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Effects of CDS and drying temperature on the flowability behavior of DDGS

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1 **TITLE: EFFECTS OF CDS AND DRYING TEMPERATURE LEVELS**
2 **ON THE FLOWABILITY BEHAVIOR OF DDGS**
3

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20

ABSTRACT

21
22 Due to increasing demand for alternative fuels and the need to reduce dependence on fossil
23 fuels, the growth of bioethanol production has been rising. One of the problems facing this
24 industry is transportation of the coproduct distillers dried grains with solubles (DDGS)
25 over long distances, because caking and agglomeration between particles can lead to bulk
26 flow problems. In this study DDGS was prepared by combining condensed distillers
27 solubles (CDS) and distillers wet grains (DWG), and then oven drying to achieve 8% (db)
28 moisture content. The effects of drying temperature (100, 200, and 300°C) and CDS (10,
29 15, and 20% wb) level on the resulting flowability behavior of the DDGS particles were
30 investigated. Statistical analyses indicated significant differences ($\alpha = 0.05$, 95%
31 confidence level) due to drying temperature and CDS main effects, and also significant
32 interaction effects between CDS level and drying temperature for many of the flow
33 parameters. Surface regression analysis of the ratio of Total Flow Index/Jenike Flow
34 Function as a function of CDS and drying temperature resulted in an R^2 value of 0.94.
35 Partial Least Squares (PLS) regression yielded an R^2 of 0.90 for the Jenike Flow Function
36 Index as a function of all flow and physical properties, using only two multivariate
37 components. Understanding the effects of varying drying temperature and CDS levels can
38 help guide efforts to overcome DDGS flowability problems.

39 **Keywords.** Agglomeration, Caking, Carr, Condensed distillers solubles, Distillers wet
40 grains, Jenike.

INTRODUCTION

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Distillers dried grains with solubles (DDGS) is a coproduct from the corn-based fuel ethanol industry, and is relatively high in protein and fiber content but low in starch. Due to its nutrient content and digestibility, it is primarily used as livestock feed for beef and dairy rations, and to some extent in swine and poultry diets. Research has also been done on using DDGS in aquaculture feed (1) As there is growing demand for fuel ethanol, there is more production of DDGS as well. It has been reported that during the fiscal year of 2008-2009, over 19 million metric tons of DDGS was produced from the ethanol industry in United States (2), and this level has risen to more than 30 million metric tons in 2010.

In order to optimize the use of DDGS in livestock feed markets, it is therefore essential to provide safe and economic handling of DDGS while it is being transported in domestic as well as international markets. Distillers dried grains with solubles storage and transportation is often problematic due to formation of particle agglomerates inside storage structures, which results in “caking” and restricts flow during discharge. Flowability problems may be due to varied environmental conditions and storage situations, such as temperature, moisture content, humidity, and storage period. Apart from environmental conditions, the inherent physical and chemical properties of the material may also affect overall flowability of DDGS (3; 4; 5). Cohesiveness and flow problems create unwanted labor and cost to unload (6).

Most organic materials (like DDGS) are hygroscopic in nature, so they have the tendency to gain or lose moisture when they are exposed to diverse humidity conditions. This can lead to possible changes in physical and chemical properties in the material itself, which in turn will affect the flowability and can cause hardening of particles.

64 Condensed Distillers Solubles (CDS), commonly known as “solubles” or “syrup,” is mixed
65 with distillers wet grains (DWG) and then dried to produce distillers dried grains with solubles
66 (DDGS). The solubles are relatively high in vitamins, fat (6-21%, db), and protein (9-12%, db),
67 but low in fiber (<5%, db). Syrup has a total digestible energy value approximately 91% that of
68 raw corn (7). DDGS typically contains approximately 86 to 93% dry matter, 3 to 13% (db) fat,
69 and 26 to 34% (db) protein (8). The high fat level in CDS may be a possible cause for DDGS
70 flowability problems, because the corn lipids may form molten bridges between the particles

71 Stickiness in corn syrup powders was determined under varying temperature and humidity
72 conditions (9). The effect of temperature, moisture content, and storage time was studied for milk
73 powder flowability and stickiness (10; 11; 12). It has been found that powder caking is often a
74 function of moisture content, and frequently there is an increase in stickiness due to an increase
75 in ambient temperature (13; 14).

