Effects of Varying Methodologies on Grain Particle Size Analysis

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Effects of Varying Methodologies on Grain Particle Size Analysis

J. R. Kalivoda¹, C. K. Jones¹, and C. R. Stark¹

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 × 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 standards S319.2 and S319.4. 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 \(\mu\)m 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 \((\text{Wondra et al., 1995})\). 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.” 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., \((2010)\) 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 \((2012)\) 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 \(\text{corn, sorghum, and wheat}\) that were ground through two mill types \(\text{hammermill or roller mill}\) with three grind settings for each mill type to create a wide range of particle sizes for each grain type. For the hammermill \((\text{Model 22115, Bliss Industries LLC.})\),

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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 ± 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 × 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 ± 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 S319.4. The Sgw results were calculated using the equations from ANSI/ASAE S319.2 and ANSI/ASAE S319.4. While the industry is more familiar with the Sgw calculated by ANSI/ASAE S319.2 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 µm and dgw equaling 591 µm.
Particle Size Range using Sgw S319.2:
\[
dgw/Sgw = \text{lower limit} \quad \text{dgw} \times Sgw = \text{upper limit}
\]
Example: 591/2.19 = 270 µm \(591 \times 2.19 = 1294 \mu m\)
1294 – 270 = 1024 µm total range for 68% of the particles

Particle Size Range using Sgw S319.4:
\[
Sgw \times 2 = \text{total range for 68% of the particles}
\]
Example: 512 µm \( \times 2 = 1024 \mu m \) 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 \(\times\) mill type \(\times\) 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 \(\mu m\) 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 \(\mu m\) 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 \(\mu m\) larger and wheat was 120 \(\mu m\) 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 \(P < 0.05\).
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.

<table>
<thead>
<tr>
<th>U.S. sieve number</th>
<th>Sieve opening, µm</th>
<th>Sieve agitator type per screen</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>3360</td>
<td>None</td>
</tr>
<tr>
<td>8</td>
<td>2380</td>
<td>None</td>
</tr>
<tr>
<td>12</td>
<td>1680</td>
<td>Three rubber balls</td>
</tr>
<tr>
<td>16</td>
<td>1190</td>
<td>Three rubber balls</td>
</tr>
<tr>
<td>20</td>
<td>841</td>
<td>Three rubber balls</td>
</tr>
<tr>
<td>30</td>
<td>595</td>
<td>One rubber ball; one bristle sieve cleaner</td>
</tr>
<tr>
<td>40</td>
<td>420</td>
<td>One rubber ball; one bristle sieve cleaner</td>
</tr>
<tr>
<td>50</td>
<td>297</td>
<td>One rubber ball; one bristle sieve cleaner</td>
</tr>
<tr>
<td>70</td>
<td>210</td>
<td>One rubber ball; one bristle sieve cleaner</td>
</tr>
<tr>
<td>100</td>
<td>149</td>
<td>One bristle sieve cleaner</td>
</tr>
<tr>
<td>140</td>
<td>105</td>
<td>One bristle sieve cleaner</td>
</tr>
<tr>
<td>200</td>
<td>74</td>
<td>One bristle sieve cleaner</td>
</tr>
<tr>
<td>270</td>
<td>53</td>
<td>One bristle sieve cleaner</td>
</tr>
<tr>
<td>Receiving pan</td>
<td>-</td>
<td>None</td>
</tr>
</tbody>
</table>

Table 2. Main effect of analytical method on geometric mean diameter and geometric standard deviation of various grains

<table>
<thead>
<tr>
<th>Method</th>
<th>Shake time, min:</th>
<th>Sieve agitator inclusion:</th>
<th>Flow agent inclusion:</th>
<th>Mean particle size (dgw), µm²</th>
<th>Standard deviation (Sgw), µm</th>
<th>SEM</th>
<th>P =</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10</td>
<td>Yes</td>
<td>No</td>
<td>586ᵇ</td>
<td>2.23ᵇ</td>
<td>0.316</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>Yes</td>
<td>No</td>
<td>554ᶜ</td>
<td>2.62ᵃ</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>Yes</td>
<td>Yes</td>
<td>615ᵃ</td>
<td>2.09ᵃ</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>Yes</td>
<td>Yes</td>
<td>576ᵇ</td>
<td>2.27ᵇ</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>Yes</td>
<td>Yes</td>
<td>540ᵇ</td>
<td>2.63ᵇ</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1 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.

2 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: $P < 0.0001$.

3 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: $P < 0.0001$.

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

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Table 3. Main effect of grain type on geometric mean particle size or standard deviation of grains¹

<table>
<thead>
<tr>
<th></th>
<th>Corn (dgw), µm</th>
<th>Sorghum (Sgw), µm</th>
<th>Wheat (Sgw), µm</th>
<th>SEM</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean particle size (dgw), µm</td>
<td>529&lt;sup&gt;a&lt;/sup&gt;</td>
<td>545&lt;sup&gt;b&lt;/sup&gt;</td>
<td>649&lt;sup&gt;a&lt;/sup&gt;</td>
<td>223</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>Standard deviation (Sgw), µm</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ANSI/ASAE S319.2</td>
<td>2.36&lt;sup&gt;b&lt;/sup&gt;</td>
<td>2.40&lt;sup&gt;a&lt;/sup&gt;</td>
<td>2.35&lt;sup&gt;b&lt;/sup&gt;</td>
<td>0.316</td>
<td>0.0245</td>
</tr>
<tr>
<td>ANSI/ASAE S319.4</td>
<td>487&lt;sup&gt;b&lt;/sup&gt;</td>
<td>492&lt;sup&gt;b&lt;/sup&gt;</td>
<td>572&lt;sup&gt;a&lt;/sup&gt;</td>
<td>116</td>
<td>&lt; 0.0001</td>
</tr>
</tbody>
</table>

¹ 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.

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.

¹ 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.