

EFFECT OF SIEVING METHODOLOGY ON DETERMINING PARTICLE SIZE OF
GROUND CORN, SORGHUM, AND WHEAT BY SIEVING

by

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Abstract

Experiments were conducted to evaluate particle size methodology and flow-ability of fractionated corn. The first experiment compared five variations of the current approved method to determine geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) described by ANSI/ASAE S319.4 “Method of determining and expressing fineness of feed materials by sieving”. This method controls many variables, including the suggested sample size and the type, number, and size of sieves. However, the method allows for variations in sieving time, sieve agitator inclusion, and the use of dispersing agent. The variations were tested with three grains (corn, wheat, and sorghum). There was no method \times grain ($P>0.05$) interaction for d_{gw} , so it was removed. Ten minute sieving time with sieve agitators and dispersing agent resulted in the lowest d_{gw} and greatest S_{gw} ($P\leq 0.05$).

The second experiment evaluated particle size analysis on ground corn using a 3-sieve method with varying sieving time (30, 60, and 90 s) with the addition of dispersing agent. The sieving time for the 3-sieve method referred to the time sieves were hand shaken side to side. Ninety seconds sieving time with dispersing agent (0.25 g) resulted in the lowest d_{gw} ($P\leq 0.05$). The 3-sieve method was not developed to calculate the S_{gw} , so means and main effects were not determined.

Experiment three evaluated particle size and flow-ability by grinding corn at two moisture (10 and 12%) with three screenings levels (0, 2.5, and 5%). Results suggested cleaning corn prior to grinding with a roller mill does not change particle size or flow-ability.

Experiment four continued the evaluation of flow-ability with corn ground to three target particle sizes (400, 500, and 600 μm) and fractionated into fine, medium, and coarse segments. Target particle size impacted d_{gw} , S_{gw} , and bulk density ($P\leq 0.05$), prior to fractionation. Based on the results of this experiment, flow-ability can be improved if fine particles ($<282 \mu\text{m}$) are removed. Results of these experiments indicated that particle size analysis should use sieve agitators, dispersing agent, and 10 or 15 min sieving time for the standard 13-sieve method and 90 s sieving time with dispersing agent for the 3-sieve method.

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Dedication

To my late grandfather, Norman and late great grandmother, Helen.

Chapter 1 - Literature Review

Introduction

The particle size of the cereal grain portion of a diet has a significant impact on animal performance. In the past, generic terms such as fine, medium, and coarse were used to define particle size. Improvements have been made to determine a numeric value by using particle size analysis to estimate the geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) of a sample. The d_{gw} is commonly referred to as the mean particle size, while the S_{gw} relates to the distribution and variation of the particles throughout the sieve stack.

The current approved method to determine d_{gw} and S_{gw} through particle size analysis of feeds and ingredients is described by ANSI/ASAE S319.4 “Method of determining and expressing fineness of feed materials by sieving”. This method controls many variables, including the suggested sample quantity and the type, number, and size of sieves. However, the method allows for variation in sieving time, sieve agitator inclusion, and the use of a dispersing agent. The most significant change in the standard method occurred between ASAE S319.2 and ANSI/ASAE S319.3, when suggested sieving time increased from 10 to 15 min. Accurate particle size analysis is important to meet required specifications and for accurate comparison between samples across laboratories, but different variations of the standard method can cause the d_{gw} to vary up to 100 μm within the same sample. Fahrenholz et al. (2010) suggested that the goal in particle size analysis is to find the lowest d_{gw} and greatest S_{gw} . Both Fahrenholz et al. (2010) and Stark and Chewing (2012) reported that the addition of sieve agitators and dispersing agent significantly changed the mean d_{gw} of a ground sample of corn, but a direct comparison using different sieving times has not been reported in various grains. Therefore, unifying the procedure used for particle size analysis would allow not only for the true d_{gw} and S_{gw} to be determined, but also for results to be accurately compared between laboratories.

Like any manufacturing process, the goal is to maximize output with the lowest costs, efficiently and effectively as possible. Therefore, if a step in the process contradicts that goal, it must be justified in the profit. In the animal, access to nutrients and

digestibility are increased with decreased particle size. However, as particle size decreases, energy consumption increases because retention time in the grinder is increased decreasing the production rate (Healy et al. 1994; Behnke, 2001). Healy et al. (1994) reported that grinding corn lowered mill throughput more and used more electrical energy than grinding sorghum. Furthermore, grinding corn to 900 μm required more electrical energy than grinding sorghum to 500 μm . Samples ground with a roller mill typically have a lower S_{gw} , indicating a narrower distribution of particles within the sample than samples ground with a hammermill to a similar d_{gw} (Goodband et al., 2006a). Energy consumption and mill throughput was overall dependent on particle size, grain type, and mill or grinder type.

Although, decreasing particle size increases digestibility, flow ability tends to decrease as a consequence. Digestibility was improved due to the increased exposed surface area to digestive enzymes while reducing selective feeding by the animal (Goodband et al., 1995). The feed industry currently has the ability to grind to a finer particle size than in the past. Currently, a mean particle size below 300 μm is considered fine, whereas past research references fine particle size as that less than 600 μm . To evaluate performance, a measure of feed efficiency is often used by determining the amount of feed it takes an animal to gain a pound. Therefore, by improving feed efficiency, feed cost and the amount excreted into the environment can be lessened. A linear relationship between decreased particle size and improved feed efficiency has been observed in swine. Research has demonstrated that swine feed efficiency is improved by 1.0 to 1.2% for every 100 μm reduction in corn particle size when ground using a hammermill (Wondra et al., 1995; De Jong et al., 2012; and Paulk et al., 2015a). Opposite of swine, broiler feed efficiency is enhanced by coarse feed particles, which were suggested to be instinctively preferred by the animal (Ferket and Gernat, 2006). Particle size is thought to be related to gizzard function and control gut motility. Even though performance can be improved, there are issues with animal health as smaller particles are thought to be a reason for an increase in stomach ulcers in swine (Wondra et al., 1995).

Particle Size Methodology

History

The first standard for estimating particle size was ASAE R246.1 “Method of determining modulus of uniformity and modulus of fineness of ground feed” published by the American Society of Agricultural and Biological Engineers (ASABE) in the 1959 Agricultural Engineers Yearbook. ASAE R246.1 (1959) determined particle size by using a 250 g sample sieved for 5 min with seven sieves (3/8, 4, 8, 14, 28, 48, and 100-mesh) with the material on screens 3/8, 4, and 8 designated as coarse, 14 and 28 as medium, and 48 and 100 as fine. The uniformity of the sample was determined by finding the proportion of particles designated as coarse, medium, and fine. The subsequent standard ASAE S319 (ASAE, 1969) calculated the particle size of ingredients based on the amount of material that was retained on a 14-sieve stack after 10 min sieving time. Due to space limitations and practicality of the Ro-Tap, or equivalent machine, most reported results were determined using 13-sieves. In these instances, the top sieve (U.S. Sieve No. 4) was removed. Pfof and Headley (1976) described the set of sieves specified in American Society for Testing and Materials E11 Standard Specifications for Wire-cloth Sieves for Testing Purposes (United States of America Standard Z23.1). ASAE S319 allowed for several variations in the analysis including: machine type, addition of sieve agitators, use of a dispersing agent, and sieving time.

Since ASAE S319 allowed for variation in the wire mesh sieve series, in the United States, there are two commonly recognized standard sieve series: Tyler and U.S., both which use a stack of sieves (each sieve possessing a different diameter opening) to separate feed particles according to size. The Tyler Standard Screen Scale sieve series was introduced in 1910 by W.S. Tyler, Inc. The original U.S. Series was proposed by the National Bureau of Standards in 1919. After some changes in wire diameter specifications, the current U.S. series was adopted by the International Standards Organization (ISO). Both sieve series meet the standards set forth in the American Standards for Testing Materials Standard E11 (Tyler, 1957). These sieve series are differentiated based on the method used to express the diameter opening. The Tyler series identifies sieves by the number of meshes (openings) per inch. The U.S. sieves are most

commonly identified by an arbitrary number that does not necessarily represent the number of meshes per inch. They also are identified by size opening in millimeters or microns. Tyler Standard Screen Scale sieves and U.S. Series sieves can be used interchangeably. Each sieve has the appropriate equivalent printed on the name plate. Half-height sieves are 1-inch in height measured from the rim to the cloth. Thus, it was recommended that the U.S. Standard, 8-inch diameter, half height sieve with a brass frame and cloth be used (Tyler, 1957).

Since there was variation in sieving time, ASAE (1969) stated that the sieve stack should be shaken until the weight of the material on the sieve, which contains material, with the smallest openings reaches equilibrium. Equilibrium was determined by weighing the sieves every five minutes, after the initial ten minute sieving time, until the weight of the material on the sieve with the smallest openings changes by 0.2 g or less. When equilibrium was achieved, it meant that all of the material reached its ideal spot in the sieve stack, theoretically. ASAE (1969) noted that dispersing agent, if required, should be added at 0.5%. Dispersing agent could be helpful in sieving high fat or similar materials. Sieve agitators, such as leather rings or small rubber balls, may be needed to break up agglomerates on finer sieves, with openings smaller than 300 μm or less than a U.S. Sieve No. 50. (ASAE, 1969). These suggestions are included in the current method. However, a majority of the research conducted over the last 30 years did not include the addition of the dispersing agent. However, recently published data observed the effect of dispersing agent on particle size analysis results and determined a consistent decrease in d_{gw} and increase in S_{gw} (Goodband et al., 2006b; Fahrenholz et al., 2010; Stark and Chewning, 2012).

The equipment required for particle size analysis included: scale, shaker, sieves, sieve cleaners, and sieve agitators. The sieve shaker was a Tyler Ro-Tap (Mentor, OH). The Ro-Tap mechanically reproduces the circular motion that occurs during hand sieving, while simultaneously tapping the sieve stack to help the particles fall through the mesh screens. Even though the standard ASAE method describes using 14 sieves, the Ro-Tap was designed to hold a maximum of 13 half height metric sieves (1-inch height, 8-inch diameter) and a pan (Tyler, 1957).

13-sieve method

The 13-sieve method is the basis of the current standard method using a sample size of 100 g. The current method stated that a dispersing agent could be used to facilitate the sieving of “high-fat” or “other materials” prone to agglomeration (ASABE, 2008 R2012). However, the terms “high-fat” and “other materials” were not defined or specified. The current method also mentioned that sieve agitators may be required to break up agglomerates on sieve less than U.S. sieve No. 50. Woodworth et al. (2002) determined that the addition of sieve agitators resulted in a lower d_{gw} and greater S_{gw} because the sieve agitators broke up agglomerates and aided in the flow ability of the sample throughout the sieve stack. ASABE (2008 R2012) did not specify the type, number, or position of sieve agitators in the stack of sieves. Woodworth et al. (2001) explained that the sieve agitators assisted in the movement of particles through the sieve openings by preventing the buildup on the sieves and passage of the particles to the next sieve, without breaking the particles into smaller pieces or forcing particles through the sieve. Woodworth et al. (2001) and Stark and Chewning (2012) both described the type and number of sieve agitators used in their respective analysis procedures. Woodworth et al. (2001) reported the tapping bar on the Ro-Tap machine had no effect on d_{gw} or S_{gw} when ground corn was either 430 or 650 μm . However, the tapping bar may effect d_{gw} and S_{gw} if a wider range of particle sizes are analyzed (Woodworth et al., 2001). The current method of sieving stated that for industrial applications, end-point determination process that uses a sieving time of 10 min can be omitted, and a sieving time of 15 min can be used (ASABE, 2008 R2012). Thinking logically, it was assumed that lower d_{gw} and higher S_{gw} values would be obtained due to an extended sieving time of an additional 5 min.

To further impact the resultant values, adding dispersing agent consistently resulted in a lower d_{gw} and greater S_{gw} (Goodband et al., 2006b; Fahrenholz et al., 2010; Stark and Chewning, 2012). Fahrenholz et al. (2010) evaluated the sieving method by the varying type of sieve shaker, sieve agitators, dispersing agent, and sieving time. Fahrenholz et al. (2010) determined that a sieving time of 15 min resulted in the lowest d_{gw} and greatest S_{gw} , and the option without sieve agitators resulted in the highest d_{gw} and the lowest S_{gw} . Goodband et al. (2006b) determined that when using a dispersing agent,

the d_{gw} value was consistently 80 μm lower than when a dispersing agent was not used in particle size analysis with strong evidence that the magnitude of difference between the two procedures increased as the S_{gw} of the sample increased. Stark and Chewning (2012) concluded that the addition of a dispersing agent better estimated the d_{gw} and S_{gw} than did the addition of sieve agitators. Even though increasing sieving time to 15 min was thought to further impact particle size results, Goodband et al. (2006b) and Stark and Chewning (2012) both used a sieving time of 10 min in their experiments, which allowed for the results to be accurately compared.

Recent research have combined the variations for the use of sieve agitators, and dispersing agent in particle size analysis. Stark and Chewning (2012) determined the effects of using sieve agitators and dispersing agent on corn, wheat, and sorghum. In both wheat and sorghum, the lowest d_{gw} and greatest S_{gw} was observed when dispersing agent and sieve agitators were used together. In corn, the lowest d_{gw} was observed when dispersing agent and sieve agitators were used together, but the greatest S_{gw} was observed with the addition of dispersing agent. The change in the d_{gw} and S_{gw} was greater in hammermill samples than in roller mill samples due to the increased amount of fine particles present in the hammermill samples (Stark and Chewning, 2012). Unfortunately, Goodband et al. (2006b) reported that previously published data on swine growth performance and diet flow ability had been conducted without the use of a dispersing agent, so an accurate comparison between results cannot be made at this time.

Alternative Methods

Alternative particle size methods have been developed to help small feed mills and swine producers monitor the particle size of ground grains. These alternative methods require less time and lower capital investment in equipment. The tradeoff is less accuracy and greater analytical variation when compared to the 13-sieve method. However, these alternative methods can be used as a quality control measurement by the mill employees to check the particle size during the grinding process. The one sieve and 3-sieve methods are commonly used by producers. The one-sieve method was developed by Iowa Farm Automation Ltd (IFA, Stanley, IA), which used a U.S. No. 14 sieve with 280 g of sample. Particle size was calculated according to the weight, to the nearest

ounce, remaining on the sieve after shaking by hand and correlated to a d_{gw} of 700 to 1,200 μm (Baldrige, et al. 2001). Even though the one sieve method cost less and was quicker than the 13-sieve method, accuracy of the true particle size was sacrificed.

The one sieve method was refined by Baldrige et al. (2001) who developed a regression equation to determine particle size using a 3-sieve method. Baldrige et al. (2001) used 50 g of sample and the sieve and sieve agitator arrangement included: U.S. No. 12 sieve with no sieve agitators, U.S. No. 30 sieve with one rubber ball and one bristle sieve cleaner, and U.S. No. 50 sieve with one rubber ball and two bristle sieve cleaners. Baldrige et al. (2001) determined that the 3-sieve method was more accurate than the one sieve method when compared to the results from the 13-sieve method without dispersing agent. The sieving time in the 3-sieve method referred to the time the sieves are shaken side to side by hand instead of being placed in the Ro-Tap machine. Bokelman et al. (2015) evaluated the accuracy of the 3-sieve method with a 90 s sieving time using different technicians, changing the top sieve (U.S. No. 16 vs. 12), addition of a dispersing agent (0.5 g), and grain type (corn, wheat, and sorghum) compared to the results from the 13-sieve method using 0.5 g of dispersing agent with sieve agitators and a 15 minute sieving time. The results of Bokelman et al. (2015) conflicted with Goodband et al. (2006b), Fahrenholz et al. (2010), and Stark and Chewning (2012) on the effect of dispersing agent in the 13-sieve method. Bokelman et al. (2015) reported no difference on d_{gw} when dispersing agent was used in the 3-sieve method. Still, Bokelman et al. (2015) observed a difference between grain types (corn, wheat and sorghum) with corn predicting a lower d_{gw} , wheat predicting a higher d_{gw} , and sorghum predicting a nearly equal result as the 13-sieve method.

Continuing with alternative methods, which required less investment of money and time, the Danish Institute of Agricultural Sciences created the Bygholm Feed Sieve, consisting of four compartments with three sieves (1000, 2000, and 3000 μm). Benz et al. (2005) indicated the sieve was shaken for about 4.5 min or until no more sample fell through the sieves. Benz et al. (2005) reported that the addition of sieve agitators mimicked the 13-sieve method, but increased the particle size because the Bygholm Feed Sieve was not calibrated for sieve agitator inclusion.

Baker and Herrman (2002) tested a short stack of 5 sieves (U.S. No. 16, 30, 50, 100, and 200) and 50 g of sample to estimate particle size using a Ro-Tap machine. The resultant d_{gw} was 40 μm less and S_{gw} 0.24 more than the results of the full stack (13-sieves) in fine ground corn (450 μm). In coarse ground corn (900 μm), the short stack yielded a d_{gw} 23 μm less and S_{gw} 0.20 more than the results from the full stack. Although alternative methods have been developed as a more cost friendly and time efficient way to estimate particle size, the accuracy is reduced when compared to the standard 13-sieve method.

