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Morphological Properties and Breakage Behavior of Three Ground Biofeedstocks by Hammermilling

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Abstract. *Size reduction is one of the most important first steps during processing of biofeedstocks for food, feed, fuel or fiber production. Size reduction not only improves conversion processes because of the creation of larger reactive surface areas, but is used to also improve material handling in the process. This study investigated the particle size and size distribution, and the morphological changes of three biofeedstocks, switchgrass, corn kernels and soybean seeds ground by hammermilling through three screen sizes, 6.4 mm, 3.2 mm and 1.6 mm. Particle sizes were significantly different among screen sizes, however significant differences among feedstock type were only found for feedstock passing through 6.4 mm and 3.2 mm hammermill screens. Of the three feedstocks, ground corn exhibited the smallest particle size as expressed by its geometric mean diameter (d_{wg}), while soybean seeds exhibited the largest d_{wg}. The morphological features of all the feedstocks expressed as circularity, roundness and aspect ratio did not change significantly as was initially hypothesized. While ground switchgrass had very high aspect ratios (10 – 12) and low circularity (0.20) and roundness (0.10), the opposite was seen for ground corn and soybean which exhibited more spherical particles. It was concluded that the breakage behavior of biofeedstocks is an inherent material property that changes very little with size reduction by hammermilling.*

Keywords. Biomass, Particle analysis, Morphology, Bulk handling, Size reduction.

Introduction

Size reduction by grinding biofeedstocks into powders or particulate solids is one of the common first steps of transformation to forms that allow for easy characterization, handling and conversion. Operations that utilize biomass feedstock for fuel, food, feed or fiber generally require size reduction as a processing step to fractionate and extract value components from them. For grain and lignocellulosic biomass feedstocks, hammermills are used to grind material to powder form, in size ranges from 0.5 mm to 25 mm or less. Grinding is a high energy intensive process, with about 5% of all electricity generated used in size reduction (Rhodes, 1998). During size reduction, particles of whatever shape and size are reduced to smaller sizes than the original. Size reduction of a particle also causes morphological changes to the particle. As particles are ground from larger solids to fine powders, it is envisaged that a larger surface area is created for each subsequent size reduction. Additionally, the shape of the particle will tend to shift towards a more spherical shape. This means a particulate feedstock with high aspect ratios, such as switchgrass would be reduced to particles that are more spherical in shape. The higher the sphericity of the particle, the less will be its tendency to interlock during bulk handling. However, because of the inherent differences in material property of various biofeedstocks, size reduction might yield different changes in particle shape, depending on the material breakage behavior. While the effect of hammermilling on particle size and particle size distribution of biofeedstocks have been investigated (Mani et al., 2004; Zhou et al., 2008; Bitra et al., 2009), studies on the effect of size reduction by hammermilling on particle shape are lacking. So does particle size reduction by hammermilling and subsequent reduction to smaller particle sizes result in spherically shaped particles? Therefore, an understanding of the effect of particle size reduction on particle shape is very important in evaluating the handling improvements of grinding on a particular type of feedstock.

The bulk properties of biomass feedstock can be highly variable due to their breakage behavior during comminution (size reduction). The behavior of bulk biomass solids is composed of the sum of its single particle characteristics, which in turn are affected by the inherent nature of the raw feedstock physical structure. The physical structure of biofeedstock does affect its breakage behavior during comminution. Drzymala (1993) stated that the size reduction of biomass changes the particle size and shape, increases bulk density, improves flow properties, increases porosity and generates new surface area. By estimating parameters from particle size distribution and the morphology of a particular biomass grind size, the breakage behavior, as well as its bulk behavior, can be evaluated. Ganesan et al. (2005) stated that particle size and distribution are significant to the bulk properties of flowability, density and compressibility because a small change in particle size will cause a significant difference in the resulting flowability. Mani et al. (2004) studied the physical properties (particle size, particle size distribution and geometric mean diameter) of switchgrass, corn stover, and wheat and barley straws of different particle sizes which were grinded by hammermilling. Results showed that all four feedstock (moistures less than 9%, wet basis) ground to sieve sizes 3.2 mm, 1.6 mm and 0.8 mm had geometric mean diameters ranges of 0.41 – 0.69 mm, 0.28 – 0.38 mm, and 0.19 – 0.32 mm, respectively. They further concluded that larger particles resulted in smaller bulk and particle densities for all four feedstock which can be attributed to the bulky nature of the larger particles as they occupy more pore volume than smaller particles. However, none of these studies analyzed the effect of size reduction on particle shape.

