in turn improves pellet quality. Briggs et al. (1999) reported that increasing retention time by 10 seconds improved PDI by an average of 4.5 points.

Most microorganisms are susceptible to high temperature, and they can be eliminated by exposure to high temperatures for certain lengths of time. With the availability of moisture and high temperature inside the conditioner, the conditioning process is considered a cooking pot for feed materials. A study by Himathongkham et al. (1996) indicated that there is a linear relationship between the logarithm of *Salmonella* survivors and the logarithm of exposure time.

They found a reduction of 10,000 fold of *Salmonella* at 93°C for 90 seconds retention time with 15% moisture content.

Long retention times may affect the activity of some enzymes and diminish the benefits of adding these products; therefore, special care must be taken if such products are included in the diet. The moisture content and temperature of the materials inside the conditioner have an influential effect on friction inside the die hole, which in turn affects the recovery of phytase after pelleting (Eeckhout, 2000).

The moisture content of the materials leaving the conditioner has been described as a lubricant (Skoch et al., 1981). Reducing the friction between materials and die holes is an important factor for reducing power consumption and increasing the throughput. This outcome is reached because less work is required by the pellet mill. Retention time and steam quality are the main factors that affect moisture content inside the conditioner. Moreover, special attention is needed for the initial moisture content of the feed materials.

Retention time measurements

Unfortunately, although retention time measurements are very important in the pelleting process, there is no formula or direct method available for such measurements. Studies on the importance of pelleting for animal performance or the factors that effect pellet quality have been done based on estimations of the retention times. Most of these studies described a long or short retention time based on the configuration of the paddles or the speed of the conditioner shaft. Briggs et al. (1999) showed two different settings for the paddles. The first setting was a 45° forward angle, and they estimated the retention time at about 5 seconds. In the second setting, all of the mixing paddles were set parallel to the mixing shaft, except the first and last set which remained the same as the first setting. They estimated this retention time at about 15 seconds.

However, changing these angles is not an easy method and is time consuming, especially in the feed industry. With the adoption of the variable speed drive, it became very easy to control the speed of the conditioner to achieve the required retention time and keep the same production rate.

Gilpin (2001) measured retention time by injecting chromic oxide and salt as tracer at the inlet of the conditioner. Samples of the discharged material were collected at time intervals and analyzed by Quantab® Chloride Titrators and color meter. After plotting the concentration with time, Gilpin then considered the peaks of the curves as the average retention time.

Special care has been taken for retention time measurements in the extrusion process, especially in food production. This is because reactions such as starch gelatinization, protein denaturation, enzyme activation, and browning are important in food production, which are affected by retention time (Yeh et al., 1998); (Kumar et al., 2006).

Methods for measuring retention time

As stated previously, there is no formula or direct method for this measurement. There are basically two methods that are usually used for research and industry purposes, which can be summarized as follow:

1. The-hold up capacity method is based on the mass that the conditioner can hold for a certain time. This follows McDonald's (2000) definition for retention time, "as the amount of material in the conditioner at any given time divided by the total flow rate through the conditioner." This concept has been described as the measurement for the mean residence time by using the physical conditions. Wolf et al. (1976) and Levenspiel (1972) both referred to the formula:

$$\bar{t} = \frac{\text{Volume}}{\text{Flow Rate}}.$$

Measuring the mass of feed materials inside the conditioner for a certain time has been accomplished in previous experiments done in the Department of Grain Science Feed Processing Center (2006). The results gave reasonable results for the mean retention time. This method included the following steps:

- The conditioner covers should be removed and the conditioner neatly cleaned of any previous residues that might affect the flow of material.
- The screw feeder and the conditioner shaft need to be run to reach steady state conditions.
- To determine the exact mass flow rate, the discharged material should be collected for a certain time in a previously scaled container. This step should be repeated to get an average mass flow rate.
- The screw feeder should be stopped, while the conditioner shaft should be kept running at the same speed. The discharged material should be collected in a previously scaled container until there is no material coming out. The collected amount of material represents the holding capacity of the conditioner.
- Applying the formula below gives the average retention time for the material inside the conditioner.

$$\bar{t} = \frac{\text{Amount of Materials inside the Conditioner (Kg)}}{\text{Mass Flow Rate (Kg/hr)}} \times 3600\,{}^{\text{sec}}\!/_{\text{hr}} = \text{Retetion Time (seconds)}$$

A disadvantage of this method that it can not be easily applied in the commercial feed industry especially with large size conditioners, since it requires collecting large amounts of materials and also stopping production many times.

2. The Pulse Input method is based on adding a tracer for a short time at the beginning of the conditioner and collecting timed samples at time zero at the discharge end of the conditioner. The concentration of the tracer increases for a certain time and then decreases until there is no residual tracer. These concentrations should be plotted with the time spent for this material to come out (Fogler, 1999). The equations required for measuring the average residence time have been derived by Levenspiel (1972) and Fogler (1999). These equations have been used for research in the extrusion field and are as follows:

$$E(t) = \frac{C}{\sum_{0}^{\infty} C.\Delta t}$$

$$F(t) = \sum_{0}^{t} E(t).\Delta t = \frac{\sum_{0}^{t} C.\Delta t}{\sum_{0}^{\infty} C.\Delta t}$$

$$\bar{t} = \frac{\sum_{0}^{\infty} t.C.\Delta t}{\sum_{0}^{\infty} C.\Delta t}$$

Where E(t): residence time distribution function.

F(t): cumulative residence time distribution function.

C: concentration distribution.

t : average sampling time.

$\bar{t}$: Average retention time.

Source: Wolf et al., 1976.

As indicated previously, Gilpin (2001) has used the pulse input method in an experiment concerning the effect of different retention times on pellet quality. However, the retention time in that study was calculated based on the peaks of the curves, which were obtained from plotting the concentrations against sampling time, without using the equations above. Smallman (1996) also indicated that retention time can be considered as the peak point of the curve.

This method is considered an acceptable method to give an approximate mean retention time. However, because the general tracer used is either NaCl (salt) or powder color dye, there is doubt because of the differences in particle sizes and densities of the tracers if compared to the feed materials that flow inside the conditioner. Tracers should have the same physical properties of the flowing materials (Fogler, 1999). Creger (1957) concluded that NaCl can be used as a tracer for mixing performance because it tends to segregate when it is mixed from particles that are larger in size. Pfost et al. (1966) mentioned that salt is difficult to mix with other feed ingredients. If this characteristic for salt is true, it will not mix with the material in the conditioner, especially when the conditioning time is short. For the latter reason, flow characteristics for salt and fine dye likely differ from those for the rest of the material.

Purpose of the study

The main reason of measuring retention time is to know the time that the conditioned material spends during conditioning, especially when dealing with heat susceptible ingredients, or to control pathogenic microorganisms and to improve pellet quality and reduce energy consumption. It is also important to understand the flow pattern of the material inside the conditioner, which may help in recognizing any defect in the equipment or in improving the design of the conditioner. Studies on the effects of particle size on the accuracy of measuring retention time are required to determine if there are differences in using particle sizes different

from the conditioned material. Since no study has been conducted on this subject, we conducted small scale studies as a reference for later research.

Experiment I

In order to compare the accuracy of using tracers with different particle sizes in measuring retention times, previously prepared tracers were used to be compared with crystalline salt, which is usually used in such studies.

Materials, Tracer Preparation, and Method

Cracked corn was prepared using the roller mill[1] in the Department of Grain Science Feed Processing Center, equipped with a 15 hp motor for the top and middle pair of rolls and a 10 hp motor for the bottom pair of rolls. The rolls were set to achieve a particle size around 700 μ. These materials were used to prepare two tracers, in addition to crystal salt, which all were used in this experiment. The first tracer was prepared by adding a salt solution (20% salt concentration) to the cracked corn ("wet mix"). The mix was then blended in a Hobart mixer for 10 minutes to ensure homogeneity. The new blend was then spread out on plastic for 48 hours to dry, assisted by fans to reduce the drying time. The second tracer was prepared by adding crystal salt directly to the cracked corn. In order to achieve the 20% salt concentration as in the first tracer, 200 grams of salt were added to 800 grams of cracked corn ("dry mix"). The new mix was blended in a Hobart mixer for 10 minutes for a homogeneous mix.

The conditioner used in this experiment was the Bliss conditioner[2] (approximately 30cm diameter and 122cm length) attached to the CPM[3] (MASTER MODEL, 1000 HD) pellet mill in the Department of Grain Science Feed Processing Center. The speed of the conditioner shaft and

[1] Model K, Roskamp Manufacturing, Cedar Falls, IA

[2] Bliss Industries., Ponca City, OK

[3] California Pellet Mill Co., Crawfordsville, IN

the screw feeder were set to achieve a steady state flow rate of 453.5 Kg/hr. The speed for both motors was the same for the rest of the treatments. When the conditioner reached a steady state, 907 g of the tracer (wet mix 910 µ) was added at the conditioner inlet after the last flight of the screw feeder. At exactly the same moment, discharged materials were collected in 3 second time intervals. The samples were collected in previously labeled containers to ensure collecting the majority of the discharged material. The samples were then transferred to plastic bags to be stored for analyses. The analytical samples were obtained by dividing the samples using a sample divider (HUMBOLDT, model: H-3964); the resulting samples were approximately 50 grams each. Chloride ion analyses were accomplished using the Quantab® titrators[4], and then the salt concentration was plotted on the Y-axis with sampling time on the X-axis. Total sampling time was two minutes for forty samples. The particle size of the materials flowing through the conditioner was 585 µ.

