The effects of physical and chemical preprocessing on the flowability of corn stover

Nathan C. Crawford^{a,*}, Nick Nagle^a, David A. Sievers^a, Jonathan J. Stickel^a

^a*National Bioenergy Center, National Renewable Energy Laboratory, Golden, CO, USA, 80401*

Abstract

Continuous and reliable feeding of biomass is essential for successful biofuel production. However, the challenges associated with biomass solids handling are commonly overlooked. In this study, we examine the effects of preprocessing (particle size reduction, moisture content, chemical additives, etc.) on the flow properties of corn stover. Compressibility, flow properties (interparticle friction, cohesion, unconfined yield stress, etc.), and wall friction were examined for five corn stover samples: ground, milled (dry and wet), acid impregnated, and deacetylated. The ground corn stover was found to be the least compressible and most flowable material. The water and acid impregnated stovers had similar compressibilities. Yet, the wet corn stover was less flowable than the acid impregnated sample, which displayed a flow index equivalent to the dry, milled corn stover. The deacetylated stover, on the other hand, was the most compressible and least flowable examined material. However, all of the tested stover samples had internal friction angles $>30^\circ$, which could present additional feeding and handling challenges. All of the “wetted” materials (water, acid, and deacetylated) displayed reduced flowabilities (excluding the acid impregnated sample), and enhanced compressibilities and wall friction angles, indicating the potential for added handling issues; which was corroborated via theoretical hopper design calculations. All of the “wetted” corn stovers require larger theoretical hopper outlet diameters and steeper hopper walls than the examined “dry” stovers.

Keywords: Biomass, Feedstock, Flowability, Shear, Rheology, Hopper

*Corresponding author. Tel.: +1 303-384-6215

Email address: nathan.crawford@nrel.gov (Nathan C. Crawford)

Abbreviations

AICS, acid impregnated corn stover; DACS, deacetylated corn stover; GCS, ground corn stover; INL, Idaho National Laboratory; IR, infrared; MCS (dry), dry milled corn stover; MCS (wet), wet milled corn stover; NREL, National Renewable Energy Laboratory.

Nomenclature

α	Maximum hopper half angle ($^{\circ}$)
A_R	Particle aspect ratio (-)
B	Minimum hopper outlet diameter (m)
C	Compressibility (%)
CAS	Critical applied stress (kPa)
f_c	Unconfined yield stress (kPa)
FF	Flow function (-)
ff	Flow factor (-)
ff_c	Flowability (or flow) index = σ_1/f_c (-)
g	Gravitational constant (9.81 m s^{-2})
μ_w	Coefficient of wall friction (-)
l	Particle length (μm)
MC	Moisture content = Moisture mass fraction, w_{H_2O} (%)
n	Number of sized particles (-)
N	Normal stress (kPa)
N_C	Pre-shear critically consolidated normal stress (kPa)
m_w	Mass of water contained in a wet sample (g)
m_t	Total mass of a wet sample (g)
ϕ_i	Angle of internal friction ($^{\circ}$)
ϕ_e	Effective angle of internal friction ($^{\circ}$)
ρ_b	Bulk density (kg m^{-3})
Ra	Surface roughness (μm)
σ_1	Major principal stress (kPa)
τ	Shear stress (kPa)
τ_0	Cohesion (kPa)
V	Sample volume at a given normal stress (cm^3)

V_0	Initial, conditioned sample volume (cm^3)
w	Particle width (μm)

1. Introduction

Continuous and stable biomass feeding is essential for successful operation of a biofuel production facility. Yet, solids feeding equipment commonly becomes jammed, bridged, or blocked with material, resulting in slowed reactor throughput and/or complete process interruption. Despite the significance of achieving uninterrupted feeding and the regularity of biomass feeding issues, more emphasis has been placed on the thermal, chemical, and biological deconstruction of biomass than on biomass handling problems. As a result, only a limited amount of information [1–3] has been published on the design and operation of biomass feeding systems.

Biomass feeding and handling is challenging for a variety of reasons. Biomass particles are inherently heterogeneous, varying greatly in size, shape, density, moisture content, and compressibility. Large particles, excessive moisture, and an insufficient (or too high) pressure differential between the feed hopper and reactor can all lead to feed system failure. In addition, feeders are commonly designed to handle a particular material under a specific set of preprocessing conditions (i.e., milling size, moisture content, chemical additive concentration, etc.). Minor alterations to the biomass, such as changes in moisture content or particle size, can significantly impact solids-handling systems. It is common for a feed system to work well for one material and completely fail for another with similar properties [4].

Further complicating this issue, biomass feedstocks are regularly preprocessed prior to entering reactor feed systems. Preprocessing can be fairly conventional, like milling to pass a certain screen size. However, preprocessing can also include chemical processing to enrich the biomass deconstruction process. Chemical additives not only increase the moisture content within the biomass, but they can potentially influence physical and chemical properties, and as a result, affect flowability.

Analyzing the flowability and physical properties of biomass is key to the design of efficient and effective biomass feeders [3]. Measuring biomass bulk density, compressibility, cohesion, internal friction, yield stress, and wall friction help determine a material’s flowability and inform proper hopper design. However, only a limited number of studies have been published on the flowability of biomass and its impact on feed system design. Fasina [5] evaluated

the affect of mill screen size on the compressibility and flowability of peanut hull, switchgrass, and poultry litter; while Chevanan et al. [6] designed, fabricated, and tested a direct shear cell to measure the shear strength and flow properties of chopped switchgrass, wheat straw, and corn stover. Zhou et al. [7] studied the affects of particle size and moisture content on particle density, bulk density, compressibility index, and porosity of two hybrid corn stovers, while Ileleji and Zhou [8] examined the effects of particle size and moisture content on the angle of repose of bulk corn stover. Samaniuk et al. [9] investigated the use of water soluble polymers as rheological modifiers to reduce the yield stress of wetted corn stover samples using a unique torque rheometer. Gil et al. [10], on the other hand, analyzed the effects of moisture content, particle size, and particle shape on the handling behavior of poplar and corn stover. Miccio et al. [11] studied the flow properties and tendency for arch formation of sawdust and ground olive husk using a ring shear apparatus and an arching tester. A recent study by Crawford et al. [12] evaluated the flowability of pure and blended biomass feedstocks, and linked the flow properties of the biomass materials to their pelleting energy requirements.