Effect of Particle Size on Flow Ability

The flow ability of feed through bins and feeders has been a growing concern for swine producers, especially as the particle size has continued to decrease over the last 10 years. One hypothesis is that poor feed flow ability is caused by greater fines and wider distribution of particles than by the lowered target d_{gw} . Goodband et al. (2006a) demonstrated that even though the d_{gw} was similar, the S_{gw} differed based on the settings applied to the grinding machine, roller mill and hammermill. Goodband et al. (2006a) stated that poor flow ability, measured by angle of repose, may be caused more by particle shape than by fine particle sizes. Goodband et al. (2006a) indicated that as particle size decreased, bulk density, production rate and flow ability decreased, while electrical energy consumption. Essentially, samples with a greater S_{gw} or particle size distribution and variation of particles have more fines, decreasing the flow ability (Goodband et al., 2006b).

Groesbeck et al. (2006) suggested that flow ability is influenced by particle size, but the behavior of the samples in bins or feeders was not observed. Goodband et al. (1995) reported that when particle size dropped below 500 μm flow ability in the bulk bins and feeders was decreased. In contrast to Goodband et al. (1995), De Jong et al. (2012, 2014a, and 2014b) reported that as particle size was decreased, angle of repose increased and bulk density decreased, which would indicate poorer flow ability, but no bridging was observed in the feeders. However, Probst et al. (2013) observed no difference for flow properties of compressibility and angle of repose when moisture content differed. Still, Probst et al. (2013) noted that an observed difference in S_{gw} may

be due to a combination of particle cohesion, reduction in brittleness, and reduction in the amount of fines due to increased moisture content. Probst et al. (2013) reported that d_{gw} of hammermill ground corn was not significant when comparing moisture content (10, 16, and 20%). Groesbeck et al. (2006) determined that hammermill ground samples had a higher angle of repose and S_{gw} compared to roller mill ground samples at similar d_{gw} due to the method of grinding, indicating poorer flow ability.

Effect of Particle Size on Swine Health and Performance

Swine Health and Digestibility

There has been much debate over the cause of ulcers in swine and how performance is affected. The effect of ulcers on pig performance were noted by (Wondra et al., 1992; Healy et al., 1994; and Wondra et al., 1995). The type of grain, particle size distribution, method of milling have been studied to determine the effect on the development of ulcers in swine. Healy et al. (1994) suggested that 300 μm corn may be more likely to cause ulcers than 300 μm sorghum in nursery pigs. Healy et al. (1994) also noted that corn maybe more ulcerogenic than sorghum as particle size decreased. Published data suggested that fine particles and differences in grain type were thought to be the culprit for ulcers.

Wondra et al., 1992; Goodband et al., 1995; Wondra et al., 1995; and Ayles et al., 1996 reported complementary results when the effect of particle size distribution was evaluated on health and digestibility. Goodband et al. (1995) reported that when particle size dropped below 500 μm , the frequency of gastric ulcers increased. Ayles et al. (1996) suggested that particle size distribution may be a contributing factor in the development of ulcers, but further investigation was needed. Still, Ayles et al. (1996) stated that feeding a diet with coarse ground corn (937 μm) caused a decrease in the severity of ulcers after they had developed. Initial research conducted by Wondra et al. (1992) determined that the fine particles in diets with a high S_{gw} did not induce formation of stomach lesions. Wondra et al. (1995) later noted, a diet with a greater S_{gw} could be more ulcerogenic than a diet with a lower S_{gw} , even if they had the same d_{gw} . In other words, increased uniformity in a sample, decreased the chance for ulcers. The particle size distribution of grain ground using a roller mill is narrower than grain ground using a

hammermill (Nir et al., 1995). Wondra et al. (1995) confirmed these statement by observing that pigs fed diets with roller mill corn had lower ulcer scores than those fed hammermill corn. Wondra et al. (1995) suggested the roller mill produced a more uniform product that improved apparent nutrient digestibility in diets and decreased undesirable changes in stomach morphology. Still, Wondra et al. (1995) found no particle size \times mill type interaction or difference in performance when corn was ground to 800 or 400 μm . Williams et al. (2010) and Paulk et al. (2015a) concluded that even though stomach lesion scores decreased linearly when cracked corn was included up to 40% in the diet only slight improvements in stomach lesion scores were observed.

Researchers have reported that nutrient digestibility of grains varies based on the mill or grinder type used to reduce the particle size of the grain. Most data presented in literature has been generated by grinding grain with either a hammermill or roller mill. Goodband et al. (1995) reported that as particle size decreases, the surface area of each particle increases allowing for greater interaction with digestive enzymes increasing nutrient digestibility and absorption. In a review, Goodband et al. (1995) discussed that as particle size decreased from 1,000 to 700 μm , digestibility of protein and energy increased. Acosta Camargo et al. (2015) evaluated the digestibility of corn and wheat in finishing pig diets at three particle sizes (300, 500, and 700 μm) ground with a hammermill or roller mill. Acosta Camargo et al. (2015) observed interactions with particle size for grain and mill type and reported an improvement in the gross energy digestibility of corn as compared to wheat. Acosta Camargo et al. (2015) determined that grinding with a roller mill had a greater impact on gross energy digestibility over a hammermill. Within each interaction, Acosta Camargo et al. (2015) reported that 300 μm was not different from 500 μm for wheat or when ground using a hammermill. Patience et al. (2011) observed a quadratic response for energy digestibility with increased S_{gw} at the same d_{gw} (550 μm) which can alter the energy digestibility of ground corn. Paulk et al. (2015b) explained that these results imply that increasing the S_{gw} may increase digestion. However, Wondra et al. (1995) determined that greater gross energy digestibility occurred with corn that was ground using a roller mill compared to a hammermill when ground to the same particle size (400 and 800 μm). Wondra et al. (1992) reported that the greatest gross energy digestibility was seen in the ground corn

sample with the lowest S_{gw} . De Jong et al. (2014a) reported a tendency for a quadratic particle size (200, 400, and 600 μm) \times wheat source (hard red winter wheat and soft white winter wheat) interaction for gross energy digestibility with the 400 μm having the greatest value. De Jong et al. (2014b) observed a linear improvement in gross energy digestibility when particle size in wheat was reduced (730, 580, and 330 μm) in finishing diets. Thus, De Jong et al. (2014b) recommended that maximum nutrient gross energy digestibility is achieved when wheat is ground to less than 400 μm .

Swine Performance

Nursery Performance

Several nursery studies have demonstrated an improvement in swine performance when the pigs were fed diets that contained grains with a particle size below 600 μm in meal diets. Recent data would suggest an improvement in efficiency in meal diets, but little to no improvement in feed efficiency for pelleted diets with decreased particle size. Goodband and Hines (1988) noted that reducing particle size improved average daily gain (ADG) and feed efficiency. Goodband et al. (2006a) determined that both energy and protein digestibility and feed efficiency were improved as particle size was decreased for corn and sorghum in nursery diets. Healy et al. (1994) determined that as particle size was reduced from 900 to 300 μm , both ADG and feed efficiency improved in nursery pigs in the first 14 days with corn greater than sorghum. Conflicting previously reported data, Rojas and Stein (2014b) observed that feed efficiency linearly worsened as particle size was decreased (339, 485, 677, and 865 μm), while ADG and feed intake saw no difference. Bokelman et al. (2014) reported that feed efficiency was improved when corn was ground to 400 versus 700 μm in meal diets with no difference in pelleted diets.

Healy et al. (1994) concluded there was a correlation between the age of the nursery pig and the optimum particle size of the grain in the diet. Mavromichalis et al. (2000) reported that feed efficiency improved as particle size decreased for the first seven days in nursery pigs, with 400 μm providing the best feed efficiency. Mavromichalis et al. (2000) concluded that overall in nursery pigs, the best ADG and feed efficiency were achieved at 600 μm . This suggests that the particle size that results in the best feed efficiency and ADG changes with the age weaning weight of the pig.

Finishing Performance

Research studies have shown that particle size, mill type, feed form, and grain type effect swine finishing performance. Contrary to nursery performance, recent data would suggest an improvement in feed efficiency in both meal and pelleted diets with decreased particle size. Confirming this statement, Goodband et al. (2006a) suggested particle size reduction of grains will improve feed efficiency regardless of age. De Jong et al. (2014b) reported that as particle size in ground wheat decreased (730, 580, and 330 μm), feed efficiency improved linearly, but no difference in ADG or intake was observed. Rojas and Stein (2014a) observed similar results with no difference in intake or ADG when particle size was reduced (865, 677, 485, and 339 μm). Healy et al. (1994) determined that a reduction in particle size improved ADG and feed efficiency more for growing-finishing pigs fed corn-based diets than for those fed sorghum-based diets with the best feed efficiency at 500 μm for all grain types. However, Paulk et al. (2015b) observed that as particle size was decreased from 800 to 400 μm in sorghum-based diets, ADG was not effected while feed efficiency improved due to a linear decrease in feed intake, De Jong et al. (2014a) reported no effect on ADG or feed efficiency when particle size was reduced from 600 to 200 μm in pelleted wheat-based diets. Still, De Jong et al. (2012) reported that feed efficiency was improved when the particle size of the corn decreased (650 to 320 μm), but observed no effect on ADG or feed intake. Conflicting other reported data, Wondra et al. (1995) observed no particle size \times mill type interaction or difference in performance when corn was ground to 800 or 400 μm using a hammermill or roller mill. Echoing Wondra et al. (1995), Laurinen et al. (2000) reported that barely-based diets ground using a roller mill increased feed intake, decreased ADG, and worsened feed efficiency, while difference in mill type had no effect on intake or performance for wheat-based diets. Paulk et al. (2015b) explained this phenomena by suggesting that as particle size is decreased in sorghum, the energy concentration of the diet increased, thus confirming that the energy value assigned to a cereal grain depended on the particle size.

Like with nursery performance, there have been conflicting reports about a possible diet form (meal or pelleted) \times particle size interaction on finishing performance. De Jong et al. (2012) reported that feeding a pelleted diet that contained finely ground

corn (320 μm) improved feed efficiency and suggested that performance can be improved through a variety of feed processing techniques. However, De Jong et al. (2012) observed that when the entire diet was finely ground and fed as a meal diet, intake decreased, but increased when pelleted. These results suggest pellets possibly improve the palatability of diets that contain finely ground grain (De Jong, et al., 2012). Still, De Jong et al. (2012) did not observe a difference in feed efficiency for pelleted versus meal diets. Nemecek et al. (2013) reported a linear response for with decreased ground corn particle size (350, 650 μm , or equal blend) \times feed form interaction decreased intake and improved feed efficiency in meal diets but not pelleted diets. Therefore, Nemecek et al. (2013) suggested that there was no benefit to decreasing particle size below 650 μm for pelleted diets. Thus, Nemecek et al. (2013) noted that more data is needed to understand why feed efficiency was improved in meal diets but not in pelleted diets with decreasing particle size.

Previous data reported the use of coarse particles, in the form of cracked corn, was thought to improve performance. Williams et al. (2010) reported no difference in ADG and feed efficiency tended to worsen as the inclusion of cracked corn (3,549 μm) increased when added up to 40% in meal diets. However, when diets with cracked corn were pelleted, Paulk et al. (2015a) observed increased ADG and improved feed efficiency. Paulk et al. (2015a) eluded to particle size distribution impacting feed efficiency. Paulk et al. (2015a) reported that there was no difference in ADG or feed intake, but feed efficiency tended to worsen linearly as the inclusion of cracked corn was increased in meal diets. In summary, adding coarse particle, in the form of cracked corn, was thought to improve performance, but yielded conflicting results.

Effect on Poultry Gut Health and Performance

In contrast to swine, a smaller particle size in broiler diets can have a negative effect on gut health and broiler performance. Although small particles can improve the digestibility of grains due to increased surface area, coarse particles are needed to stimulate and maintain a healthy gizzard. Coarse particles in the feed increase the activity of the gizzard, which results in increased reverse peristalsis in the digestive tract, increasing the retention time and therefore the digestibility. Xu et al. (2015a) suggested

that coarse ground grain should be mixed with fine ground grain to stimulate the gizzard and slow passage rate while fine particles increase digestibility. The feed efficiency of a broiler is related to gizzard function, which is thought to control gut motility, and is enhanced by coarse feed particles, which are instinctively preferred by the animal (Ferket and Gernat, 2006).

Similar to swine, age was thought to impact performance and digestibility, depending on particle size. Chewning et al. (2012) and Xu et al. (2015a) observed that feeding a high percentage of large particles of corn to broiler chicks the first 14 days decreased feed intake. Jacobs et al. (2010) reported a linear decrease in feed efficiency as corn particle size increased from 557 to 1387 μm . Xu et al. (2015b) suggested that the gizzard of a broiler chick may not have the ability to grind large particles. Chewning et al. (2012) also reported heavier gizzard weights in birds fed diets that contained 600 μm versus 300 corn. Researchers suggested increasing the percentage of coarse particles ($> 1,000 \mu\text{m}$) in the diet to stimulate gizzard development after 14 days (Xu et al., 2015b). Jacobs et al. (2010) observed the greatest increase in gizzard with 1387 μm ground corn. In summary, Ferket and Gernat (2006) stated that for every 100 μm reduction in feed particle size, intake was decreased on average by 4%.

Previous research has evaluated animal performance in grain type (corn and sorghum), mill type (hammermill and roller mill), and feed form (meal or pellet). Nir et al. (1995) evaluated wheat and sorghum ground using a hammermill or roller mill on broiler performance. Nir et al. (1995) determined that grain ground using a roller mill improved performance more than grain ground using a hammermill, further stressing the importance of less variation and narrower distribution of particle size. However, Douglas et al. (1990) observed no interactions for grain (corn and sorghum), grind size (fine and coarse), and feed form (meal or pelleted) on bird performance. When analyzed as main effects, Douglas et al. (1990) reported that coarse ground grain decreased ADG and resulted in poorer feed efficiency in meal diets, while pelleting had the opposite effects. However, Chewning et al. (2012) observed no difference in feed efficiency when particle was reduced from 600 to 300 μm in pelleted corn diets, but 300 μm corn in meal diets resulted in the best feed efficiency. Conflicting data has been reported relating poultry performance to particle size, grain type, mill type, and feed form.

Conclusion

Review of literature found that animal health, animal performance, and flow ability were impacted by particle size. Although particle size can positively impact animal performance, there are drawbacks. Decreasing particle size tends to improve performance in swine, but increases the cases and severity of ulcers. However, in poultry, coarse particles are required to maintain proper gizzard function. Therefore accurate estimation through particle size analysis is necessary. However, variations within the ANSI/ASAE S319.4 standard method can cause the mean particle size result to vary significantly. These variations include: sieving time, sieve agitators, and dispersing agent. Most samples are analyzed using a Ro-Tap machine with 13-sieves, but there are alternative methods that require less time and cost investment. These alternative methods include: Bygholm Feed Sieve, one-sieve method, and 3-sieve method. There is little published data describing the method for particle size analysis used in animal feeding trials. Therefore, many variations are unknowingly used for particle size analysis that are unable to be accurately compared.

Based on this review, variations of the standard method need to be evaluated to determine which method most accurately estimates particle size. Also, alternative methods such as the 3-sieve method need to be evaluated on how they compare to the 13-sieve method for accuracy. Debate still remains on what causes flow ability issues so it is suspected that by removing the fines from a sample, flow ability can be improved. However, the change in nutrient content could become a concern and should be taken into account. In conclusion, there are variations in particle size analysis that need to be unified and obtain a better understanding of the reasoning for flow ability issues in order for particle size to have a positive impact on animal health and performance.

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Chapter 2 - Effects of varying methodologies on grain particle size

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Abstract

Particle size reduction is an important component of feed manufacturing that impacts pellet quality, feed flow ability, and animal performance. The estimation of particle size is an important quality control measurement for feed manufacturers, nutritionists, and producers. ANSI/ASAE S319.4 is the current approved method for determining the geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) of grains. This method controls many variables, including the suggested quantity of initial material and the type, number, and size of sieves. However, the method allows for variation in sieving time, sieve agitators, and the use of a dispersing agent. Therefore, the objective of this experiment were to determine which method of particle size analysis best estimates the particle size of various cereal grains. Eighteen samples of either corn, sorghum, or wheat were ground and analyzed using different variations of the standard particle size analysis method. Treatments were arranged in a 5×3 factorial arrangement with 5 sieving methods: 1) 10 minute sieving time with sieve agitators and no dispersing agent, 2) 10 minute sieving time with sieve agitators and dispersing agent, 3) 15 minute sieving time with no sieve agitators or dispersing agent, 4) 15 minute sieving time with sieve agitators and no dispersing agent, or 5) 15 minute sieving time with sieve agitators and dispersing agent conducted in 3 grains types (corn, sorghum, or wheat). There were four replicates per treatment. Results for d_{gw} and S_{gw} were calculated according to both standard methods S319.2 and S319.4. The analytical method that resulted in the lowest d_{gw} and greatest S_{gw} was considered desirable because it was presumably representative of increased movement of particles to their appropriate sieve.