The same procedure was repeated twice for the other two tracers (dry mix tracer at 700 µ and salt tracer at 425 µ). In order to achieve the same salt concentration, the amount of salt added as a tracer in the last run was calculated based on the total amount of materials inside the conditioner.

Results and Discussion

Mean retention time was calculated for the control treatment, using the hold up capacity and applying the formula:

$$\bar{t} = \frac{\text{Amount of Materials inside the Conditioner (Kg)}}{\text{Mass Flow Rate (Kg/hr)}} \times 3600\ {}^{\text{sec}}\!/_{\text{hr}} = \text{Retetion Time (seconds)},$$

which was 61.25 seconds without adding the tracer, 68.4 seconds after adding the tracer in phase

[4] Quantab Chloride Titrators procedure (Quantab®, Hach Co., Loveland, CO)

two, and 62.67 seconds with the crystal salt addition as a tracer in the third phase. Treatments one, two and three are calculated by the formula: $\bar{t} = \frac{\sum_{0}^{\infty} C.t}{\sum_{0}^{\infty} C}$. where, C is the tracer concentration, $\bar{t}$ is the average retention time, and t is the average sampling time. Average retention times for treatment one and two were 69.26 seconds with the wet mix as a tracer, and 71.63 seconds when the dry mix was used as a tracer. However during the use of crystal salt in treatments three retention time was 73.93 seconds.

It is obvious that measuring retention time with relatively large particles was closer to the hold up capacity method. Small particles tend to spend more time inside the conditioner than large particles. This outcome is because the small size of the particle reduces the probability of being hit by the paddle and moved from one point to another.

By looking to the plots between salt concentration and sampling times (Figures 2-1, 2-2, and 2-3), there is a uniform distribution for the wet mix tracer, while the distribution for the other two tracers was inconsistent.

Experiment II

Crystal salt and colored tracers are used widely for retention time measurements for both research and industry purposes; however, there are no published studies about the affect of particle size of these tracers on the accuracy of these measurements. The purpose of this experiment was to study the effects of using tracers with different particle size from the main materials flowing in the conditioner.

Materials, Tracer Preparation, and Method

Since soybean meal (SBM) and corn are included in most formulations for animal feed, we selected a formula of 75% corn and 25% SBM for our experiment. The grains were ground to a particle size of approximately 700 µ, which is the typical size of most materials prepared to produce pelleted feed. After testing particle size for the material by using "method of Determining and Expressing Fineness of Feed Materials by Sieving" (ASAE S319.3), we prepared our tracer for this experiment, which was red salty mash (20% salt and 2.5% red dye). The salt and dye[5] were dissolved in distilled water (400 ml water /100 gram salt) at room temperature, then the solution was added to the required amount of mash. A food mixer (Hobart) was used for 10 minutes to mix the mash with the solution in order to allow the color penetrate the particles and produce the desired color. In order to get the required concentration, the test batches were 775 gram of material and 800 ml (200 gram salt and 25 gram dye) of the red dye/salt solution. The batches were then spread on plastic to dry using a fan for the first 16 hours. The agglomerated materials (Figure 2-4) were rubbed by hand to break the adhesiveness between particles and avoid further reducing particle size. All materials that did not pass through a US sieve number 8 were rubbed again by hand to get a size approximately the same as the original. A thin layer of the red mash (Figure 2-5) was spread on plastics for 48 hours to let them equilibrate and have the same moisture content as the original material. The test mash was again assayed for particle size, which was around 770 µ. Also, a Quantab® (low rang & high range) test was done for the test mash to distinguish the final salt concentration, which was targeted at 20%. Because of the high level of salt in the test mash, a dilution factor of 99:1 was used rather than the normal one (9:1) as applied in the recommended Quantab® procedure.

[5] Powder Red Dye No. 40, FD & C (Warner Jenkinson, St. Louis, MO)

The experiment consisted of three phases run under atmospheric conditions and without any addition of steam (dry basis). This limited the affect on material transfer to only the rotation of the conditioner paddles and the material flow rate. Before starting the first phase, the conditioner cover was opened and the conditioner was cleaned from any old residues as shown in Figure 2-6. The three phases were then conducted as described below:

Phase one:

In order to measure the amount of material inside the conditioner for a certain production rate and type of material, the hold up capacity method was used in phase one. Screw feeder and conditioner speeds were recorded to be used in later stages. This phase consisted of the following steps:

1. Operating the pellet mill screw feeder to accomplish the production rate of 453.5 Kg/hr, the screw feeder speed was recorded, which was 3.4 RPM, 5.3 Hz.
2. The conditioner shaft was operated at 75.3 RPM, 15.1 Hz to accomplish an approximate retention time of 60 seconds based on previous studies.
3. The material at the end of the conditioner was collected to be reused in later phases.
4. At a steady state condition, the pellet mill screw feeder was stopped while the conditioner shaft was kept running. At the same time, the discharged material from the conditioner was collected in previously scaled container until there was no more material coming out. This material represents the hold up capacity for the conditioner, which was 7.4 Kg.
5. The conditioner shaft was then stopped, the conditioner cover opened, and all material inside was vacuumed and weighed. This material represents the residues in the conditioner, which was 5.08 Kg.

6. The material collected in steps 4 & 5 represent an approximation of the total amount of material inside the conditioner for certain conditions. The total capacity (12.48 Kg) was used in later phases to determine the concentration of the tracer to be used.
7. The following formula was used to calculate an approximate retention time:

$$\frac{\text{Material collected in step 4 (Kg)}}{\text{Production rate (Kg/hr)}} \times 3600\ (\text{sec/hr}) = \text{Time (Sec)}$$, which was 58.86 seconds.

Phase Two:

The objective of this phase was to measure the retention time through the use of the test mash as a tracer, which had approximately the same particle size as the original material. Similar particle sizes ensure even distribution of the test mash within the original mash. Testing the salt concentration (Quantab® titrators) gave us an idea about the material flow patterns inside the conditioner and also enabled us to measure the retention time. A description for the second phase is as follows:

1. Both the pellet mill screw feeder and the conditioner shaft were run at the same speeds described in phase one.
2. At a steady state condition, 907 g of the test mash was introduced to the conditioner through an opening at the top of the conditioner, which is located at the same point that the feeder feeds the material to the conditioner. At the same time of adding the test mash, samples were taken in three second time intervals at the discharge end of the conditioner (pellet mill die side). The samples were collected in sequentially numbered containers. The reason for using these containers was to ensure collecting all the materials coming out.

3. The samples were then transferred to plastic bags and mixed to ensure an even distribution for the test mash with the original material.
4. In order to measure the salt concentration, a Quantab® test was used for the collected samples from step 3.
5. A plot of the sampling time on the X-axis and the salt concentration on the Y-axis is shown in Figure 2-7.
6. The formula: $\bar{t} = \frac{\sum_{0}^{\infty} C.t}{\sum_{0}^{\infty} C}$ was used to determine the residence time distribution.

Phase Three:

In previous research for measuring retention time, a powdered dye mixed with salt is usually used. In order to make a comparison between previous studies and the current study, a mix of powdered red dye and salt was added to the conditioner following the same procedure in phase two. The added amount supplies the same concentration of salt and dye that was used in phase two. After collecting the samples, the same procedure that was used in phase two was applied again for phase three.

Results and Discussion

Retention time was calculated for the control treatment as described in previous sections. It was 58.86 seconds for the hold up capacity method (phase one) without adding the tracer, and 66.06 seconds after the addition of 907 g of tracer. Phase two measurements using the pulse input method indicate that retention time for the wet mix is approximately 72.52 seconds with a difference of 6 seconds from phase one. Phase three, using crystal salt as a tracer, results for retention time came as 80.76 seconds, with a difference of 20 seconds. A plot that is shown in

Figure 2-7 between the sampling time on the X-axis and salt concentration on the Y-axis for phase two show a uniform flow. The highest concentration was at 66 seconds, which came to agree with Gilpin (2001) who considered the highest concentration point as the retention time. The plot that represents phase three (Figure 2-8), shows that the differences in salt concentration were not as uniform as in phase two. Salt concentration in the last sample was lower in phase two than that in phase three, which indicates that crystal salt residuals were higher than those for the test mash.

Summery and Conclusion

Based on these results, it appears that using tracers with similar particle sizes resulted in more accurate measurements for retention time than using different particle sizes. One reason for this outcome is that at using materials with different particle sizes tend to segregate and have different flow characteristics. In addition to that, small particles have less surface area, which reduce the probability of being carried or hit by the paddles inside the conditioner. The high residuals for crystal salt in the last sample is because salt has a higher bulk density than the rest of material, which makes it settle in the conditioner and not flow like the rest of material.

