Size and shape related characteristics of alfalfa grind

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Yang, W., Sokhansanj, S., Crerar, W.J. and Rohani, S. 1996. Size and shape related characteristics of alfalfa grind. Can. Agric. Eng. 38:201-205. Particle size distribution of a commercial alfalfa grind was determined by sieving test. Shape characteristics of alfalfa grind were examined using scanning electron microscope and image processing technique. The particle size was best described by the log-normal distribution based on the curve fitting results of various distribution functions. The median size (geometric mean diameter) of alfalfa grind was 238 μm with a standard deviation of 166 μm. Particle density measured by air comparison pycnometer ranged from 1390 to 1599 kg/m3 (average 1453 ± 62 kg/m3). Surface area of the alfalfa grind measured using the BET method with nitrogen sorption and calculated following the ASAE S319.2 was 0.750 ± 0.259 m2/g and 0.0215 m2/g, respectively. Scanning electron microscopy (SEM) and computer-aided image analysis were used to characterize the particles retained on individual test sieves of nominal apertures from 20 to 850 μm. It was found that the mean particle projection length ranged from 0.242 to 0.979 mm, width from 0.034 to 0.425 mm, area from 0.002 to 0.295 mm2, perimeter from 0.188 to 2.421 mm, and sphericity (roundness) from 0.54 to 0.64. A statistical regression analysis showed that these parameters were linear with sieve openings. Keywords: alfalfa grind, sieving test, particle characterization, SEM, image processing.

INTRODUCTION

Alfalfa grind is an intermediate product derived after hammer milling the dehydrated alfalfa. The size of alfalfa grind varies with the size of screen used in a hammer mill. It also changes with other factors such as moisture content of the alfalfa chops, the type of blade assembly, screen wear, and hammer rotational speed (Sokhansanj et al. 1992; Pulkinen 1994).

Information on size and shape of alfalfa grind is important. When evaluating a hammer mill, particle size distribution reflects on the performance of the mill. Particle size distribution data also are required for the design of pneumatic conveyors and cyclones. For the steam conditioning of alfalfa grind prior to pelleting, basic material properties such as specific surface area, particle density, and particle shape factor determine the rate of steam application. Particle size and shape also highly relate to the feed intake, digestion, and metabolic products of ruminants (Troelsen and Campbell 1968; Woodford and Murphy 1988; Luginbuhl et al. 1989).

The international standard ISO 2591-1 (ISO 1988) and its equivalent ASAE S319.2 (ASAE 1995) specify test methods for measuring particle size distribution by sieving tests. Finding particle size and shape through other means, such as projector and image analysis (Patil and Sokhansanj 1992), also have been reported. Scanning electron microscope (SEM) also provides an excellent alternative for examining size and shape for finer particles.

The objectives of this paper are: 1) to determine particle size distribution of alfalfa grind by sieving tests, and 2) to determine the size and shape related characteristics of alfalfa particles using SEM and image analysis.

MATERIALS AND METHODS

Alfalfa grind

The alfalfa grind used in this study was collected from an alfalfa processing plant during multiple shifts over a 24 h period. It was transported to the laboratory before it was subdivided using the "cone-and-quartering" method (Woodcock and Mason 1986) to obtain samples for the sieving test and other analysis. The moisture content of the alfalfa grind was 5.3 %wb.

Sieving tests

The sieving test was performed in a Gilsonic Autosiever Model GA-6 (Gilson Co., Inc., Worthington, OH). The test sieves were the wire-cloth type with a 100-mm frame diameter. The opening of the wire-cloth followed ASTM specifications, ranging from 20 to 1700 μm. The autosiever chamber held a nest of five sieves plus a pan. A charge of 45 g was initially loaded to the top sieve. The sieving test lasted 42 min, i.e., 2 min for the vibration amplitude to increase.
from zero to its maximum, 30 min to remain at the peak amplitude, and 10 min to decrease from maximum to zero. At the end of each cycle, each sieve was unloaded from the autosiever chamber and weighed. The material in the pan was weighed and transferred to the next series of five sieves at finer nominal openings. Three parallel sieving tests were performed for the same conditions. Both the mass percentage retained on each sieve and the cumulative percent undersize were plotted against sieve openings to examine the size distribution characteristics of alfalfa grind.

**Measurement of the specific surface area**

The Brunauer-Emmett-Teller (BET) method (Brunauer et al. 1938) with nitrogen sorption was used to measure the surface area of the alfalfa grind. According to the BET theory, the partial pressure of an adsorbate relates to its amount of sorption as:

$$\frac{1}{w \left( \frac{p}{p_0} - 1 \right)} = \frac{1}{w_m C} + \frac{C - 1}{w_m C} \left( \frac{p}{p_0} \right)$$  \hspace{1cm} (1)

where:

- $w$: mass of adsorbate in the material (g),
- $p$: partial pressure of adsorbate (Pa),
- $p_0$: saturation pressure of adsorbate (Pa),
- $w_m$: mass of adsorbate in monolayer (g), and
- $C$: constant.