There was no analytical method \times grain type interaction ($P < 0.05$) for d_{gw} , so it was removed from the model. Analytical method affected ($P \leq 0.05$) d_{gw} and S_{gw} measured by both standards. Inclusion of sieve agitators and dispersing agent resulted in

the lowest d_{gw} , regardless of sieving time. Inclusion of dispersing agent reduced ($P < 0.05$) the mean d_{gw} by 32 or 36 μm when shaken for 10 or 15 min, respectively, compared to the same sample analyzed without dispersing agent. The addition of the dispersing agent also increased the S_{gw} . The dispersing agent increased the quantity of very fine particles collected in the pan, so therefore S_{gw} was substantially greater ($P < 0.05$). Corn and sorghum ground using the same mill parameters had similar ($P > 0.05$) d_{gw} , but wheat ground using the same mill parameters was 120 to 104 μm larger ($P \leq 0.05$) compared to corn and sorghum, respectively.

In conclusion, both sieve agitators and dispersing agent should be included when conducting particle size analysis. The results of this study indicate that 10 or 15 min of sieving time produced similar results. Wheat ground using the same mill parameters as corn and sorghum had 120 to 104 μm larger d_{gw} compared to corn and sorghum, respectively.

Keywords: Corn, feed, grain, methodology, particle size analysis

Introduction

Research has demonstrated that swine feed efficiency is improved by 1.0 to 1.2% for every 100 μm reduction in corn particle size or geometric mean diameter (d_{gw}) ground with a hammermill (Wondra et al., 1995; De Jong et al., 2012; Paulk et al., 2015). Accurate particle size analysis is important to meet quality control specifications in the feed mill, as well as compare samples across laboratories. However, allowable variations within the standard method used to determine the mean d_{gw} can result in differences of up to 100 μm for the same sample. The current approved method used to determine d_{gw} and the geometric standard deviation (S_{gw}) of feeds and ingredients is described by ANSI/ASAE S319.4 “Method of determining and expressing fineness of feed materials by sieving”. This method controls many variables, including the suggested quantity of initial material and the type, number, and size of sieves. However, the method allows for variations in sieving time, sieve agitator inclusion, and the use of a dispersing agent. The most significant change in the standard method occurred between ASAE S319.2 and ANSI/ASAE S319.3, when sieving time increased from 10 to 15 min. Fahrenholz et al.

(2010) suggested that the goal in particle size analysis is to find the lowest d_{gw} and greatest S_{gw} . Both Fahrenholz et al. (2010) and Stark and Chewing (2012) reported that the addition of agitators and dispersing agent significantly changed the mean d_{gw} of a ground sample of corn, but a direct comparison using different sieving times has not been reported in various grains. Therefore, the objective of this experiment was to determine which method of particle size analysis best estimates the particle size of various cereal grains.

Materials and Methods

Material Preparation

A total of 360 particle size analytical procedures were conducted in this experiment, stemming from 18 different samples of ground grain. These samples represented 3 grain types (corn, wheat, and sorghum), 2 mill types (hammermill and roller mill), and 3 grind sizes (coarse, medium, and fine). The hammermill (Model 22115, Bliss Industries LLC., Ponca City, OK) was equipped with 1.59 mm, 4.76 mm, and 6.35 mm screens for fine, medium and coarse grinds, respectively. The roller mill (Model 924, RMS Roller Grinder, Harrisburg, SD) rolls were 2.36 and 2.36 corrugations/cm, 4.72 and 5.51 corrugations/cm, and 6.30 and 7.09 corrugations/cm roll on the top, middle, and bottom roll pairs, respectively. The hammermill screen sizes and roll gap settings were kept constant for each cereal grain. Samples were ground at the Kansas State University O.H. Kruse Feed Technology Innovation Center in Manhattan. The differences in type of mill type and grind size was intended to create a robust set of ground grain samples, but were made random effects due to their natural confounding with the response criterion.

Sample Analysis

Samples were divided using a riffle divider to approximately 100 ± 5 g. The weighed samples were then analyzed using different variations of the ANSI/ASAE S319.4 standard method for particle size analysis in the Swine Laboratory at Kansas State University. Particle size analysis was conducted with two stainless steel sieve stacks (13-sieves) to prevent the residual dispersing agent present on the sieve from affecting subsequent samples without the dispersing agent. Both sieve stacks contained sieve

agitators with bristle sieve cleaners and rubber balls measuring 16 mm in diameter (Table 2.1). Sieves were cleaned after each analysis with compressed air and a stiff bristle sieve cleaning brush.

Each sieve was individually weighed with the sieve agitators to obtain a tare weight. The 100 ± 5 g sample was then placed on the top sieve. If dispersing agent (Model SSA-58, Gilson Company, Inc., Lewis Center, OH) was required, (0.5 g) it was mixed into the sample prior to placing the mixture on the top sieve. The sieve stack was then placed in the Ro-Tap machine (Model RX-29, W. S. Tyler Industrial Group, Mentor, OH) and ran for the specified time. Once completed, each sieve was weighed with the sieve agitator(s) to obtain the weight of the sample on each sieve. The amount of material on each sieve was used to calculate the d_{gw} and S_{gw} . When dispersing agent was used its weight was not subtracted from the weight of the pan as specified in the ANSI/ASAE S319.4. Calculations were performed according to the equations listed and described in ANSI/ASAE standard S3219.4 (Eq. 2.1 to 2.4) for d_{gw} and S_{gw} and ASAE standard S319.2 for S_{gw} (Eq. 2.3). Equations 2.5 and 2.6 depict how to calculate the range for 68% of the particles in a sample. Equation 2.5 and 2.6 use the d_{gw} calculated with Eq. 2.1 and 2.2. Equation 2.5 uses the S_{gw} calculated with Eq. 2.3, while Eq. 2.6 uses the S_{gw} calculated with Eq. 2.4.

$$d_{gw} = \log^{-1} \left[\frac{\sum_{i=1}^n (W_i \log \bar{d}_i)}{\sum_{i=1}^n W_i} \right] \quad (\text{Eq. 2.1})$$

where W_i is mass on i^{th} sieve, g

d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} is geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 2.2

n is number of sieves +1 (pan)

$$\bar{d}_i = (d_i \times d_{i+1})^{1/2} \quad (\text{Eq. 2.2})$$

where d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{i+1} is nominal sieve aperture size in next larger than i^{th} sieve (just above in a set), mm

$$S_{\log} = \left[\frac{\sum_{i=1}^n W_i (\log \bar{d}_i - \log d_{gw})^2}{\sum_{i=1}^n W_i} \right]^{1/2} = \frac{S_{\ln}}{2.3} \quad \text{(Eq. 2.3)}$$

where W_i is mass on i^{th} sieve, g

d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} is geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 2.2

S_{\log} is geometric standard deviation of log-normal distribution by mass in ten-based logarithm, dimensionless

S_{\ln} is geometric standard deviation of log-normal distribution by mass in natural logarithm, dimensionless

n is number of sieves +1 (pan)

$$S_{gw} \approx \frac{1}{2} d_{gw} \left[\log^{-1} S_{\log} - (\log^{-1} S_{\log})^{-1} \right] \quad \text{(Eq. 2.4)}$$

where W_i is mass on i^{th} sieve, g

d_i =nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} = geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 2.2

S_{\log} is geometric standard deviation of log-normal distribution by mass in ten-based logarithm, dimensionless

S_{gw} is geometric standard deviation of particle diameter by mass, mm

$$\frac{d_{gw}}{S_{gw}} = \text{lower limit} \quad d_{gw} \times S_{gw} = \text{upper limit} \quad \text{(Eq. 2.5)}$$

where 68% of the particles are determined by finding the difference between the upper and lower limits using the S_{gw} from Eq. 2.3

d_{gw} = geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 2.2

$$\sum_{gw} \times 2 = 68\% \text{ of particles} \quad (\text{Eq. 2.6})$$

Where 68% of the particles are determined using the S_{gw} from Eq. 2.4

Experimental Design

Analytical methods were chosen based on the five most common variations currently used in the feed manufacturing industry. These variations in the ANSI/ASAE S319 standard method were evaluated by versions ASAE S319.2 and ANSI/ASAE S319.4 for a method x grain interaction effect and main effects for d_{gw} and S_{gw} for method and grain. Treatments were arranged in a 5×3 factorial arrangement with 5 sieving methods and 3 grain types:

- 1) 10 minute sieving time with sieve agitators and no dispersing agent
- 2) 10 minute sieving time with sieve agitators and dispersing agent
- 3) 15 minute sieving time with no sieve agitators and no dispersing agent
- 4) 15 minute sieving time with sieve agitators and no dispersing agent
- 5) 15 minute sieving time with sieve agitators and dispersing agent

The 5 sieving methods were repeated four times for each of the 18 samples, comprised of 3 grain types (corn, wheat, and sorghum), 2 mill types (hammermill and roller mill), and 3 grind sizes (coarse, medium, and fine) with a different technician conducting the procedure for each of the 4 replicates with random effects being grind size and mill type. Fixed and random effects were included in experimental design and initial reports of data. Data were analyzed using GLIMMIX procedure of SAS (SAS Institute, Inc., Cary, NC). Samples were blocked by day and technician. Interactions were removed from the model if $P > 0.05$. Results were considered significant if $P \leq 0.05$, and a tendency if $0.05 \leq P \leq 0.10$. Orthogonal contrasts were used to evaluate differences in time (10 vs. 15 min), sieve agitators, and dispersing agent. The least significance difference test was used to determine differences between sieving method and grain. The CORR procedure of SAS was used to determine Pearson Correlation Coefficients for d_{gw} and S_{gw} to compare when dispersing agent was subtracted from the weight of the pan for each grain.

Results and Discussion

Technician was intended to be a fixed effect in this experiment, but the variable was removed from the model due to insignificance for d_{gw} ($P > 0.05$) and S_{gw} ($P > 0.05$). The method \times grain interaction for d_{gw} ($P > 0.05$) was not significant (Table 2.2). The method \times grain interaction for S_{gw} method ASAE S319.2 calculated with Eq. 2.3 was significant ($P \leq 0.05$) due to the differences within each grain for each method. A similar trend among grains was observed across all methods when S_{gw} was calculated using method ASAE S319.2. The S_{gw} method ANSI/ASAE S319.4 calculated with Eq. 2.4 eliminated the method \times grain interaction ($P > 0.05$; Table 2.2), while main effects for method ($P \leq 0.05$; Table 2.3) and grain ($P \leq 0.05$; Table 2.4) were significant. Differences were observed when d_{gw} was evaluated for different grains ground using the same mill parameters ($P \leq 0.05$). When compared to corn (529 μm), the d_{gw} of sorghum was 16 μm (545 μm) larger and wheat was 120 μm (649 μm) larger ($P \leq 0.05$).

The main effects of method and grain were significant for d_{gw} ($P \leq 0.05$). The d_{gw} was lowest when both sieve agitators and dispersing agent were included in the analysis. The addition of dispersing agent reduced the mean d_{gw} by 32 μm (586 to 554 μm) with a 10 minute sieving time ($P \leq 0.05$). The addition of dispersing agent with a 15 minute sieving time, reduced the mean d_{gw} 36 μm (576 to 540 μm) ($P \leq 0.05$). However, the difference in d_{gw} with increasing sieving time from 10 to 15 min was not significant ($P > 0.05$). Adding sieve agitators reduced d_{gw} by 39 μm (615 to 576 μm) with a 15 minute sieving time ($P \leq 0.05$).

Research consistently has demonstrated the addition of sieve agitators and dispersing agent lowered d_{gw} and increased S_{gw} (Goodband et al., 2006; Fahrenholz et al., 2010; Stark and Chewing, 2012). Woodworth et al. (2002) determined that the addition of sieve agitators resulted in a lower d_{gw} and greater S_{gw} because the sieve agitators broke up agglomerates and aided in the flow ability of the sample throughout the sieve stack. ASABE (2008 R2012) did not specify the type, number, or position of sieve agitators in the stack of sieves. Woodworth et al. (2001) explained that the sieve agitators assisted in the movement of particles through the sieve openings by preventing the buildup on the sieves and passage of the particles to the next sieve, without breaking the particles into smaller pieces or forcing particles them through the sieve. Woodworth et al. (2001) and

Stark and Chewning (2012) both described the type and number of sieve agitators used in their respective analysis procedures.

In agreement with the results of this experiment, Goodband et al. (2006), Fahrenholz et al. (2010), and Stark and Chewning (2012) also reported decreased d_{gw} and increased S_{gw} with the use of sieve agitators and dispersing agent in ground corn samples. Fahrenholz et al. (2010) evaluated the sieving method using the following options: sieve shaker, sieve agitators, dispersing agent, and sieving time. Fahrenholz et al. (2010) determined that a sieving time of 15 min resulted in the lowest d_{gw} and greatest S_{gw} , while the option without sieve agitators resulted in the highest d_{gw} and the lowest S_{gw} . Fahrenholz et al. (2010) reported 74 μm (560 to 486 μm) decrease with dispersing agent, 101 μm (624 to 523 μm) decrease when using sieve agitators, and 42 μm (523 to 481 μm) decrease when sieving time was increased from 10 to 15 min for particle size analysis. Goodband et al. (2006) noted a consistent 80 μm decrease in d_{gw} with the use of dispersing agent in samples ranging from 400 to 1000 μm with strong evidence that the magnitude of difference between the two procedures increased as the S_{gw} of the sample increased. Stark and Chewning (2012) observed 76 μm (554 to 478 μm), 49 μm (659 to 610 μm), and 54 μm (886 to 832 μm) decreases when using sieve agitators and decreases of 149 μm (554 to 329 μm), 203 (659 to 407 μm), and 184 μm (886 to 648 μm) when using dispersing agent on fine, medium, and coarse hammermill ground corn, respectively. Thus, Stark and Chewning (2012) concluded that the addition of a dispersing agent better estimated the d_{gw} and S_{gw} than did the addition of sieve agitators.

The method for calculating the S_{gw} of samples was changed between ASAE S319.2 and ANSI/ASAE S319.3. ANSI/ASAE S319.4 used the method described in ANSI/ASAE S319.3. Although the method to calculate S_{gw} has changed, the range for 68% of the particles has remained the same in both methods. There was significant differences in main effects of method ($P \leq 0.05$) and grain ($P > 0.05$) for the S_{gw} according to ASAE S319.2 (Table 2.3), calculated using Eq. 2.3. The S_{gw} according ANSI/ASAE S319.4, calculated using Eq. 2.4, was also significant for method ($P \leq 0.05$) and grain ($P \leq 0.05$). The S_{gw} indicated the distribution of particles throughout the sieve stack so a greater the S_{gw} value indicates a greater distribution. The range for 68% of the particles describes the range within one standard deviation of the d_{gw} . The range

and variation of the particles increased with the use of sieve agitators and dispersing agent because sieve agitators and dispersing agent both facilitated the movement of small particles to the pan. This created a S_{gw} substantially greater ($P \leq 0.05$) when one or both were included in the analysis. Figure 2.1 illustrates the increase in range of particles facilitated by the addition of dispersing agent on moving particles to screens with small openings with the amount in the pan ($< 53 \mu\text{m}$) increasing by 10%. The same effect was observed throughout all samples with dispersing agent and was further supported by an increased S_{gw} .

The S_{gw} increased 0.39 (2.23 to 2.62) according to S319.2 and $94 \mu\text{m}$ (485 to 579 μm) according to S319.4 ($P \leq 0.05$) when both sieve agitators and dispersing agent were included with 10 min sieving time. With 15 min sieving time, S_{gw} increased 0.36 (2.27 to 2.63) according to S319.2 and $80 \mu\text{m}$ (487 to 567 μm) according to S319.4 ($P \leq 0.05$). However, there was no significant change in S_{gw} according to S319.2 or S319.4 when sieving time increased from 10 min to 15 min. Fahrenholz et al. (2010) reported the addition of sieve agitators increased S_{gw} according to ASAE S319.2 by 0.40 (2.00 to 2.40), dispersing agent increased by 0.36 (2.10 to 2.46) and a 0.16 (2.40 to 2.56) increase when sieving time was increased from 10 to 15 min. Goodband et al. (2006) reported that the addition of dispersing agent also increased the S_{gw} , calculated using ASAE S319.2, significantly ($P \leq 0.05$) in samples with a d_{gw} of 400 to 1,000 μm .

Due to the difference in how the S_{gw} was calculated in the current study, the differences among the grains changed. The S_{gw} according to ASAE S319.2, resulted in corn (2.36) and wheat (2.35) being similar, but significantly different from sorghum (2.40; $P \leq 0.05$). However, when S_{gw} was evaluated by ANSI/ASAE S319.4, corn (487 μm) and sorghum (492 μm) were similar, but different from wheat (572 μm ; $P \leq 0.05$).

Pearson Correlation Coefficients compared the goodness-of-fit for d_{gw} and S_{gw} for each grain to when d_{gw} and S_{gw} were calculated by subtracting the weight of dispersing agent from the weight of the pan (Table 2.5). For the reported means in the current study, dispersing agent, was not subtracted from the weight of the pan, as described in ANSI/ASAE S319.4. All of the dispersing agent was verified in the current study to reach the pan with 99.7% recovery ($n = 3$). Still, debate remained if the d_{gw} and S_{gw} were significantly different when the weight of the dispersing agent was subtracted versus

when it was not subtracted from the weight of the pan. Correlations were evaluated using the means and data from the method with the 10 minute sieving time with sieve agitators and dispersing agent. All grains were highly correlated for d_{gw} ($P \leq 0.05$; $r = 1.0000$), S_{gw} S319.2 ($P \leq 0.05$; $r > 0.9993$), and S_{gw} 319.4 ($P \leq 0.05$; $r > 0.9971$) with corn having the highest correlation (Table 2.5). Differences for d_{gw} were: 6, 7, and 9 μm for corn, sorghum, and wheat, respectively. Differences for S_{gw} S319.2 were 0.04 for corn, sorghum, and wheat. Differences for S_{gw} S319.4 were: 5, 11, and 13 μm for corn, sorghum, and wheat, respectively.