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Figures and Tables

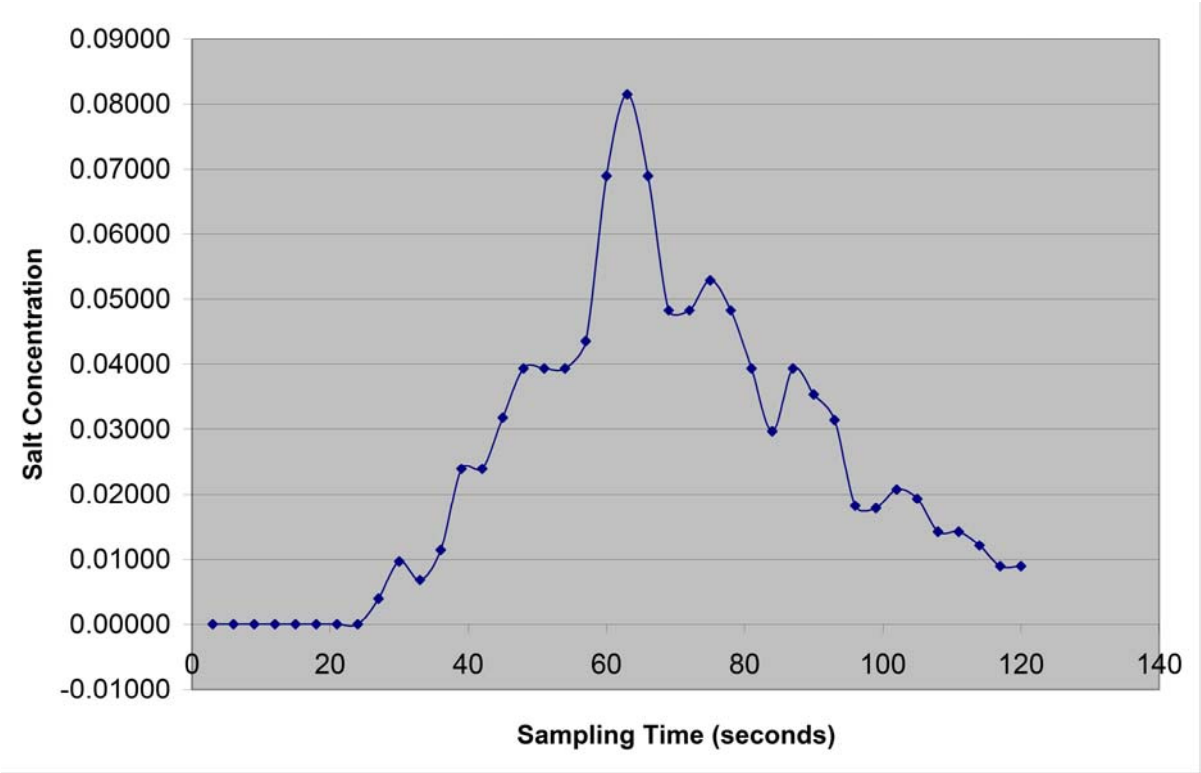

Figure 2-1: Salt analyses for cracked corn with salt solution (wet mix) as a tracer.

Sample	Time In	Time out	Av time	C	tC
1	0	3	1.5	0	0.0000
2	3	6	4.5	0	0.0000
3	6	9	7.5	0	0.0000
4	9	12	10.5	0	0.0000
5	12	15	13.5	0	0.0000
6	15	18	16.5	0	0.0000
7	18	21	19.5	0	0.0000
8	21	24	22.5	0	0.0000
9	24	27	25.5	0.11	2.8050
10	27	30	28.5	0.27	7.6950
11	30	33	31.5	0.19	5.9850
12	33	36	34.5	0.32	11.0400
13	36	39	37.5	0.67	25.1250
14	39	42	40.5	0.67	27.1350
15	42	45	43.5	0.89	38.7150
16	45	48	46.5	1.10	51.1500
17	48	51	49.5	1.10	54.4500
18	51	54	52.5	1.10	57.7500
19	54	57	55.5	1.22	67.7100
20	57	60	58.5	1.93	112.9050
21	60	63	61.5	2.28	140.2200
22	63	66	64.5	1.93	124.4850
23	66	69	67.5	1.35	91.1250
24	69	72	70.5	1.35	95.1750
25	72	75	73.5	1.48	108.7800
26	75	78	76.5	1.35	103.2750
27	78	81	79.5	1.10	87.4500
28	81	84	82.5	0.83	68.4750
29	84	87	85.5	1.10	94.0500
30	87	90	88.5	0.99	87.6150
31	90	93	91.5	0.88	80.5200
32	93	96	94.5	0.51	48.1950
33	96	99	97.5	0.50	48.7500
34	99	102	100.5	0.58	58.2900
35	102	105	103.5	0.54	55.8900
36	105	108	106.5	0.40	42.6000
37	108	111	109.5	0.40	43.8000
38	111	114	112.5	0.34	38.2500
39	114	117	115.5	0.25	28.8750
40	117	120	118.5	0.25	29.6250
			Total	27.9800	1937.9100
				RTD	69.2605

Table 2-1: Salt Concentration distribution for cracked corn with salt solution as a tracer.

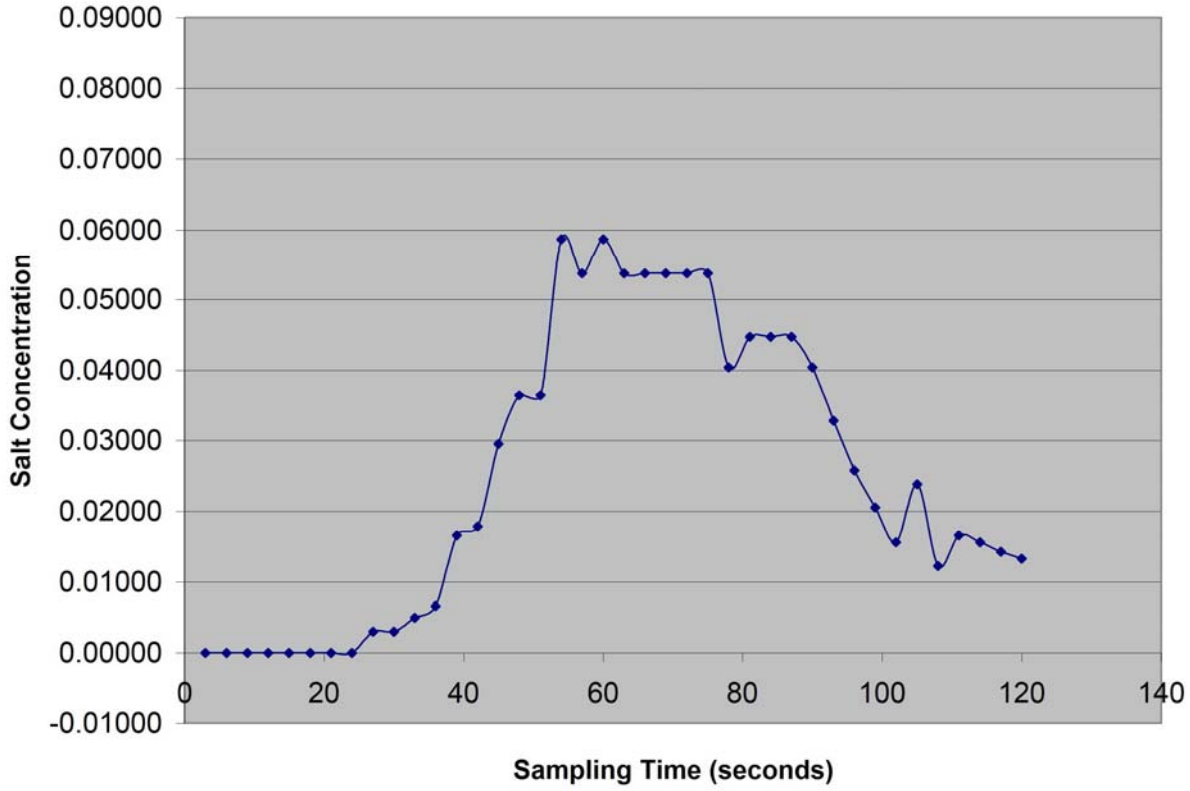

Figure 2-2: Salt analyses for cracked corn with dry salt (dry mix) as a tracer.

Sample	Time In	Time out	Av time	C	tC
1	0	3	1.5	0	0.0000
2	3	6	4.5	0	0.0000
3	6	9	7.5	0	0.0000
4	9	12	10.5	0	0.0000
5	12	15	13.5	0	0.0000
6	15	18	16.5	0	0.0000
7	18	21	19.5	0	0.0000
8	21	24	22.5	0	0.0000
9	24	27	25.5	0.09	2.2950
10	27	30	28.5	0.09	2.5650
11	30	33	31.5	0.15	4.7250
12	33	36	34.5	0.20	6.9000
13	36	39	37.5	0.50	18.7500
14	39	42	40.5	0.54	21.8700
15	42	45	43.5	0.89	38.7150
16	45	48	46.5	1.10	51.1500
17	48	51	49.5	1.10	54.4500
18	51	54	52.5	1.77	92.9250
19	54	57	55.5	1.62	89.9100
20	57	60	58.5	1.77	103.5450
21	60	63	61.5	1.62	99.6300
22	63	66	64.5	1.62	104.4900
23	66	69	67.5	1.62	109.3500
24	69	72	70.5	1.62	114.2100
25	72	75	73.5	1.62	119.0700
26	75	78	76.5	1.22	93.3300
27	78	81	79.5	1.35	107.3250
28	81	84	82.5	1.35	111.3750
29	84	87	85.5	1.35	115.4250
30	87	90	88.5	1.22	107.9700
31	90	93	91.5	0.99	90.5850
32	93	96	94.5	0.78	73.7100
33	96	99	97.5	0.62	60.4500
34	99	102	100.5	0.47	47.2350
35	102	105	103.5	0.72	74.5200
36	105	108	106.5	0.37	39.4050
37	108	111	109.5	0.50	54.7500
38	111	114	112.5	0.47	52.8750
39	114	117	115.5	0.43	49.6650
40	117	120	118.5	0.40	47.4000
			Total	30.1600	2160.5700
				RTD	71.6369

Table 2-2: Salt concentration distribution for cracked corn with dry salt as a tracer.