76 Powder flowability is most commonly assessed using Carr (15) and Jenike (16) shear test
77 procedures. Parameters for flowability in the Carr (15) test procedure include Angle of Repose
78 (AoR), Aerated and Packed Bulk Density (ABD and PBD), Compressibility (Cc), Angle of
79 Spatula (AoS), Angle of Difference (AoD), Uniformity, Angle of Fall (AoF), Dispersibility, and
80 Total Flow and Flood Indices. Hausner Ratio is defined as the ratio of tapped bulk density to the
81 aerated (or apparent) bulk density (i.e., PBD/ABD). Values less than 1.25 typically indicate good
82 flow, whereas values greater than 1.25 generally indicate poor flow (17). Angle of Spatula is
83 measured by inserting a flat blade into a pile of powder and then lifting it up. After the
84 evaluation of the above properties (i.e., Angle of Repose through Uniformity) (excluding
85 Hausner Ratio), the index values are combined to provide an overall flowability index value for

86 the powder/bulk solid under investigation. The smaller the uniformity value, the more
87 homogenous the particle shapes are, and typically the less the flow problem (15, 17).

88 After the overall (or total) flowability index has been determined, then the floodability
89 assessment is done. The Angle of Fall, the Angle of Difference, and Dispersibility is measured as
90 part of floodability index. The above properties (Angle of Fall, Angle of Difference, and
91 Dispersibility, in addition to Flow Index) are numerically combined to calculate the Total
92 Floodability Index of the bulk solid (by summation of the individual indices). The angle of fall is
93 the new angle of repose that is formed after impaction has been applied to the material. More
94 detailed information on these properties and testing procedures can be found in Carr (15) and
95 Bhadra et al. (3).

96 Another procedure which is used commonly to assess flow behavior is Jenike (16) shear
97 testing. In this type of test, when the powder is subjected to a normal stress (σ), there will be a
98 particular shear stress (τ) which causes bulk failure (i.e., flow). This data gives the yield locus
99 curve which can be used to compute the angle of internal friction (Φ , degrees), effective angle of
100 internal friction (δ , degrees), major consolidation stress (σ_1 , kPa), and unconfined yield strength
101 (σ_c , kPa).

102 Unconfined yield strength is a measure of the compressive strength (kPa) of the granular
103 solid (18). Major consolidation stress is determined as the point of intersection between the Mohr
104 circle (drawn with a shear and normal stress plot) and the stress x-axis. Flow Function Index
105 (dimensionless) is the ratio of the major consolidation stress to unconfined yield strength.
106 Depending on the value of this index, the flow behavior of a material can be categorized as
107 “good flow”, “fair to passable flow,” or “cohesive flow.” More details on this can be found in
108 Jenike (16) and Bhadra et al. (3).

109 Some work has previously examined DDGS flowability, including use of flow agents,
110 measuring physical and chemical properties and correlating them with the Carr and Jenike shear
111 test properties (19; 20; 21; 22). Reduced fat and normal DDGS samples have been studied to
112 understand flow properties, and reduced fat DDGS had slightly better flow than normal DDGS
113 (5). Previous work has also shown the effects of moisture content and CDS addition levels on
114 DDGS flowability (19). Development of a predictive flowability model ($R^2 = 0.94$) using
115 exploratory data analysis techniques was accomplished (21). A comprehensive dynamic water
116 adsorption model (GRM model, $R^2 = 0.94$) incorporating varying CDS, relative humidity, and
117 drying temperature levels has been developed (22). A model ($R^2 = 0.94$) to predict the sorption
118 isotherm behavior of DDGS with varying CDS and equilibrium moisture content levels was also
119 developed (23). Various studies have also revealed the typical ranges of DDGS chemical,
120 physical, and flowability properties (3; 4; 24).

121 The functionality and properties of bulk solids and granular materials are greatly
122 influenced by drying conditions during the manufacturing process (46, 47, 48, 49). Two studies
123 examined drying rate and moisture desorption during DDGS production, using varying CDS and
124 drying temperature levels (25; 26). Those studies were able to establish regression models of the
125 drying kinetics and moisture desorption behavior; but no correlational studies between the
126 resulting flowability properties and the effects of drying temperature and CDS addition levels
127 were performed. Understanding these effects on physical and flow properties is an essential step
128 toward improving DDGS flowability.

129 Therefore, the objectives of this study were: 1) to prepare DDGS samples under
130 laboratory conditions using CDS and DWG using multiple ratios of CDS:DWG and multiple
131 drying temperatures; 2) to measure several physical and flow properties (both Carr and Jenike

132 flow properties) of the prepared DDGS samples; 3) to determine the effects of drying
133 temperature and CDS addition levels on the resulting properties of the DDGS; and 4) to examine
134 predictive regression models of various DDGS properties to fully understand the effects CDS
135 level and drying temperature.