The results of this experiments present a challenge for feed and animal industries when comparing particle size research without knowing the method used to determine d_{gw} . The increase in sieving time (10 to 15 min) that occurred in ANSI/ASAE S319.3 (ASAE, 2007) was not widely adopted by the feed industry. Furthermore, recent scientific publications (Pacheco, et al., 2014; Paulk et al., 2015; Xu, et al., 2015) reported the use of a 10 minute sieving time. With the exception of Fahrenholz et al. (2010), all known reported particle size data has used a sieving time of 10 min. A literature review by Goodband et al. (2006) did not find reports or an indication that dispersing agent was used when reporting the d_{gw} of ground grains used in swine research studies on the effect of particle size reduction. While past research on animal performance has not reported the use of a dispersing agent, recent scientific publications have reported the use of dispersing agent in particle size analysis (De Jong et al., 2014; Pacheco, et al., 2014; and Xu, et al., 2015). Woodworth et al. (2001), Goodband et al. (2006), and Stark and Chewing (2012) described the type and arrangement of sieve agitators used in their respective analytical methods research. However, De Jong, et al. (2014) and Xu et al. (2015) were among the first researchers to report the use of sieve agitators used in animal research trials. Similar to the findings of Goodband et al., (2006) the number, type, and arrangement of the agitators on the sieves are not typically reported in animal research studies related to particle size.

Conclusion

In conclusion, results of this experiment indicated that sieve agitators and dispersing agent best facilitated the movement of material through the sieves and reduce

the agglomeration of fine particles on sieves with small openings. The data from this experiment suggests the use of sieve agitators arranged on sieves as depicted in Table 2.1 and the addition of 0.5 g dispersing agent provide a better estimate of particle size with a 10 min sieving time. Furthermore, a 15 min sieving time as described in ANSI/ASAE S319.4 may not be required when a dispersing agent is added to the sample. In order to accurately compare particle size analysis results and animal research related to difference in ground materials, the method must be accurately described.

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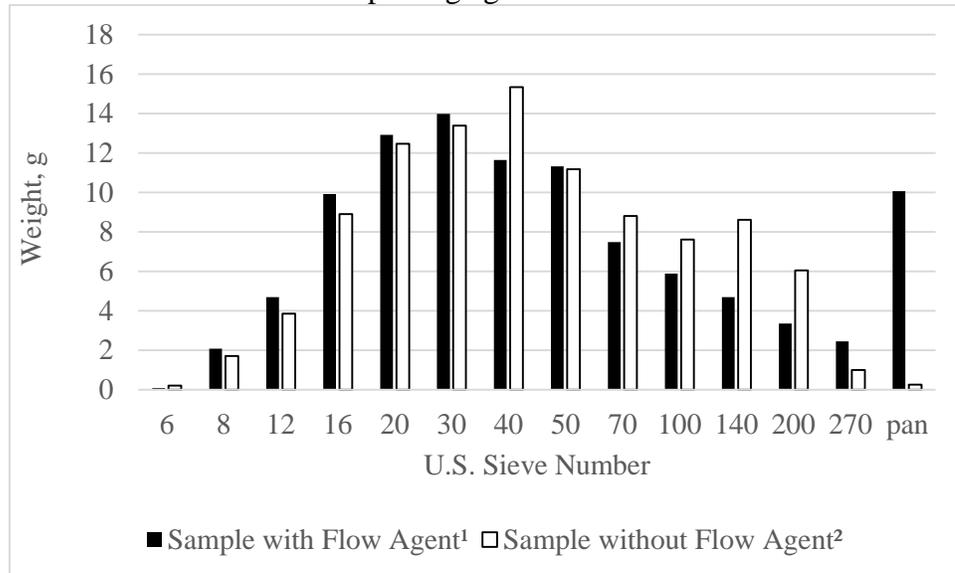
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Figures and Tables

Figure 2.1 Particle size distribution graph of a hammermill ground corn sample with and without the addition of a dispersing agent.



¹ d_{gw} : 402 μm ; S_{gw} (calculated using ANSI/ASAE S319.2): 3.11; S_{gw} (calculated using ANSI/ASAE S319.4): 561 μm .

² d_{gw} : 448 μm ; S_{gw} (calculated using ANSI/ASAE S319.2): 2.50; S_{gw} (calculated using ANSI/ASAE S319.4): 470 μm .

Table 2.1 Sieve and sieve agitator arrangement

U.S. sieve number	Sieve opening (μm)	Sieve agitator(s)
6	3,360	None
8	2,380	None
12	1,680	Three rubber balls
16	1,190	Three rubber balls
20	841	Three rubber balls
30	595	One rubber ball; one bristle sieve cleaner
40	420	One rubber ball; one bristle sieve cleaner
50	297	One rubber ball; one bristle sieve cleaner
70	210	One rubber ball; one bristle sieve cleaner
100	149	One bristle sieve cleaner
140	105	One bristle sieve cleaner
200	74	One bristle sieve cleaner
270	53	One bristle sieve cleaner
Pan	-	None

Table 2.2 Interaction effects of method \times grain type on geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw})¹

	Method					SEM	P =
	10	10	15	15	15		
Sieving time, min							
Sieve agitator inclusion	Yes	Yes	No	Yes	Yes		
Dispersing agent inclusion	No	Yes	No	No	Yes		
Geometric mean diameter (d_{gw}), μm						225	0.172
Corn	544	498	586	530	486		
Wheat	656	640	682	647	623		
Sorghum	559	524	577	551	512		
Geometric standard deviation (S_{gw}), μm							
ASAE S319.2						0.32	< 0.0001
Corn	2.20 ^{de}	2.67 ^a	1.97 ^f	2.26 ^{cd}	2.69 ^a		
Wheat	2.26 ^{cd}	2.55 ^b	2.16 ^e	2.27 ^c	2.55 ^b		
Sorghum	2.24 ^{cd}	2.65 ^a	2.16 ^e	2.29 ^c	2.65 ^a		
ANSI/ASAE S319.4						117	0.931
Corn	452	554	427	459	542		
Wheat	548	631	528	540	614		
Sorghum	455	552	448	462	545		

¹A total of 360 particle size analytical procedures were conducted in this experiment, with 18 samples each of corn, sorghum, and wheat. Subsamples of each grain type were then analyzed using 5 different variations of the ANSI/ASAE S319.4 standard particle size analysis method. There were 4 replicates per method.

^{abcdef}Means with different superscripts differ $P < 0.05$.

Table 2.3 Main effect of analytical method on geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) of various grains¹

	Method					SEM	P =	Orthogonal contrasts		
	10	10	15	15	15			Sieving time	Sieve agitators	Dispersing agent
Sieving time, min	10	10	15	15	15					
Sieve agitator inclusion	Yes	Yes	No	Yes	Yes					
Dispersing agent inclusion	No	Yes	No	No	Yes					
Geometric mean diameter (d_{gw}), μm^2	586 ^b	554 ^c	615 ^a	576 ^b	540 ^c	223	<0.0001	0.125	<0.0001	<0.0001
Geometric standard deviation (S_{gw}) ³										
ASAE S319.2	2.23 ^b	2.62 ^a	2.09 ^c	2.27 ^b	2.63 ^a	0.32	<0.0001	<0.0001	<0.0001	<0.0001
ANSI/ASAE S319.4, μm^4	485 ^{bc}	579 ^a	467 ^c	487 ^b	567 ^a	116	<0.0001	N/A	N/A	N/A

¹A total of 360 particle size analytical procedures were conducted in this experiment, with 18 samples each of corn, sorghum, and wheat. Subsamples of each grain type were then analyzed using 5 different variations of the ANSI/ASAE S319.4 standard particle size analysis method. There were 4 replicates per method.

²Orthogonal contrasts included sieving time 10 vs. 15 min; with or without sieve agitators; and with or without dispersing agent.

³Orthogonal contrasts included sieving time 10 vs. 15 min; with or without sieve agitators; and with or without dispersing agent.

⁴Orthogonal contrasts were not determined because calculations were not conducted at the time of analysis.

^{abc}Means within a row without common superscripts differ $P < 0.05$.

Table 2.4 Main effect of grain type on geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) of grains¹

	Corn	Sorghum	Wheat	SEM	<i>P</i> =
Geometric mean diameter (d_{gw}), μm	529 ^c	545 ^b	649 ^a	223	< 0.0001
Geometric standard deviation (S_{gw})					
ASAE S319.2	2.36 ^b	2.40 ^a	2.35 ^b	0.32	0.025
ANSI/ASAE S319.4, μm	487 ^b	492 ^b	572 ^a	116	< 0.0001

¹A total of 360 particle size analytical procedures were conducted in this experiment, with 18 samples each of corn, sorghum, and wheat. Subsamples of each grain type were then analyzed using 5 different variations of the ANSI/ASAE S319.4 standard particle size analysis method. There were 4 replicates per method.

^{abc}Means within a row without common superscripts differ $P < 0.05$.

Table 2.5 Pearson Correlation Coefficients for d_{gw} and S_{gw} using the means and data from the method with 10 minute sieving time with sieve agitators and dispersing agent compared to when dispersing agent was subtracted from the weight of the pan^{1, 2}

	Grain Type					
	Corn		Sorghum		Wheat	
	With	Without	With	Without	With	Without
Geometric mean diameter (d_{gw}), μm	511	517	519	526	644	653
Pearson Correlation Coefficient						
$P =$	< 0.0001		< 0.0001		< 0.0001	
$r =$	1.0000		1.0000		1.0000	
Geometric standard deviation (S_{gw})						
ASAE S319.2	2.67	2.63	2.66	2.62	2.52	2.48
Pearson Correlation Coefficient						
$P =$	< 0.0001		< 0.0001		< 0.0001	
$r =$	0.9995		0.9994		0.9993	
ANSI/ASAE S319.4, μm	564	559	557	546	627	614
Pearson Correlation Coefficient						
$P =$	< 0.0001		< 0.0001		< 0.0001	
$r =$	0.9997		0.9996		0.9971	

¹A total of 360 particle size analytical procedures were conducted in this experiment, with 18 samples each of corn, sorghum, and wheat. Subsamples of each grain type were then analyzed using 5 different variations of the ANSI/ASAE S319.4 standard particle size analysis method. There were 4 replicates per method.

²Pearson Correlation Coefficients evaluated the goodness-of-fit for each grain compared to when the weight of dispersing agent was subtracted from the pan weight for the method with a 10 minute sieve time with sieve agitators and dispersing agent

Chapter 3 - Effects of varying methodologies on grain particle size using a three sieve method for particle size analysis

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Abstract

Particle size reduction is an important component of feed manufacturing that impacts pellet quality, feed flow ability, and animal performance. The estimation of particle size is an important quality control measurement for feed manufacturers, nutritionists, and producers. ANSI/ASAE S319.4 is the current approved method for determining the geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) of grains. This method controls many variables, including the suggested quantity of initial material and the type, number, and size of sieves. However, the method allows for variation in sieving time, sieve agitators, and the use of a dispersing agent. While this method works well in a laboratory setting, it may not be practical for quality control measurement in a feed mill due to the absence of an on-site lab and initial cost of investment. The objective of this experiment was to determine which alternative method best estimates the particle size of ground corn compared to the 13-sieve method. Treatments were arranged in a 3×2 factorial arrangement with fixed effects of 3 sieving times (30, 60, and 90 s) with or without dispersing agent. For the 3-sieve method, sieving time referred to the time sieves were shaken side to side by hand. Random effects were 3 grind sizes (coarse, medium, or fine) and 2 mill types (hammermill or roller mill). Results for d_{gw} were calculated two ways: 1) according to standard method ANSI/ASAE S319.4, and 2) the regression equation developed by Baldrige et al. (2001).

There was no sieving time \times dispersing agent interaction for d_{gw} ($P > 0.05$) so it was removed. The main effects of sieving time and dispersing agent level differed ($P < 0.05$) for d_{gw} . Increasing sieving time resulted in decreases of 23 μm and 17 μm as sieving time increased from 30 to 60 to 90 s, respectively. This resulted in an overall decrease of 40 μm when sieving time increased from 30 to 90 s. Adding dispersing agent (0.25 g), resulted in a 27 μm decrease in d_{gw} . The regression equation developed by

Baldrige et al. (2001) was not developed to calculate S_{gw} , so main effects and means were not determined. Pearson Correlation Coefficients compared the goodness-of-fit of both analytical models compared to the ASAE Standard 13-sieve method. Both methods were highly correlated ($P \leq 0.01$; $r > 0.97$), with the standard method ANSI/ASAE S319.4 having the greatest correlation value.

The results of this experiment indicate that when performing particle size analysis using the 3-sieve method, the sample should be shaken side to side by hand for 90 s with sieve agitators and 0.25 g dispersing agent. The data confirmed that both the regression equation and ANSI/ASAE S319.4 standard method were slightly correlated with the 13-sieve method for 200 to 1,000 μm samples.

Keywords: Corn, feed, grain, methodology, particle size analysis

Introduction

Particle size reduction is an important component of feed manufacturing that impacts pellet quality, feed flow ability, and animal performance. The estimation of particle size is an important quality control measurement for feed manufacturers, nutritionists, and producers. ANSI/ASAE S319.4 is the current approved method for determining the geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) of grains. This method controls many variables, including the suggested quantity of initial material and the type, number, and size of sieves. However, the method allows for variation in sieving time, sieve agitators, and the use of a dispersing agent. While this method works well in a laboratory setting, it may not be practical for quality control measurement in a feed mill due to the absence of an on-site lab and initial cost of investment. Therefore, a 3-sieve method was developed by Kansas State University to allow for a quick and easy estimation of particle size to be determined. The geometric mean diameter (d_{gw}), commonly termed particle size, is determined with the 13-sieve standard method (ANSI/ASAE S319.4). The 3-sieve method was developed to allow mill operators to quickly estimate the d_{gw} while setting the roller mill or hammermill. Baldrige et al. (2001) refined the one-sieve method developed by Iowa Farm Automation Ltd (IFA, Stanley, IA) to develop a regression equation to predict the particle

size using 3 sieves for 600 μm corn samples. However, the addition of dispersing agent with varying sieving times when shaken by hand had not been previously reported. Therefore, the objective of this experiment was to determine which alternative method best estimates the particle size of ground corn compared to the 13-sieve method. Results for d_{gw} were calculated two ways: 1) according to the standard method ANSI/ASAE S319.4, and 2) the regression equation developed by Baldrige et al. (2001). The standard 13-sieve used sieve agitators, dispersing agent, and a sieving time of 10 min for particle size analysis.

Materials and Methods

Material Preparation

Whole corn was ground using 2 mill types consisting of a hammermill (Model 22115, Bliss Industries LLC., Ponca City, OK) and roller mill (Model 924, RMS Roller Grinder, Harrisburg, SD). The hammermill was equipped with 1.59 mm, 4.76 mm, and 6.35 mm screens for fine, medium and coarse grinds, respectively. The roller mill rolls were 2.36 and 2.36 corrugations/cm, 4.72 and 5.51 corrugations/cm, and 6.30 and 7.09 corrugations/cm roll on the top, middle, and bottom roll pairs, respectively. Samples were prepared and collected at the O.H. Kruse Feed Technology Innovation Center at Kansas State University. The differences in mill type and grind size was intended to create a robust set of ground corn samples, but were made random effects due to their natural confounding with the response criterion.

Sample Analysis

Particle size was determined according to the ANSI/ASAE S319.4 standard method with 10 minute sieving time, sieve agitators, and dispersing agent in the Swine Laboratory at Kansas State University. Samples were divided using a riffle divider to 100 ± 5 g. The analysis was conducted with a stainless steel sieve stack (13-sieves). The sieve stack contained sieve agitators with bristle sieve cleaners and rubber balls measuring 16 mm in diameter (Table 3.1). Sieves were cleaned after each analysis with compressed air and a stiff bristle sieve cleaning brush.

Each sieve was individually weighed with the sieve agitators to obtain a tare weight. The 100 ± 5 g sample was then placed on the top sieve. Dispersing agent (Model SSA-58, Gilson Company, Inc., Lewis Center, OH) was added (0.5 g) and mixed into the 100 ± 5 g sample and placed on the top sieve. The sieve stack was then placed in the Ro-Tap machine (Model RX-29, W. S. Tyler Industrial Group, Mentor, OH) and ran for 10 min. Once completed, each sieve was individually weighed with the sieve agitator(s) to obtain the weight of the sample on each sieve. The weight of the dispersing agent was not subtracted from the weight of the pan, as specified in the ANSI/ASAE S319.4. The results for d_{gw} were calculated according to ANSI/ASAE S319.4.