Although previous studies have investigated the effects of milling screen size and moisture content on the flow properties of various feedstocks, no study has specifically examined how preprocessing conditions, including chemical additives, affect the flowability of a single biomass species. In this manuscript, our objectives were to: 1) measure the compressibility and flow properties of corn stover under various processing conditions, 2) evaluate how moisture content, particle size, and chemical additives influence corn stover flowability, and 3) use the compressibility, shear cell, and wall friction measurements for theoretical hopper design calculations (see Section Appendix A for details) as a relative method to compare handling strategies for the preprocessed corn stover feedstocks.

2. Materials and Methods

2.1. Materials

Corn stover is a crop residue consisting of all portions of the plant left after corn grain harvest (the stock, stem, leafs, and cob). Corn stover is a desirable biofuel feedstock because it is not used as human or animal food, and large quantities are available as a byproduct of corn agriculture. The

Figure 1: Digital images of the preprocessed corn stovers (scale bar = 2.54 cm).

70 corn stover examined in this study was provided by Idaho National Labo-
 71 ratory (INL, Idaho Falls, ID, USA). All of the milling and/or grinding of
 72 the corn stover was performed by, or contracted out through, INL. The corn
 73 stover was single-pass harvested in Boone County, IA (USA) in the fall of
 74 2011. However, detailed information on the chain of custody for this specific
 75 substrate was unavailable. Therefore, the cultivar cannot be specified and
 76 while the authors believe that this work illustrates the difference between
 77 preprocessing strategies, unknown substrate factors may have influenced the
 78 obtained results.

79 All chemical processing or wetting of the stover was performed at the
 80 National Renewable Energy Laboratory. The following subsections describe
 81 the preprocessing conditions for the five tested corn stover samples.

82 2.1.1. *Ground*

83 Bales of corn stover were fed through a two-stage, full-scale grinding
 84 process using the Feedstock Process Demonstration Unit (PDU) as part of
 85 DOE's Biomass Feedstock National User Facility (BFNUF) located at Idaho
 86 National Laboratory (Idaho Falls, ID, USA). First, material was processed
 87 through a Vermeer BG-480 (Pella, IA, USA), which has two horizontal grind-
 88 ing drums with swinging hammers powered by two, 149 kW motors [13], and
 89 passed through a 5 cm screen. Then, the 5 cm screened material was pro-

90 cessed through a Bliss hammer mill (Ponca City, OK, USA) with a 6.35 mm
91 screen. The ground corn stover (GCS) sample was prepared for a separate
92 feedstock pelleting study.

93 2.1.2. Milled (dry)

94 Baled corn stover was milled in a single-stage milling process using a Bliss
95 hammer mill equipped with a 6.35 mm screen. The dry milled corn stover
96 (MCS (dry)) sample was used as the base feedstock for the remaining corn
97 stover samples (wet milled, acid impregnated, and deacetylated).

98 2.1.3. Milled (wet)

99 DI water was added to a subsample of MCS (dry) material to a target
100 moisture mass fraction of 50 %. The “wet” milled corn stover (MCS (wet))
101 sample was saturated to 50 % total solids in order to match the moisture con-
102 tents of the following preprocessed corn stover materials (acid impregnated
103 and deacetylated).

104 2.1.4. Acid Impregnated

105 The dry milled corn stover was soaked with a 2.0 % mass fraction sulfuric
106 acid (H_2SO_4) in water solution at a total mass fraction of solids of 8 % for 2
107 hours. The acid impregnated corn stover (AICS) slurry was then transferred
108 to a continuous screw press (Vincent Corporation, Model CP10, Tampa, FL,
109 USA) to remove excess liquor and achieve a target moisture mass fraction
110 of ~ 50 %. The selected acid impregnation conditions correspond with pilot-
111 scale dilute acid pretreatment for the biochemical conversion of lignocellulosic
112 biomass to biofuels [14, 15].

113 2.1.5. Deacetylated

114 Dry milled corn stover was soaked in a 0.4 % mass fraction of sodium
115 hydroxide (NaOH) in water solution at a total solids mass fraction of 8 %.
116 The corn stover slurry was mixed continuously at 80°C for 2 hours. The
117 slurry was drained overnight, allowing the free-liquid fraction to be removed.
118 The corn stover solids were then washed in DI water (10 % mass fraction of
119 solids) and mixed for 1 hour. Again, the free-liquid was removed via draining.
120 The washed corn stover solids were then dilute acid impregnated with 0.8 %
121 mass fraction of H_2SO_4 in water at a total solids mass fraction of 8 % and
122 thoroughly mixed at ambient conditions for 2 hours. The deacetylated and
123 acid treated corn stover (DACS) was then pumped to a continuous screw

124 press to remove excess free-liquid and achieve a total mass fraction of corn
125 stover solids of 45 to 50 %. The chosen deacetylation conditions correspond
126 with the State of Technology for pilot-scale deacetylation preprocessing for
127 the biochemical conversion of lignocellulosic biomass to biofuels [16].

128 2.2. Moisture Content

129 The moisture contents of the preprocessed corn stover samples were deter-
130 mined using a semi-automated infrared (IR) balance (Mettler Toledo, model
131 HR83 Halogen, Columbus, OH, USA). The IR balance has two separate op-
132 erating procedures designed specifically for measuring the dry matter content
133 of both wet and “dry” biomass feedstock samples. The oven operated at a
134 maximum temperature of 105 °C and dried the corn stover samples until less
135 than 0.01 % change in dry matter content was detected over a 1 min interval.
136 Moisture content (MC) was reported as the percent mass fraction of water
137 (w_{H_2O}) per the total wet mass of the sample. Sample size ranged from 0.5
138 to 1.0 g, and the samples were evenly distributed across the drying pan. All
139 moisture content measurements were repeated in triplicate and measurement
140 variability was reported as a single standard deviation.

141 2.3. Microscopy

142 Particle size parameters for the preprocessed corn stover samples were ob-
143 tained using optical microscopy and quantitative image analysis (Leica DM
144 3000, Clemex Technologies Inc., Longueuil, Quebec, Canada; data analysis
145 via Clemex Vision PE software, version 6). The optical micrographs not only
146 allowed for sizing of the corn stover materials (particle length and width),
147 but also particle dimensional analysis, namely aspect ratio. Particle dimen-
148 sions of individual particles were measured and categorized into geometrically
149 spaced bins ranging from 25 to 55,500 μm for length (20 total bins) and 25
150 to 5,000 μm for width (14 total bins), which provided full particle length and
151 width distributions. The particle length and width histograms were reported
152 on a number basis, while average particle dimensions were computed on a
153 volume basis [17, 18]. Number based dimensional analysis was avoided due
154 to the high number of fine particles quantified via microscopy, which biases
155 average particle dimensions towards smaller values. In addition, analyzing
156 microscopy results on a volume basis allows for a more direct comparison be-
157 tween other commonly used biomass sizing techniques (i.e., laser diffraction
158 and sieving).