Feedstock grinding equipment also has an effect on the breakage behavior and ultimately efficient handling of biomass in bulk form. Smeenk and Brown (1998) reported the effects of switchgrass preparation for power generation in the Chariton Valley Project; the original approach employed preparation that involved shredding switchgrass bales in a tub grinder which produced non-uniform material breakage resulting in varying lengths of switchgrass from less than 0.5 cm to as long as 20 cm. Ultimately this resulted in a difficulty in feeding material that nested and easily clogged up the handling and discharge systems. A more successful approach which was used processed the switchgrass with a hammermill resulting in more efficient breakage. For example, a 2.5 cm screen resulted in 95% of the switchgrass having a length less than 2.5 cm.

Three common biofeedstocks under the categories of starchy grains, oilseeds and lignocellulosics are corn, soybean and switchgrass, respectively. These feedstocks undergo grinding during their conversion process to a feed, fuel, fiber or value-added products. Switchgrass (*Panicum vigratum, L.*), a perennial warm-season grass and dedicated energy crop can be liquefied, gasified, pelletized or burned directly in biorefineries or existing coal operating units. They are harvested as long-cut baled forage feedstock having low density and poor flowing needle-like particles when ground compared to powders and granular solids. Corn (*Zea mays L.*) kernels are a natural flowable pellet with many industrial applications such as food, feed, fuel ethanol, chemicals, and biopolymers. Soybean (*Glycine max*) seeds are also a natural flowable pellet, but more circular in appearance than corn kernels, with applications for biodiesel production, animal feed, cooking (oils, shortenings and margarines) and other industrial uses such as lubricants.

The overall goal of this study was to understand the breakage behavior of three different types of biofeedstocks. We hypothesized that as particle size is reduced by grinding using a hammermill, the morphological characteristics of particles will move from less spherical towards sphericity. This means that as particle size reduces, the aspect ratio of particles will decrease toward one (more spherical), thereby improving the flowability of the bulk. The primary objectives pursued were: (1) characterize particle size and size distribution of corn kernels, soybean seeds and switchgrass particles reduced by hammermilling through 6.4 mm , 3.2 mm and 1.6 mm screens, and (2) evaluate the morphological changes of corn kernels, soybean seeds and switchgrass particles ground by hammermilling and screened through 6.4 mm, 3.2 mm to 1.6 mm.

Materials and Methods

Sample Preparation

The three biomass feedstock used in this study were switchgrass, corn kernels and soybean seeds. Switchgrass samples were taken from a bale which was stored under a tarp in outdoor storage. Corn kernels and soybean seeds were collected from Purdue's Agronomy Farm as whole kernels/seeds in bulk. The varieties of these feedstocks were not particularly noted, except for switchgrass which was Cave 'N' Rock. Corn (No. 2 yellow shelled corn) and soybean used were assumed to be of regular commodity grain and oilseeds. The initial moisture contents of all feedstocks investigated were determined prior to grinding. Moisture content of switchgrass was determined according to ASAE Standard S358.2 (ASAE, 2004) by weighing about 25 grams of material and drying it in an air oven for 24 hours at 103°C. For corn and soybean, moisture content was determined according to ASAE Standard S352.1 (ASAE, 2004) by weighing about 15 grams of material and drying it in an air oven for 72 hours at 103°C. All moisture content results were determined on a percent (%) wet basis.

Grinding of the feedstocks was performed using a hammermill (Glenn Mills Inc. model 1200, Clifton, NJ). During grinding, particles were reduced by hammermilling and screening through

6.4 mm, 3.2 mm and 1.6 mm (1/4, 1/8 and 1/16 inch) hammermill screens that were installed just below the rotating hammers. The grind sizes were selected on the basis of the work conducted by Zhou et al. (2008) for corn stover and Zhou and Ileleji (2005) for corn stover and switchgrass. Unlike the grinding procedure used by Zhou et al. (2008) which grinded corn stover in sucession by passing ground feedstock from one screen size through to the next, moving from the largest 6.4 mm to the smallest 1.6 mm, the ground feedstocks for each screen size in this study was screened through that screen size once from the beginning. Feedstocks were manually fed into the hammermill feed hopper and a 1100 cfm JET Dust Collector (model DC-1100A, Taiwan) air-assist dust vacuum was used to aid the pulling of ground material from the grinding chamber through the hammermill screens. Each feedstock preparation resulted in three separate grind sizes which were produced by running three grinding trials (three replications) in succession.