For the 3-sieve particle size analysis method, samples were divided using a riffle divider to 50 ± 5 g. Each sieve was individually weighed with the sieve agitators to obtain a tare weight. The sieve agitators were bristle sieve cleaners and rubber balls measuring 16 mm in diameter (Table 3.2). The 50 ± 5 g sample was then placed on the top sieve. If dispersing agent (Model SSA-58, Gilson Company, Inc., Lewis Center, OH) was required (0.25 g), it was mixed into the sample prior to placing the mixture on the top sieve. The sieve stack was then hand shaken side-to-side by a technician for the specified amount of time. Once completed, each sieve was weighed individually again with the sieve agitator(s) to obtain the weight of the sample on each sieve. To maintain consistency with the 13-sieve method, the weight of dispersing agent was not subtracted from the weight of the pan. Sieves were cleaned after each analysis with a stiff bristle sieve cleaning brush. Sieves and sieve agitators were cleaned with soapy water and allowed to dry to prevent residual dispersing agent affecting the results of subsequent samples without dispersing agent. The amount of material on each sieve was then used to calculate the d_{gw} .

The d_{gw} results were calculated two ways, 1) ANSI/ASAE standard S319.4 method (Eq. 3.1); and 2) the regression equation developed by Baldrige et al. (2001) (Eq. 3.2).

$$d_{gw} = \log^{-1} \left[\frac{\sum_{i=1}^n (W_i \log \bar{d}_i)}{\sum_{i=1}^n W_i} \right] \quad (\text{Eq. 3.1})$$

where W_i is mass on i^{th} sieve, g

d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} is geometric mean diameter or median size of particles by mass, mm, or

geometric mean diameter or median size of particles on i^{th} sieve, mm

n is number of sieves +1 (pan)

$$d_{\text{gw}} = (18.832 \times A) + (10.870 \times B) + (1.1827 \times C) - 149.978 \quad (\text{Eq. 3.2})$$

where d_{gw} is the geometric mean diameter or median size of particles by mass, mm,

or geometric mean diameter or median size of particles on i^{th} sieve, mm

A represents the percentage of sample on the number 12 sieve

B represents the percentage of sample on the number 30 sieve

C represents the percentage of sample on the number 50 sieve

Experimental Design

Treatments were arranged in a 3×2 factorial arrangement with fixed effects being 3 sieving times (30, 60, and 90 s) with or without dispersing agent. Random effects were 3 grind sizes (coarse, medium, or fine) and 2 mill types (hammermill or roller mill). The results from this experiment were compared to the results when samples were analyzed using the ANSI/ASAE S319.4 Standard 13-sieve method.

Fixed and random effects were included in experimental design. Data were analyzed using GLIMMIX procedure of SAS (SAS Institute, Inc., Cary, NC). Interactions were removed from the model if $P > 0.05$. Results were considered significant if $P \leq 0.05$, and a tendency if $0.05 \leq P \leq 0.10$. The CORR procedure of SAS was used to determine Pearson Correlation Coefficients for d_{gw} results using the ANSI/ASAE S319.4 Standard 13-sieve method. Linear regression by the REG procedure of SAS was used to develop equations to predict the mean particle size of the 3-sieve method compared to the standard 13-sieve method for both 1) the regression equation developed by Baldrige et al. (2001) and 2) the standard ANSI/ASAE standard method S319.4.

Results and Discussion

The sieving time \times dispersing agent interaction for d_{gw} ($P > 0.05$) was not significant (Table 3.3), so it was removed. In contrast to Bokelman et al. (2015), results from this experiment determined that results for d_{gw} on ground corn samples differed based on sieving time (30, 60, and 90 s) and with the inclusion of dispersing agent (0.25

g). The main effects of sieving time and dispersing agent level differed when calculated according to the regression equation developed by Baldrige, et al. (2001) ($P \leq 0.05$; Table 3.3). Increasing sieving time resulted in decreases of 23 μm (491 to 467 μm) and 17 μm (467 to 451 μm) when sieving time increased from 30 to 60 to 90 s, respectively. This resulted in an overall decrease of 40 μm (491 to 451 μm) when sieving time increased from 30 to 90 s. The d_{gw} decreased 27 μm (483 to 456 μm) with the addition of dispersing agent.

Previous research also observed that the addition of dispersing agent consistently resulted in a lower d_{gw} in the 13-sieve method (Goodband et al., 2006; Fahrenholz et al., 2010; Stark and Chewning, 2012). Goodband et al. (2006) determined that when using a dispersing agent, the d_{gw} value was consistently 80 μm less than when a dispersing agent was not used in particle size analysis for the 13-sieve method. Similar to the results of this experiment, Woodworth et al. (2002) determined that the addition of sieve agitators resulted in a lower d_{gw} because the sieve agitators broke up agglomerates and aided in the flow ability of the sample throughout the sieve stack. Woodworth et al. (2001) explained that the sieve agitators assisted in the movement of particles through the sieve openings by preventing the buildup on the sieves and passage of the particles to the next sieve, without breaking the particles into smaller pieces or forcing particles them through the sieve. The sieve agitator used in particle size analysis has not been frequently described in previous research. However, Woodworth et al. (2001) and Stark and Chewning (2012) both described the type and number of sieve agitators used in their respective analysis procedures, which allowed for more accurate comparisons to be made.

Results from this experiment were used to perform linear regression comparing results from the 13-sieve method to the 3-sieve method calculated two ways: 1) according to standard method ANSI/ASAE S319.4, and 2) the regression equation developed by Baldrige et al. (2001) (Fig. 3.1). Figure 3.1 depicts that both calculation methods resulted in similar accuracy at 600 to 800 μm , which is logical since the regression equation developed by Baldrige et al. (2001) was for samples at 600 μm . As particle size increased to 1000 μm , the difference in the calculations increased slightly. As particle size decreased to 400 μm , the results between the calculations began to separate slightly and completely separated at 200 μm . The regression equation developed by

Baldrige et al. (2001) underestimated particle size while the standard ANSI/ASAE method equation overestimated particle size at 200 μm .

Pearson Correlation Coefficients compared the goodness-of-fit of both analytical models compared to the ASAE Standard 13-sieve method. Both models were highly correlated ($P < 0.05$; $r > 0.97$; Table 3.3), with the standard ANSI/ASAE S319.4 method having the greatest correlation value. The regression equation developed by Baldrige et al. (2001) was not developed to calculate S_{gw} , so main effects and means were not determined.

Conclusion

The results of this experiment indicate the sample should have a sieving time of 90 s with sieve agitators and 0.25 g dispersing agent for 3-sieve particle size analysis. The data confirmed that both the regression equation and ANSI/ASAE S319.4 standard method were highly correlated with the 13-sieve results for samples from 200 to 1,000 μm , with the ANSI/ASAE S319.4 standard method having a slightly higher correlation value.

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Figures and Tables

Figure 3.1 Relationship in prediction of geometric mean diameter (d_{gw}) using a 3-sieve method compared to ANSI/ASAE Standard 13-sieve method

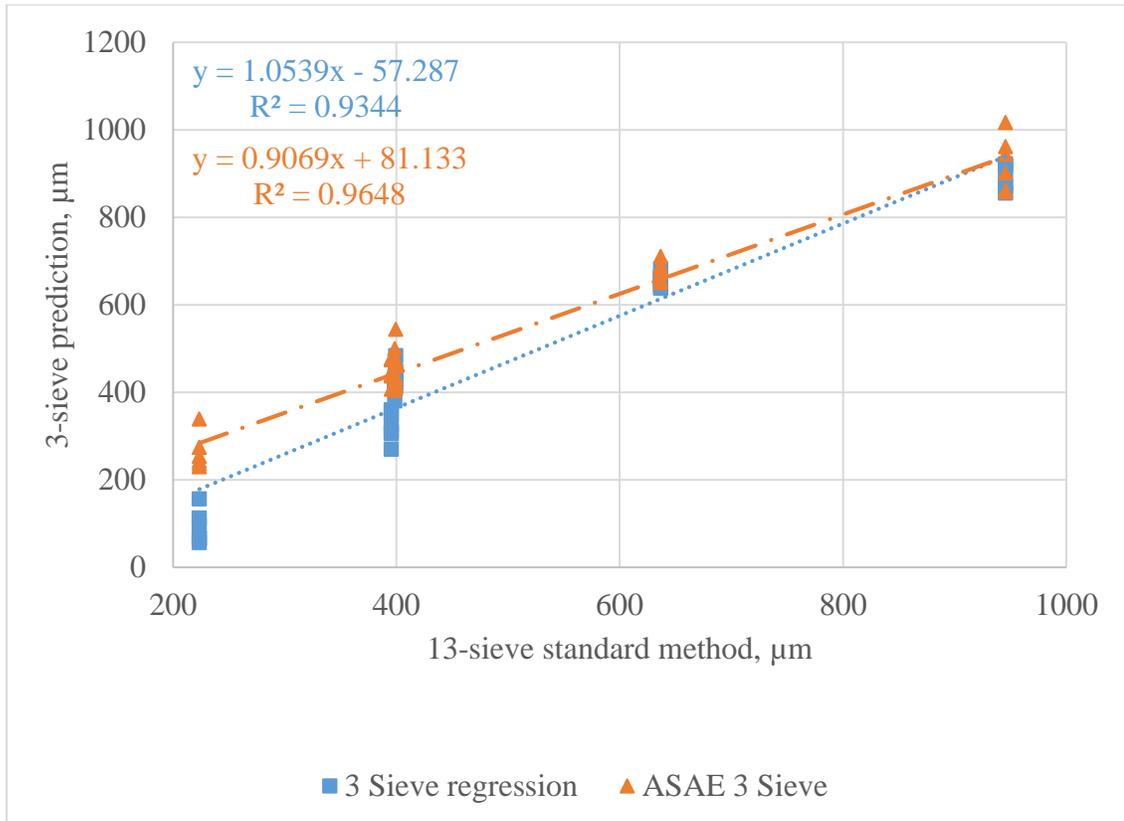


Table 3.1 Sieve and sieve agitator arrangement for ANSI/ASAE Standard 13-sieves method

U.S. sieve number	Sieve opening (μm)	Sieve agitator(s)
6	3,360	None
8	2,380	None
12	1,680	Three rubber balls
16	1,190	Three rubber balls
20	841	Three rubber balls
30	595	One rubber ball; one bristle sieve cleaner
40	420	One rubber ball; one bristle sieve cleaner
50	297	One rubber ball; one bristle sieve cleaner
70	210	One rubber ball; one bristle sieve cleaner
100	149	One bristle sieve cleaner
140	105	One bristle sieve cleaner
200	74	One bristle sieve cleaner
270	53	One bristle sieve cleaner
Pan	-	None

Table 3.2 Sieve and sieve agitator arrangement for 3-sieve method

U.S. sieve number	Sieve opening (μm)	Sieve agitator(s)
12	1,680	None
30	595	One rubber ball; one bristle sieve cleaner
50	297	One rubber ball; two bristle sieve cleaners
Pan	-	None

Table 3.3 Main effects of analytical method on geometric mean diameter (d_{gw})^{1,2}

	Sieving time, s			SEM	<i>P</i> =	Dispersing agent			SEM	<i>P</i> =
	30	60	90			Yes	No			
Geometric mean diameter (d_{gw}), μm	491 ^a	467 ^b	451 ^c	203	< 0.0001	456 ^b	483 ^a	203	< 0.0001	
	Sieving time \times Dispersing agent									
	Sieving time, s	30	60	90	30	60	90			
	Dispersing agent	Yes	Yes	Yes	No	No	No	SEM	<i>P</i> =	
Geometric mean diameter (d_{gw}), μm		477	452	440	505	483	462	203	0.765	

¹A total of 36 particle size analytical procedures were conducted in this experiment with varying sieving time and the addition of dispersing agent on ground corn samples. Results were calculated according to the regression equation developed by Baldrige, et al. (2001).

²Pearson Correlation Coefficients for the results from using the 13-sieve method to regression equation developed by Baldrige, et al. (2001) ($r = 0.9709$) and equation from ANSI/ASAE S319.4 ($r = 0.9948$).

^{abc}Means within a row without common superscripts differ $P < 0.05$.

Chapter 4 - Effects of grain particle size and flow ability from differing levels of added screenings

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Abstract

Corn cleaning prior to grinding to remove the cobs and stalks, otherwise known as screenings, may vary based on mill operating procedures. Grates over the receiving pit remove large physical hazards of foreign material, while magnets are placed in the receiving flow to remove ferrous metals. The combination of grates and magnets help prevent damage to the equipment and harm to animals and employees.

The objective of this experiment was to compare the particle size and flow ability characteristics of ground corn at two moisture contents with three levels of screenings. The particle size or geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) were determined since they are often used as quality control measurements. The d_{gw} was determined according to standard method ANSI/ASAE S319.4 and S_{gw} according to standard methods ASAE S319.2 and ANSI/ASAE S319.4. The flow ability characteristics analyzed included: compressibility, angle of repose, critical orifice diameter, shear, composite flow index (CFI), and bulk density. Treatments were arranged in a 2×3 factorial arrangement with 2 moisture contents, (12 and 10%), and 3 levels of screenings (0, 2.5, and 5%). Data were analyzed using the GLIMMIX procedure of SAS with three replicates per treatment.

There was no moisture \times screenings interaction ($P > 0.05$), so it was removed. The level of screenings was not significant for d_{gw} , S_{gw} , and measures of flow ability ($P > 0.05$). Moisture content was not significant for d_{gw} and measures of flow ability ($P > 0.05$). However, moisture content was significant for S_{gw} method S319.2 ($P \leq 0.05$) and S_{gw} method S319.4 ($P \leq 0.05$). The S_{gw} method S319.2 had a mean estimate of 2.72 for

12% moisture and 2.45 for 10% moisture. The S_{gw} method S319.4 had a mean estimate of 744 μm for 12% moisture and 630 μm for 10% moisture. When analyzed for moisture content, moisture content was significant ($P \leq 0.05$), but was not significant for screenings ($P > 0.05$).

The results of this experiment suggest that cleaning corn prior to grinding with a roller mill does not change particle size or flow ability characteristics. The S_{gw} reflected the distribution and variation of the particles with a greater S_{gw} indicating a wider distribution of particles. While the d_{gw} was not significant for moisture or screenings, it should still be used as a quality control measurement in the grinding process.

Key Words: corn, feed, grain, flow ability, particle size analysis

Introduction

The particle size or geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) are often used as quality control measurements in the grinding process. Maintaining a consistent product is especially difficult when grains are ground using a roller mill due to variations in grain, such as: kernel size, moisture content, source, and amount of handling. Equipment manufacturers have recommended to check grain particle size periodically while grinding to maintain consistency (RMS, 2012). External factors, separate from the grain itself that impacted grain particle size include: equipment operator, facility design, and environmental conditions.

Grates over the receiving pit remove large physical hazards of foreign material, while magnets placed in the receiving flow remove ferrous metals. The combination of grates and magnets help prevent damage to equipment and harm to animals and employees. Cleaning corn before grinding to remove the cobs and stalks, otherwise known as screenings, may vary based on equipment type and mill operating procedures. Roller mills typically have a scalper (12.7 to 25.4 mm) above the rolls to remove cobs and stalks but are not designed to remove fine particles that would be removed by a grain cleaner.

The objective of this experiment was to compare the particle size and flow ability characteristics of ground corn at two moisture contents from two crop years with three

levels of screenings. The flow ability characteristics analyzed included: compressibility, angle of repose, critical orifice diameter, shear, composite flow index (CFI), and bulk density.

Flow ability is affected more by physical characteristics than by chemical properties (Haque, 2010). Physical characteristics that affect the flow ability include: d_{gw} , S_{gw} , particle shape, and electrostatic charge (Haque, 2010). Composite flow index (CFI) is calculated by combining the results for angle of repose, compressibility, and critical orifice diameter (Horn, 2008). Angle of repose is defined and calculated by determining the angle between the free standing platform of the sample pile and the height of the pile (Fig. 4.1; Appel, 1994). According to a suggested scale developed by Horn (2008), flow ability decreases as CFI and angle of repose increase. Compressibility and shear are analyzed using a Powder Rheometer (Model FT4, Freeman Technologies, Gloucestershire, United Kingdom) with shear measured according to ASTM Standard D7891. Shear determines the behavior of a sample as it transitions from a non-flowing state to a flowing state (Freeman Technology, 2015). Compressibility measures the change in volume of the sample when increasing levels of compressive force are applied, and is influenced by factors including: particle size distribution, particle shape, and particle texture (Freeman Technology, 2015). The critical orifice diameter is determined using a powder flow ability test instrument (Flodex Model WG-0110, Paul N. Gardner Company, Inc., Pompano Beach, FL). Bulk density reflects how the sample behaves when it transitions from a non-flowing state to a flowing state back to a non-flowing state.