159 2.4. Powder Rheometer

160 The FT4 Powder Rheometer (Freeman Technology, Worcestershire, UK)
161 was used to measure the flow properties of the preprocessed corn stover
162 feedstocks. The FT4 Powder Rheometer is a comprehensive powder tester,
163 capable of measuring compressibility and rotational shear properties, includ-
164 ing wall friction. A full description of the FT4’s capabilities can be found
165 elsewhere [19, 20].

166 Testing was performed with the 50 mm diameter, borosilicate test vessel
167 (85 cm³ test volume). Prior to all testing, samples were conditioned using
168 the 23.5 mm blade to standardize the packing state of each sample (larger
169 diameter blades were avoided because the biomass particles became entangled
170 with the blade leading to a torque overload). The conditioning step consisted
171 of an axial traverse of the blade downward and upward through the full height
172 of the sample. During the upward traverse, the blade lifts the particles and
173 lightly drops them behind the blade. Conditioning aerates and loosens the
174 granular sample, removing any trapped air and/or any pre-consolidation that
175 may have occurred during filling of the vessel.

176 All test methodologies, compressibility, shear cell, and wall friction, were
177 repeated in triplicate (at minimum) to assess measurement variability, with
178 all error bars reported as a single standard deviation.

179 2.4.1. Compressibility

180 The compressibility test involved the compression of a preconditioned
181 sample bed with a vented piston which permits entrained air to escape. Each
182 sample was slowly compacted across a range of normal stresses (N) from 0.5
183 to 21 kPa, the change in bed height was measured at 11 distinct intervals. The
184 compressibility of a sample (C) is defined as the percent change in volume
185 after compression, $C = (V_0 - V)/V_0$; where V_0 is the initial, conditioned
186 volume and V is the volume at a specific normal stress value.

187 2.4.2. Shear Cell

188 Shear cell testing was executed with the 48 mm shear head in the normal
189 force control mode, so as the shear head was rotated, torque was measured
190 while the normal force remained constant (i.e., the shear head displaced
191 vertically during testing). During the shear cell test, the sample was first
192 compacted with the vented piston to a specific pre-shear normal load (3, 6,
193 9, or 15 kPa). After compaction, the piston was removed and the shear head
194 was attached. The shear head was then lowered and its blades were inserted

195 into the sample until the pre-shear normal force was again achieved. The
 196 head was then slowly rotated at 5 mrad s^{-1} , inducing a shear stress. The
 197 sample was sheared continuously until the shear stress reached steady state,
 198 whereupon the sample was considered “critically consolidated” [21]. Next,
 199 the normal stress was reduced, which allowed room for the particles to distend
 200 and move past one another. At that point, a yield stress could be measured.
 201 The pre-shear/shear sequence was repeated five times (as recommended by
 202 Carson and Wilms [22]) at decreasing normal stress values (the sample was
 203 always pre-sheared to the critically consolidated state prior to measurement).
 204 A plot of the normal stress and shear stress pairings was then attained,
 205 referred to as the yield locus. Yield loci are unique and contingent upon
 206 the pre-shear history of the sample. Each yield locus was independently
 207 evaluated using Mohr stress circle analysis to obtain values of: the major and
 208 minor principal stresses, unconfined yield stress, cohesion, and interparticle
 209 friction, as described by Carson and Wilms [22] and Freeman [19].

210 In order to characterize a material’s flowability, Jenike [21] proposed using
 211 the ratio of the major principal stress to the unconfined yield stress, called
 212 the flowability index (ff_c). Based on the flow index value, materials can be
 213 classified as non-flowing, very cohesive, cohesive, easy flowing, or free flowing
 214 [23].

215 *2.4.3. Wall Friction*

216 Wall friction properties are essential for understanding how a consolidated
 217 granular material will interact with the wall material of a storage vessel or
 218 processing equipment. Wall friction tests are similar to shear cell testing.
 219 Instead of shearing the sample against itself (acquiring properties like inter-
 220 particle friction or cohesion), the wall friction test shears the sample along
 221 a disc of material with well defined surface properties. Essentially, the shear
 222 head was replaced with a 48 mm stainless steel coupon (316 grade, surface
 223 roughness, $R_a = 1.2 \text{ } \mu\text{m}$). During wall friction tests, the samples were com-
 224 pacted and pre-sheared at a given normal stress (3, 9, or 15 kPa). As with the
 225 shear cell test, the normal force was decreased and the sample was sheared
 226 until a maximum shear stress was reached (i.e., a wall yield stress was mea-
 227 sured). The shear stress and normal force pairs were then plotted to obtain
 228 a wall friction yield locus. Wall friction coefficients, or angles, were then
 229 extracted from the loci. Given that the structural integrity of the sample
 230 was not destroyed during shearing, the wall friction test requires only one
 231 pre-shear step and five successive shear steps.

232 3. Results and Discussion

233 3.1. Particle Morphology

234 The particle size distributions of the ground and milled corn stover (GCS
235 and MCS (dry), respectively) appear to be significantly different (Figure
236 2a and b). The MCS (dry) material has a much broader particle length
237 distribution with $\sim 35\%$ of its particles longer than $1000\ \mu\text{m}$. The GCS
238 material, on the other hand, has a narrower particle length distribution with
239 only $\sim 5\%$ of its particles longer than $1000\ \mu\text{m}$ (Figure 2a). The particle
240 width distributions for the ground and milled corn stover are fairly similar,
241 but the MCS (dry) sample still has significantly more wide particles. The
242 MCS (dry) material has $\sim 20\%$ of its particles wider than $300\ \mu\text{m}$, while
243 the GCS feedstock has only $\sim 8\%$ of its particles wider than $300\ \mu\text{m}$ (Figure
244 2). The large fraction of long particles found in the MCS (dry) sample are a
245 direct result of milling conditions. The MCS (dry) feedstock was just hammer
246 milled to a $6.35\ \text{mm}$ screen size, while the GCS material was first ground and
247 then hammer milled using a $6.35\ \text{mm}$ screen. The grinding process used to
248 generate the GCS material was more effective at eliminating large particles
249 and producing a more uniform size distribution.

250 The volume-averaged particle lengths of all the materials are provided in
251 Table 2. Again, the GCS material was much smaller than the other examined
252 corn stover samples. Although the milled and chemically processed samples
253 (MCS dry and wet, AICS, and DACS) have slightly different particle lengths
254 (especially the AICS sample), the large variance in the particle length values
255 keep them from being statistically different from one another. In addition, the
256 milled and chemically treated corn stover materials also have similar particle
257 widths that are significantly larger than the ground corn stover sample (Table
258 2).