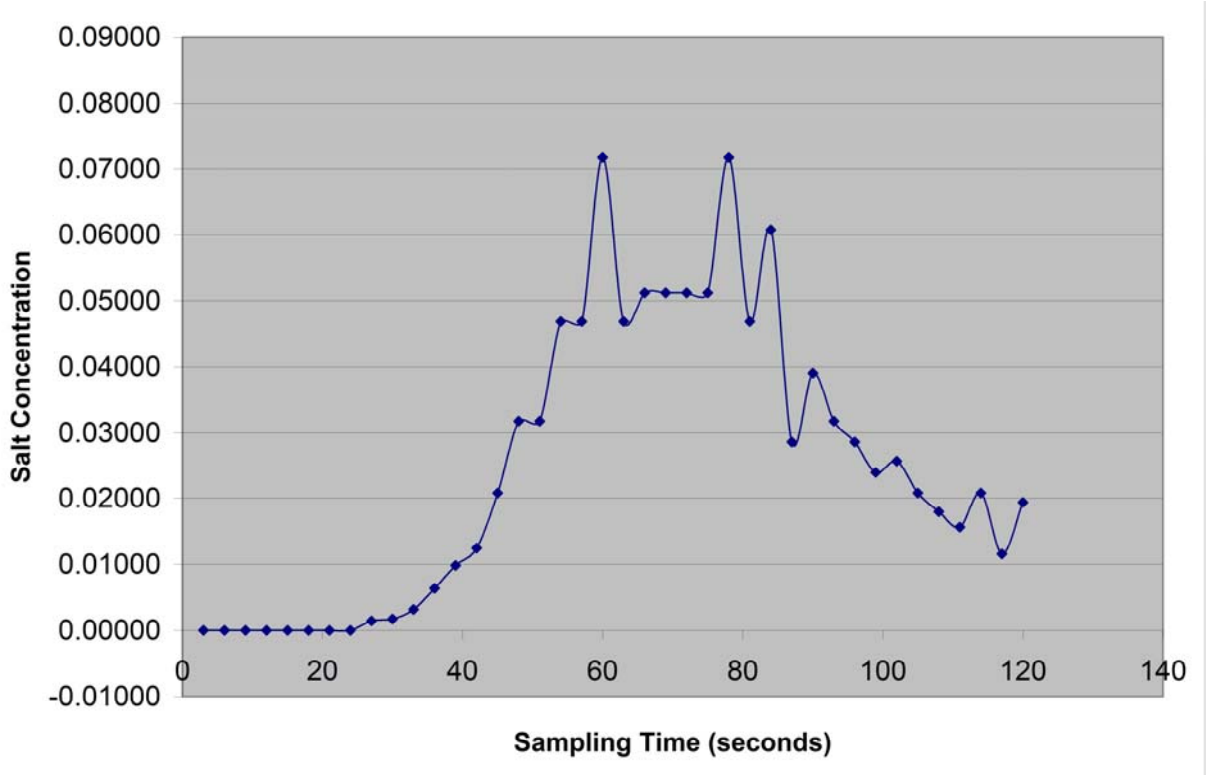

Figure 2-3: Salt analyses for crystal salt as a tracer.

Sample	Time In	Time out	Av time	C	tC
1	0	3	1.5	0	0.0000
2	3	6	4.5	0	0.0000
3	6	9	7.5	0	0.0000
4	9	12	10.5	0	0.0000
5	12	15	13.5	0	0.0000
6	15	18	16.5	0	0.0000
7	18	21	19.5	0	0.0000
8	21	24	22.5	0	0.0000
9	24	27	25.5	0.05	1.2750
10	27	30	28.5	0.06	1.7100
11	30	33	31.5	0.11	3.4650
12	33	36	34.5	0.22	7.5900
13	36	39	37.5	0.34	12.7500
14	39	42	40.5	0.43	17.4150
15	42	45	43.5	0.72	31.3200
16	45	48	46.5	1.10	51.1500
17	48	51	49.5	1.10	54.4500
18	51	54	52.5	1.62	85.0500
19	54	57	55.5	1.62	89.9100
20	57	60	58.5	2.48	145.0800
21	60	63	61.5	1.62	99.6300
22	63	66	64.5	1.77	114.1650
23	66	69	67.5	1.77	119.4750
24	69	72	70.5	1.77	124.7850
25	72	75	73.5	1.77	130.0950
26	75	78	76.5	2.48	189.7200
27	78	81	79.5	1.62	128.7900
28	81	84	82.5	2.10	173.2500
29	84	87	85.5	0.99	84.6450
30	87	90	88.5	1.35	119.4750
31	90	93	91.5	1.10	100.6500
32	93	96	94.5	0.99	93.5550
33	96	99	97.5	0.83	80.9250
34	99	102	100.5	0.89	89.4450
35	102	105	103.5	0.72	74.5200
36	105	108	106.5	0.62	66.0300
37	108	111	109.5	0.54	59.1300
38	111	114	112.5	0.72	81.0000
39	114	117	115.5	0.40	46.2000
40	117	120	118.5	0.67	79.3950
			Total	34.5700	2556.0450
				RTD	73.9382

Table 2-3: Salt concentration distribution for crystal salt as a tracer.

Figure 2-4: The agglomerated materials (dry).

Figure 2-5: Thin layers of material spread to equilibrate.

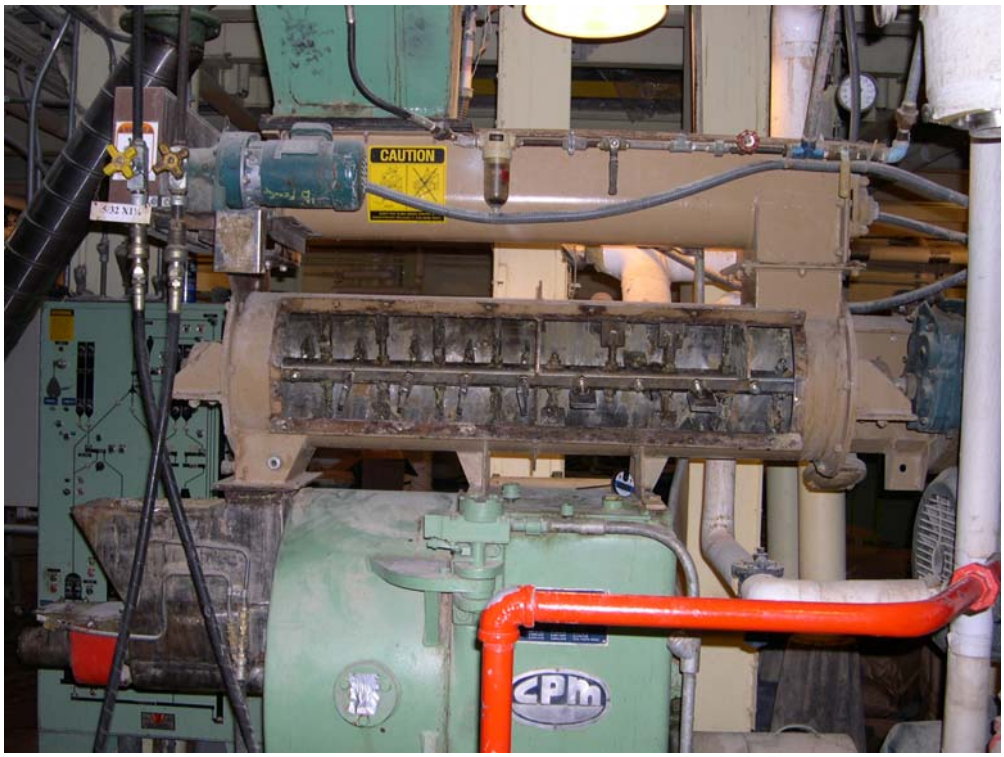

Figure 2-6: Covers opened and conditioner cleaned from old residue.