For most substances, linearity results if $[w(p_0/p - 1)]^{-1}$ is plotted against relative pressure $p/p_0$ for $p/p_0 < 0.3$. The resultant slope $\alpha$ and intercept $\beta$ are:

$$\alpha = \frac{C - 1}{w_m C} \quad \text{and} \quad \beta = \frac{1}{w_m C}$$

Rearranging these expressions gives:

$$w_m = \frac{1}{\alpha + \beta}$$  \hspace{1cm} (2)

The total surface area is then calculated as:

$$S_t = w_m N A_{cs} / M$$  \hspace{1cm} (3)

where:

- $N$: Avogadro's number ($6.023 \times 10^{23}$ molecules/mol),
- $M$: molecular weight of adsorbate (nitrogen),
- $A_{cs}$: cross-sectional area of nitrogen (0.162 nm² at 77 K).

The specific surface area is defined as:

$$S = S_t / W_t$$  \hspace{1cm} (4)

where $W_t$: sample mass (g).

**Particle characterization by SEM and image analysis**

A scanning electron microscope (Philips SEM 505, Eindhoven, The Netherlands) was used to capture the image of alfalfa particles retained on each sieve. Sample preparation involved spreading adhesive tape used specially for SEM analysis on top of a sample-holding button and inserting the button into the bulk of samples for the surface of the adhesive tape to collect particles. Extra layers of adhered particles were removed with a soft brush in order for the button to form a single layer of separate particles. The image of the particles was video taped during microscopic examination.

The image analysis system consisted of a Panasonic VCR that played back the video tape to a Macintosh Ilfx computer through an image analysis software (MacRail 7.2, Automatic, Inc., Billerica, MA). Because the images of video-taped-alfalfa particles from the SEM were too noisy for the image analysis program to capture the particles exactly, the image of particles was manually traced to facilitate image analysis. This was done by printing the played-back image on a laser printer and then tracing out the printed image of particles on a transparent drawing paper put on top of the printed image using a drawing pen with a fine tip. The traced image was in turn subjected to the image processing system for analysis. The parameters examined were mean particle projection length, width, area, perimeter, and sphericity (roundness). These parameters were statistically correlated to the nominal sieve openings.

**Size and shape related parameters**

The following size and shape related parameters of alfalfa grind were determined: median size (geometric mean diameter) of alfalfa particles and its standard deviation; surface area of alfalfa particles; number of particles per unit mass; and mean particle projection length, width, area, perimeter, and sphericity.

A number of distribution functions were fitted to the data of mass fraction on each sieve vs sieve aperture size. The non-linear regression was performed by means of a computer software called 'TABLECURVE' developed by Jandel Scientific (1994). Using the model constants obtained, the median particle size and the standard deviation of the log-normal distribution were determined.

Based on the determined median size and the standard deviation of the log-normal distribution, the surface area and the number of particles per unit mass were calculated using Eqs. 5 and 6 (ASAE 1995), respectively:

$$S = \frac{\beta_s W_t}{\beta_v} \exp \left[ 0.5 \left( \ln \sigma \right)^2 \right]$$  \hspace{1cm} (5)

where:

- $S$: surface area of sample (m²),
- $\beta_s$: shape factor for calculating surface area of particles.
The particle density of alfalfa grind, \( \rho \), was measured by an air comparison pycnometer (Model 930, Beckman Instruments, Inc., Fullerton, CA). Twelve replications were taken.

The mean particle projection length, width, area, perimeter, and sphericity for the alfalfa particles on each individual sieve were determined using SEM and the image analysis system. Particle length and width were defined as the length and width of a rectangular box circumscribing the projected area of a particle and oriented along the major and minor axis. Since the particles were randomly oriented during SEM imaging, the overall roundness of alfalfa particles actually reflected their sphericity. Therefore, particle sphericity was calculated using Eq. 7 that was available in the manual of MacRail 7.2 (Automatix, Inc., Billerica, MA):

\[
R = \frac{4\pi A}{P^2}
\]

where:
- \( R \) = sphericity of the projected area of a particle (between 0 and 1),
- \( A \) = projected area of a particle, and
- \( P \) = perimeter of the projected area of a particle.