259 The average aspect ratio (defined as the length to width ratio) was be-
260 tween 9 to 14 for all four of the milled and/or treated corn stover feedstocks,
261 with large maximum measured aspect ratios of ~ 80 to 190 (parentheses in
262 Table 2). However, the GCS material had an aspect ratio of ~ 4 with a max-
263 imum value of ~ 26 . Once again, the ground corn stover feedstock was more
264 uniform in shape and appeared to be devoid of multiple large, string-like
265 particles that bias the particle aspect ratio towards larger values.

266 Overall, the milled and chemically treated corn stover samples (MCS dry
267 and wet, AICS, and DACS) have nearly identical particle sizes (length and
268 width) and size distributions, and fairly similar aspect ratios. The MCS dry

Figure 2: Geometric particle (a) length and (b) width distributions for ground and milled corn stover (GCS and MCS (dry), respectively) using optical microscopy. The solid lines are from a cubic interpolation function to help guide the eye.

Table 2: Moisture content (MC) and volume average particle dimensions of the preprocessed corn stovers via optical microscopy (n = the number of particles sized, l = particle length, w = particle width, and A_R = aspect ratio). The “ $\pm$ ” indicates a single standard deviation, while the parentheses denote a maximum measured aspect ratio.

Corn stover	Abbreviation	MC (%)	n (-)	l (μm)	w (μm)	A_R (-)
Ground	GCS	6.7 ± 0.6	4432	1668 ± 901	257 ± 97	4.3 (26)
Milled (dry)	MCS (dry)	5.2 ± 0.2	692	6205 ± 3218	424 ± 295	9.0 (171)
Milled (wet)	MCS (wet)	49.9 ± 0.5	460	6097 ± 2897	433 ± 230	11.3 (86)
Acid Impregnated	AICS	50.8 ± 1.1	1802	4615 ± 1534	480 ± 194	11.6 (81)
Deacetylated	DACS	44.8 ± 0.8	2165	5960 ± 1728	296 ± 205	13.5 (190)

and wet, AICS, and DACS materials differ only by moisture content and chemical additive. Therefore, differences in their compressibility and flowability should be a direct result of their moisture content and/or chemical processing history, and not due to differences in particle size or shape. The ground corn stover feedstock (GCS), on the other hand, has much smaller particles, more narrow particle length and width distributions, and a more uniform particle shape (as described by the aspect ratio). Thus, any potential differences in the compressibility and flowability of the GCS feedstock will most likely be due to its reduced particle size and increased particle homogeneity (particularly when compared to the MCS (dry) sample).

3.2. Compressibility

Compressibility is a bulk property that quantifies a material’s propensity to become compacted when subjected to a normal force. The compressibility measurement is not a direct evaluation of flowability, but it can reveal the strength of adhesive forces between particles (which are inversely related to flowability). For example, poor flowing bulk solids typically have strong interparticle adhesive forces, which creates large voids within the particle network. These large voids will collapse under compression, leading to increased material compressibility. Therefore, it is anticipated that highly compressible bulk solids will also be poor flowing.

The compressibilities of the preprocessed corn stover feedstocks were as-

Figure 3: Compressibility (% change in volume) of the preprocessed corn stovers as a function of applied normal stress.

290 sessed from 0.5 to 21 kPa (Figure 3). The DACS sample was by far the most
 291 compressible, compressing by over 75 % at 21 kPa (Figure 3). The AICS and
 292 MCS (wet) materials were less compressible and exhibited nearly identical
 293 compressibilities (both compressing to ~62 % at 21 kPa); while the dry MCS
 294 sample was even less compressible, compressing by 49 % at 21 kPa. The GCS
 295 feedstock was the least compressible, compressing only 38 % at 21 kPa.

296 The additional compressibility of the deacetylated corn stover was most
 297 likely due to the alkaline soaking step in the deacetylation process (see Sec-
 298 tion 2.1.5 for details), especially since the acid impregnated material was less
 299 compressible than the DACS sample. Alkaline treatment is used in biomass
 300 processing to remove acetyl groups from hemicellulose [24, 25] and to par-
 301 tially solubilize lignin [26, 27], a complex biopolymer responsible for giving
 302 strength to the secondary cell wall in most terrestrial plants. Treating with
 303 NaOH has been reported to swell lignocellulosic biomass (leading to increased
 304 internal surface area) [26–29], as well as decrease its crystallinity [26, 28, 29],
 305 disrupt the structure of lignin [26–29], and break bonds between hemicel-
 306 lulose and lignin [29]. If a portion of the lignin in the DACS sample was
 307 solubilized or disrupted, then the internal structure of the corn stover would
 308 be weakened, making it more flexible and more susceptible to collapse under
 309 compression. In addition, alkaline preprocessing could also alter the sur-

face chemistry of the corn stover particles; making them more adhesive (or “sticky”), which would also enhance its compressibility (as discussed above). However, qualitatively, the DACS particles appeared much more flexible and “fluffy” in comparison to the other preprocessed corn stover materials.

In contrast, acid impregnation of corn stover does not appear to greatly affect its internal structure or surface chemistry (unlike the deacetylation process). The AICS material was more compressible than the MCS (dry) sample, but was equally as compressible as the MCS (wet) material. Therefore, the increase in compressibility via acid impregnation seems to be a result of increased moisture content and not a consequence of acid-aided physical or chemical alteration. The AICS and MCS (wet) feedstocks both had moisture contents of $\sim 50\%$, while the MCS (dry) material had a moisture content of only $\sim 7\%$ (Table 2). Increased moisture can lead to a “softening” of the biomass and act a lubricant during compaction, both resulting in enhanced compressibility.

However, all four of the milled and treated corn stover feedstocks (including the MCS (dry) material) were more compressible than the ground corn stover. The increased compressibility of the milled materials was most likely due to their higher fraction of large particles. The large particles in the MCS (dry and wet), AICS, and DACS samples generate collapsible void spaces within the solids network. During compaction, smaller particles rearrange and fill in the void spaces surrounding the larger particles [5, 30]. As a result, materials with broader size distributions are typically more compressible [5, 31].

3.3. Shear Testing

The yield stress values for the five preprocessed feedstocks, GCS, MCS (wet and dry), AICS, and DACS, at a pre-shear normal stress value of 9 kPa, decreased with decreasing normal stress (Figures 4a and b). The MCS (wet) and DACS materials displayed relatively high, yet similar, yield strengths, cohesion, and interparticle friction (Figure 4a). The MCS (dry) and AICS feedstocks, on the other hand, had slightly lower, but similar, yield stress, cohesion, and interparticle friction values; whereas the GCS material had the lowest yield stress values, as well as the lowest interparticle cohesion and friction (Figure 4b).

