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# Effects of Varying Methodologies on Grain Particle Size Analysis

J. R. Kalivoda<sup>1</sup>, C. K. Jones<sup>1</sup>, and C. R. Stark<sup>1</sup>

## Summary

Particle size reduction is an important component of feed manufacturing that impacts pellet quality, feed flowability, and pig feed efficiency. The correct determination of particle size is important for feed manufacturers, nutritionists, and pork producers to meet target specifications. The current method for determining the geometric mean diameter (dgw) and geometric standard deviation (Sgw) of grains has been published by the ANSI/ASAE S319.4. 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 shake time, sieve agitators, and the use of a flow agent. Therefore, the objectives of this experiment were: 1) to determine which method of particle size analysis best estimates the particle size of various cereal grains, and 2) assess analytical variation within each method. Eighteen samples of corn, sorghum, or wheat were ground and analyzed using different variations of the standard particle size analysis method. Treatments were arranged in a  $5 \times 3$  factorial design with five sieving methods: 1) 10-minute shake time with sieve agitators and no flow agent; 2) 10-minute shake time with sieve agitators and flow agent; 3) 15-minute shake time with no sieve agitators or flow agent; 4) 15-minute shake time with sieve agitators and no flow agent; or 5) 15-minute shake time with sieve agitators and flow agent conducted in three grains - corn, sorghum, or wheat. There were four replicates per treatment. Results for dgw and Sgw were calculated according to both standard methods \$319.2 and \$319.4. The analytical method that resulted in the finest dgw and greatest Sgw was considered desirable because it is presumably representative of the largest quantity of particles moved through the appropriate sieve.

There was no analytical method × grain type interaction for dgw, so it was removed from the model. Analytical method affected (P < 0.0001) dgw and Sgw measured by both standards. Inclusion of sieve agitators and flow agent resulted in the finest dgw, regardless of sieving time. Inclusion of flow agent reduced (P < 0.05) the mean particle size by 32 or 36 µm when shaken for 10 or 15 minutes, respectively, compared to the same sample analyzed without flow agent. Flow agent was also an important factor to alter Sgw. Because the flow agent increased the quantity of very fine particles collected in the pan, Sgw was substantially greater (P < 0.05) when flow agent was included in the method. Particle size of corn and sorghum ground using the same mill parameters was

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similar (P > 0.05), but wheat ground using the same mill parameters was 120 to 104 µm larger (P < 0.05) compared to corn or sorghum, respectively.

In conclusion, both sieve agitators and flow agent should be included when conducting particle size analysis, but only 10 minutes of shake time is required. Wheat ground using the same hammermill settings as corn and sorghum is approximately 100  $\mu$ m larger in particle size.

Key words: 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 micron reduction in corn particle size ground with a hammermill (Wondra et al., 1995)<sup>2</sup>. Accurate particle size analysis is important to meet required specifications and compare samples across laboratories, but different variations of the standard method used to determine the average particle size can result in a variation of up to 100  $\mu$ m in the same sample. The current approved method used to determine particle size of feeds and ingredients is described by ANSI/ASAE S319.4 "Method of determining and expressing fineness of feed materials by sieving."<sup>3</sup> 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 shake time, sieve agitator inclusion, and the use of a flow agent. The most significant change in the standard method occurred between ASAE S319.2 and ANSI/ASAE S319.3, when shaking time increased from 10 to 15 minutes. Fahrenholz et al., (2010)<sup>4</sup> suggested that the goal in particle size analysis is to find the lowest geometric mean diameter (dgw) and highest geometric standard deviation (Sgw). Both Fahrenholz et al. (2010) and Stark and Chewning (2012)<sup>5</sup> reported that the addition of agitators and flow agent significantly changed the average particle size of a ground sample of corn, but a direct comparison using different shaking times has not been reported in various grains. Therefore, the objectives of this experiment were: 1) to determine which method of particle size analysis best estimates the particle size of various cereal grains, and 2) assess analytical variation within each method.

## Procedures

A total of 360 particle size analyses were conducted in this experiment, stemming from 18 different samples of ground grain. The 18 samples represented three grain types (corn, sorghum, and wheat) that were ground through two mill types (hammermill or roller mill) with three grind settings for each mill type to create a wide range of particle sizes for each grain type. For the hammermill (Model 22115, Bliss Industries LLC.,

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<sup>&</sup>lt;sup>2</sup> Wondra, K. J., J. D. Hancock, K. C. Behnke, and C. R. Stark. 1995. Effects of mill type and particle size uniformity on growth performance, nutrient digestibility, and stomach morphology on finishing pigs. J. Anim. Sci. 73:2564-2573.

<sup>&</sup>lt;sup>3</sup> ASABE. 2012. Method of determining and expressing fineness of feed materials by sieving. Am. Soc. Agric. Eng. St., Joseph, MI.