Sieve Analysis

Particle size (PS) and particle size distribution (PSD) were determined according to ASAE S319.3 (*ASAE Standards*, 2004). To determine PS and PSD after grinding to each size of ground feedstock, a Ro-Tap shaker (RX-29, Tyler Inc., OH) composed of U.S. standard sieves nos. 3 to 270 (4.76 mm to 0.053 mm sieve opening) was filled with a charge of ground material. The amount (charge) of ground corn kernels and soybean seeds placed in the Ro-Tap shaker were 100 grams per grind size each as recommended by the standard procedure. However, due to the bulky nature of switchgrass, volumetric limitations of the sieves allowed only 40 grams to be used for PS and PSD analysis.

The mass of material retained on each of the sieves in the nest after shaking using the Ro-Tap shaker was used to determine the geometric mean size (d_{wg}), geometric standard deviation (S_{wg}). One-way analysis of variance (ANOVA) using PROC GLM procedure (at $\alpha = 0.05$) on SAS software (SAS, 1999) was conducted among the screen sizes for each feedstock type and among feedstock types in each screen size to detemine whether there were significant differences among the particle sizes through screens 6.4 mm, 3.2 mmand 1.6 mm. Additionally, Tukey tests was conducted using SAS to rank the particle sizes among screen sizes for a feedstock type and among feedstock types for a screen size.

Image Analysis

Feedstock material of all three biofeedstock types used for image analysis was determined based on U.S. sieve nos. that contained particles making at least 75% of the bulk. For switchgrass, the sieves that made up 75% (30 grams) of the bulk of the 6.4 mm grind size were U.S. sieve nos. 12, 16, 20 and 30. For the 3.2 mm grind size, U.S. sieve nos. 16, 20, 30 and 40 represented 75% of the bulk and for the 1.6 mm grind size, U.S. sieve nos. 20, 30, 40, 50 and 70 represented 75% of the bulk. In the case of corn kernels, the sieves which made up 75% (75 grams) of the bulk for the 6.4 mm grind size were from U.S. sieve nos. 12, 16, 20, 30, 40 and 50, for the 3.2 mm grind size the U.S. sieve nos. were 12, 16, 20, 30, 40 and 70 and for the 1.6 mm grind size the U.S. sieve nos. were 20, 40, 50 and 70. For soybean seed grinds, the 75% bulk from the 6.4 mm U.S. sieve nos. were 8, 12, 16, 20 and 30, for the 3.2 mm grind size the U.S. sieve nos. were 12, 16, 20, 30 and 50, and for the 1.6 mm grind size the U.S. sieve nos. were 20, 30, 40, 50 and 70.

In order to extract the morphological features of the grind particles, 300 particles from each of the sieves making 75% of the bulk of each feedstock type were captured by imaging using a Nikon stereo microscope and analyzed using the IMAGEJ software program (NIH *Image*, http://rsbweb.nih.gov/ij/). IMAGEJ is a free software program used to analyze images and is offered on different operating systems (OS) platforms. Particle imaging was conducted using a digital camera mounted on the microscope (0.5x objective lens) to capture live images of a few particles at a time which were placed under the microscope's lens. After adjusting the particles under the microscope to achieve the best possible image of each individual particle, the image was captured and saved as a tiff file. This process was repeated until images of 300 particles were captured from each selected sieve. The Photoshop program (Adobe Photoshop 6.0, Adobe Systems, San Jose, CA) was used to clean the background of the images, making the background white and the particles black as seen in Figure 1. This was done so that the IMAGEJ program could easily differentiate the exact particles that needed to be analyzed for geometry.

