Size and shape related characteristics of alfalfa grind

W. YANG[1], S. SOKHANSANJ[1], W.J. CRERAR[1] and S. ROHANI[2]

[1]Department of Agricultural and Bioresource Engineering; and [2]Department of Chemical Engineering, University of Saskatchewan, Saskatoon, SK, Canada S7N 5A9. *Received 5 June 1995; accepted 27 May 1996.*

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/m^3 (average 1453 ± 62 kg/m^3). 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 m^2/g and 0.0215 m^2/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 mm^2, 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

Une analyse granulométrique d'une mouture commerciale de luzerne a été faite avec la méthode des tamis. La forme des particules de la mouture de luzerne a été examinée avec un microscope électronique à balayage et un analyseur d'image. La granulométrie était le mieux décrite par une distribution log-normale. La grosseur médiane des particules (moyenne géométrique des diamètres) était de 238 μm avec un écart-type de 166 μm. La densité réelle mesurée avec des pycnomètres à air s'étalait de 1390 à 1599 kg/m^3 (moyenne de 1453 ± 62 kg/m^3). L'aire des particules mesurée avec la méthode BET utilisant la sorption d'azote était de 0.750 ± 0.259 m^2/g alors qu'on a calculé une valeur de 0.0215 m^2/g avec la méthode ASAE S319.2. Le microscope à balayage électronique et l'analyseur d'image ont servi à caractériser les particules retenues sur les tamis dont les ouvertures allaient de 20 à 850 μm. On a trouvé des valeurs pour les différents paramètres qui variaient de 0.242 à 0.979 mm pour la longueur de projection moyenne, de 0.034 à 0.425 mm pour la largeur, de 0.002 à 0.295 mm^2 pour l'aire, de 0.188 à 2.421 mm pour le périmètre, et de 0.54 à 0.64 pour ce qui est du coefficient de sphéricité. Une analyse de régression a montré que ces paramètres variaient de façon linéaire avec la dimension des ouvertures des tamis.

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 s 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:

$$\frac{1}{w\left(\frac{p_o}{p}-1\right)}=\frac{1}{w_mC}+\frac{C-1}{w_mC}\left(\frac{p}{p_o}\right) \quad (1)$$

where:

w = mass of adsorbate in the material (g),
p = partial pressure of adsorbate (Pa),
p_o = saturation pressure of adsorbate (Pa),
w_m = mass of the adsorbate formed in monolayer (g), and
C = constant.

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

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

Rearranging these expressions gives:

$$w_m = 1/(\alpha+\beta) \quad (2)$$

The total surface area is then calculated as:

$$S_t = w_m N A_{cs}/M \quad (3)$$

where:

N = Avogadro's number (6.023 x 10^{23} molecules/mol),
M = molecular weight of adsorbate (nitrogen), and
A_{cs} = cross-sectional area of nitrogen (0.162 nm^2 at 77 K).

The specific surface area is defined as:

$$S = S_t/W_t \quad (4)$$

where W_t = sample mass (g).

Measurement was conducted in a device called Autosorp-1 (Quantachrome Corp., Syosset, NY). The device is capable of automatically determining surface area, porosity, and isotherms and carrying out pore structure analysis for powders. The procedure involved five steps: 1) weighing (accurate to 0.0001 g) a sample of 0.2-0.3 g; 2) loading the sample into a sample cell; 3) outgassing under 1.33 Pa vacuum for 22 h with insertion of a steel rod on top of the sample to prevent it from elutriation during evacuation; 4) automatic isotherm measurement at low relative pressure range; and 5) programmed data reduction. The system was connected to a CompuAdd-810 computer and the resultant specific surface area was printed out automatically at the end of a test. Before the tests, the device was calibrated according to its operation manual. Six replicates were carried out.

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 IIfx computer through an image analysis software (MacRail 7.2, Automatix, 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:

$$S=\frac{\beta_s W_t}{\beta_v \rho \mu}\exp\left[0.5(\ln\sigma)^2\right] \quad (5)$$

where:

S = surface area of sample (m^2),
β_s = shape factor for calculating surface area of particles

(cubical, $\beta_s = 6$; spherical, $\beta_s = \pi$),
β_v = shape factor for calculating volume of particles (cubical, $\beta_s = 1$; spherical, $\beta_s = \pi/6$),
ρ = particle density of material (kg/m^3),
μ = median size of particles (m),
$\ln\sigma$ = standard deviation of log-normal distribution, and
W_t = mass of a charge (kg).

$$N_t = \frac{W_t}{\beta_v \rho \mu^3} \exp\left[4.5(\ln\sigma)^2\right] \quad (6)$$

where N_t = number of particles in a mass of W_t.

The particle density of alfalfa grind, ρ, 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):

$$R = \frac{4\pi A}{P^2} \quad (7)$$

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 µm. 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.