Using Mohr circle analysis, the yield loci from Figures 4a and b can be extrapolated to produce values of the unconfined yield stress (f_c) and the major principal stress (σ_1). Plotting f_c as a function of σ_1 generates a

Figure 4: Steady shear stress (open symbols) and yield stress (filled symbols) at a consolidation normal stress (N_C) of 9 kPa as a function of applied normal stress for: (a) MCS (dry and wet) and DACS and (b) MCS (dry), AICS, and GCS (MCS (dry) was plotted on both figures for reference). The dashed lines are linear fits to the yield stress data ($R^2 > 0.98$ for all fits).

linearized relationship, deemed the flow function. The inverse of the flow function slope is referred to as the flowability index (ff_c). Moving from the bottom-right to the upper-left corner in Figure 5, the values of the flowability index decrease from the free flowing region to the non-flowing regime.

The flow functions for all of the preprocessed corn stover feedstocks yielded linear increases in f_c with increasing σ_1 (Figure 5); with MCS (dry) and AICS both falling in the cohesive flow regime. Alternatively, DACS and MCS (wet) had flow indices categorized as very cohesive; while GCS was the most flowable material with a flow function in the easy flowing region. Flowability of a material is dependent on its physical properties, as well as the environmental conditions and the equipment used for handling, storing, and processing [32]. Both adding water and deacetylating the milled corn stover lead to a reduction in flowability. Interestingly, acid impregnating the corn stover did not significantly alter its flowability. However, reducing the particle size and narrowing the size distribution via grinding, enhanced the flowability of corn stover.

A summary of all flowability parameters, including: static and effective angles of interparticle friction (ϕ_i and ϕ_e , respectively), cohesion (τ_0), unconfined yield stress (f_c), and major principal stress (σ_1) for pre-shear normal stress (N_C) values of 3 to 15 kPa are reported in Table 3. In general, the cohesive strengths and unconfined yield stresses of the MCS (wet) and DACS

Figure 5: Unconfined yield stress versus major principal stress for the preprocessed corn stover feedstocks. The solid lines represent the boundaries between flowability designations, while the dashed lines are linear fits to the individual data sets.

materials were found to be the highest; while MCS (dry) and AICS both had slightly lower cohesion and unconfined yield stress values. The GCS sample, on the other hand, had significantly lower values of both cohesion and unconfined yield stress. All of the preprocessed stovers had internal friction angles greater than 30° . Commonly, bulk materials with interparticle cohesion less than 2 kPa and internal friction angles less than 30° can be handled using gravity alone [6, 33]. Unfortunately, it appears that all of the examined preprocessed stovers will present challenges during the feeding, passage, and discharge of process vessels.

The measured flow indices (ff_c) of the preprocessed corn stover samples ranged from 1.7 to 6.2. The ground corn stover (GCS) had a ff_c of 5.0 ± 0.8 , which was the largest flow index for all of the examined corn stovers and classifies GCS as an “easy flowing” material. The MCS (dry) and AICS displayed identical ff_c values of 3.0 ± 0.4 and 3.0 ± 0.3 , respectively; designating them both as “cohesive” materials. The MCS (wet) had an average flow index of 2.2 ± 0.2 , which was slightly lower than the ff_c for MCS (dry) and AICS, but still classifies MCS (wet) as “cohesive.” Conversely, the DACS sample had the smallest flow index at 1.9 ± 0.2 , which places the deacetylated material in the “very cohesive” regime.

There is a limited archive of literature reporting on the flowability pa-

Table 3: Shear testing results for the preprocessed corn stover feedstocks at pre-shear normal stress (N_C) values of 3, 6, 9, and 15 kPa (static and effective angles of interparticle friction (ϕ_i and ϕ_e , respectively), cohesion (τ_0), unconfined yield stress (f_c), and major principal stress (σ_1)). The “ $\pm$ ” indicates a single standard deviation.

Material	N_C (kPa)	ϕ_i ($^\circ$)	ϕ_e ($^\circ$)	τ_0 (kPa)	f_C (kPa)	σ_1 (kPa)
GCS	3	41.2 $\pm$ 1.1	45.2 $\pm$ 0.2	0.28 $\pm$ 0.06	1.2 $\pm$ 0.2	7.2 $\pm$ 0.1
	6	41.3 $\pm$ 0.7	46.8 $\pm$ 0.6	0.85 $\pm$ 0.07	3.8 $\pm$ 0.3	15.6 $\pm$ 0.9
	9	41.6 $\pm$ 1.3	47.8 $\pm$ 2.0	1.36 $\pm$ 0.30	5.3 $\pm$ 0.3	23.3 $\pm$ 0.5
	15	40.9 $\pm$ 0.8	48.0 $\pm$ 1.1	2.50 $\pm$ 0.32	9.1 $\pm$ 0.5	35.7 $\pm$ 1.8
MCS (dry)	3	46.5 $\pm$ 1.6	53.5 $\pm$ 1.3	0.55 $\pm$ 0.05	2.8 $\pm$ 0.3	8.8 $\pm$ 0.2
	6	42.4 $\pm$ 2.3	51.4 $\pm$ 2.7	1.42 $\pm$ 0.20	6.2 $\pm$ 0.3	18.1 $\pm$ 0.9
	9	39.2 $\pm$ 3.7	49.8 $\pm$ 2.5	2.50 $\pm$ 0.59	9.2 $\pm$ 1.0	26.0 $\pm$ 0.6
	15	40.1 $\pm$ 4.5	50.6 $\pm$ 3.8	3.78 $\pm$ 1.40	13.0 $\pm$ 3.7	38.5 $\pm$ 4.8
MCS (wet)	3	48.6 $\pm$ 3.2	58.5 $\pm$ 3.9	0.79 $\pm$ 0.09	4.1 $\pm$ 0.9	10.0 $\pm$ 0.7
	6	46.5 $\pm$ 2.8	57.7 $\pm$ 1.9	1.64 $\pm$ 0.26	8.7 $\pm$ 1.0	19.8 $\pm$ 1.8
	9	45.2 $\pm$ 3.0	57.3 $\pm$ 1.1	3.01 $\pm$ 0.35	14.8 $\pm$ 0.6	32.1 $\pm$ 1.7
	15	46.7 $\pm$ 2.5	56.8 $\pm$ 4.2	4.12 $\pm$ 0.53	20.7 $\pm$ 0.8	47.7 $\pm$ 1.3
AICS	3	47.8 $\pm$ 1.7	56.1 $\pm$ 2.3	0.66 $\pm$ 0.09	3.5 $\pm$ 0.4	9.6 $\pm$ 0.3
	6	45.6 $\pm$ 4.0	56.1 $\pm$ 2.9	1.82 $\pm$ 0.18	6.1 $\pm$ 0.5	18.8 $\pm$ 1.1
	9	47.7 $\pm$ 3.1	56.8 $\pm$ 3.5	2.10 $\pm$ 0.36	9.4 $\pm$ 2.7	27.5 $\pm$ 3.3
	15	50.4 $\pm$ 3.1	61.4 $\pm$ 0.8	4.69 $\pm$ 1.41	16.3 $\pm$ 5.6	51.1 $\pm$ 4.5
DACS	3	45.7 $\pm$ 2.6	60.0 $\pm$ 3.2	1.05 $\pm$ 0.17	5.2 $\pm$ 1.1	9.1 $\pm$ 1.1
	6	44.0 $\pm$ 3.3	56.6 $\pm$ 1.8	1.71 $\pm$ 0.12	8.1 $\pm$ 0.6	16.2 $\pm$ 1.7
	9	41.8 $\pm$ 1.5	56.4 $\pm$ 1.2	3.09 $\pm$ 0.42	13.2 $\pm$ 1.5	24.9 $\pm$ 1.0
	15	39.3 $\pm$ 6.5	52.5 $\pm$ 5.6	4.20 $\pm$ 0.45	17.8 $\pm$ 2.1	36.4 $\pm$ 2.7