<sup>&</sup>lt;sup>4</sup> Fahrenholz, A. C., L. J. McKinney, C. E. Wurth, and K. C. Behnke. 2010. The importance of defining the method in particle size analysis by sieving. Feed Management. 261-264.

<sup>&</sup>lt;sup>5</sup> Stark, C. R. and C. G. Chewning. 2012. The effect of sieve agitators and dispersing agent on the method of determining and expressing fineness of feed materials by sieving. Anim. Prod. Sci. 52:69-72.

Ponca City, OK), grain was ground through a 4/64-inch, 12/64-inch, or 16/64-inch screen. For the roller mill (Model 924, RMS Roller Grinder, Harrisburg, SD), settings were achieved by adjusting the rolls (top: 6 corrugations/inch; middle: 12 and 14 corrugations/inch; and bottom: 16 and 18 corrugations/inch roll arrangements). All grains were ground at Kansas State University's O. H. Kruse Feed Technology Innovation Center in Manhattan. The differences in type of mill and grind size were intended to create a robust set of ground grain samples, but neither were fixed effects due to their natural confounding with the response criterion.

Each of the 18 ground samples was subdivided into twenty  $100 \pm 5$  g subsamples using a riffle divider. Subsamples were then analyzed using different variations of the ANSI/ ASAE S319.4 standard particle size analysis method. Treatments were arranged in a  $5 \times 3$  factorial design with five sieving methods:

- 1) 10-minute shake time with sieve agitators and no flow agent;
- 2) 10-minute shake time with sieve agitators and flow agent;
- 3) 15-minute shake time with no sieve agitators and no flow agent;
- 4) 15-minute shake time with sieve agitators and no flow agent;
- 5) 15-minute shake time with sieve agitators and flow agent;

and three grains — corn, sorghum, and wheat. The analysis used two stainless steel sieve stacks (13 sieves + pan) with bristle sieve cleaners and 13 mm rubber balls arranged as depicted in Table 1. Sieves were cleaned with compressed air and a stiff bristle sieve cleaning brush after each analysis. Each sieve was individually weighed with the sieve agitators to obtain a tare weight. The  $100 \pm 5$  g subsample was then placed on the top sieve. If flow agent (Model SSA-58, Gilson Company, Inc., Lewis Center, OH) was required, 0.5 g was mixed into the sample prior to placing the mixture on the top sieve by stirring the flow agent with the mixture for 5 s. The sieve stack was then placed in a Ro-Tap machine (Model RX-29, W. S. Tyler Industrial Group, Mentor, OH) and shaken for 10 or 15 minutes, according to the analytical method treatment. Once completed, each sieve was individually weighed with the sieve agitators to obtain the weight of the sample on each sieve. The quantity of material on each sieve was then entered into a spreadsheet to calculate the dgw and Sgw. The spreadsheet calculations were reviewed, and there was no discernable difference in response criterion if the 0.5 g of flow agent was subtracted from the net weight of the pan; therefore the flow agent weight was not subtracted to simplify the process. The dgw results were calculated using the traditional formula in the standard ANSI/ASAE \$319.4. The Sgw results were calculated using the equations from ANSI/ASAE \$319.2 and ANSI/ASAE \$319.4. While the industry is more familiar with the Sgw calculated by ANSI/ASAE \$319.2 with standard deviation values typically ranging from 1.8 to 2.4, the ANSI/ASAE S319.4 method revised the calculation for Sgw so it represents the spread of particles with geometric standard deviation values in microns. We chose to depict both values for this paper. The following examples depict how to calculate the particle size range to represent 68% of the particles in a sample with Sgw S319.2 equaling 2.19, Sgw S319.4 equaling  $512 \,\mu m$  and dgw equaling 591 μm.

Particle Size Range using Sgw S319.2:

dgw/Sgw = lower limit	$dgw \times Sgw = upper limit$
Example: 591/2.19 = 270 μm	$591 \times 2.19 = 1294 \mu m$
$1294 - 270 = 1024 \mu\text{m}$ total	range for 68% of the particles

Particle Size Range using Sgw S319.4:

Sgw  $\times$  2 = total range for 68% of the particles Example: 512  $\mu$ m  $\times$  2 = 1024  $\mu$ m total range for 68% of the particles

Although the method to calculate Sgw has changed, the calculated total representative range for 68% of the particles has remained the same in both methods.

The five different particle size analysis procedures were repeated four times for each of the 18 grain × mill type × grind setting combinations with a new technician conducting the procedure for each of the four replicates. We intended technician to be a fixed effect in this experiment, but the variable was removed from the model due to insignificance for dgw (P = 0.7414) and Sgw (P = 0.3098). 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 and Discussion**

Variations in the ANSI/ASAE S319 method affected (P < 0.0001) dgw and Sgw evaluated by both S319.2 and S319.4 (Table 2). The dgw was finest when both sieve agitators and flow agent were included in the analysis, with the addition of flow agent reducing (P < 0.05) the mean particle size by 32 or 36 µm when shaken for 10 or 15 minutes, respectively, compared to the same sample analyzed without flow agent. Interestingly, increasing the shake time from 10 to 15 minutes did not further improve (P = 0.1247) dgw. Adding sieve agitators alone reduced dgw, where the mean particle size was reduced (P < 0.05) by 39 µm when sieves included agitators and were shaken for 15 minutes.