**RESULTS AND DISCUSSION**

**Particle size distribution**

Figure 1 shows the distribution of the percent mass retained on each test sieve in relation to the nominal sieve openings for three repeated trials (no measurement was done between 850 and 1700 sieve openings). It shows the skewness in shape that is typical of ground agricultural products such as flour, soybean meal, and ground corn (Pfost and Headley 1976). Most particles were in the range of 200 to 400 \( \mu \)m. Figure 2 shows particle size distribution when the abscissa was transformed to the log scale. The corresponding cumulative undersize curve (mean of three replications) was also calculated and plotted in Fig. 2. The experimental data of Fig. 1 were used to estimate constants in 19 distribution functions. A log-normal distribution function (Eq. 8) was the best fitted equation among all \( (R^2 = 0.81) \). Equation 8 is plotted as "estimated" in Fig. 2. The fitted curve in Fig. 2 did not show a sharp peak to cover the three data points. No intermediate data were available to force the curve to go through these points.

\[
Y = 11.74 \exp \left[ -0.5 \left( \frac{\ln \left( \frac{X}{238.01} \right)}{0.65} \right)^2 \right]
\]

where:
- \( Y \) = mass percentage retained on sieves, and
- \( X \) = nominal sieve openings (\( \mu \)m).

The median size and the standard deviation for the log-normal distribution were found to be 238.01 \( \mu \)m and 0.65, respectively. The standard deviation of the particle size was calculated at 165.83 \( \mu \)m based on the formula proposed by Sokhansanj and Yang (1996). The relatively small standard deviation of log-normal distribution, i.e., 0.65, signified that most particles of the alfalfa grind were in the sizes close to their median size and that the uniformity of particle size was good.
Particle density
Table I lists the results of twelve measurements of particle density. These measurements ranged from 1390.1 to 1598.6 kg/m³ at the moisture content of 5.3 %wb. The average particle density was 1453.4 ± 61.5 kg/m³.

Specific surface area of particles
The measured and calculated specific surface areas were 0.750 m²/g and 0.021 m²/g, respectively. The number of particles per gram charge were calculated to be approximately 652,000 (spheres) and 342,000 (cubes) using Eq. 6.

The specific surface area is known to increase if a geometric shape deviates from spherical, since a sphere has the minimum specific surface area of all geometric configurations. Thus, the calculated specific area, assuming that alfalfa particles are spherical, should be less than the measured value. However, the measured specific surface area (0.750 m²/g) was substantially higher than the calculated value (0.021 m²/g). The possible reason was that the particles of alfalfa grind were porous in nature and full of cracks and fissures on their surface due to the abrasion and shear during size reduction. The measured surface area should therefore be understood as the total surface area that included the external surface area plus that of the internal pores, cracks, or fissures. The calculated area accounted for only the external surface area of geometric particle shape. To study this further, we checked the derivation of Eq. 5 as well as Eq. 6 and proved the correctness of these two equations. We also examined the data of some soil samples that had been measured in the Autosorp-1, such as those by Violante and Huang (1992) and Dynes and Huang (1995), and found that they experienced similar results, i.e., measured values were higher than the calculated ones. The six replications of alfalfa sample yielded surface area ranging from 0.266 to 1.072 m²/g. It seemed unlikely that the deviation was due to the inaccuracy of the Autosorp-1. Despite the above discussion, the true reasons for the deviation needs further investigation.

Particle characterization
In Fig. 3, the projected image of the particles retained on the sieve of 90-μm opening is shown. Most particles were in the shape of parallelopipeds and not spheres. Table II was based on the image analysis, where five parameters including the mean particle projection length, width, area, perimeter, and particle sphericity retained on individual sieves were given. The length, width, area, and perimeter were in the ranges of 0.074-0.979 mm, 0.034-0.425 mm, 0.002-0.295 mm², and 0.0.188-2.421 mm, respectively, in the sieve openings from 20 to 850 μm. Regression analysis showed that these parameters followed a linear relationship with sieve openings (R² ranging from 0.96 to 0.99). The data showed that particles on