rameters of corn stover. Chevanan et al. [6] used a custom fabricated linear shear box to determine the flow properties of chopped corn stover. They reported ϕ_i values of 46-50° and ff_c values of 2.2-3.4, respectively, for corn stover. Gil et al. [10], on the other hand, used a Jenike shear device to measure the flowability of corn stover milled to target particle sizes of 2 and 5 mm with varying moisture contents (7-33 %). They reported ϕ_i and ff_c values of 26-33° and 3.6-10.1 for corn stover. It was difficult to make a direct comparison between these past studies and our current work due to the different shearing devices used and the fact that no two corn stover samples are alike. However, the flow properties reported here for the preprocessed corn stover materials are still in agreement with this limited set of literature values.

3.4. Wall Friction

Wall friction testing involves measuring a wall yield locus, which describes the shear stress required to move granular solids continuously across a wall surface under a specified normal stress. From the wall yield loci, wall friction angles (or coefficients) can be determined.

The measured angles of wall friction (at a surface roughness, R_a , of 1.2 μm) for the preprocessed corn stover materials ranged from 10 to 32° (Figure 6). The two dry corn stover feedstocks, GCS and MCS (dry), both had wall friction angles of $\sim 10^\circ$. A wall friction angle of 10° (denoted with a dashed line in Figure 6) is considered relatively low, indicating that the dry feedstocks should not experience discharge problems due to material wall binding. The AICS and DACS materials, on the other hand, displayed wall friction angles of $\sim 23^\circ$; which are significant enough to lead to material holdup issues during vessel discharge. The MCS (wet) sample exhibited a wall friction angle of $\sim 32^\circ$. An angle of wall friction $\geq 30^\circ$ (dashed line in Figure 6) signifies a high potential for material binding to vessel walls during discharge. As a result, the “wetted” corn stover samples (MCS (wet), AICS, and DACS), may present additional handling issues due to their moderate-to-high affinity to bind to wall surfaces.

3.5. Hopper Design

Jenike’s hopper design methodology (see Section Appendix A for details) was used to estimate the maximum hopper half angle (α) and minimum hopper opening diameter (B) for mass flow in a conical hopper for each preprocessed corn stover sample. Measured values of bulk density as a function

Figure 6: Average wall friction angles ($Ra = 1.2 \mu\text{m}$) for the preprocessed corn stover feedstocks across consolidation normal stresses of 3, 9, and 15 kPa.

of applied normal stress, effective angle of internal friction (ϕ_e), unconfined yield strength (f_c), major principal stress (σ_1), and wall friction angle (ϕ_w) were used in Jenike's hopper analysis. It is worth noting that Jenike's hopper design method can sometimes produce irrational values for the size of a hopper opening, as his analysis relies heavily on where the flow function intersects the unconfined yield stress (f_c) axis. Poor-flowing materials with a steep flow function will intersect the f_c axis near the origin, resulting in an undersized hopper opening. Free-flowing materials with relatively flat flow functions, on the other hand, will result in large f_c axis intercept values, leading to larger than necessary hopper openings [34]. Therefore, Jenike's hopper design protocol should be applied with caution and its results may not reflect true or suitable hopper design. However, if properly applied, Jenike's design methodology can be used as a relative assessment of material hopper discharge.

The calculated maximum hopper half angle (α) for the corn stover feedstocks varied from 16 to 36° (right axis, Figure 7). Commonly, a hopper half angle of 20° is often used as a rule of thumb for achieving mass flow of powders [34]. However, a hopper half angle of 20° is inadequate for the wet or chemically impregnated corn stover materials, MCS (wet), AICS, and DACS, which require α values between 16 to 18°. The hopper half angles

Figure 7: Theoretical minimum hopper outlet diameter (left axis) and maximum hopper half angle (right axis) to achieve mass flow during discharge of the examined preprocessed corn stover feedstocks.

for the two dry corn stover samples, GCS and MCS (dry), were 36° and 33° , respectively, and should be easily discharged from a hopper with an α of 20° .

The minimum hopper outlet diameter (B), which can be a less reliable metric than α , ranged from 0.46 to 1.42 m for the preprocessed corn stover materials (left axis, Figure 7). The B values for the GCS and MCS (dry) corn stover samples were 0.46 and 0.65 m, respectively. The calculated hopper diameters for the MCS (wet) and AICS feedstocks were correspondingly 0.90 and 0.80 m. The DACS material, on the other hand, had an estimated B value of 1.42 m, which was the largest required hopper opening for all of the preprocessed corn stover materials.

4. Conclusions

The compressibility, flow properties, and wall friction angles were measured for five preprocessed corn stovers: ground (GCS), milled (MCS (dry) and MCS (wet)), acid impregnated (AICS), and deacetylated (DACS). Of the tested samples, the ground corn stover was the least compressible and most flowable material. The deacetylated stover, on the other hand, was the most compressible and least flowable of the examined stovers. All of the impregnated corn stovers (wet, acid, and deacetylated) had elevated wall friction

462 angles and reduced flow indices (excluding AICS). The reduced flowabili-
463 ties and enhanced compressibilities and wall friction angles of the “wetted”
464 corn stovers indicate the potential for additional feeding and handling is-
465 sues; which was corroborated via theoretical hopper design calculations. All
466 of the “wetted” materials (MCS (wet), AICS, and DACS) required larger
467 hopper outlet diameters and steeper hopper walls (in theory) than the dry
468 stovers (GCS and MCS (dry)), suggesting that feedstock preprocessing can
469 significantly impact the design and operation of solids-handling systems.