136 MATERIALS AND METHODS

137 138 **Sample Collection and Preparation**

139 Samples of distillers wet grains (DWG) and condensed distillers solubles (CDS) were
140 collected from a commercial fuel ethanol plant in South Dakota and were stored under
141 refrigerated conditions ($10 \pm 1^\circ\text{C}$). The initial moisture content of the DWG and CDS samples
142 were between 45% (db) to 47% (db). Prior to the drying experiments, CDS was added to DWG
143 at several predetermined levels (10, 15, and 20%, wb), and then thoroughly mixed in a laboratory
144 mixer (model D300, Hobart Corporation, Troy, OH) for 5 min. After blending, each combined
145 sample (approximately 350 g of mixed DWG and CDS) was spread uniformly on a thin
146 aluminum plate ($38\text{ cm} \times 27\text{ cm} \times 2\text{ cm}$) and then dried in a laboratory-scale oven (model 838F,
147 Fisher Scientific, Pittsburg, PA). For a single drying temperature and CDS combination, three
148 aluminum trays were used. Drying was done at three selected temperatures (100, 200, and
149 300°C) for each DWG/CDS mixture. Temperature selection was based on interviews and
150 discussions with industry experts, and also based on our previous studies of drying rate and
151 moisture content of DDGS (25; 26). For each temperature/CDS combination, drying was done to
152 reduce all the experimental samples to a target of 8% (db) moisture content, in order to have
153 common baseline moisture content and eliminate its possible influence on the flowability
154 behavior. To achieve this, each treatment combination was dried at different drying times. The
155 drying time slightly varied while changing the CDS levels: it was around 60 min, 35 min, and 15

156 min for drying temperatures of 100, 200, and 300°C, respectively. The drying continued until the
157 final blend moisture contents reached 8% (db). This target was based on previous research by
158 Bhadra et al. (24) and Rosentrater (27), and upon discussions with industry representatives
159 (unpublished), which indicated a typical average moisture content of approximately 8 % (db) in
160 the marketplace. Preparation of the dried DDGS samples (9 total treatments) was done with
161 three replications.

162

163 **Experimental Design**

164 Experiments were conducted using a 3*3 full factorial design, with 3 drying temperatures
165 (100, 200, and 300°C) and 3 CDS addition levels (10, 15, and 20%, wb), yielding a total of 9
166 treatment combinations. These treatment combinations were implemented using a completely
167 randomized design. DDGS samples were prepared in three replications, thus yielding $3*3*3 = 27$
168 experimental runs. Each physical and flow property was determined using three replicate
169 measurements for each treatment combination.

170 Once the drying was completed, the granular particles of each sample were cooled for 6
171 to 8 h under ambient conditions ($\sim 25^{\circ}\text{C}$), and then placed in polyethylene bags and stored at
172 room temperature ($\sim 25^{\circ}\text{C}$), throughout the duration of the study. After the drying was completed
173 for all 27 experimental runs, the physical and flow properties were then measured.

174 **Flowability Property Measurement**

175 A powder characteristics tester (Model PTR, Hosokawa Micron Powder Systems, Summit,
176 NJ) was used to measure the Carr (15) flow properties of the DDGS, following the procedures
177 described by ASTM D6393 (28). The Carr flow properties included AoR, ABD, PBD,

178 Compressibility, Uniformity, AoF, AoS, AoD, and Dispersibility. These parameters were then
179 used to determine both the Total Flow Index and Total Floodability Index.

180 AoR is defined as the angle formed between the slope of a pile of material and a horizontal
181 plane. ABD and PBD are used to assess compressibility and the ability of the material to entrap
182 air in pores between particles (29). Packed (or tapped) density is an actual representation of a
183 material's bulk density when it is stored in bins or transported over large distances (i.e., in a rail
184 car as entrained air is forced out). Angle of Spatula is measured by inserting a flat blade into a
185 pile of material and then lifting it up. The new Angle of Repose which the material forms relative
186 to the horizontal plane of the blade gives the Angle of Spatula. Uniformity is the ratio of the
187 width of the sieve opening that will allow 60% of the material to pass to the width of sieve
188 opening that will only allow 10% of the sample to pass. Uniformity thus gives a relative measure
189 of the homogeneity of the size and shape of the particles. Uniformity is the ratio obtained
190 between the width of sieve opening that will pass 60% of the sample to the width of sieve
191 opening that will pass only 10% of the sample. Particle size distributions were determined using
192 standard US sieves, from no. 4 (pore opening size of 4.76 mm) to 270 (pore opening size of 53
193 μm). Thus, the Total Flow Index was determined by adding the Angle of Repose, Uniformity,
194 Compressibility, and Angle of Spatula.