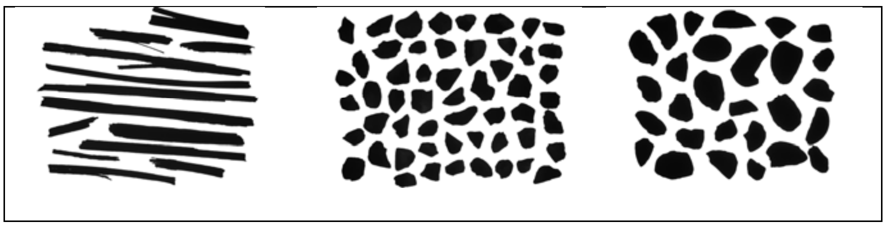

Figure 1. Images of ground switchgrass, corn kernels and soybean seeds (L-R) after background cleaning.

The photo of a stage micrometer was taken for each sieve to calibrate the IMAGEJ program. This was conducted using the number of pixels in the photo of the stage micrometer and the length shown on the stage micrometer. After the scale was set, the image threshold was adjusted and the measurements were set to measure the area, circularity, perimeter (p) and fit ellipse. Equations for each parameter are displayed in equations 1-3. A range was selected for the measurements prior to generating a results table for the particles. Each image was numbered and corresponded to the results table. This process was repeated for each image that was taken from each sieve for all three hammermill screen sizes.

$$\text{Aspect Ratio} = \frac{d_{max}}{d_{min}} \qquad \text{(Eq. 1)}$$

$$\text{Roundness} = \frac{4A}{\Pi d_{max^2}} \qquad \text{(Eq. 2)}$$

$$\text{Circularity (FF)} = 4\Pi \frac{A}{p^2} \qquad \text{(Eq. 3)}$$

Results and Discussions

Particle Size and Distribution Analysis

The initial moisture contents of the ground feedstocks, switchgrass, corn kernels and soybean seeds were 10.0, 9.0 and 10.3%, respectively. Also note that the initial particle sizes of switchgrass (straws) was quite different and larger than that of corn kernels or soybean seeds. Particle size and distribution analysis were evaluated based on the geometric mean diameter

(d_{wg}) and histogram of PSD of ground feedstocks through 6.4 mm, 3.2 mm and 1.6 mm hammermill screens shown in Tables 1 and Figs. 2, 3 and 4, for switchgrass, corn and soybean, respectively. For all three biofeedstocks, there were significant differences among particle sizes based on the d_{wg} between screen sizes of the same feedstock type and between feedstocks of the same screen size. The d_{wg} of switchgrass ground through 6.4 mm and 3.2 mm screens were similar and both significantly larger than particles screened through 1.6 mm. The d_{wg} of all hammermill screen sizes for both ground corn and soybean were significantly different, with 6.4 mm having the largest particles and 1.6 mm the smallest particles. Comparing d_{wg} among feedstock types for a given screen size, only feedstock types ground through the 6.4 and 3.2 mm screen were significantly different. In addition, trends from Tukey ranking tests showed a gradual increase in d_{wg} uniformity between feedstock types. For both 6.4 and 3.2 mm screen sizes, ground soybean had the largest d_{wg}, while ground corn had the lowest d_{wg}. However, switchgrass had the largest d_{wg} for 1.6 mm, while corn still had the lowest d_{wg}. These results indicate that corn is an easier feedstock to grind and less energy may be expended per given change in surface area for corn kernels than for soybean or switchgrass. Further work which takes into account the energy expended during grinding would confirm this finding.

Table 1. Summary of geometric mean diameter and geometric standard deviation for ground switchgrass, corn and soybean through hammermill screens 6.4 mm, 3.2 mm and 1.6 mm.

Feedstock	6.4 mm[1]	3.2 mm	1.6 mm	F value	P-value
Switchgrass	0.872 (0.556) a[2]y	0.804 (0.505) ax	0.435 (0.281) b	153.73	< .0001
Corn	0.757 (0.714) az[3]	0.669 (0.598) by	0.395 (0.232) c	161.89	< .0001
Soybean	1.142 (0.921) ax	0.850 (0.657) bx	0.409 (0.304) c	761.57	< .0001
F value	146.39	34.42	1.73		
P-value	< .0001	0.0005	0.2551		