Materials and Methods

Material Preparation

Whole corn was cleaned using a grain cleaner (Model S 206, Carter Day International, Inc., Minneapolis, MN) upon receiving. Screening treatments were based on the amount of screenings obtained while receiving the whole corn. Screenings were added back into whole corn, mixed, and then ground using a roller mill (Model 924, RMS Roller Grinder, Harrisburg, SD). The roller mill rolls were 2.36 and 2.36 corrugations/cm, 4.72 and 5.51 corrugations/cm, and 6.30 and 7.09 corrugations/cm roll

on the top, middle, and bottom roll pairs, respectively. The roll gap settings and feed rate were kept constant for all samples. Production rate was calculated and obtained by recording the amount of time required to grind each sample. Specific energy consumption (SEC_E) was calculated using Eq. 4.1 Stark (1994) based on production rate and total amperage for each motor on the 3-high roller mill for each replication.

$$SEC_E = \frac{I \times E \times PF \times 1.73}{PR \times 1000} \quad \text{Eq. 4.1}$$

where I is amperage

E is voltage

PF is power factor set to 0.85

PR is production rate expressed in Mton/hr

Sample Analysis

Each ground sample was divided using a riffle divider to approximately 500 ± 5 g for each analysis. The 500 ± 5 g sample was then divided using a riffle divider to reach the appropriate sample size requested for each analysis. Particle size was determined according to ANSI/ASAE S319.4. Calculations were performed according to the equations listed and described in ANSI/ASAE standard S3219.4 for d_{gw} and S_{gw} and ASAE standard S319.2 for S_{gw} . Flow ability characteristics analyzed included: compressibility, angle of repose, critical orifice diameter, shear, CFI, and bulk density.

Composite flow index was calculated by combining the results for angle of repose, compressibility, and critical orifice diameter (Horn, 2008). Angle of repose was determined by weighing 200 ± 5 g of sample and allowing it to flow from a funnel 15.24 cm above a free standing platform, 13 cm in diameter. The angle between the free standing platform of the sample pile and the height of the pile was calculated by taking the inverse tangent of the height of the pile divided by the platform radius. (Fig. 4.1; Appel, 1994). The critical orifice diameter was determined using a powder flow ability test instrument (Flodex Model WG-0110, Paul N. Gardner Company, Inc., Pompano Beach, FL). Fifty grams of sample was measured and allowed to flow through a stainless steel funnel into a cylinder. The sample rested for 30 s in the cylinder, and then evaluated based on the flow through an opening in a horizontal disc. The discs were 6 cm in diameter and the interior hole diameter ranged from 4 to 34 mm. A negative result was

recorded when the sample did not flow through the opening in the disc or formed a cylindrical hole (Fig. 4.2). The disc hole size diameter was then increased by one disc size until a positive result was observed. A positive result was recorded when the material flowed through the disc opening forming an inverted cone shape (Fig. 4.3). If a positive result was observed, the disc hole size diameter was decreased until a negative result was observed. Three positive results were used to determine the critical orifice diameter. Compressibility and shear were analyzed using a Powder Rheometer (Model FT4, Freeman Technologies, Gloucestershire, United Kingdom). The sample for these analyses was placed in a 50 mm × 85 mL glass split vessel. The compressibility analysis used a vented piston to compress the sample to achieve 15 kPa of consolidating stress and held for 60 s and the percentage change in volume was calculated (Freeman Technology, 2006). Shear stress was measured according to ASTM Standard D7891, which analyzed the stress acting parallel to the surface of a plane and determined how the sample behaved by applying different states of strain and stress (ASTM, 2014). Before each analysis, a twisted blade was used to condition the sample and generate repeatable stress conditions within the sample. Shear stress analysis used a shear head consisting of 18 blades to generate shearing within the sample. The shear head moved at a rate of 0.5 mm/s until it reached the surface of sample. The speed was decreased to 0.08 mm/s until the 15 kPa of consolidating stress was reached again and held for 60 s. Shear results were expressed as a flow function, which depicted how flow ability changed as the compaction load changed (McGregor, 2010). On the x-axis was the control parameter that regulated the test, principal consolidation stress, which was directly related to the applied compaction load (McGregor, 2015). On the y-axis was the unconfined failure strength, which was directly related to the stress required to cause the particles to flow against each other (McGregor, 2015).

The d_{gw} and S_{gw} of the sample were determined according to the ANSI/ASAE S319.4 standard particle size analysis method. A 100 ± 5 g sample was sieved with a stainless steel sieve stack (13-sieves) containing sieve agitators with bristle sieve cleaners and rubber balls measuring 16 mm in diameter (Table 4.1). Each sieve was individually weighed with the sieve agitators to obtain a tare weight. Dispersing agent (Model SSA-58, Gilson Company, Inc., Lewis Center, OH; 0.5 g) was mixed with the sample and then

placed on the top sieve. The sieve stack was placed in the Ro-Tap machine (Model RX-29, W. S. Tyler Industrial Group, Mentor, OH) and run for 15 min. Once completed, each sieve was individually weighed with the sieve agitator(s) to obtain the weight of sample on each sieve. The weight of the dispersing agent was not subtracted from the weight of the pan as specified in the ANSI/ASAE S319.4. Sieves were cleaned after each analysis with compressed air and a stiff bristle sieve cleaning brush. Calculations were performed according to the equations listed and described in ANSI/ASAE standard S3219.4 (Eq. 4.1 to 4.4) for d_{gw} and S_{gw} and ASAE standard S319.2 for S_{gw} (Eq. 4.3). Equations 4.5 and 4.6 depict how to calculate the range for 68% of the particles with both using the d_{gw} calculated with Eq. 4.1 and 4.2. Equation 4.5 uses the S_{gw} calculated with Eq. 4.3, while Eq. 4.6 uses the S_{gw} calculated with Eq. 4.4.

$$d_{gw} = \log^{-1} \left[\frac{\sum_{i=1}^n (W_i \log \bar{d}_i)}{\sum_{i=1}^n W_i} \right] \quad (\text{Eq. 4.1})$$

where W_i is mass on i^{th} sieve, g

d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} is geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 4.2

n is number of sieves +1 (pan)

$$\bar{d}_i = (d_i \times d_{i+1})^{1/2} \quad (\text{Eq. 4.2})$$

where d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{i+1} is nominal sieve aperture size in next larger than i^{th} sieve (just above in a set), mm

$$S_{\log} = \left[\frac{\sum_{i=1}^n W_i (\log \bar{d}_i - \log d_{gw})^2}{\sum_{i=1}^n W_i} \right]^{1/2} = \frac{S_{\ln}}{2.3} \quad (\text{Eq. 4.3})$$

where W_i is mass on i^{th} sieve, g

d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} is geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 4.2

S_{\log} is geometric standard deviation of log-normal distribution by mass in ten-based logarithm, dimensionless

S_{\ln} is geometric standard deviation of log-normal distribution by mass in natural logarithm, dimensionless

n is number of sieves +1 (pan)

$$S_{gw} \approx \frac{1}{2} d_{gw} \left[\log^{-1} S_{\log} - (\log^{-1} S_{\log})^{-1} \right] \quad \text{(Eq. 4.4)}$$

where W_i is mass on i^{th} sieve, g

d_i =nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} = geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 4.2

S_{\log} is geometric standard deviation of log-normal distribution by mass in ten-based logarithm, dimensionless

S_{gw} is geometric standard deviation of particle diameter by mass, mm

$$\frac{d_{gw}}{S_{gw}} = \text{lower limit} \quad d_{gw} \times S_{gw} = \text{upper limit} \quad \text{(Eq. 4.5)}$$

where 68% of the particles are determined by finding the difference between the upper and lower limits using the S_{gw} from Eq. 4.3

d_{gw} = geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 4.2

$$S_{gw} \times 2 = 68\% \text{ of particles} \quad \text{(Eq. 4.6)}$$

Where 68% of the particles are determined using the S_{gw} from Eq. 4.4

Bulk density was determined using a quart test weight cup (Seedboro Equipment Company, Des Plaines, IL) that met the specifications of Grain Inspection, Packers and Stockyards Administration (GIPSA). The sample was placed into a stainless steel funnel and dropped into the bulk density cup until it overflowed around the circumference of the

cup. A wood leveling stick was used to remove excess sample and level the sample with the top of the cup. The weight of the sample in the cup was recorded.

Moisture content was determined according to AOAC Official Method 930.15. Air oven temperature was regulated to 135 ± 2 °C. Two grams of sample were weighed and shaken until evenly distributed in an Al dish, 50 mm in diameter and 40 mm deep. The samples were placed in the oven separately and allowed to dry for $2 \text{ h} \pm 5 \text{ min}$. The samples were transferred to a desiccator to cool. Loss in weight on drying (LOD) was calculated as an estimate of moisture.

Experimental Design

Treatments were arranged in a 2×3 factorial arrangement with fixed effects of moisture contents (12 or 10%), determined by using corn from two different crop years and screenings (0, 2.5, and 5%). Three replications of each treatment were ground and analyzed for flow ability characteristics and particle size. Flow ability characteristics analyzed included: compressibility, angle of repose, critical orifice diameter, shear, CFI, and bulk density. Particle size calculations were performed according to the equations listed and described in ANSI/ASAE standard S3219.4 for d_{gw} and S_{gw} and ASAE standard S319.2 for S_{gw} . Data were analyzed using GLIMMIX procedure of SAS (SAS Institute, Inc., Cary, NC) with three replicates per treatment. Interactions were removed from the model if $P > 0.05$. Results were considered significant if $P \leq 0.05$, and a tendency if $0.05 \leq P \leq 0.10$. The least significance difference test was used to evaluate differences in means.

Results and Discussion

The SEC_E was significant for moisture content ($P \leq 0.05$) with 10% moisture having a lower SEC_E , but no difference was observed for the amount of screenings ($P > 0.05$; Table 4.2). Production rate tended to differ for the amount of screenings ($P < 0.10$) and moisture content ($P < 0.10$). As the amount of screenings increased, production rate increased linearly. However, production rate decreased as moisture content increased.

The amount of screenings was not significant for d_{gw} , S_{gw} , or measures of flow ability ($P > 0.05$; Table 4.3). Moisture content was not significant for d_{gw} or measures of flow ability ($P > 0.05$; Table 4.4). The S_{gw} method S319.2 ($P \leq 0.05$) and S_{gw} method

S319.4 ($P \leq 0.05$) were both significant for moisture content. The S_{gw} method S319.2 had a mean estimate of 2.72 for 12% moisture corn and 2.45 for 10% moisture corn. The S_{gw} method S319.4 had a mean estimate of 744 μm for 12% moisture and 630 μm for 10% moisture.

According to a scale developed by Horn (2008), flow ability decreases as CFI and angle of repose increase. Even though CFI was not significant for this experiment, the results were considered fair for flow description as described by Horn (2008). USP (2004) described that as angle of repose and compressibility increase, flow ability decreases and indicated these experimental results as fair for angle of repose and good for compressibility. For shear analysis, flow function increased with flow ability (Jenike, 1964). According to Jenike (1964), the shear results for this experiment were classified as easy flowing.

Limited data has been published that evaluated the differences in moisture and screenings for ground corn. For particle size analysis, past research determined similar results to this experiment. Probst et al. (2013) reported that d_{gw} of hammermill ground corn was not significant when comparing different moisture contents (10, 16, and 20%). Probst et al. (2013) noted that the observed difference in S_{gw} may be due to a combination of particle cohesion, reduction in brittleness, and reduction in the amount of fines due to higher moisture.

Researchers have observed similar flow ability results as the current study (Groesbeck et al., 2006; Probst et al., 2013). Probst et al. (2013) observed no difference in the flow properties of compressibility and angle of repose when moisture content differed in hammermill ground corn. Groesbeck et al. (2006) suggested that flow ability is influenced by particle size. Groesbeck et al. (2006) determined that hammermill ground samples had a higher angle of repose and S_{gw} compared to roller mill ground samples at similar particle size due to the method of grinding, indicating poorer flow ability.

Conclusion

The results of this experiment suggest that cleaning corn prior to grinding with a roller mill does not change particle size or flow ability characteristics. When moisture

content differed, S_{gw} showed the only significant difference with 12% moisture having a greater S_{gw} than 10% moisture. Based on the results of this experiment, there was no benefit to cleaning the corn to remove the screenings prior to grinding with a roller mill.

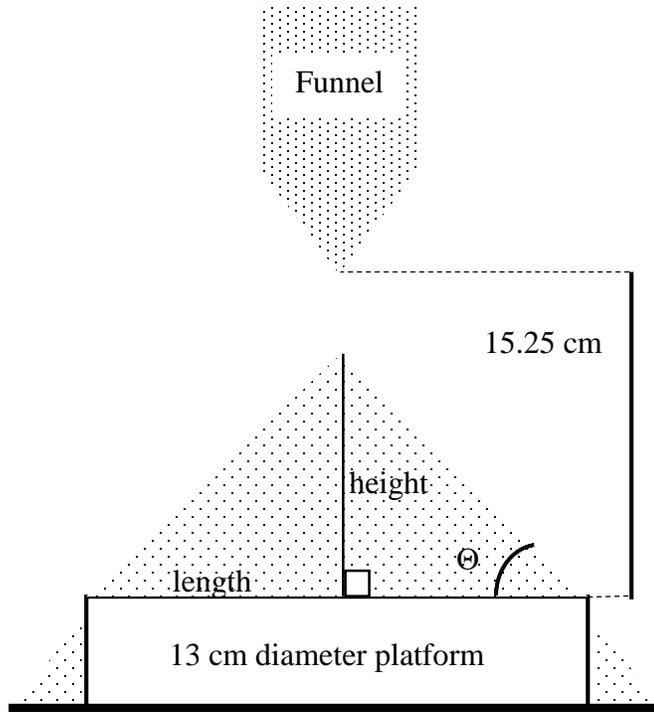
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Figures and Tables

Figure 4.1 Static angle of repose method (adapted from Juliano and Barbosa-Cánovas, 2005)¹



¹The angle of repose = \tan^{-1} (height of pile/radius of platform) (Appel, 1994).

Figure 4.2 Depiction of negative result for critical orifice diameter



Figure 4.3 Depiction of positive result for critical orifice diameter



Table 4.1 Sieve and sieve agitator arrangement

U.S. sieve number	Sieve opening (μm)	Sieve agitator(s)
6	3,360	None
8	2,380	None
12	1,680	Three rubber balls
16	1,190	Three rubber balls
20	841	Three rubber balls
30	595	One rubber ball; one bristle sieve cleaner
40	420	One rubber ball; one bristle sieve cleaner
50	297	One rubber ball; one bristle sieve cleaner
70	210	One rubber ball; one bristle sieve cleaner
100	149	One bristle sieve cleaner
140	105	One bristle sieve cleaner
200	74	One bristle sieve cleaner
270	53	One bristle sieve cleaner
Pan	-	None

Table 4.2 Specific energy consumption (SEC_E) and production rate for screenings level and moisture content¹

	Screenings level, %			SEM	<i>P</i> =	Moisture content, %			SEM	<i>P</i> =
	0	2.5	5			10	12			
Specific energy consumption (SEC _E), kWh/Mton	13.3	13.0	12.5	1.30	0.873	13.3 ^a	12.5 ^b	0.37	< 0.0001	
Production rate, Mton/hr	2.94	2.97	3.09	0.05	0.095	3.06	2.95	0.04	0.074	

¹A total of 18 samples of corn were ground using a roller mill for 3 levels of screenings and 2 moisture contents with 3 replicates per treatment.

^{ab}Means within a row that do not share a common superscript differ $P < 0.05$.

Table 4.3 Main effect for increasing amounts of screenings¹

	Screenings level, %			SEM	P =
	0	2.5	5		
Geometric mean diameter (d_{gw}), μm	630	644	622	29	0.869
Geometric standard deviation (S_{gw})					
ASAE S319.2	2.56	2.55	2.65	0.11	0.798
ANSI/ASAE S319.4, μm	675	687	699	27	0.826
Compressibility, %	11.87	12.67	13.60	1.55	0.549
Angle of repose, degrees	39.76	39.48	39.51	0.39	0.851
Critical orifice diameter, mm	20	21	21	2	0.773
Shear	5.84	5.37	5.24	0.26	0.257
Composite flow index (CFI)	68	66	65	3	0.742
Bulk density, kg/m^3	540.30	534.53	532.79	0.31	0.408

¹A total of 18 samples of corn at 2 moisture contents with 3 levels of screening and 3 replicates per treatment. Moisture \times screenings interaction was removed ($P = 0.152$).

Table 4.4 Main effect for moisture content¹

	Moisture content, %		SEM	<i>P</i> =
	12	10		
Geometric mean diameter (d_{gw}), μm	638	626	23	0.734
Geometric standard deviation (S_{gw})				
ASAE S319.2	2.72 ^a	2.45 ^b	0.08	0.028
ANSI/ASAE S319.4, μm	744 ^a	630 ^b	8	<0.0001
Compressibility, %	13.36	12.06	0.87	0.309
Angle of repose, degrees	39.59	39.57	0.31	0.964
Critical orifice diameter, mm	21	20	2	0.759
Shear	5.33	5.63	0.22	0.356
Composite flow index (CFI)	66	67	2	0.603
Bulk density, kg/m^3	536.90	534.84	0.26	0.671

¹A total of 18 samples of corn at 2 moisture contents with 3 levels of screenings and 3 replicates per treatment. Moisture \times screenings interaction was removed ($P = 0.152$).

^{ab}Means within a row that do not share a common superscript differ $P < 0.05$.