195 After the overall flowability index has been determined, then floodability is assessed. The
196 Angle of Fall is the new angle of repose that is formed after impaction (by the impactor device
197 provided on the Hosokawa Micron Powder System Unit) has been applied to the material. It is
198 done to simulate the disturbance due to vibrations and transport effects on bulk solids (15). . The
199 Angle of Difference is then calculated by subtracting the Angle of Fall from the Angle of
200 Repose. Dispersibility is measured by discharging a specified amount (10 g) of material through

201 a column onto a watch glass 98 cm diameter); the quantity of material left behind on the watch
202 glass quantifies how disperse the material is. Hence, Total Floodability Index was determined by
203 adding the Angle of Fall, Angle of Difference, Dispersibility, and Total Flow Index.

204 The other flowability behavior is measured by Jenike (16) shear testing. This method uses
205 a split, horizontal testing container, and various normal stresses and shear stresses are applied to
206 the top half, while the lower half is kept stationary. In this type of test, when the powder is
207 subjected to a normal stress (σ), there will be a particular shear stress (τ) which causes bulk
208 failure (i.e., flow). This data gives the yield locus curve, which can then be used to compute the
209 angle of internal friction (Φ , degrees), effective angle of internal friction (δ , degrees), major
210 consolidation stress (σ_1 , kPa), and unconfined yield strength (σ_c , kPa). Angle of internal friction
211 is the inter-particle friction as the bulk solid tends to slide on itself at the onset of flow. Effective
212 angle of internal friction is measured during flow when granular solids are constantly exposed to
213 pressures. The major pressure acting on a particle element is denoted by σ_1 while the minor
214 pressure is termed σ_2 . The relationship between these two pressures varies slightly with changes
215 in temperature for most bulk solids (1964). Their relationship can be expressed as:

216

$$217 \quad \frac{\sigma_1}{\sigma_2} = \frac{1 + \sin \delta}{1 - \sin \delta} \quad (1)$$

218

219 This equation is called the effective yield function. It describes inter-particle kinematic
220 particle friction which exists during steady flow. Unconfined yield strength is a measure of the
221 compressive strength (kPa) of the granular solid (18). Major consolidation stress is determined as
222 the point of intersection between the Mohr circle and the stress x-axis. Major consolidation stress

223 (σ_1 , kPa) was calculated from the Mohr circles, which are drawn from the equation of effective yield
224 function (i.e., equation 1). The point where the largest Mohr circle intersects the normal stress axis (i.e.,
225 x-axis) gives σ_1 . Additionally, σ_c (unconfined yield strength, kPa) was determined from the Mohr circle as
226 well. It is the intersection of the smaller Mohr circle on the normal stress axis. Flow Function Index
227 (dimensionless) is the ratio of the major consolidation stress (σ_1 , kPa) to unconfined yield
228 strength (σ_c , kPa).

229 Jenike (16) shear testing was then performed to quantify the instantaneous shear behavior
230 for each DDGS sample, including angle of internal friction, effective angle of internal friction,
231 major consolidation stress, unconfined yield strength, and Jenike Flow Index. A Jenike shear cell
232 unit was used (Model ST-5, Jenike and Johansson Co., Westford, MA) following the procedures
233 described in ASTM D6128 (30). Jenike compressibility testing was also performed using the
234 same shear cell unit, but using the technique discussed in ASTM D6683 (31).

235 **Physical Property Measurement**

236 Color and thermal properties are not directly correlated with flowability properties of
237 DDGS however, there can be some indirect effects. For example, color and brightness of a product
238 can indicate the level of nutrients (carbohydrates, lipids, fiber, etc.), which can, in turn, affect the
239 flow properties of DDGS. Similarly, thermal properties can correlate to frictional properties,
240 which, in turn, affect flow behavior. Water activity changes can indicate shelf life. PDI provides
241 a measure of protein solubility, which interacts with surface moisture films between DDGS
242 particles.

243 Color was measured using a spectrophotometer (LabScan XE, Hunter Associates
244 Laboratory, Reston, VA) with the L-a-b color scale (Hunter Associates Laboratory Universal
245 Software Manual V. 2.5; Reston, VA). Water activity was measured using a calibrated water

246 activity meter (AW Sprint TH 500, Novasina, Talstrasse, Switzerland) using factory-supplied
247 standards. Thermal properties (conductivity, diffusivity, and resistivity) were determined with a
248 thermal properties meter (KD2, Decagon Devices, Pullman, WA) that utilized the line heat
249 source probe technique (32). Geometric mean diameter (GMD) (average particle size) was
250 determined by ASAE standard method S319.3 (33) with a Rotap sieve shaker (model RX-29,
251 Tyler Manufacturing, Mentor, OH). Porosity of the DDGS samples was calculated from the
252 method described in Sahin and Sumnu (34) using a multivolume pycnometer (model 1305,
253 Micromeritics, Norcross, GA). The protein dispersibility index (PDI) was calculated using
254 AACC method 46-24 (35).