1 d_{wg} (S_{wg})
2 Values with different letters, a, b, and c are comparisons of d_{wg} between screen sizes for each feedstock which are significantly different at $p > 0.05$
3 Values with different letters, x, y, and z are comparisons of d_{wg} between feedstock types for each screen size which are significantly different at $p > 0.05$

The histogram of the particle size distribution in Figure 2 shows that the particle distribution of switchgrass ground through 6.4 and 3.2 mm screens produced similar distributions. Percent retained on U.S. sieve no. 20 were 33 and 34% for samples ground through 6.4 mm and 3.2 mm, respectively. Switchgrass ground through the 1.6 mm screen is skewed to the right as seen by the retention of 42% of particles on both sieve nos. 40 and 50 combined.

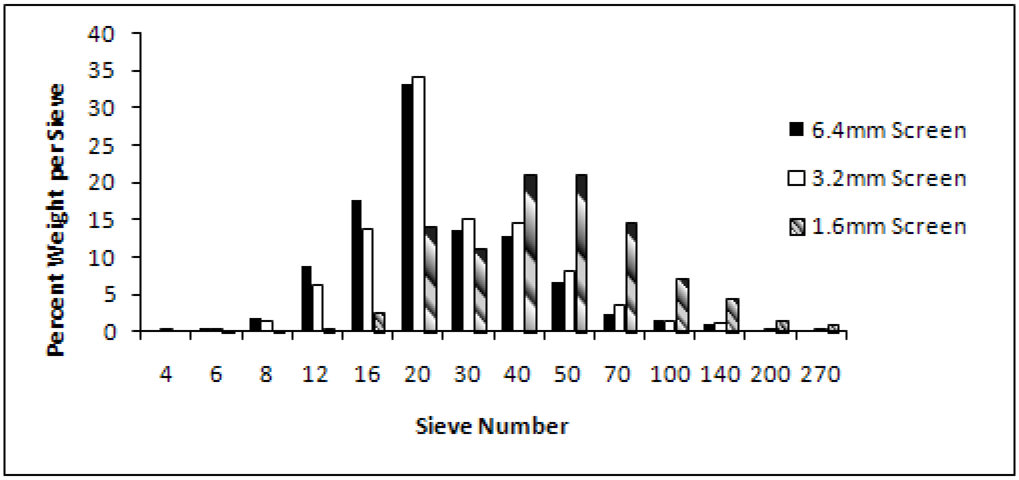

Figure 2. Histogram of particle size distribution of ground switchgrass screened through 6.4 mm, 3.2 mm and 1.6 mm.

PSD of ground corn from all three screen sizes in Figure 3 followed a similar trend as those of ground switchgrass. However, the distributions of ground corn for all screen sizes were more spread than was observed for switchgrass. A lower percent of ground material was retained on U.S. sieve no. 20, about 21 and 23% for ground material through 6.4 mm and 3.2 mm, respectively.

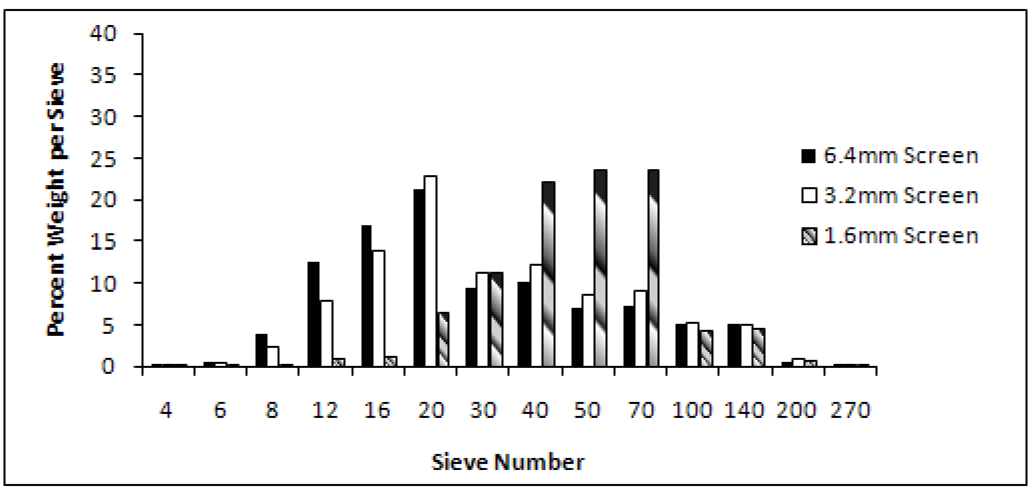

Figure 3. Histogram of particle size distribution of ground corn screened through 6.4 mm, 3.2 mm and 1.6 mm.