Chapter 5 - Effect of ground corn fractionation on flow ability

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Abstract

Particle size reduction is an important component of feed manufacturing that impacts pellet quality, feed flow ability, and animal performance. However, reducing particle size too fine often results in reduced flow ability of the ground corn and finished feed, which creates potential handling and storage concerns at the feed mill and farm. The objective of this experiment was to determine how fractionation affected flow ability of ground corn. Whole corn was received from a single source and ground to achieve 3 target particle sizes, 400, 500, and 600 μm with actual results of 469, 560, and 614 μm . Each target particle size was fractionated into three segments: fine ($< 282 \mu\text{m}$), medium ($\leq 630 \mu\text{m}$ and $\geq 282 \mu\text{m}$), and coarse ($> 630 \mu\text{m}$), particles using a vibratory separator (model LS18SP3, SWECO, Florence, KY). Within particle size treatment, the percentage of sample obtained for each fraction was: 400 μm : 4.9, 34.2, and 60.9% for fine, medium, and coarse, respectively; 500 μm : 1.9, 31.3, and 66.9% for fine, medium, and coarse, respectively; and 600 μm : 1.0, 24.4, and 74.7% for fine, medium, and coarse, respectively. When the fractions were separated, their particle sizes were: 400 μm : 94, 269, and 744 μm for fine, medium, and coarse, respectively; 500 μm : 96, 253, and 815 μm for fine, medium, and coarse, respectively; and 600 μm : 99, 220, and 898 μm for fine, medium, and coarse, respectively. Fractionated samples were analyzed for multiple flow ability characteristics, including: angle of repose, critical orifice diameter, composite flow index (CFI), bulk density, and compressibility. Treatments were arranged in a nested model with three replicates per treatment. Data were analyzed using the GLIMMIX procedure of SAS. When fraction was nested within particle size for each treatment, the fine fraction ($< 282 \mu\text{m}$) of the 400 μm corn had the poorest CFI ($P \leq 0.05$). Whereas the coarse fraction ($> 630 \mu\text{m}$) of the 600 μm corn had the best CFI. The nutrient content of the fractions was greatest in the medium fraction ($\leq 630 \mu\text{m}$ and $\geq 282 \mu\text{m}$) for crude protein, fat, and acid detergent fiber (ADF). In conclusion, reducing

particle size resulted in the ground corn having poorer flow ability characteristics, caused predominantly by particles that passed through a 282 μm screen. Based on this data, producers may potentially grind corn to a lower particle size while maintaining flow ability if fine particles ($< 282 \mu\text{m}$) are removed.

Key Words: corn, flow ability, particle size analysis

Introduction

The particle size or geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) are often used as quality control measurements in the grinding process. Maintaining a consistent product is especially difficult when grains are ground using a roller mill due to variations in grain, such as: kernel size, moisture, source, and amount of handling. External factors, separate from the grain itself that impacted grain particle size include: equipment operator, facility design, and environmental conditions. Equipment manufacturers have recommended to check grain particle size periodically while grinding to maintain consistency (RMS, 2012).

The objective of this experiment was to determine how fractionation affected flow ability of ground corn. Composite samples as well as representative samples from each fraction were analyzed for particle size, flow ability characteristics, and nutritive value. The flow ability characteristics analyzed included: compressibility, angle of repose, critical orifice diameter, composite flow index (CFI), and bulk density. Nutritive value was evaluated using chemical analysis which included: moisture, crude protein, fat, and acid detergent fiber (ADF).

Haque, (2010) suggested that flow ability was affected more by physical characteristics than by chemical properties. Physical characteristics that affect material flow ability include: d_{gw} , S_{gw} , particle shape, and electrostatic charge (Haque, 2010). According to a scale developed by Horn (2008), flow ability decreases as CFI and angle of repose increase. CFI was calculated by combining the results for angle of repose, compressibility, and critical orifice diameter (Horn, 2008). Angle of repose is defined and calculated by determining the angle between the free standing platform of the sample pile and the height of the pile (Fig. 5.1; Appel, 1994). Compressibility measured the change in

volume of the sample from initial to final tapped volume, and is influenced by factors including: particle size distribution, particle shape, and particle texture (Freeman Technology, 2015). The critical orifice diameter was determined using a powder flow ability test instrument (Flodex Model WG-0110, Paul N. Gardner Company, Inc., Pompano Beach, FL). Bulk density reflects how the sample behaves when it transitions from a non-flowing state to a flowing state back to a non-flowing state.

Materials and Methods

Material Preparation

Whole corn was received from a single source to reduce variation. Corn was ground to 3 target particle sizes (400, 500, and 600 μm) using a roller mill (Model 924, RMS Roller Grinder, Harrisburg, SD). The roller mill rolls were 2.36 and 2.36 corrugations/cm, 4.72 and 5.51 corrugations/cm, and 6.30 and 7.09 corrugations/cm roll on the top, middle, and bottom roll pairs, respectively. The feed rate was kept constant for all samples. Production rate was calculated and obtained by recording the amount of time required to grind each sample. Specific energy consumption (SEC_E) was calculated using Eq. 5.1 developed by Stark (1994) using the production rate and total amperage for each motor on the 3-high roller mill as each sample was ground.

$$SEC_E = \frac{I \times E \times PF \times 1.73}{PR \times 1000} \quad \text{Eq. 5.1}$$

where I is amperage

E is voltage

PF is power factor set to 0.85

PR is production rate expressed in Mton/hr

Each target particle size was fractionated into three segments: coarse ($> 630 \mu\text{m}$), medium ($\leq 630 \mu\text{m}$ and $\geq 282 \mu\text{m}$), and fine ($< 282 \mu\text{m}$) particles using a vibratory separator (Model LS18SP3, SWECO, Florence, KY).

Sample Analysis

Fractionated samples were analyzed for particle size, flow ability characteristics, and nutritive value. Each sample was divided using a riffle divider to approximately 500

± 5 g for each analysis. Each 500 ± 5 g sample was then divided using a riffle divider to reach the appropriate sample size requested for each analysis. Particle size was determined according to ANSI/ASAE S319.9. Calculations were performed according to the equations listed and described in ANSI/ASAE standard S3219.4 for d_{gw} and S_{gw} and ASAE standard S319.2 for S_{gw} . Flow ability characteristics analyzed included: compressibility, angle of repose, critical orifice diameter, shear, CFI, and bulk density. Nutritive value was evaluated using chemical analysis which included: moisture, crude protein, fat, and ADF (Ward Laboratories, Inc., Kearney, NE).

The CFI was calculated by combining the results for angle of repose, compressibility, and critical orifice diameter (Horn, 2008). Angle of repose was determined by weighing 200 ± 5 g of sample and allowing it to flow from a funnel 15.24 cm above a free standing platform, 13 cm in diameter. The angle between the free standing platform of the sample pile and the height of the pile was calculated by taking the inverse tangent of the height of the pile divided by the platform radius. (Fig. 5.1; Appel, 1994) The critical orifice diameter was determined using a powder flow ability test instrument (Flodex Model WG-0110, Paul N. Gardner Company, Inc., Pompano Beach, FL). Fifty grams of sample was allowed to flow through a stainless steel funnel into a cylinder. The sample rested for 30 s in the cylinder, and then evaluated based on the flow through an opening in a horizontal disc. The discs were 6 cm in diameter and the interior hole diameter ranged from 4 to 34 mm. A negative result was recorded when the sample did not flow through the opening in the disc or formed a cylindrical hole (Fig. 5.2). The disc hole size diameter was then increased by one disc size until a positive result was observed. A positive result was recorded when the material flowed through the disc opening forming a cone shape (Fig. 5.3). If a positive result was observed, the disc hole size diameter was decreased until a negative result was observed. Three positive results were used to determine the critical orifice diameter. Compressibility was determined by measuring the initial and final tapped volume. The sample was poured into a 250 mL graduated cylinder and the initial volume was recorded. This initial volume was referred as the unsettled apparent volume. The cylinder was tapped until no further change in the volume was observed. The final volume was recorded and change in compressibility calculated. The change in compressibility, expressed as a percentage, was

calculated by finding the difference between the initial and final volume, dividing by the initial volume, and multiplying by 100.

The d_{gw} and S_{gw} of the sample were determined according to the ANSI/ASAE S319.4 standard particle size analysis method. A 100 ± 5 g sample was sieved with a stainless steel sieve stack (13-sieves) containing sieve agitators with bristle sieve cleaners and rubber balls measuring 16 mm in diameter (Table 5.1). Each sieve was individually weighed with the sieve agitators to obtain a tare weight. Dispersing agent (Model SSA-58, Gilson Company, Inc., Lewis Center, OH; 0.5 g) was mixed into the sample and then placed on the top sieve. The sieve stack was placed in the Ro-Tap machine (Model RX-29, W. S. Tyler Industrial Group, Mentor, OH) and ran for 10 min. Once completed, each sieve was individually weighed with the sieve agitator(s) to obtain the weight of sample on each sieve. The amount of material on each sieve was used to calculate the d_{gw} and S_{gw} . The weight of the dispersing agent was not subtracted from the weight of the pan as specified in the ANSI/ASAE S319.4. Sieves were cleaned after each analysis with compressed air and a stiff bristle sieve cleaning brush. Calculations were performed according to the equations listed and described in ANSI/ASAE standard S3219.4 (Eq. 5.1 to 5.4) for particle size and ASAE standard S319.2 for S_{gw} (Eq. 5.3). Equations 5.5 and 5.6 depict how to calculate the range for 68% of the particles with both using the d_{gw} calculated with Eq. 5.1 and 5.2. Equation 5.5 uses the S_{gw} calculated with Eq. 5.3, while Eq. 5.6 uses the S_{gw} calculated with Eq. 5.4.

$$d_{gw} = \log^{-1} \left[\frac{\sum_{i=1}^n (W_i \log \bar{d}_i)}{\sum_{i=1}^n W_i} \right] \quad \text{(Eq. 5.1)}$$

where W_i is mass on i^{th} sieve, g

d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} is geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 5.2

n is number of sieves +1 (pan)

$$\bar{d}_i = (d_i \times d_{i+1})^{1/2} \quad \text{(Equation 5.2)}$$

where d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{i+1} is nominal sieve aperture size in next larger than i^{th} sieve (just above in a set), mm

$$S_{\log} = \left[\frac{\sum_{i=1}^n W_i (\log \bar{d}_i - \log d_{gw})^2}{\sum_{i=1}^n W_i} \right]^{1/2} = \frac{S_{\ln}}{2.3} \quad \text{(Equation 5.3)}$$

where W_i is mass on i^{th} sieve, g

d_i is nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} is geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 5.2

S_{\log} is geometric standard deviation of log-normal distribution by mass in ten-based logarithm, dimensionless

S_{\ln} is geometric standard deviation of log-normal distribution by mass in natural logarithm, dimensionless

n is number of sieves +1 (pan)

$$S_{gw} \approx \frac{1}{2} d_{gw} \left[\log^{-1} S_{\log} - (\log^{-1} S_{\log})^{-1} \right] \quad \text{(Equation 5.4)}$$

where W_i is mass on i^{th} sieve, g

d_i =nominal sieve aperture size of the i^{th} sieve, mm

d_{gw} = geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 5.2

S_{\log} is geometric standard deviation of log-normal distribution by mass in ten-based logarithm, dimensionless

S_{gw} is geometric standard deviation of particle diameter by mass, mm

$$\frac{d_{gw}}{S_{gw}} = \text{lower limit} \quad d_{gw} \times S_{gw} = \text{upper limit} \quad \text{(Equation 5.5)}$$

where 68% of the particles are determined by finding the difference between the upper and lower limits using the S_{gw} from equation 5.3

d_{gw} = geometric mean diameter or median size of particles by mass, mm, or geometric mean diameter or median size of particles on i^{th} sieve, mm, or Eq. 5.2

$$S_{gw} \times 2 = 68\% \text{ of particles} \quad (\text{Eq. 5.6})$$

where 68% of the particles are determined using the S_{gw} from Eq. 5.4

Bulk density was determined using a quart test weight cup (Seedboro Equipment Company, Des Plaines, IL) that met the specifications of Grain Inspection, Packers and Stockyards Administration (GIPSA). The sample was placed into a stainless steel funnel and dropped into the bulk density cup until it overflowed around the circumference of the cup. A wood leveling stick was used to remove excess sample and level the sample with the top of the cup. The weight of the sample in the cup was recorded.

Nutritive value was determined using chemical analysis. Moisture content was determined and calculated according to AOAC Official Method 930.15 with the revision in air oven temperature regulated to 105 °C for 3 hours. Crude protein was determined and calculated according to AOAC Official Method 990.03 combustion method that ignited the dried sample at 1050 °C. This method quantified the amount of nitrogen in the sample using a resistance furnace and a thermal conductivity detector. Fat and ADF were analyzed using the Ankom method. Fat was extracted from the sample using filter bag technology and an Ankom fat extractor. Lower percent ADF indicates increased amount and digestibility of energy. ADF is the portion of cellulose, lignin, heat damaged protein, cell wall protein, and ash that remain after cell solubles, hemicellulose, and soluble minerals have been removed.

Experimental Design

Particle size was analyzed as a main effect with three replicates. Fraction was nested with particle size to form a nested model with three replicates per treatment. Replications of each treatment were analyzed for particle size, flow ability characteristics, and nutritive value. Particle size analysis was done according to ANSI/ASAE S319.4. Calculations were performed according to the equations listed and described in ANSI/ASAE standard S3219.4 for d_{gw} and S_{gw} and ASAE standard S319.2 for S_{gw} . Flow ability characteristics analyzed included: compressibility, angle of repose,

critical orifice diameter, CFI, and bulk density. Nutritive value was evaluated using chemical analysis which included: moisture, crude protein, fat, and ADF. Data were analyzed using GLIMMIX procedure of SAS (SAS Institute, Inc., Cary, NC) with three replicates per treatment. Results were considered significant if $P \leq 0.05$, and a tendency if $0.05 \leq P < 0.10$. The least significance difference test was used to evaluate differences in means. Orthogonal contrasts were used to compare target particle sizes to their respective fractionated particle sizes.

Results and Discussion

The SEC_E and production rate were significant for each ground particle size ($P \leq 0.05$; Table 5.2). As particle size was decreased, SEC_E increased while production rate decreased. SEC_E was expressed in kWh/Mton and production rate expressed in Mton/hour.

When particle size was analyzed as a main effect, d_{gw} differed ($P \leq 0.05$) with actual results of 469, 560, and 614 μm for target particle sizes 400, 500, and 600 μm , respectively. When S_{gw} was calculated using the standard ANSI/ASAE S319.4 method, particle size differed with 400 μm (525 μm) having the lowest S_{gw} and 600 μm (687 μm) having the greatest ($P \leq 0.05$; Table 5.3). Particle size tended to affect S_{gw} when calculated using the standard ASAE S319.2 method, with 500 μm (2.55) having the lowest value, and 400 and 600 μm (2.62) having equal values ($P \leq 0.10$; Table 5.3). Particle size had no significant effect when analyzed for the composite samples on compressibility, angle of repose, critical orifice diameter and CFI ($P > 0.05$; Table 5.3). Bulk density was affected by particle size ($P \leq 0.05$; Table 5.3). The 400 μm (508 kg/m^3) sample yielded the lowest value which differed from the 600 μm (525 kg/m^3) sample, but not significantly from the 500 μm (517 kg/m^3) sample. The 500 and 600 μm samples were not significantly different. Particle size had no significant effect due on nutritive value for moisture, crude protein, and fat ($P > 0.05$; Table 5.4). However, ADF was significant for particle size ($P \leq 0.05$; Table 5.4). The 400 and 500 μm (1.9) had equal values and were lower than 600 μm (2.4). This likely occurred because larger foreign material of corn cobs and corn stalks was not ground as fine in 600 μm as it was in 400 μm .

Within particle size treatment, the percentage of ground corn for each fraction was: 400 μm : 4.6, 32.3, and 57.5% for fine, medium, and coarse, respectively; 500 μm : 1.80, 30.1, and 64.4% for fine, medium, and coarse, respectively; and 600 μm : 0.90, 23.2, and 71.2% for fine, medium, and coarse, respectively. When the fractions were separated, their particle sizes were: 400 μm : 94, 269, and 744 μm for fine, medium, and coarse, respectively; 500 μm : 96, 253, and 815 μm for fine, medium, and coarse, respectively; and 600 μm : 99, 220, and 898 μm for fine, medium, and coarse, respectively. When fraction was nested within particle size, it impacted all measures of flow ability, with the fine fraction ($< 282 \mu\text{m}$) of the 400 μm corn having the poorest flow ability ($P \leq 0.05$; Table 5.5). Nutritive value was also affected for crude protein, fat, ADF ($P \leq 0.05$), and moisture ($P \leq 0.05$; Table 5.6) with the fine fraction ($< 282 \mu\text{m}$) of the 400 μm corn having the lowest moisture.

According to a suggested scale developed by Horn, et al. (2008), flow ability decreased as CFI and angle of repose increased. These experimental results for CFI were considered fair for flow description when particle size was analyzed as a main effect as described by Horn (2008). However, when target particle sizes were fractionated, CFI ranged from passable for fine, fair for medium, and good for coarse. USP (2004) and Haque (2010) described that as angle of repose and compressibility increased, flow ability decreased. For angle of repose, these experimental results were passable when target particle size differed according to USP (2004). When target particle sizes were fractionated, these results ranged from poor for fine, passable for medium, and fair to passable for coarse (USP, 2004). For compressibility, target particle sizes were determined to be fair according to USP (2004). When target particle sizes were fractionated, these results ranged from poor to very poor for fine, fair to passable for medium, and good for coarse.