470 Continuous feeding of biomass into, through, and out of reactors is essen-
471 tial for optimal conversion and for avoiding costly process stoppages. Moving
472 forward, the methods and analysis used here can be applied to other ligno-
473 cellulosic feedstocks to evaluate how preprocessing strategies impact their
474 flowability. However, every feedstock is unique, and the relationships be-
475 tween preprocessing and flowability will be highly dependent on the nature
476 of the feedstock (e.g., lignin content and structure, crystallinity, fiber con-
477 tent, etc.). Therefore, the feeding and handling of a potential feedstock must
478 be evaluated (not just material conversion and product yield) when assessing
479 the economic feasibility of a given pretreatment strategy.

480 **Acknowledgments**

481 This work was funded by the U.S. Department of Energy under Contract
482 No. DE-AC36-08-GO28308 with the National Renewable Energy Laboratory
483 and through the Office of the Biomass Program. The authors thank Allison
484 E. Ray and Neal A. Yancey (INL) for supplying the milled and ground corn
485 stovers for this study. The U.S. Government and the publisher, by accepting
486 the article for publication, acknowledges that the U.S. Government retains a
487 nonexclusive, paid up, irrevocable, worldwide license to publish or reproduce
488 the published form of this work, or allow others to do so, for U.S. Government
489 purposes.

490 **Appendix A. Hopper Design Calculations**

491 The discharge of bulk solids from hoppers was classified by Jenike [21]
492 into two major flow regimes: mass flow and funnel flow. Mass flow is when
493 every particle in a storage vessel moves downward in unison towards the
494 vessel opening (i.e., the “first-in, first-out” principle). Mass flow is considered
495 the ideal flow pattern for most applications. Funnel flow or “rat-holing”,

on the other hand, is when only a portion of the solids (typically near the vessel center) move downward during discharge and stagnant particle regions develop along the vessel walls. Funnel flow can be problematic and can commonly lead to material loss and/or degradation, unpredictable discharge patterns, flooding, and particle segregation (to name a few) [35]. However, if a hopper is designed properly, funnel flow can be avoided entirely [25, 35–37].

Along with categorizing the flow patterns of bulk solids, Jenike also developed a procedure for designing hoppers to ensure mass flow during discharge [21]. Jenike’s methodology is still considered standard practice for hopper design [34]. A full description of Jenike’s analysis is beyond the scope of this paper, but in general his approach involves determining two parameters: the flow function (FF) and flow factor (ff). FF depends solely on the shear strength of the bulk solids and describes the relationship between consolidating stress and shear strength of a material (i.e., unconfined yield stress vs. major principal stress; as plotted in Figure 5 in Section 3.3). Conversely, ff depends on the characteristics of the hopper (material of construction, shape, wall friction, etc.) as well as the solids, and describes the ability of a hopper to discharge consolidated (or compacted) solids via gravity. For mass flow to occur, ff must be stronger than FF (i.e., the applied hopper discharge stress must be greater than the unconfined yield strength of the material).

Jenike’s design procedure allows for the determination of the maximum hopper half angle, α (the angle between the vertical vessel wall and the sloped hopper outlet) in order to avoid “rat-holes”, and the minimum hopper outlet size, B , which prevents arching or bridging of material at the vessel opening. The first step in custom hopper design is to quantify the flow properties of the bulk solids. For proper design, a material’s bulk density as a function of applied normal stress, effective angle of internal friction (ϕ_e), unconfined yield strength (f_C), major principal stress (σ_1), and wall friction angle (ϕ_w) must be measured. The effective angle of internal friction is needed for the design of gravity-driven hoppers because it determines the lateral (or outward) stress acting on the vessel walls [32]. The angle of wall friction, on the other hand, describes the strength of interaction between the bulk solids and the vessel wall material (the higher the angle the more difficult it is to move the particles along the wall surface). The unconfined yield stress is also an important parameter for hopper design because it indicates a material’s arching strength.

Jenike’s hopper design protocol involves the following steps:

Figure A.8: Diagram of a conical hopper depicting the hopper half angle (α) and hopper outlet diameter (B).

1. Obtain values of the effective angle of internal friction (ϕ_e) and wall friction angle (ϕ_w) and use them to calculate the hopper half angle (α) and flow factor (ff); Jenike published charts that relate ϕ_e and ϕ_w to both α and ff (mathematical representations of these charts are now available [38]).
2. Then determine the critical applied stress (CAS) from the intersection of the measured flow function and calculated flow factor lines. The flow factor line is a straight line through the origin with a slope equal to $1/\text{ff}$.
3. Next, calculate the minimum hopper outlet size (B) using values of CAS , hopper half angle (α), and bulk density (ρ_b). The outlet diameter for a conical hopper, B , is given by:

$$B = \frac{H(\alpha)CAS}{\rho_b g}, \quad (\text{A.1})$$

$$H(\alpha) = 2 + \frac{\alpha}{60}, \quad (\text{A.2})$$

where g is the gravitational constant. The bulk density must correspond to the consolidating stress at the hopper outlet.

548 References

- 549 [1] Babu SP, Whaley TP. IEA biomass thermal gasification project.
550 Biomass Bioenerg. 1992;2(1-6):299–6. International Energy Agency
551 Bioenergy Agreement Progress and Achievements 1989-1991.
- 552 [2] Cummer KR, Brown RC. Ancillary equipment for biomass gasification.
553 Biomass Bioenerg. 2002;23(2):113–28.
- 554 [3] Dai J, Cui H, Grace JR. Biomass feeding for thermochemical reactors.
555 Prog Energy Comb Sci. 2012;38(5):716–36.
- 556 [4] Edwards D. Scaling Up Bioenergy Technologies. Chem Eng Prog.
557 2015;111(3):58–1.
- 558 [5] Fasina OO. Flow and physical properties of switchgrass, peanut hull,
559 and poultry litter. Trans ASABE. 2006;49(3):721–8.
- 560 [6] Chevanan N, Womac AR, Bitra VSR, Yoder DC, Sokhansanj S. Flowa-
561 bility parameters for chopped switchgrass, wheat straw and corn stover.
562 Powder Tech. 2009;193(1):79–6.
- 563 [7] Zhou B, Ileleji KE, Ejeta G. Physical property relationships of bulk corn
564 stover particles. Trans ASABE. 2008;51(2):581–90.
- 565 [8] Ileleji KE, Zhou B. The angle of repose of bulk corn stover particles.
566 Powder Tech. 2008;187(2):110–18.
- 567 [9] Samaniuk JR, Scott CT, Root TW, Klingenberg DJ. Rheological mod-
568 ification of corn stover biomass at high solids concentrations. J Rheol.
569 2012;56(3):649–65.
- 570 [10] Gil M, Schott D, Arauzo I, Teruel E. Handling behavior of two milled
571 biomass: SRF poplar and corn stover. Fuel Process Tech. 2013;112:76–5.
- 572 [11] Miccio F, Barletta D, Poletto M. Flow properties and arching behavior
573 of biomass particulate solids. Powder Tech. 2013;235(0):312–21.
- 574 [12] Crawford NC, Ray AE, Yancey NA, Nagle N. Evaluating the pelleti-
575 zation of “pure” and blended lignocellulosic biomass feedstocks. Fuel
576 Process Tech. 2015;140:46–6.