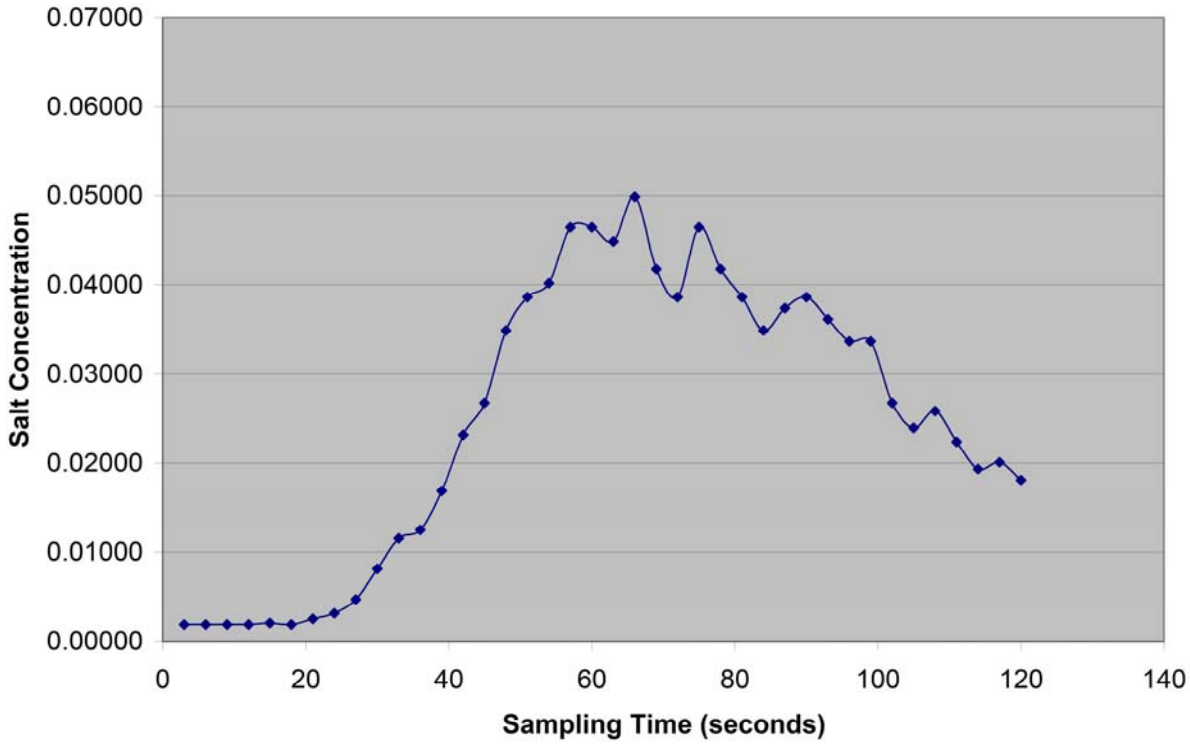

Figure 2-7: Salt analyses for test mash as a tracer.

Sample	Time In	Time out	Av time	C	Tc
1	0	3	1.5	0.0600	0.0900
2	3	6	4.5	0.0600	0.2700
3	6	9	7.5	0.0600	0.4500
4	9	12	10.5	0.0600	0.6300
5	12	15	13.5	0.0650	0.8775
6	15	18	16.5	0.0600	0.9900
7	18	21	19.5	0.0800	1.5600
8	21	24	22.5	0.1000	2.2500
9	24	27	25.5	0.1500	3.8250
10	27	30	28.5	0.2600	7.4100
11	30	33	31.5	0.3700	11.6550
12	33	36	34.5	0.4000	13.8000
13	36	39	37.5	0.5400	20.2500
14	39	42	40.5	0.7450	30.1725
15	42	45	43.5	0.8600	37.4100
16	45	48	46.5	1.1200	52.0800
17	48	51	49.5	1.2400	61.3800
18	51	54	52.5	1.2900	67.7250
19	54	57	55.5	1.4900	82.6950
20	57	60	58.5	1.4900	87.1650
21	60	63	61.5	1.4400	88.5600
22	63	66	64.5	1.6000	103.2000
23	66	69	67.5	1.3400	90.4500
24	69	72	70.5	1.2400	87.4200
25	72	75	73.5	1.4900	109.5150
26	75	78	76.5	1.3400	102.5100
27	78	81	79.5	1.2400	98.5800
28	81	84	82.5	1.1200	92.4000
29	84	87	85.5	1.2000	102.6000
30	87	90	88.5	1.2400	109.7400
31	90	93	91.5	1.1600	106.1400
32	93	96	94.5	1.0800	102.0600
33	96	99	97.5	1.0800	105.3000
34	99	102	100.5	0.8600	86.4300
35	102	105	103.5	0.7700	79.6950
36	105	108	106.5	0.8300	88.3950
37	108	111	109.5	0.7200	78.8400
38	111	114	112.5	0.6200	69.7500
39	114	117	115.5	0.6450	74.4975
40	117	120	118.5	0.5800	68.7300
				32.0950	2327.4975
				RTD	72.5190

Table 2-4: Salt concentration distribution for test mash as a tracer.

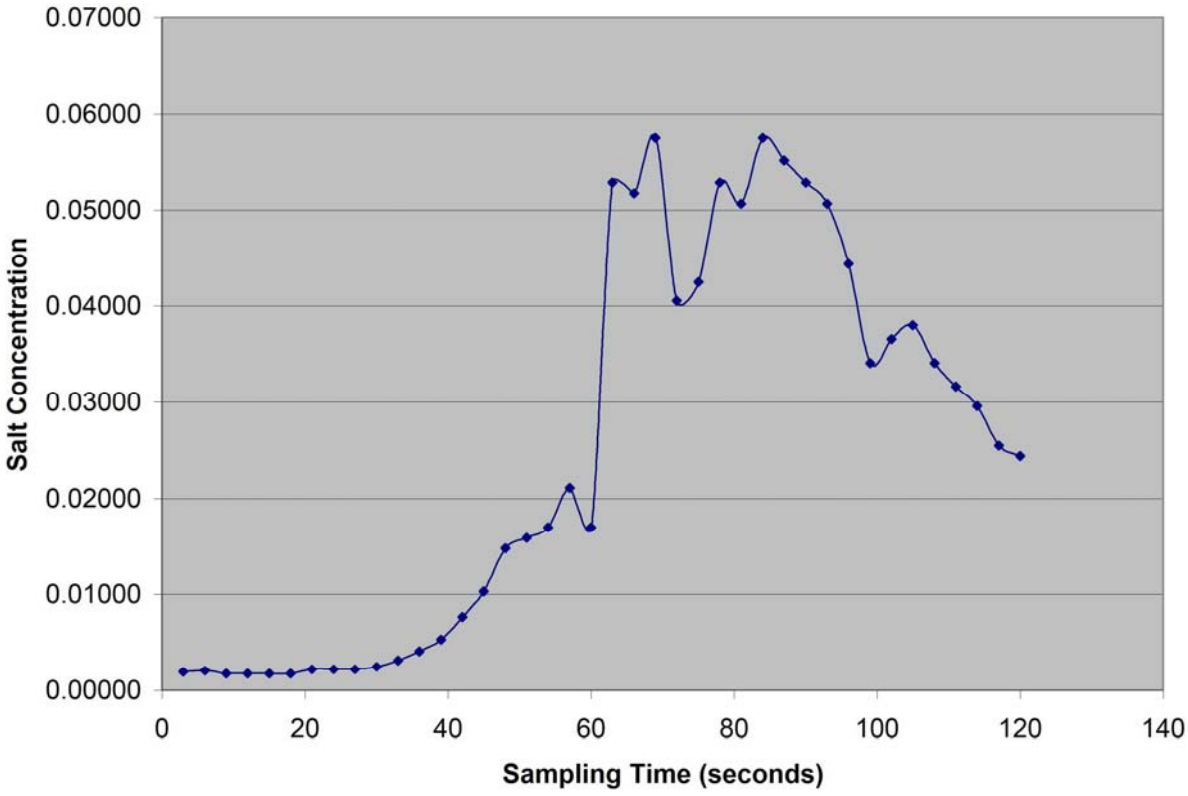

Figure 2-8: Salt analyses for crystal salt as a tracer.

Sample	Time In	Time out	Av time	C	Tc
1	0	3	1.5	0.0700	0.1050
2	3	6	4.5	0.0750	0.3375
3	6	9	7.5	0.0650	0.4875
4	9	12	10.5	0.0650	0.6825
5	12	15	13.5	0.0650	0.8775
6	15	18	16.5	0.0650	1.0725
7	18	21	19.5	0.0800	1.5600
8	21	24	22.5	0.0800	1.8000
9	24	27	25.5	0.0800	2.0400
10	27	30	28.5	0.0900	2.5650
11	30	33	31.5	0.1150	3.6225
12	33	36	34.5	0.1500	5.1750
13	36	39	37.5	0.1950	7.3125
14	39	42	40.5	0.2800	11.3400
15	42	45	43.5	0.3775	16.4213
16	45	48	46.5	0.5400	25.1100
17	48	51	49.5	0.5800	28.7100
18	51	54	52.5	0.6200	32.5500
19	54	57	55.5	0.7700	42.7350
20	57	60	58.5	0.6200	36.2700
21	60	63	61.5	1.9300	118.6950
22	63	66	64.5	1.8900	121.9050
23	66	69	67.5	2.1000	141.7500
24	69	72	70.5	1.4800	104.3400
25	72	75	73.5	1.5500	113.9250
26	75	78	76.5	1.9300	147.6450
27	78	81	79.5	1.8500	147.0750
28	81	84	82.5	2.1000	173.2500
29	84	87	85.5	2.0150	172.2825
30	87	90	88.5	1.9300	170.8050
31	90	93	91.5	1.8500	169.2750
32	93	96	94.5	1.6200	153.0900
33	96	99	97.5	1.2450	121.3875
34	99	102	100.5	1.3350	134.1675
35	102	105	103.5	1.3875	143.6063
36	105	108	106.5	1.2450	132.5925
37	108	111	109.5	1.1550	126.4725
38	111	114	112.5	1.0800	121.5000
39	114	117	115.5	0.9300	107.4150
40	117	120	118.5	0.8900	105.4650
				36.4950	2947.4175
				RTD	80.7622

Table 2-5: Salt concentration distribution for crystal salt as a tracer.