255 **Statistical Analysis**

256 Formal statistical data analyses were performed using Microsoft Excel v.2003 (Microsoft
257 Corp., Redmond, WA) and SAS v.8 (SAS Institute, Cary, NC) software. Analyses included
258 summary statistics, analysis of variance (at $\alpha = 0.05$), and Least Significant Difference (LSD) (at
259 $\alpha = 0.05$) testing; these were performed to determine significant differences and interaction
260 effects between the main effects and treatment combination effects due to drying temperature
261 and CDS level. Pearson linear correlation analysis among all properties was performed to
262 examine relationships at the 95% confidence level. TableCurve 3D v.4.0.01 (SYSTAT Software,
263 Inc., San Jose, CA) was then used to develop regression equations and response surface models.
264 Partial Least Squares (PLS) and Principal Component Analysis (PCA) tests were then performed
265 using Minitab v.14 software (Minitab, State College, PA).

266

267 **RESULTS AND DISCUSSION**

268

269 *Main Effects, Interaction Effects and Treatment Combination Effects*

270 Table 1 gives the summary of the statistical output obtained after examining main effects
271 on all of the physical and flow properties. It was observed that there were significant differences
272 in the main effects for both CDS addition and drying temperature levels using the Least
273 Significant (LSD) test. To verify these differences, Tukey's significance test (TSD) at $\alpha = 0.05$
274 was also performed, and similar significant differences (although they are not reported here) was
275 observed (36). Even for a conservative test like TSD, significant differences were observed for
276 most of the physical and flowability properties. Flowability of DDGS is a multivariate
277 phenomenon, as reflected by these results. Considerable variability in physical and flowability
278 properties was also found in commercial DDGS samples collected from various ethanol plants
279 (27).

280 These results also verify our hypothesis that drying temperature and CDS level results in
281 significant differences in DDGS properties. For example, there was a significant increase in
282 geometric mean diameter when drying temperature increased from 200 (0.78 mm) to 300°C
283 (0.99 mm). Even though the DDGS moisture content was constant for all the samples produced
284 (8% db, as stated earlier), higher drying temperatures produced larger particle sizes. Large
285 particle sizes generally result in decreased flow problems in bulk solids. Increasing CDS levels
286 from 10% (0.91 mm) to 15% (0.75 mm) resulted in a significant decrease in particle size,
287 indicating possible flow problems. However, geometric mean diameter showed an interaction
288 effect between CDS and temperature. CDS*temperature increased the particle size overall (0.67
289 to 1.31 mm), except at 300°C (from 15 and 20% CDS), where particle size was slightly reduced
290 to 0.81 mm. Angle of Repose increased (42.06° to 47.16°) with increased CDS*temperature
291 combinations; higher angles indicate poorer flow. For water activity, CDS*temperature led to a

292 decrease in value (from 0.71 to 0.23) as the temperature was increased. More details of
293 CDS*temperature interaction effects are discussed in later sections.

294 As shown in Table 2, there were also significant interaction effects ($p < 0.05$) for varying
295 CDS levels and drying temperatures for most of the physical and flowability properties, except
296 for Hausner Ratio (HR, -) and Carr Compressibility (%).

297 There were significant differences among treatment combinations (Table 3) observed for
298 most of trials. For a drying temperature of 100°C, HR, PDI, and Carr Compressibility did not
299 show any significant differences among the CDS levels (as indicated in bold letters). For a
300 drying temperature of 200°C, HR, AoF, Hunter L (-) (which measures the brightness or
301 luminosity of a product), and thermal conductivity did not show significant differences among
302 the CDS levels. For drying temperature of 300°C, HR, Carr compressibility, Total Flow Index,
303 angle of internal friction, major consolidation stress, Hunter a(-) (which refers to the redness or
304 greenness of a product), and thermal conductivity did not shown any significant differences
305 among the CDS levels. Thus, it can be concluded that at lower drying temperatures, changes in
306 CDS levels can result in more variability in DDGS properties (both flow and physical) , which
307 could lead to potential flow problems (3).

308 ***Property Relationships***

309 Pearson product moment linear correlation analysis (37) was performed for the properties
310 in this study (Table 4). The correlation coefficient quantifies how closely two properties are
311 related to each other by a linear relationship. Only 19 combinations had p values less than $\alpha =$
312 0.05 (i.e., were significant correlations) and had correlation coefficient (r) values greater than
313 0.65. Out of these 19 combinations, 10 combinations had r values from |0.7| to |0.8|; 7 variable
314 combinations had correlation coefficients from |0.8| to |0.9|; 2 combinations had r values from

315 |0.9| to |1.0|. A closer examination provides several insights. HR had a high correlation with Carr
316 compressibility (C_c), which was anticipated because HR is calculated using PBD and ABD,
317 which are basically the same parameters that are used to calculate C_c . Angle of Repose, an
318 important parameter for flowability assessment, showed moderate correlation with uniformity;
319 AoR is strongly dependent on the particle size and shape, and uniformity is measured via particle
320 size. Higher drying temperature will cause the moisture to evaporate more effectively, thereby
321 yielding lower water activity values, and hence improving the shelf life of the biomaterial.
322 However, higher drying temperatures can increase utility costs in bioethanol plants.