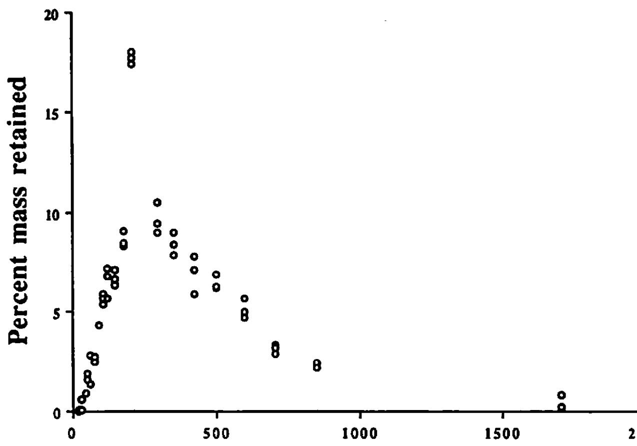

Fig. 1. The mass percentage of alfalfa grind retained on individual test sieves in relation to sieve openings.

$$Y = 11.74 \exp\left[-0.5\left(\frac{\ln\left[\frac{X}{238.01}\right]}{0.65}\right)^2\right] \quad (8)$$

where:
Y = mass percentage retained on sieves, and
X = nominal sieve openings (µm).

The median size and the standard deviation for the log-normal distribution were found to be 238.01 µm and 0.65, respectively. The standard deviation of the particle size was calculated at 165.83 µm 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.

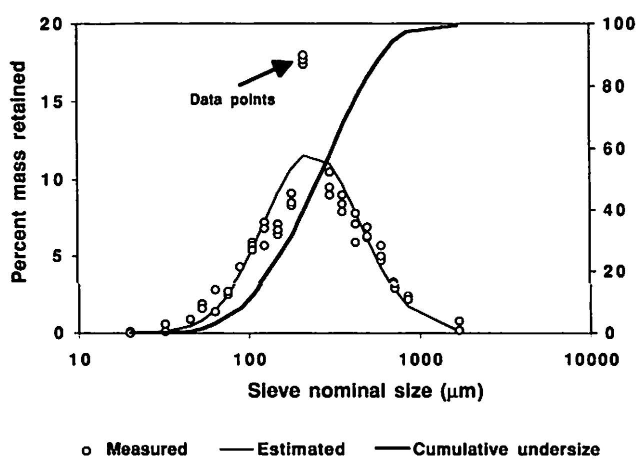

Fig. 2. Log-normal and cumulative undersize distributions of alfalfa grind particles used in this study.

Particle density

Table I lists the results of twelve measurements of particle density. These measurements ranged from 1390.1 to 1598.6 kg/m^3 at the moisture content of 5.3 %wb. The average particle density was 1453.4 ± 61.5 kg/m^3.

Specific surface area of particles

The measured and calculated specific surface areas were 0.750 m^2/g and 0.021 m^2/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 m^2/g) was substantially higher than the calculated value (0.021 m^2/g). The possible reason was that the particles of alfalfa grind were porous in nature and full of cracks and fissures on their surface because of 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 m^2/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.

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

Sample mass (g)	Measured volume (10^{-6} m^3)	Particle density (kg/m^3)
6.1926	4.42	1401.0
8.8688	6.38	1390.1
11.8041	8.52	1385.5
14.8468	10.44	1422.1
16.2840	11.20	1453.9
17.5124	11.61	1508.4
19.2472	12.04	1598.6
19.4781	13.67	1424.9
19.5827	13.82	1417.0
24.1570	16.32	1480.2
25.0132	17.15	1458.5
25.0344	16.68	1500.9
	Mean	1453.4
	Standard deviation	61.4

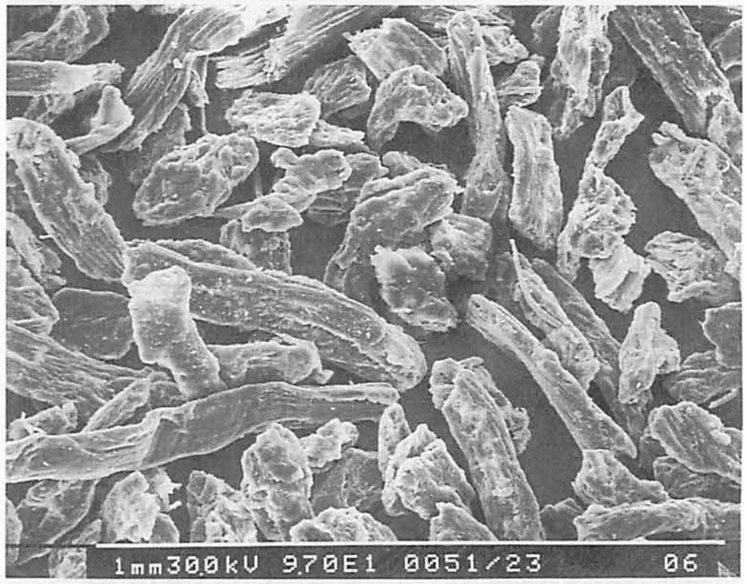

Fig. 3. A SEM micrograph showing the alfalfa particles retained on the sieve of 90-μm opening.

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 mm^2, 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 (R^2 ranging from 0.96 to 0.99). The data showed that particles on

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

Aperture (μm)	Length (mm)	Width (mm)	Area (mm^2)	Perimeter (mm)	Sphericity (roundness)
20	0.074	0.034	0.002	0.188	0.568
45	0.098	0.045	0.003	0.246	0.609
63	0.144	0.066	0.006	0.363	0.602
90	0.242	0.104	0.016	0.591	0.594
125	0.274	0.145	0.028	0.717	0.612
180	0.268	0.150	0.031	0.885	0.540
300	0.350	0.194	0.044	0.934	0.638
425	0.510	0.234	0.071	1.328	0.549
600	0.780	0.346	0.187	1.927	0.619
850	0.979	0.425	0.296	2.421	0.597

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 μm with standard deviation of 166 μm.
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 μm. 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.

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