PSD of ground soybean for all screen sizes (Figure 4) was different than ground switchgrass and corn. The percent grinds retained on U.S. sieves seem quite different than what was observed for ground switchgrass and corn. The percent ground soybean retained on sieves showed more variation among sieve sizes compared to switchgrass and corn kernels. The largest percent of ground soybean through 6.4 mm screen was retained on U.S. sieve no. 12 (23%) and U.S. sieve no. 16 (21%). For ground soybean through 3.2 mm, U.S. sieve nos. 16

and 20 retained the largest amounts; 20% and 27%, respectively. For ground soybean through 1.6 mm, the largest amount (23%) was retained on U.S. sieve no. 40.

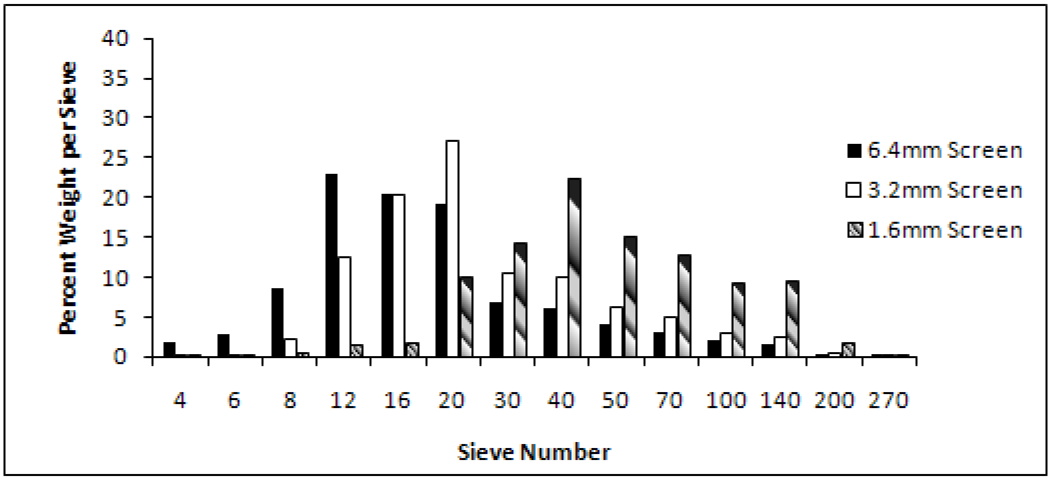

Figure 4. Histogram of particle size distribution of ground soybean screened through 6.4 mm, 3.2 mm and 1.6 mm.

Morphology and Image Analysis

Mean and standard deviation values for image analysis parameters of circularity, aspect ratio and roundness for the three grind sizes taken from 75% of the bulk of ground switchgrass, corn kernel and soybean are presented in Tables 2, 3 and 4, respectively. Statistical analysis, using SAS Proc GLM was used to determine significant differences among these parameters. 0

Image analysis results for switchgrass in Table 2 indicate that the key morphology features (circularity, roundness and the aspect ratio) did not change as the material was ground from 6.4 to 1.6 mm ($\alpha > 0.05$). The low circularity and roundness value and high aspect ratio is indicative of the very poor flow characteristics that ground switchgrass exhibits. Microscopic observations of the particles revealed that ground switchgrass tends to break along its longitudinal axis instead of its latitudinal axis. This unique breakage characteristic coupled with the reduction in particle length preserves the aspect ratio of d_{max} to d_{min}, thereby causing the particles to remain needle shaped with high aspect ratios throughout size reduction.

Table 2. Mean and standard deviation values and ANOVA summary of switchgrass from image analysis.