323 Drying temperature was found to correlate with most of the properties. These tie in to our
324 previous studies (25, 26) where mathematical modeling based on the same drying temperatures
325 and CDS levels were used. In these studies, it was found that temperature had more significant
326 effects on the drying behavior than CDS addition levels. It thus appears that drying temperature
327 is mainly responsible for differences in physical and flow properties as well.

328 ***Effect of Drying Temperature and CDS Levels on Hausner Ratio (HR)***

329 Hausner Ratio (-) values ranged from 1.05 to 1.25, depending on the drying temperature
330 and CDS level (Figure 1). HR depends on the friction in a moving powder mass (i.e., internal
331 friction) during the compaction of powders (38). Higher HR (>1.25) generally indicates poor
332 flowability. In this study, the HR values were mostly below 1.25. As the drying temperature
333 increased from 100 to 300°C, the HR values decreased, indicating that higher temperatures
334 yielded better DDGS flowability, for all CDS levels. For the 10% CDS level, the R^2 value (0.16)
335 obtained from the regression equation was much less than other two CDS levels, which was
336 probably due to the fact that there was substantial scatter in the data points at 100 and 300°C.
337 More extensive study with greater replications may provide a better regression equation.

338 However, for all CDS levels, the HR (-) decreased linearly with an increase in drying
339 temperature, indicating better flow DDGS behavior.

340 *Effect of Drying Temperature and CDS Levels on Jenike Flow Index*

341 Figure 2 presents the flowability behavior of the DDGS samples based on Jenike Flow
342 Index (16). In these flow functions, lines lying towards the bottom of the graph represent easy
343 flow, while more difficult flow is indicated by lines lying near the top and the left of the graph;
344 flowability worsens as the flow function moves upwards in an anticlockwise direction (5). At
345 lower CDS levels (10%, wb), for 100°C, the flow function line lies near the shear stress axis (y-
346 axis), but for 200°C and 300°C, it moves towards the normal stress axis (x-axis). This indicates
347 that DDGS with 10% CDS level and dried at 100°C had a higher compressive strength, and thus
348 greater ability to obstruct flow (i.e., was least free flowing). But higher drying temperatures (200
349 and 300°C) yielded better flowing DDGS with the same CDS level (10%, wb). For higher CDS
350 levels (15 and 20%, wb), a shift in the flow function line towards the x-axis was observed,
351 indicating better flowability, especially for the 200°C and 300°C drying temperatures. Generally,
352 higher CDS levels will result in higher fat content among the DDGS particles, which may hinder
353 flow, by forming bridges (i.e., molten or solidified fat layers) between particles, depending upon
354 temperature. In this study, there were slight shifts of the flow function lines towards the x-axis,
355 indicating better flow instead of flow obstruction. In some instances it has been found that higher
356 CDS levels in DDGS may lubricate the materials and create easy flow (5). For all 3 CDS levels,
357 the drying temperature showed similar behavior; higher temperature yielded better Jenike Flow
358 Function Index line mostly inclined to x-axis, indicating good flow.

359 *Effect of Drying Temperature and CDS Levels on Particle Size and Bulk Density*

360 Figure 3 shows the effect of drying temperature on particle size and aerated bulk density
361 of the DDGS samples for different CDS addition levels. With an increase in the drying
362 temperature from 100 to 300°C the bulk density slightly increased as did particle size, except for
363 20% (wb) CDS, where a decrease in the particle size from 100 to 200°C was observed. There
364 were also significant interactions between drying temperature and CDS (Table 3), thus the trend
365 observed in particle size is not solely due to the main effect of drying temperature alone, but
366 CDS and drying temperature together. These changes in particle size (due to the rapid formation
367 of dried layers on the particle surfaces) with drying temperature were similar to findings by
368 Chegini and Ghobadian (39). The particle size increase could be due to case hardening of the
369 droplets at the higher temperatures, which leads to the formation of vapor-impermeable films on
370 the drop surface, followed by the formation of vapor bubbles, and consequently droplet
371 expansion. This hardened skin does not allow the moisture to exit from the droplet, and as a
372 consequence the particle size is increases (39).