Table I: Particle density of alfalfa grind by air comparison pycnometry

<table>
<thead>
<tr>
<th>Sample mass (g)</th>
<th>Measured volume (10⁻⁶ m³)</th>
<th>Particle density (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.1926</td>
<td>4.42</td>
<td>1401.0</td>
</tr>
<tr>
<td>8.8688</td>
<td>6.38</td>
<td>1390.1</td>
</tr>
<tr>
<td>11.8041</td>
<td>8.52</td>
<td>1385.5</td>
</tr>
<tr>
<td>14.8468</td>
<td>10.44</td>
<td>1422.1</td>
</tr>
<tr>
<td>16.2840</td>
<td>11.20</td>
<td>1453.9</td>
</tr>
<tr>
<td>17.5124</td>
<td>11.61</td>
<td>1508.4</td>
</tr>
<tr>
<td>19.2472</td>
<td>12.04</td>
<td>1598.6</td>
</tr>
<tr>
<td>19.4781</td>
<td>13.67</td>
<td>1424.9</td>
</tr>
<tr>
<td>19.5827</td>
<td>13.82</td>
<td>1417.0</td>
</tr>
<tr>
<td>24.1570</td>
<td>16.32</td>
<td>1480.2</td>
</tr>
<tr>
<td>25.0132</td>
<td>17.15</td>
<td>1458.5</td>
</tr>
<tr>
<td>25.0344</td>
<td>16.68</td>
<td>1500.9</td>
</tr>
</tbody>
</table>

Mean 1453.4
Standard deviation 61.4

Table II: The mean projection length, width, area, perimeter, and sphericity (roundness) of alfalfa particles in the sieve openings from 20 to 850 μm

<table>
<thead>
<tr>
<th>Aperture (μm)</th>
<th>Length (mm)</th>
<th>Width (mm)</th>
<th>Area (mm²)</th>
<th>Perimeter (mm)</th>
<th>Sphericity (roundness)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>0.074</td>
<td>0.034</td>
<td>0.002</td>
<td>0.188</td>
<td>0.568</td>
</tr>
<tr>
<td>45</td>
<td>0.098</td>
<td>0.045</td>
<td>0.003</td>
<td>0.246</td>
<td>0.609</td>
</tr>
<tr>
<td>63</td>
<td>0.144</td>
<td>0.066</td>
<td>0.006</td>
<td>0.363</td>
<td>0.602</td>
</tr>
<tr>
<td>90</td>
<td>0.242</td>
<td>0.104</td>
<td>0.016</td>
<td>0.591</td>
<td>0.594</td>
</tr>
<tr>
<td>125</td>
<td>0.274</td>
<td>0.145</td>
<td>0.028</td>
<td>0.717</td>
<td>0.612</td>
</tr>
<tr>
<td>180</td>
<td>0.268</td>
<td>0.150</td>
<td>0.031</td>
<td>0.885</td>
<td>0.540</td>
</tr>
<tr>
<td>300</td>
<td>0.350</td>
<td>0.194</td>
<td>0.044</td>
<td>0.934</td>
<td>0.638</td>
</tr>
<tr>
<td>425</td>
<td>0.510</td>
<td>0.234</td>
<td>0.071</td>
<td>1.328</td>
<td>0.549</td>
</tr>
<tr>
<td>600</td>
<td>0.780</td>
<td>0.346</td>
<td>0.187</td>
<td>1.927</td>
<td>0.619</td>
</tr>
<tr>
<td>850</td>
<td>0.979</td>
<td>0.425</td>
<td>0.296</td>
<td>2.421</td>
<td>0.597</td>
</tr>
</tbody>
</table>

Fig. 3. A SEM micrograph showing the alfalfa particles retained on the sieve of 90-μm opening.
each sieve had similar mean sphericity (in the range of 0.54-0.64), although other parameters varied widely. The average sphericity calculated from a total of about 2130 particles retained on all sieves was 0.601 (standard deviation 0.044).

CONCLUSIONS
The following conclusions are made about the alfalfa grind used in this study:

1. Particle size of alfalfa grind could be best described by a log-normal distribution. The median particle size was 238 \( \mu m \) with standard deviation of 166 \( \mu m \).
2. The particle density was in the range of 1390 to 1599 kg/m\(^3\) at 5.3\%wb moisture content. The average particle density was 1453 kg/m\(^3\) with a standard deviation of 62 kg/m\(^3\).
3. The measured and calculated surface area of alfalfa particles were 0.750 m\(^2\)/g (standard deviation 0.259) and 0.0215 m\(^2\)/g, respectively.
4. Based on the results of SEM and image analysis, the mean particle length, width, area, and perimeter were in the ranges of 0.074-0.979 mm, 0.034-0.425 mm, 0.002-0.295 mm\(^2\), 0.188-2.421 mm, respectively, in the sieve openings from 20 to 850 \( \mu m \). These parameters followed fairly good linear relationships with sieve openings. The sphericity of alfalfa particles ranged from 0.54 to 0.64. The mean sphericity calculated from 2132 particles was 0.601 with a standard deviation of 0.044. The sphericity remained relatively constant over all sieve openings involved.

ACKNOWLEDGMENT
The authors thank Dr. P.M. Huang, Department of Soil Science, University of Saskatchewan for his assistance in particle surface area measurement and Mr. M. Romanik, Department of Electrical Engineering, University of Saskatchewan for his assistance in the image analysis.

REFERENCES