Hammermill Screen Size	Circularity	Aspect Ratio	Roundness
6.4 mm	0.255 ± 0.042	9.723 ± 1.371	0.144 ± 0.02
3.2 mm	0.208 ± 0.017	12.088 ± 0.660	0.107 ± 0.004
1.6 mm	0.258 ± 0.05	10.276 ± 2.456	0.148 ± 0.04
F value	0.04	0.10	0.08
P-value*	0.8426	0.7636	0.7799

For ground corn, there was significant difference in only circularity among screen sizes (p-value = 0.0017), while no change occurred for aspect ratio or roundness (Table 3). Ground corn is more spherical in shape than switchgrass as shown by its circularity and roundness being close to unity and low aspect ratio compared to switchgrass. Compared with switchgrass, the circularity and roundness of ground corn were 3 times higher and aspect ratios were up to 8 times smaller than that of switchgrass. According to Table 3, the circularity of ground corn reduced slightly as particle size decreased, but the aspect ratio and roundness did not show any relationship pattern with particle size.

Table 3. Mean and Standard deviation values and ANOVA summary of corn samples from image analysis.

Hammermill Screen Size	Circularity	Aspect Ratio	Roundness
6.4 mm	0.773 $\pm$ 0.015	1.456 $\pm$ 0.073	0.717 $\pm$ 0.025
3.2 mm	0.748 $\pm$ 0.016	1.433 $\pm$ 0.115	0.728 $\pm$ 0.039
1.6 mm	0.740 $\pm$ 0.008	1.463 $\pm$ 0.131	0.716 $\pm$ 0.049
F value	15.06	0.0025	0.0049
P-value	0.0017	0.9627	0.9460

Table 4 shows results of image analysis of ground soybean. The results were quite close to those of ground corn kernels. No significant differences were seen among screen sizes for all three morphological characteristics. The morphological features of ground soybean for the three hammermill screens were very similar to those of ground corn. Overall, it was surprising to see that the aspect ratios of ground switchgrass, corn and soybean did not decrease with subsequent size reduction by hammermilling. Neither was the circularity and roundness of these three feedstocks increased by subsequent size reduction. Thus, our initial hypothesis that size reduction decreased the aspect ratio, causing feedstocks to be more circular and round with each size reduction by hammermilling did not hold true for switchgrass, corn and soybean. It appears that each of these biofeedstocks have their inherent breakage behavior which is retained through each subsequent size reduction step.

Table 4. Mean and Standard deviation values and ANOVA summary of soybean from image analysis.

Hammermill Sieve Size	Circularity	Aspect Ratio	Roundness
Soybean 6.4 mm	0.766 $\pm$ 0.006	1.438 $\pm$ 0.065	0.722 $\pm$ 0.027
Soybean 3.2 mm	0.772 $\pm$ 0.013	1.474 $\pm$ 0.116	0.711 $\pm$ 0.044
Soybean 1.6 mm	0.750 $\pm$ 0.015	1.519 $\pm$ 0.135	0.694 $\pm$ 0.047
F value	4.39	1.60	1.30
P-value	0.0563	0.2282	0.2754

Conclusion

This study investigated the breakage behavior of switchgrass, corn kernels and soybean seeds ground through 6.4 mm, 3.2 mm and 1.6 mm screens by hammermilling. For all screen sizes, ground corn had the smallest geometric mean particle size, while ground soybean had the largest geometric mean particle size. Significant differences in particle sizes were seen for all screen sizes as feedstocks were reduced from 6.4 mm to 1.6 mm. Surprisingly, the morphological features analyzed in this study, circularity, roundness and aspect ratio of switchgrass, corn kernels and soybean did not result in significant differences as the feedstock grind size was reduced. Thus, our initial hypothesis that stated as the particle size reduced, the particle tended toward sphericity was not correct. It appears there is an inherent morphological feature exhibited by these biofeedstocks upon breakage, which changed very little between each hammermill screen size and change in surface area. The conclusion is that size reduction will not greatly enhance a poor flowable feedstock having very irregular morphological features that do not favor bulk flow such as a high aspect ratio. It should also be noted that every biofeedstock will exhibit its own unique breakage behavior given the material properties of initial moisture content, feedstock variety and particle size. Therefore, caution should be made in making general assumptions. Future work is recommended to incorporate the measurement of energy expended in grinding thorough quantitative PSD analysis and the use of population balance modeling to evaluate the breakage behavior of these biofeedstocks.

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