373 In terms of flowability, particle size plays an important role in the compressibility of
374 powders. An increase in the particle size can lead to a reduction in the bulk density of the
375 material, due to more entrapped void spaces (40) ^{but} for our samples, the bulk density slightly
376 increased. This is may be due to the fact that increase in the particle size increased the mass of
377 the solid, which was due to CDS. Thus, the overall bulk density increased. Lower particle sizes
378 yield greater cohesive strength due to an increase in the surface/volume ratio between the
379 particles (41). Thus, again from the particle size and bulk density perspective, similar results: the
380 flowability was better with an increase in the drying temperatures at each CDS level, was
381 noticed.

382 *Effect of Drying Temperature and CDS Levels on Protein Dispersibility Index (PDI)*

383 Protein Dispersibility Index estimates of the amount of water-soluble protein present in
384 the sample. From Figure 4, a decrease in the PDI with an increase in the drying temperature for
385 each CDS level was observed. Regression equations with R^2 values from 0.70 to 0.79 were
386 obtained for all treatment combinations. Similar results of a decrease in PDI with an increase in
387 processing temperature were obtained by Thomas et al., (42) and Qin et al., (43). In Thomas et
388 al., (42) the decrease in PDI was linear with an increase in temperature for soy grits, similar to
389 what was found in DDGS. But for Qin et al., (43) the decrease in PDI was exponential for full-
390 fat soybeans collected from different origins. The decrease in PDI is due to the fact that at higher
391 temperatures, the denaturation of protein occurs, and hence it changes the protein's biochemical
392 and solubility properties. It was observed that heat processed soy flour had lower PDI values, but
393 high nutritional content, and high consumption and marketability (44).

394 With respect to DDGS flowability problems, water soluble protein side chains may
395 facilitate the formation of hydrogen bonds with the associated moisture film present between the
396 particles, thus facilitating liquid bridging among particles. Increasing the drying temperature led
397 to greater denaturing of the protein, which in turn reduced the water-soluble side chains and
398 hence lowered PDI. But from a flowability perspective, lower PDI may mean a lower propensity
399 to form liquid bridges, and hence, less particle caking..

400 *Effect of Drying Temperature and CDS Levels on Flowability Indicator (ζ)*

401 Figure 5 indicates the relationships between drying temperature and CDS addition levels
402 with the “flowability indicator” parameter, ζ . This parameter was developed by Ganesan et al.
403 (5), who established a predictive model for DDGS flowability based on combining Jenike and
404 Carr data using Exploratory Data Analysis techniques:

405

406
$$\zeta = f(\text{Hausner Ratio}) \quad (2)$$

407 or

408
$$\zeta = \frac{C_c}{\text{Dispersibility}} \times \frac{\delta}{\Phi} \quad (3)$$

409

410 Where, C_c represents Carr compressibility, δ represents the effective angle of friction, and Φ
411 represents the angle of internal friction. Generally, it has been found that lower regions in a
412 flowability indicator plot indicate good flow. In Ganesan et al., (5) the flowability indicator plot
413 for varying CDS and moisture contents resulted in a power law fit of $R^2 = 0.94$. For this study,
414 higher drying temperature treatments occupied a position towards the origin of the plot,
415 indicating better flow for those treatments. For 15% (wb) CDS addition levels, an R^2 value of
416 0.90 was obtained, but for 10% and 20% CDS levels, high R^2 values were not observed with a
417 power law regression model. Similar results were obtained when fitting exponential regression
418 equations to the data set, as indicated in Figure 6. Overall, the lower the drying temperature, the
419 worse the DDGS flowability.

420 ***Regression Modeling and Multivariate Analysis***

421 Table 5 provides regression output for various combinations of physical and flow
422 properties, and provides predictive models for flowability parameters as functions of drying
423 temperature and CDS levels. The ratio of Total Flow Index/Jenike Flow Function Index (-)
424 yielded the highest R^2 (0.943), whereas the ratio of Total Flow Index/Total Flood Index (-) yields
425 a slightly lower R^2 (0.920), but has the lowest standard error value (0.031). Thus, from a
426 standard error point of view, Total Flow Index/Total Floodability Index = f (drying temperature,
427 CDS levels) resulted in a better model for flowability than the Total Flow Index/Jenike Flow
428 Function Index = f (drying temperature, CDS levels).

429 Both of these new response variables are dimensionless, and therefore yield versatile
430 models for DDGS flowability. Additionally, these dimensionless parameters can overcome
431 limitations imposed by the units. Techniques for combining two or more properties with similar
432 units in order to achieve dimensionless parameters are often used in dimensional analysis and
433 Exploratory Data Analysis (EDA) (21). Extensive work by others regarding flowability
434 examined the dimensionless parameter ζ (equation 2), and obtained an R^2 value of about 0.92
435 (21). However, for our case, $R^2 = 0.64$ was obtained, as indicated in Table 5. Angle of repose (R^2
436 = 0.88) and Jenike Flow Function Index ($R^2 = 0.80$) yielded a better prediction of flowability
437 than ζ . The Hausner Ratio, which is often used as a robust parameter to describe flowability, did
438 not give promising results in this study, with $R^2 = 0.601$.