CHAPTER 3 - The effects of using similar and small particle sizes tracers on measuring retention time

Introduction

The cost of producing pelleted feed is likely to increase in the next few years, similar to other industries dependent on petrol products, because of the dramatic increase in oil prices. In addition to the production cost, transporting feed material is another area, which will be affected by these prices. As one of the physical properties, the relatively high bulk density of pellets plays an important role in decreasing the cost of transportation for animal feed. Since the pelleting process is considered the most expensive process in feed manufacturing, special care must be taken for pellet quality to accomplish the benefits of this process. Behnke (1994) states that the factors that affect pellet quality are:

- Formulation
- Particle size
- Conditioning
- Mash moisture
- Retention time and conditioner design
- Steam properties.

Diet formula, particle size, mash moisture, and steam properties all are identified before the pelleting process starts. This limits the pelleting factors to the conditioning process, which is affected by retention time and conditioner design. Conditioner design includes dimensions, number of paddles, angle of paddles, and the steam inlets (Gilpin, 2001). If there is any variance

in these variables, material retention time will be different. Briggs et al., (1999) reported that changing the angle of paddles resulted in a retention time difference of about ten seconds and pellet quality was improved by 4.5 points (PDI) in favor of the longer retention time.

In addition to improving pellet quality, retention time is an important factor in eliminating microorganisms (Himathongkham et al., 1996). Care must be taken if enzymes are included in the diet formula because exposure to high temperatures for long periods of time may reduce the activity of some enzymes (Eeckhout, 2000). Although it is an important factor in the pelleting process, unfortunately there is no formula that can be applied directly to calculate retention time.

By adding a colored dye or salt at the inlet throat of the conditioner, researchers have estimated retention time inside the conditioner. Samples are collected in time intervals at the die end. The highest concentration of the tracer is usually considered an average retention time.

Retention time measurements receive special care in the extrusion industry, because reactions such as protein denaturation, starch gelatinization, and browning are very important in the food industry (Kumar et al., 2006). These measurements are usually obtained using the pulse input method, which consists of introducing a colored dye at the feed throat and collecting samples in time intervals at the end of the conditioner or the extruder barrel. Samples are then analyzed for color concentration. Cumulative quantity of the tracer is then considered to calculate retention time (Kumar et al., 2006).

Purpose of the study

Tracers should have the same physical properties of the flowing materials. (Fogler, 1999) Flow and mixing characteristics of materials depend on the physical properties of these materials. Particle size is considered one of these properties (Pfost et al., 1966). Materials with

different particle sizes have different flow patterns and tend to segregate, particularly if the differences are large. Measuring retention time using tracers with small particle sizes might not represent the actual flow of materials inside the conditioner. Also, these tracers might spend different periods of time from those that the original materials spend. In two experiments done on this subject, there were differences in retention time measurements, more than 20% between small and large particle sizes. This study was conducted by combining two tracers in the experiment for three replications to achieve more information on this subject

Materials and Method

Materials used in this experiment were 75% ground corn and 25% soybean meal (SBM). Corn was ground with a 30 Hp hammermill (Model P-240)[1]. The final particle size of materials (corn and SBM) was approximately 750µ. The experiment was conducted using a Bliss conditioner[2] (approximately 30cm diameter and 122cm length)without steam addition. Speed of the conditioner and screw feeder before the conditioner were 75 RPM and 4.1 RPM, respectively for all replications. The mass flow rate of materials was 453.5 Kg/hr, and was calculated by collecting materials for 30 seconds in a previously scaled container.

A tracer was prepared by mixing the materials with previously prepared salt and red dye solution. The concentration of the tracer was 20% salt and 2.5% red dye[3]. The mixing procedure (for the tracer) was done using a Hobart mixer for ten minutes to achieve a homogenous mix of the tracer that had similar particle size and density to the main materials.

The experiment basically consisted of three phases; the first phase was to measure retention time using the hold up capacity method as a control treatment. This method is based on

[1] Jacobson Machine Works, Minneapolis, MN

[2] Bliss Industries., Ponca City, OK

[3] Powder Red Dye No. 40, FD & C (Warner Jenkinson, St. Louis, MO)

measuring the amount of material coming out from the conditioner for certain density and speeds, and then by using the formula:

$$\bar{t} = \frac{\text{Amount of Materials inside the Conditioner (Kg)}}{\text{Mass Flow Rate (Kg/hr)}} \times 3600\ {}^{\text{sec}}\!/_{\text{hr}} = \text{Retetion Time (seconds)}.$$

Phase two was done using the pulse input method, which is usually used in such measurements. At a steady state condition, 907g of the prepared tracer was introduced into the conditioner at the last flight of the screw feeder. This location ensures a tracer flow rate similar to the main materials and prevents any dead spots that might hold the tracer if introduced directly to the conditioner. At the same moment and with 3 second time intervals, samples were collected from the other side of the conditioner (pellet mill die side) in previously numbered containers. These containers were used to ensure that all the materials coming out were sampled. These samples were then transferred to plastic bags, which had been divided into analytical samples for later analyses. Quantab® Titrators[4] were used for chloride ion analyses. Color analyses were done using a spectrophotometer (SHIMADZU, UV-1601PC). One gram of material was dissolved in ten milliliters of distilled water, then filtered using a syringe filter (CORNING, Steril Syring Filter, 0.20μm) to ensure the clarity of the solutions. Solutions were then analyzed with the spectrophotometer using a wave length 450 nm. Average retention time was calculated using the formula: $\bar{t} = \frac{\sum_{0}^{\infty} C.t}{\sum_{0}^{\infty} C}$.

Phase three was basically repeating phase two with the use of crystal salt and powder red dye as tracer after mixing them together without using the main material as a carrier. The amounts of salt and dye were 180 g and 22 g, respectively, to ensure similar concentrations as in

[4] Quantab Chloride Titrators Procedure (Quantab®, Hach Co., Loveland, CO)

phase two. Analyses and retention time measurements were also done similarly to that in phase two.

Results and discussion

Statistical analyses for the experiment indicated that there was a significant difference between the standard control (60.3 seconds) and both of the treatments, P value was <0.05. Since the control treatment was calculated based on the holding capacity of the conditioner, adjusted control should be calculated for statistical analyses purposes because the amount of material flowing through the conditioner is different with adding different amounts of tracers. Based on the previous statement, two adjusted retention times were calculated based on the amount of tracers added to the conditioner. The adjusted retention time with the addition of 907g of the test mash was 67.5 seconds, while the addition of salt and dye directly (202 g) resulted in 61.92 seconds retention time. Similar results were obtained when comparing the two treatments with their adjusted retention times. There was a significant difference for both treatments and P value was <0.05. Average retention time using the test mash as a tracer was 76.84 seconds (NaCl) and 75.42 seconds (red dye). There was no significant difference and P value was >0.05. Contrary, there was significant difference between NaCl and red dye (P<0.05) when used directly without using a carrier similar to the test mash. This result indicates that tracers with different particle sizes spent a different periods inside the conditioner. In another comparison of the differences between treatments and their related controls, there was significant difference (P<0.05). However, the P-value for the red dye comparison (0.0126) was higher than that of salt (0.0026), which adds density as another effective factor that affects retention time measurement.

It is also clear that measuring retention times using the test mash was closer to the adjusted retention time, with approximately 7 seconds difference. The differences were about 15

seconds greater when salt and red dye were used directly without carrier, which is about 25% higher than the adjusted retention time (61.92 seconds). These results suggest that materials with different particle sizes tend to segregate or may have different flow characteristics and do not spend the same amount of time inside the conditioner.

Plots, between sampling time on the X-axis and tracer concentrations on the Y-axis, have shown that color concentration in the final sample of powder dye was less than that for the test mash. This indicates that the light dye particles spent less time and there were fewer residues because light material is easier to be carried by the material flowing inside the conditioner. On the other hand salt residues for the test mash were fewer than those of salt as a tracer, which confirm the density as an influential factor that may affect these measurements.

Summary and Conclusion

Based on these results, it is clear that both particle size and density have effects on measuring retention time. Since retention time and conditioning have an important role in the production of good quality pellets and safe feed, special care should be taken for tracers' physical properties.

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Figures and Tables

Sample	Time In	Time out	Av time	C	tC
1	0	3	1.5	0.00	0.0000
2	3	6	4.5	0.00	0.0000
3	6	9	7.5	0.00	0.0000
4	9	12	10.5	0.00	0.0000
5	12	15	13.5	0.00	0.0000
6	15	18	16.5	0.00	0.0000
7	18	21	19.5	0.25	4.8100
8	21	24	22.5	0.28	6.3750
9	24	27	25.5	0.37	9.4350
10	27	30	28.5	0.45	12.9200
11	30	33	31.5	0.50	15.7500
12	33	36	34.5	0.60	20.8150
13	36	39	37.5	0.87	32.7500
14	39	42	40.5	0.86	34.8300
15	42	45	43.5	1.33	58.0000
16	45	48	46.5	1.79	83.0800
17	48	51	49.5	1.79	88.6050
18	51	54	52.5	1.78	93.6250
19	54	57	55.5	2.30	127.8350
20	57	60	58.5	2.05	120.1200
21	60	63	61.5	1.94	119.1050
22	63	66	64.5	1.79	115.4550
23	66	69	67.5	1.87	126.0000
24	69	72	70.5	1.98	139.5900
25	72	75	73.5	2.02	148.4700
26	75	78	76.5	2.03	155.5500
27	78	81	79.5	1.94	154.4950
28	81	84	82.5	1.84	151.8000
29	84	87	85.5	1.79	152.7600
30	87	90	88.5	1.85	164.0200
31	90	93	91.5	1.67	152.8050
32	93	96	94.5	1.76	166.3200
33	96	99	97.5	1.62	157.9500
34	99	102	100.5	1.66	166.8300
35	102	105	103.5	1.57	162.4950
36	105	108	106.5	1.36	145.1950
37	108	111	109.5	1.35	147.4600
38	111	114	112.5	1.24	139.1250
39	114	117	115.5	1.19	137.0600
40	117	120	118.5	1.08	127.9800
			Total	48.7833	3639.4150
				RTD	74.6037

Table 3-1: Average color distribution for powder dye.