Limited data has been published that evaluated the differences in flow ability characteristics and nutritive value when samples of ground corn were fractionated. Abdullah and Geldart (1999) suggested that flow ability tends to increase with increasing particle size and then reaches a point where it plateaus. The results for this experiment determined no difference for critical orifice diameter or CFI when target particle size was changed, indicating a plateau was reached between 469 and 614 μm . However, when

target particle size was fractionated, critical orifice diameter and CFI decreased from fine ($< 282 \mu\text{m}$), to medium ($\leq 630\mu\text{m}$ and $\geq 282 \mu\text{m}$), to coarse ($> 630 \mu\text{m}$), which indicates particle size was not great enough to cause a plateau. In contrast to the results of this experiment, Yan and Barbosa-Cánovas (1997) noted that the greater the particle size, the greater the compressibility. Goodband et al. (2006) discussed that flow ability may be caused by particle shape in fine particle sizes. Even though the d_{gw} was similar, the S_{gw} differed based on the settings applied to the grinding machine, roller mill and hammermill (Goodband et al., 2006). Essentially, samples with a greater S_{gw} and particle size distribution and variation of particles have more fines, decreasing the flow ability.

Although differences in nutrient value and flow ability were not evaluated for animal performance or using different mill types, past studies have been conducted. Wondra et al. (1992) determined that greater nutrient digestibility occurred with grain that was milled using a roller mill and the greatest nutrient digestibility was seen in the grain with the lowest S_{gw} . According to Wondra et al. (1995) increased particle size uniformity and using a roller mill to grind grain improved apparent nutrient digestibility in diets and decreased undesirable changes in stomach morphology.

Conclusion

In conclusion, reducing particle size resulted in the ground corn having poorer flow ability characteristics, caused predominantly by particles that passed through a $282 \mu\text{m}$ screen. Nutritive value analyzed through chemical analysis differed when target particle sizes were fractionated in to fine, medium, and coarse. Based on this data, producers may potentially grind corn to a lower particle size while maintaining flow ability if fine particles ($< 282 \mu\text{m}$) are removed.

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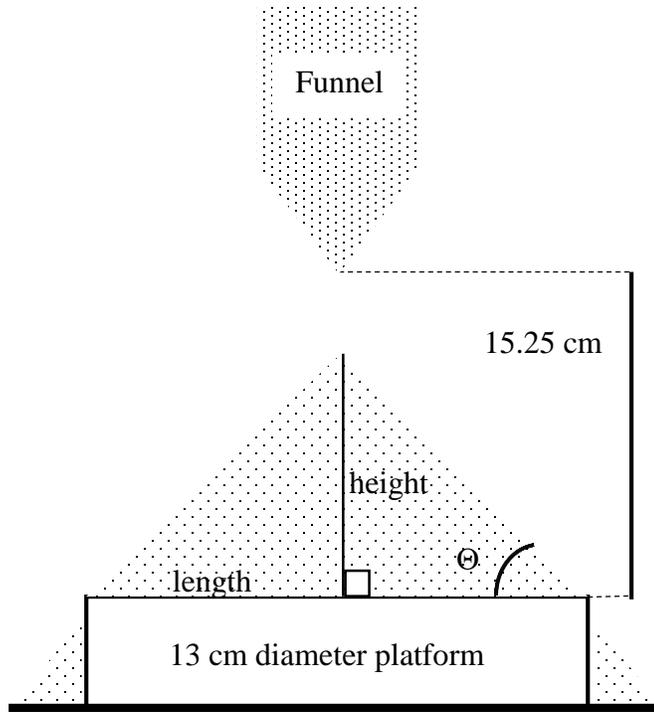
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Figures and Tables

Figure 5.1 Static angle of repose method (adapted from Juliano and Barbosa-Cánovas, 2005)¹



¹The angle of repose = \tan^{-1} (height of pile/radius of platform) (Appel, 1994).

Figure 5.2 Depiction of negative result for critical orifice diameter



Figure 5.3 Depiction of positive result for critical orifice diameter



Table 5.1 Sieve and sieve agitator arrangement

U.S. sieve number	Sieve opening (μm)	Sieve agitator(s)
6	3,360	None
8	2,380	None
12	1,680	Three rubber balls
16	1,190	Three rubber balls
20	841	Three rubber balls
30	595	One rubber ball; one bristle sieve cleaner
40	420	One rubber ball; one bristle sieve cleaner
50	297	One rubber ball; one bristle sieve cleaner
70	210	One rubber ball; one bristle sieve cleaner
100	149	One bristle sieve cleaner
140	105	One bristle sieve cleaner
200	74	One bristle sieve cleaner
270	53	One bristle sieve cleaner
Pan	-	None

Table 5.2 Specific energy consumption (SEC_E) and production rate for particle size¹

	Particle size, μm			SEM	$P =$
	400	500	600		
Specific energy consumption (SEC _E), kWh/Mton	11.2 ^a	8.8 ^b	6.9 ^c	0.77	< 0.0001
Production rate, Mton/hr	3.96 ^c	4.80 ^b	5.50 ^a	0.118	< 0.0001

¹A total of 36 samples were analyzed for physical, chemical, and flow ability characteristics. Three replicates of samples with target particle sizes of 400, 500, and 600 μm were each analyzed prior to fractionation and then fractionated into fine (< 282 μm), medium ($\leq 630 \mu\text{m}$ and $\geq 282 \mu\text{m}$), and coarse (> 630 μm) fractions.

^{abc}Means within a row that do not share a common superscript differ $P < 0.05$.

Table 5.3 Main effect of particle size on physical characteristics^{1, 2}

	Particle size, μm			SEM	<i>P</i> =
	400	500	600		
Geometric mean diameter (d_{gw}), μm	469 ^c	560 ^b	614 ^a	5.3	<0.0001
Geometric standard deviation (S_{gw})					
ASAE S319.2	2.62	2.55	2.62	0.008	0.094
ANSI/ASAE S319.4, μm	525 ^c	602 ^b	687 ^a	5.2	<0.0001
Compressibility, %	15	15	15	0.0	0.845
Angle of repose, degrees	44.6	43.9	43.2	0.50	0.224
Critical orifice diameter, mm	21	21	23	1.3	0.729
Composite flow index (CFI)	71	71	70	1.3	0.872
Bulk density, kg/m^3	508 ^b	517 ^{ab}	525 ^a	0.2	0.014

¹A total of 36 samples were analyzed for physical, chemical, and flow ability characteristics. Three replicates of samples with target particle sizes of 400, 500, and 600 μm were each analyzed prior to fractionation and then fractionated into fine (< 282 μm), medium ($\leq 630 \mu\text{m}$ and $\geq 282 \mu\text{m}$), and coarse (> 630 μm) fractions.

²Means were determined for the composite samples.

^{abc}Means within a row without common superscripts differ $P < 0.05$.

Table 5.4 Main effect of particle size on chemical analysis (as fed)¹

	Particle size, μm			SEM	<i>P</i> =
	400	500	600		
Moisture	13.5	13.7	13.7	0.12	0.843
Crude Protein	7.4	7.4	7.4	0.03	0.775
Fat	3.2	3.1	2.8	0.12	0.125
Acid Detergent Fiber (ADF)	1.9 ^b	1.9 ^b	2.4 ^a	0.11	0.021

¹A total of 36 samples were analyzed for physical, chemical, and flow ability characteristics. Three replicates of samples with target particle sizes of 400, 500, and 600 μm were each analyzed prior to fractionation and then fractionated into fine ($< 282 \mu\text{m}$), medium ($\leq 630 \mu\text{m}$ and $\geq 282 \mu\text{m}$), and coarse ($> 630 \mu\text{m}$) fractions.

^{ab}Means within a row without common superscripts differ $P < 0.05$.

Table 5.5 Effect of fraction nested with particle size on physical characteristics¹

	Fraction (particle size, μm)												SEM	P =
	Composite			Fine			Medium			Coarse				
	400	500	600	400	500	600	400	500	600	400	500	600		
Geometric mean diameter (d_{gw}), μm^2	469 ^f	560 ^e	614 ^d	94 ⁱ	96 ⁱ	99 ⁱ	269 ^g	253 ^g	220 ^h	744 ^c	815 ^b	898 ^a	10.7	<0.0001
Geometric standard deviation (S_{gw}) ²														
ASAE S319.2	2.62 ^a	2.55 ^b	2.62 ^a	1.65 ^h	1.63 ^h	1.66 ^h	2.01 ^e	2.10 ^d	2.17 ^c	1.90 ^f	1.81 ^g	1.80 ^g	0.017	<0.0001
ANSI/ASAE S319.4, μm	525 ^d	602 ^b	687 ^a	49 ^f	49 ^f	52 ^f	203 ^e	206 ^e	188 ^e	512 ^d	513 ^d	556 ^c	10.4	<0.0001
Compressibility, % ²	16 ^d	16 ^d	15 ^d	33 ^a	28 ^b	31 ^a	20 ^c	21 ^c	20 ^c	13 ^e	11 ^e	12 ^e	0.01	<0.0001
Angle of repose, degrees ³	44.6 ^{bc}	43.9 ^{bc} ^d	43.2 ^d	50.8 ^a	50.1 ^a	49.7 ^a	44.4 ^{bcd}	43.5 ^{cd}	45.2 ^b	41.1 ^e	40.6 ^e	40.4 ^e	0.42	<0.0001
Critical orifice diameter, mm^2	21 ^c	21 ^c	23 ^c	30 ^a	27 ^b	29 ^{ab}	27 ^b	29 ^{ab}	28 ^{ab}	15 ^d	14 ^{de}	13 ^e	0.8	<0.0001
Composite flow index (CFI) ²	71 ^c	71 ^c	70 ^c	57 ^g	61 ^{ef}	59 ^{fg}	65 ^d	63 ^{de}	63 ^{de}	80 ^b	82 ^{ab}	83 ^a	1.0	<0.0001
Bulk density, kg/m^3 ^{3 4}	508 ^{abc}	517 ^{ab}	525 ^a	425 ^f	432 ^f	N/A ⁵	505 ^{bc}	500 ^{bc}	482 ^{de}	471 ^e	491 ^{cd}	509 ^{abc}	0.5	<0.0001

¹A total of 36 samples were analyzed for physical, chemical, and flow ability characteristics. Three replicates of samples with target particle sizes of 400, 500, and 600 μm were each analyzed prior to fractionation and then fractionated into fine (< 282 μm), medium ($\leq 630 \mu\text{m}$ and $\geq 282 \mu\text{m}$), and coarse (> 630 μm) fractions.

² Orthogonal contrasts were conducted comparing the composite sample to each fraction ($P < 0.0001$ for fine, medium, and coarse).

³ Orthogonal contrasts were conducted comparing the composite sample to each fraction ($P < 0.0001$; 0.1998; < 0.0001 for fine, medium, and coarse, respectively).

⁴ Orthogonal contrasts were conducted comparing the composite sample to each fraction ($P < 0.0001$; 0.0006; and < 0.0001 for fine, medium, and coarse, respectively).

⁵ Sufficient sample was not available to conduct analysis.

^{abcde fghi} Means within a row without common superscripts differ $P < 0.05$.

Table 5.6 Effect of fraction nested with particle size on chemical analysis (as fed)¹

	Fraction (particle size, μm)												SEM	<i>P</i> =
	Composite			Fine			Medium			Coarse				
	400	500	600	400	500	600	400	500	600	400	500	600		
Moisture ²	13.5 ^{cde}	13.7 ^{bcd}	13.7 ^{bcd}	12.8 ^{efg}	14.1 ^{abc}	14.4 ^{ab}	12.3 ^g	13.2 ^{def}	13.4 ^{cde}	12.6 ^{fg}	13.8 ^{abcd}	14.5 ^a	0.25	0.006
Crude protein ³	7.4 ^d	7.4 ^d	7.4 ^d	5.6 ^e	5.3 ^f	5.1 ^f	8.8 ^a	8.4 ^b	7.9 ^c	4.4 ^g	4.3 ^{gh}	5.1 ^f	0.06	<0.0001
Fat ³	3.1 ^b	3.1 ^b	2.8 ^c	1.8 ^d	1.5 ^e	1.2 ^f	4.0 ^a	3.8 ^a	3.0 ^{bc}	1.3 ^{ef}	1.0 ^f	0.5 ^g	0.10	<0.0001
Acid detergent fiber (ADF) ⁴	1.9 ^b	1.9 ^b	2.4 ^a	0.6 ^{de}	0.9 ^{cd}	1.0 ^c	2.4 ^a	2.3 ^a	2.6 ^a	0.5 ^e	0.4 ^e	0.5 ^e	0.12	<0.0001

¹A total of 36 samples were analyzed for physical, chemical, and flow ability characteristics Three replicates of samples with target particle sizes of 400, 500, and 600 μm were each analyzed prior to fractionation and then fractionated into fine (< 282 μm), medium (\leq 630 μm and \geq 282 μm), and coarse (> 630 μm) fractions.

²Orthogonal contrasts were conducted comparing the composite sample to each fraction (*P* = 0.4861; 0.0030; and 0.9308 for fine, medium, and coarse, respectively).

³Orthogonal contrasts were conducted comparing the composite sample to each fraction (*P* < 0.0001 for fine, medium, and coarse).

⁴Orthogonal contrasts were conducted comparing the composite sample to each fraction (*P* < 0.0001; 0.0010; and < 0.0001 for fine, medium, and coarse, respectively).

^{abcde}Means within a row without common superscripts differ *P* < 0.05.

Chapter 6 - Summary of Findings

The results of these experiments determined the particle size methodology and flow ability of fractionated corn. The first experiment compared 5 variations of the current approved method used to determine the geometric mean diameter (d_{gw}) and geometric standard deviation (S_{gw}) described by ANSI/ASAE S319.4 “Method of determining and expressing fineness of feed materials by sieving”. The variations were tested with three grains (corn, wheat, and sorghum). There was no method \times grain ($P > 0.05$) interaction for d_{gw} , so it was removed from the model. A 15 minute sieving time with sieve agitators and dispersing agent resulted in the lowest d_{gw} and greatest S_{gw} ($P \leq 0.05$). Results indicated that sieve agitators and dispersing agent best facilitated the movement of material through the sieves and reduced the agglomeration of fine particles on sieves with small openings. The data from this experiment suggests the use of sieve agitators arranged on sieves as depicted in Table 2.1 and the addition of 0.5 g dispersing agent provide a better estimate of particle size with a 10 min sieving time. Furthermore, a 15 min sieving time as described in ANSI/ASAE S319.4 may not be required when a dispersing agent is added to the sample. In order to accurately compare particle size analysis results and animal research related to difference in ground materials, the method must be accurately described.

The second experiment evaluated particle size analysis on ground corn using a 3-sieve method for particle size with varying sieving time (30, 60, and 90 s) along with the addition of dispersing agent. The sieving time for the 3-sieve method referred to time the sieves were shaken side to side by hand. A 90 s sieving time with 0.25 g dispersing agent resulted in the lowest d_{gw} ($P \leq 0.05$). Results for d_{gw} were calculated two ways: 1) according to standard method ANSI/ASAE S319.4, and 2) the regression equation developed by Baldrige, et al. (2001). The regression equation developed by Baldrige et al. (2001) was never developed to calculate S_{gw} , so main effects and means were not determined. Pearson Correlation Coefficients compared the goodness-of-fit of both alternative methods for the tested analytical model to the results from the standard ANSI/ASAE 13-sieve method. Both alternative methods were slightly correlated ($P \leq 0.01$; $r > 0.97$), with the standard method ANSI/ASAE S319.4 having the greatest value.

The 13-sieve method used sieve agitators, 0.5 g dispersing agent, and 10 min sieving time.

Experiment three evaluated particle size and flow ability by grinding corn at two moisture contents (10 and 12%) with three levels of screenings (0, 2.5, and 5%). Moisture content was significant for S_{gw} with 12% moisture corn having a higher value ($P \leq 0.05$). The results of this experiment suggested that cleaning corn prior to grinding with a roller mill does not change particle size or measures of flow ability, which included: bulk density, critical orifice diameter, compressibility, angle of repose, shear, and composite flow index (CFI).

Experiment four continued the evaluation of flow ability with samples of corn ground to three target particle sizes (400, 500, and 600 μm) and fractionated into fine, medium, and coarse segments. Prior to fractionation, each target particle size was analyzed and significant for d_{gw} , S_{gw} , and bulk density ($P \leq 0.05$). When fraction was nested within particle size, it impacted ($P \leq 0.05$) all measures of flow ability, which included: bulk density, critical orifice diameter, compressibility, angle of repose, and CFI.

The results of these experiments indicated that particle size analysis should use sieve agitators and dispersing agent with 10 or 15 min sieving time for the standard 13-sieve method and 90 s sieving time with 0.25 g dispersing agent for the 3-sieve method. Cleaning whole grain before grinding had no significant effect on particle size or flow ability. This data suggested that producers may potentially grind corn to a lower particle size while maintaining flow ability by removing fine particles.