- 577 [13] Yancey NA, Wright CT, Conner CC, Tumuluru JS. Optimization of Pre-
578 processing and Densification of Sorghum Stover at Full-Scale Operation.
579 Proc ASABE, 2011 Annu Int Meet. 2011;.
- 580 [14] Schell D, Farmer J, Newman M, McMillan J. Dilute-sulfuric acid pre-
581 treatment of corn stover in pilot-scale reactor - Investigation of yields,
582 kinetics, and enzymatic digestibilities of solids. Appl Biochem Biotech.
583 2003;105:69–85.
- 584 [15] Tucker MP, Kim KH, Newman MM, Nguyen QA. Effects of temperature
585 and moisture on dilute-acid steam explosion pretreatment of corn stover
586 and cellulase enzyme digestibility. Appl Biochem Biotech. 2003;105(1-
587 3):165–77.
- 588 [16] Shekiri I Joseph, Kuhn EM, Nagle NJ, Tucker MP, Elander RT, Schell
589 DJ. Characterization of pilot-scale dilute acid pretreatment performance
590 using deacetylated corn stover. Biotech Biofuels. 2014;7(1).
- 591 [17] Trottier R, Dhodapkar S. A Guide to Characterizing Particle Size and
592 Shape. Chem Eng Prog. 2014;110(7):36–6.
- 593 [18] International Organization for Standardization. ISO Standard 9276-
594 2:2014 - Representation of results of particle size analysis – Part 2: Cal-
595 culation of average particle sizes/diameters and moments from particle
596 size distributions; 2014.
- 597 [19] Freeman R. Measuring the flow properties of consolidated, conditioned
598 and aerated powders - A comparative study using a powder rheometer
599 and a rotational shear cell. Powder Tech. 2007;174(1-2):25–3.
- 600 [20] Freeman RE, Cooke JR, Schneider LCR. Measuring shear properties and
601 normal stresses generated within a rotational shear cell for consolidated
602 and non-consolidated powders. Powder Tech. 2009;190(1-2):65–9.
- 603 [21] Jenike AW. Storage and flow of solids. Salt Lake City, Utah: Bull. Utah
604 Eng. Exp. Stat; 1964.
- 605 [22] Carson JW, Wilms H. Development of an international standard for
606 shear testing. Powder Tech. 2006;167(1):1–9.
- 607 [23] Tomas J, Schubert H. Particle characterization. Partec. 1979;79:301–19.

- 608 [24] Chen X, Shekiro J, Elander R, Tucker M. Improved Xylan Hydrolysis
609 of Corn Stover by Deacetylation with High Solids Dilute Acid Pretreat-
610 ment. *Ind Eng Chem Res.* 2012;51(1):70–6.
- 611 [25] Chen P, Yuan Z, Shen X, Zhang Y. Flow properties of three fuel powders.
612 *Particuology.* 2012;10(4):438–43.
- 613 [26] Camesasca L, Ramirez MB, Guigou M, Ferrari MD, Lareo C. Eval-
614 uation of dilute acid and alkaline pretreatments, enzymatic hydrolysis
615 and fermentation of napiergrass for fuel ethanol production. *Biomass*
616 *Bioenerg.* 2015;74(0):193–1.
- 617 [27] Agbor VB, Cicek N, Sparling R, Berlin A, Levin DB. Biomass pretreat-
618 ment: Fundamentals toward application. *Biotech Adv.* 2011;29(6):675–
619 85.
- 620 [28] Balat M, Balat H, Oz C. Progress in bioethanol processing. *Prog Energ*
621 *Comb Sci.* 2008;34(5):551–73.
- 622 [29] Kumar P, Barrett DM, Delwiche MJ, Stroeve P. Methods for Pretreat-
623 ment of Lignocellulosic Biomass for Efficient Hydrolysis and Biofuel Pro-
624 duction. *Ind Eng Chem Res.* 2009;48(8):3713–29.
- 625 [30] Barbosa-Canovas GV, Juliano P. Compression and compaction char-
626 acteristics of selected food powders. vol. 49. New York, NY: Kluwer
627 Academic Plenum Publishers; 2005.
- 628 [31] Mani S, Tabil L, Sokhansanj S. Grinding performance and physical prop-
629 erties of wheat and barley straws, corn stover and switchgrass. *Biomass*
630 *Bioenerg.* 2004;27(4):339–52.
- 631 [32] Ganesan V, Rosentrater KA, Muthukumarappan K. Flowability and
632 handling characteristics of bulk solids and powders - A review with im-
633 plications for DDGS. *Biosyst Eng.* 2008;101(4):425–35.
- 634 [33] Puri V. Characterizing powder flowability. *Chem Process.* 2002;65:29–
635 42.
- 636 [34] Fitzpatrick JJ, Barringer SA, Iqbal T. Flow property measurement of
637 food powders and sensitivity of Jenike’s hopper design methodology to
638 the measured values. *J Food Eng.* 2004;61(3):399–5.

- 639 [35] Johanson J. Troubleshooting bins, hoppers and feeders. Chem Eng
640 Prog. 2002;98(4):24–6.
- 641 [36] Purutyan H, Pittenger B, Carson J. Solve solids handling problems by
642 retrofitting. Chem Eng Prog. 1998;94(4):27–39.
- 643 [37] Schulze D. Powders and Bulk Solids: Behavior, Characterization, Stor-
644 age and Flow. 1st ed. Springer - Verlag Berlin Heidelberg; 2008.
- 645 [38] McGlinchey D. Characterisation of Bulk Solids. Wiley; 2009.