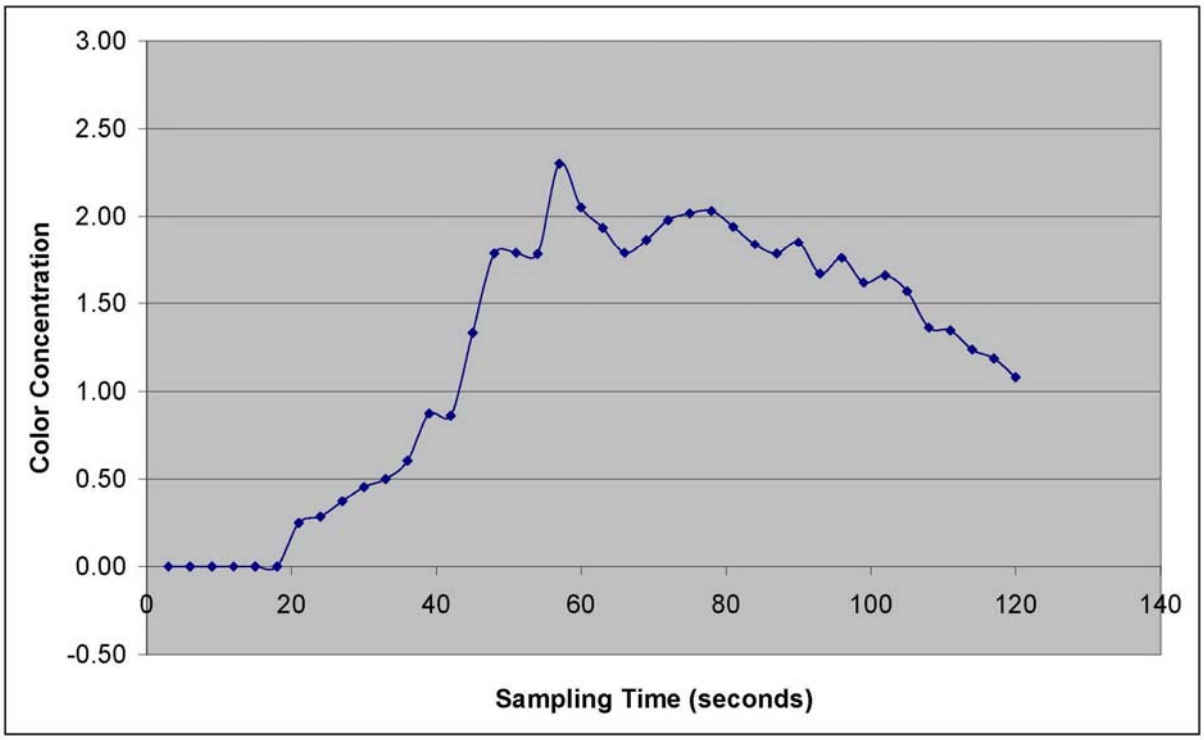

Figure 3-1: Color analyses for powder dye

Sample	Time In	Time out	Av time	C	tC
1	0	3	1.5	0.00	0.0000
2	3	6	4.5	0.00	0.0000
3	6	9	7.5	0.00	0.0000
4	9	12	10.5	0.00	0.0000
5	12	15	13.5	0.00	0.0000
6	15	18	16.5	0.00	0.0000
7	18	21	19.5	0.17	3.3150
8	21	24	22.5	0.46	10.2750
9	24	27	25.5	0.45	11.4750
10	27	30	28.5	0.58	16.4350
11	30	33	31.5	0.54	16.9050
12	33	36	34.5	0.72	24.8400
13	36	39	37.5	0.93	34.8750
14	39	42	40.5	1.31	52.9200
15	42	45	43.5	1.30	56.5500
16	45	48	46.5	1.92	89.2800
17	48	51	49.5	2.13	105.2700
18	51	54	52.5	1.80	94.5000
19	54	57	55.5	2.35	130.4250
20	57	60	58.5	2.36	137.8650
21	60	63	61.5	2.51	154.5700
22	63	66	64.5	2.49	160.3900
23	66	69	67.5	2.39	161.5500
24	69	72	70.5	2.67	188.2350
25	72	75	73.5	2.71	198.9400
26	75	78	76.5	2.66	203.2350
27	78	81	79.5	2.47	196.6300
28	81	84	82.5	2.47	203.7750
29	84	87	85.5	2.37	202.6350
30	87	90	88.5	2.55	225.9700
31	90	93	91.5	2.14	195.5050
32	93	96	94.5	2.18	206.0100
33	96	99	97.5	2.02	196.6250
34	99	102	100.5	1.90	190.6150
35	102	105	103.5	1.83	189.0600
36	105	108	106.5	1.82	193.8300
37	108	111	109.5	1.63	178.4850
38	111	114	112.5	1.81	204.0000
39	114	117	115.5	1.46	168.6300
40	117	120	118.5	1.22	144.5700
				60.2933	4548.1900
				RTD	75.4344

Table 3-2: Average color distribution for test mash.

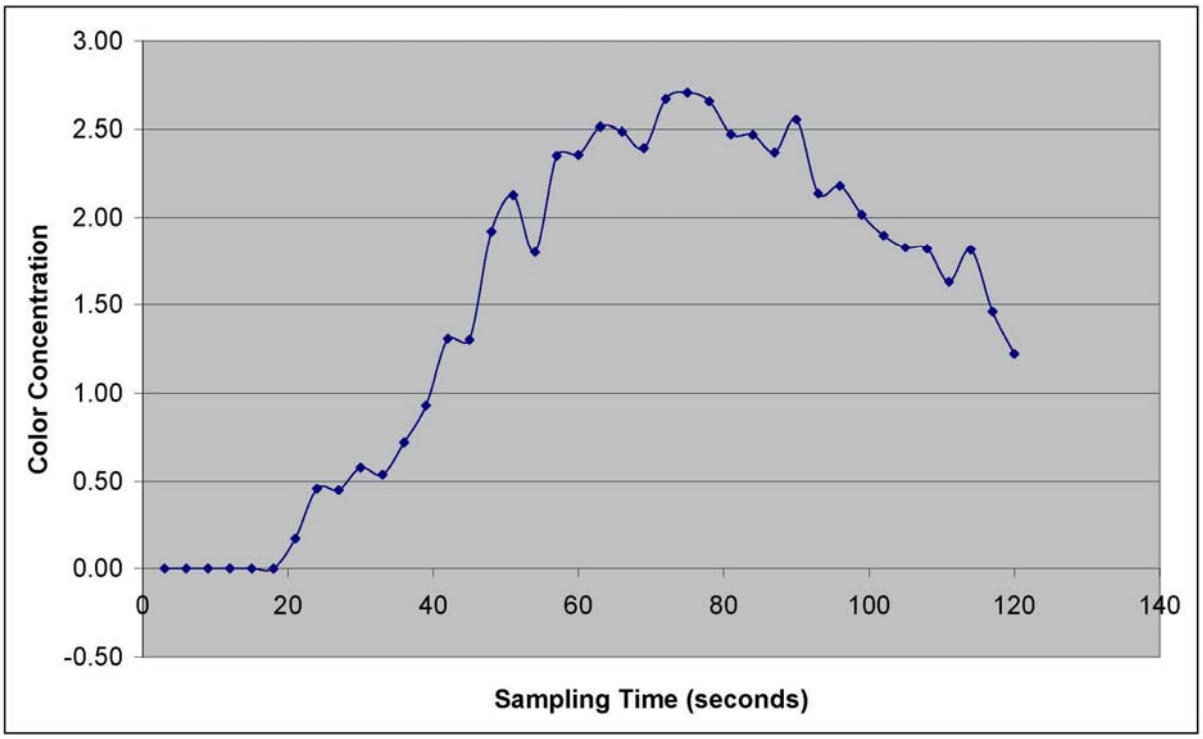

Figure 3-2: Color analyses for test mash.