Because sieve agitators and flow agent both increase the quantity of very fine particles collected in the pan, Sgw was substantially greater (P < 0.05) when one or both were included in the procedures. Figures 1 and 2 depict the shift facilitated by the addition of flow agent on moving particles to screens with small openings.

The Sgw according to S319.4 was again maximized (P < 0.05) when both sieve agitators and flow agent were included. Furthermore, there is an advantage to a 15-minute shake time to increase (P < 0.0001) Sgw according to both S319.2 and S319.4. However, this increased shake time may not be practical, as it did not impact dgw and may substantially reduce the efficiency of particle size analysis laboratories.

The dgw of grains ground using the same parameters differed (P < 0.0001). When compared to corn, sorghum was 16 µm larger and wheat was 120 µm larger (P < 0.05) when ground using the same mill settings. Due to the variability from calculating Sgw, grains differed from each other. Corn was similar to wheat for Sgw evaluated by ASAE S319.2, but had lower Sgw than sorghum (P < 0.05). When Sgw was evaluated by ANSI/ASAE S319.4, corn was similar to sorghum and lower than wheat (P < 0.05).

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In conclusion, results of this experiment indicate that sieve agitators and flow agent best facilitate the movement of material through the sieves and reduce the agglomeration of fine particles on sieves with small openings. Thus, it is our recommendation to use sieve agitators arranged on sieves as depicted in Table 1, 0.5 g of flow agent in particle size analysis, and a sieving time of at least 10 minutes.

U.S. sieve number	Sieve opening, µm	Sieve agitator type per screen
6	3360	None
8	2380	None
12	1680	Three rubber balls
16	1190	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
Receiving pan	-	None

Table 1. Sieve type and agitator arrangement

# Table 2. Main effect of analytical method on geometric mean diameter and geometric standard deviation of various grains<sup>1</sup>

	Method					_	
Shake time, min:	10	10	15	15	15		
Sieve agitator inclusion:	Yes	Yes	No	Yes	Yes		
Flow agent inclusion:	No	Yes	No	No	Yes	SEM	P =
Mean particle size (dgw), μm²	586 <sup>b</sup>	554°	615ª	576 <sup>b</sup>	540°	223	< 0.0001
Standard deviation (Sgw), μm							
ANSI/ASAE \$319.2 <sup>3</sup>	2.23 <sup>b</sup>	2.62ª	2.09°	2.27 <sup>b</sup>	2.63ª	0.316	< 0.0001
ANSI/ASAE \$319.4 <sup>3</sup>	485 <sup>bc</sup>	579ª	467°	487 <sup>b</sup>	567ª	116	< 0.0001

<sup>1</sup> A 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 five different variations of the ANSI/ASAE S319.4 standard particle size analysis method. There were four replicates per method.

<sup>2</sup>Orthogonal contrasts included shake time 10 vs. 15 min: P = 0.1247, with or without sieve agitators: P < 0.0001, and with or without flow agent: P < 0.0001.

<sup>3</sup>Orthogonal contrasts included shake time 10 vs. 15 min: P < 0.0001, with or without sieve agitators: P < 0.0001, and with or without flow agent: P < 0.0001.

<sup>abc</sup> Means within a row without common superscripts differ P < 0.05.

Table 3. Main effect	of grain	type on	geometric mean	particle size of	r standard (	deviation o	of grains <sup>1</sup>
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	-			_	
	Corn	Sorghum	Wheat	SEM	P =
Mean particle size (dgw), μm	529°	545 <sup>b</sup>	649ª	223	< 0.0001
Standard deviation (Sgw), μm					
ANSI/ASAE S319.2	2.36 <sup>b</sup>	$2.40^{a}$	2.35 <sup>b</sup>	0.316	0.0245
ANSI/ASAE S319.4	487 <sup>b</sup>	492 <sup>b</sup>	572ª	116	< 0.0001

<sup>1</sup> A 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 five different variations of the ANSI/ASAE S319.4 standard particle size analysis method. There were four replicates per method.

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



Figure 1. Distribution graph depicting the quantity of particles collected on each sieve for a corn sample medium ground using a hammermill, comparing when flow agent was used and not used.

<sup>1</sup> dgw: 402 μm; Sgw (calculated with ANSI/ASAE S319.2): 3.11; Sgw (calculated using ANSI/ ASAE S319.4): 560.48 μm; 45% of the particles were below 300 μm.

 $^{2}$  dgw: 448 μm; Sgw (calculated with ANSI/ASAE S319.2): 2.50; Sgw (calculated using ANSI/ASAE S319.4): 470.40 μm; 44% of the particles were below 300 μm.