Sample	Time In	Time out	Av time	C	tC
1	0	3	1.5	0.00	0.0000
2	3	6	4.5	0.00	0.0000
3	6	9	7.5	0.00	0.0000
4	9	12	10.5	0.00	0.0000
5	12	15	13.5	0.00	0.0000
6	15	18	16.5	0.00	0.0000
7	18	21	19.5	0.00	0.0000
8	21	24	22.5	0.03	0.7500
9	24	27	25.5	0.06	1.6150
10	27	30	28.5	0.07	2.0900
11	30	33	31.5	0.14	4.3050
12	33	36	34.5	0.21	7.2450
13	36	39	37.5	0.32	11.8750
14	39	42	40.5	0.42	17.1450
15	42	45	43.5	0.65	28.2750
16	45	48	46.5	0.88	41.0750
17	48	51	49.5	0.99	49.0050
18	51	54	52.5	1.03	53.9000
19	54	57	55.5	1.15	63.6400
20	57	60	58.5	1.32	77.4150
21	60	63	61.5	1.49	91.6350
22	63	66	64.5	1.49	96.1050
23	66	69	67.5	1.59	107.3250
24	69	72	70.5	1.80	126.6650
25	72	75	73.5	1.54	112.9450
26	75	78	76.5	1.42	108.3750
27	78	81	79.5	1.54	122.1650
28	81	84	82.5	1.63	134.4750
29	84	87	85.5	1.54	131.3850
30	87	90	88.5	1.41	124.4900
31	90	93	91.5	1.40	127.7950
32	93	96	94.5	1.35	127.5750
33	96	99	97.5	1.11	108.2250
34	99	102	100.5	1.03	103.1800
35	102	105	103.5	1.16	119.7150
36	105	108	106.5	0.88	94.0750
37	108	111	109.5	0.81	88.6950
38	111	114	112.5	0.75	84.3750
39	114	117	115.5	0.65	74.6900
40	117	120	118.5	0.63	75.0500
				32.47	2517.2750
				RTD	77.5262

Table 3-3: Average salt distribution for crystal salt.

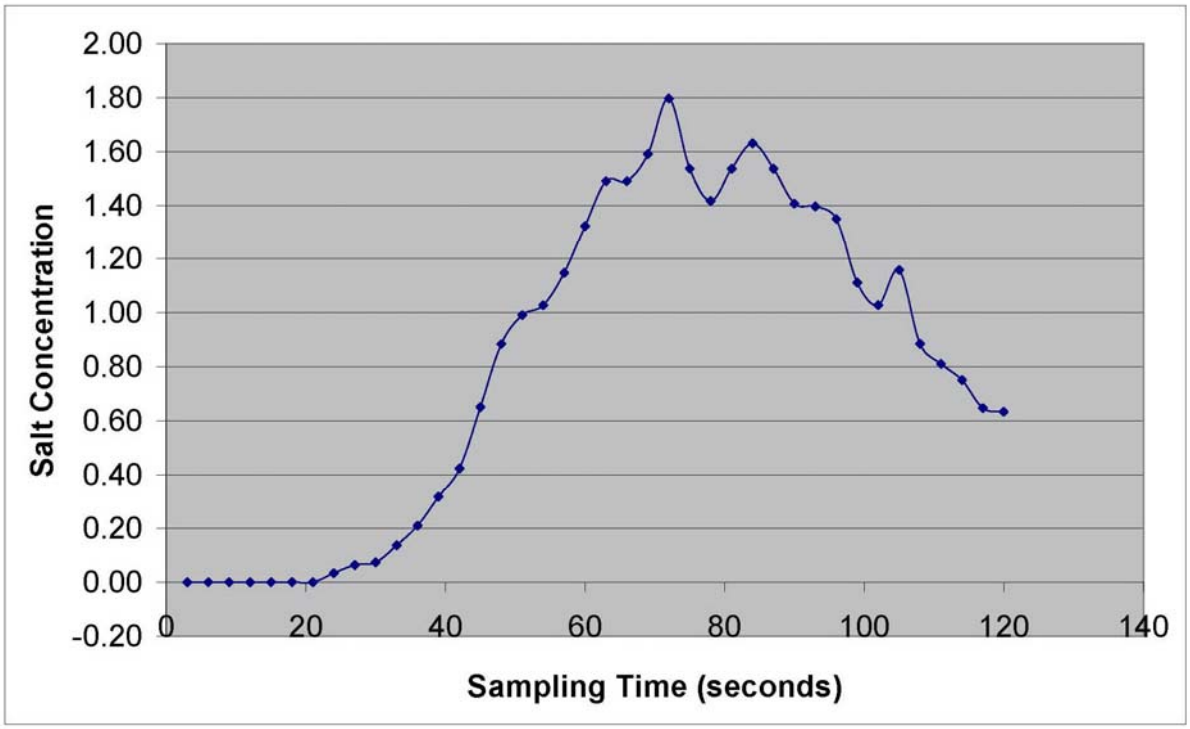

Figure 3-3: Salt analyses for crystal salt.

Sample	Time In	Time out	Av time	C	tC
1	0	3	1.5	0.00	0.0000
2	3	6	4.5	0.00	0.0000
3	6	9	7.5	0.00	0.0000
4	9	12	10.5	0.00	0.0000
5	12	15	13.5	0.00	0.0000
6	15	18	16.5	0.00	0.0000
7	18	21	19.5	0.00	0.0000
8	21	24	22.5	0.03	0.7500
9	24	27	25.5	0.05	1.1900
10	27	30	28.5	0.08	2.1850
11	30	33	31.5	0.13	4.0950
12	33	36	34.5	0.18	6.0950
13	36	39	37.5	0.22	8.1250
14	39	42	40.5	0.33	13.2300
15	42	45	43.5	0.47	20.3000
16	45	48	46.5	0.62	28.9850
17	48	51	49.5	0.69	34.1550
18	51	54	52.5	0.89	46.5500
19	54	57	55.5	0.95	52.9100
20	57	60	58.5	1.18	69.2250
21	60	63	61.5	1.36	83.6400
22	63	66	64.5	1.54	99.1150
23	66	69	67.5	0.95	64.3500
24	69	72	70.5	1.22	86.2450
25	72	75	73.5	1.43	105.1050
26	75	78	76.5	1.35	103.5300
27	78	81	79.5	1.35	107.5900
28	81	84	82.5	1.54	126.7750
29	84	87	85.5	1.23	105.1650
30	87	90	88.5	1.23	108.8550
31	90	93	91.5	0.95	87.2300
32	93	96	94.5	0.81	76.8600
33	96	99	97.5	0.81	79.3000
34	99	102	100.5	0.75	75.0400
35	102	105	103.5	0.78	81.0750
36	105	108	106.5	0.62	66.0300
37	108	111	109.5	0.61	66.4300
38	111	114	112.5	0.58	64.8750
39	114	117	115.5	0.51	58.9050
40	117	120	118.5	0.50	58.8550
				25.93667	1992.7650
				RTD	76.8320

Table 3-4: Average salt distribution for test mash.

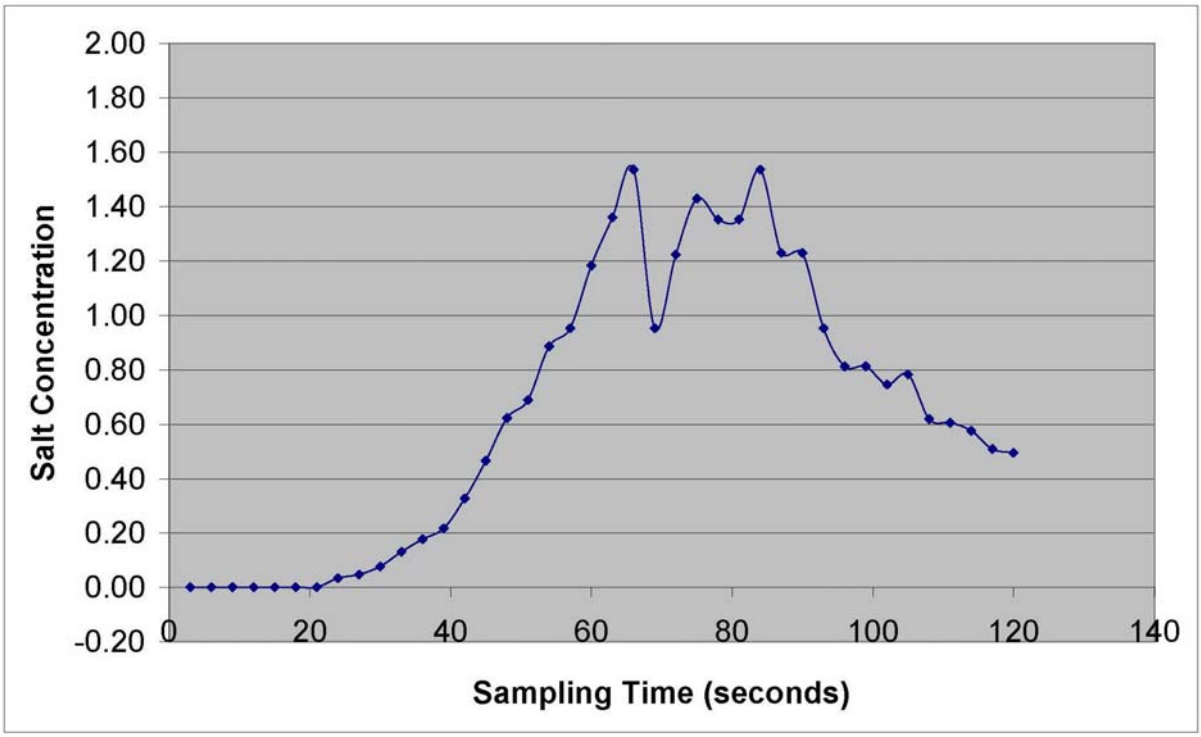

Figure 3-4: Salt analyses for test mash.