439 Response surface plots for AoR, HR, ζ , Jenike Flow Function, Total Flow Index/Jenike
440 Flow Function, and Total Flow Index/Total Flood Index as functions of drying temperature and
441 CDS levels are provided in Figures 7 through 12. For “good” flow behavior in DDGS, it was
442 predicted that the ratio of Total Flow Index/Total Flood Index (-) should be from 1.25 to 1.29,
443 which indicates that drying temperature should be $>225^\circ\text{C}$ and CDS $< 14\%$ (wb) or $>17.5\%$ (wb)
444 (Figure 11). Perhaps higher CDS levels ($>17.5\%$, wb) lubricates the material and produces better
445 DDGS flow. Additionally, for “good” flow behavior in DDGS, it was predicted that the ratio of
446 Total Flow Index/Jenike Flow Index (-) should be from 22.50 to 24.50, which means that the
447 drying temperature should be between 200 to 260°C and CDS $< 14\%$ (wb) (Figure 12).

448 Another way to look at the data is by multivariate analysis. Figure 13a and Figure 13b
449 represent the loading plot and model selection plot obtained from Partial Least Squares (PLS)
450 regression for Jenike Flow Function Index as a function of all physical and flow variables, as
451 listed in Table 1. Simple correlations between the original and the new variables are called the

452 “loadings,” and they indicate to what extent the original variables are influential in forming the
453 new set of Principal Components. In other words, the higher the loading value of the variable,
454 the more influential it is in forming the principal component scores.

455 Table 6 presents the summary results for the PLS regression modeling of Jenike Flow
456 Index as a function of all other properties. It was observed that a high R^2 value of 0.90 using only
457 2 components was possible. Jenike Flow Index can easily be calculated from linear regression
458 modeling whose coefficients are listed in Table 6. Therefore, the labor intensive task of the
459 Jenike shear test procedure could be avoided.

460 From Figure 13a, for 2 components only, AoS, Compressibility, Total Flow Index,
461 Hausner Ratio, and water activity were the variables which most influenced Jenike Flow Index.
462 Figure 13b indicates $R^2 = 0.9$ using two principal components only, although 6 components
463 resulted in an R^2 of nearly 0.97. Our experimental design had 26 dependent variables and 2
464 independent variables. PLS regression was found to be effective in reducing the
465 multidimensional dataset to a fewer number of components without loss of information (45).

466

467

CONCLUSIONS

468

469 Based on our results, the flowability parameters such as Hausner Ratio, Jenike Flow
470 Function, PDI, and ζ (a dimensionless flowability indicator) showed better flowability at higher
471 drying temperatures. Also, at higher drying temperatures, fewer significant differences were
472 observed in the flow and physical parameters among the CDS levels. Non-linear regression
473 analyses developed with dimensionless flowability parameters resulted in $R^2 > 0.90$, and
474 adequately represented the effects of CDS and drying temperature. Partial Least Squares (PLS)
475 regression could effectively summarize the data with only two components, and provided a

476 model for predicting Jenike Flow Index (-) as a function of all other flow and physical
477 parameters. Thus, future flowability studies may be able to avoid performing the labor-intensive
478 Jenike shear tests. Further studies with a larger sample size and more elaborate drying
479 temperatures and CDS addition levels should be investigated. Additional studies to quantify
480 flowability changes with varying cooling temperatures and times during storage should also be
481 pursued in order to more fully understand DDGS flowability behavior.

482

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484

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490

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Table 1: Main effects of drying temperature and CDS levels on resulting flow and physical properties of DDGS.*

Properties	Temperature (°C)			CDS (% wb)		
	100	200	300	10	15	20
Angle of Repose (°)	44.47b (0.35)	44.86b (0.71)	46.36a (0.56)	42.86c (0.07)	45.69b (1.10)	47.12a (1.32)
Hausner Ratio (-)	1.17a (0.01)	1.15a (0.01)	1.09b (0.03)	1.11b (0.07)	1.14ab (0.02)	1.15a (1.00)
Carr Compressibility (%)	14.71a (3.53)	12.80a (2.56)	7.88b (3.72)	9.68b (2.71)	12.17ab (2.13)	13.54a (1.73)
Angle of Spatula (°)	53.95a (1.07)	48.67c (1.10)	49.26b (0.97)	50.21c (1.21)	50.96a (1.32)	50.70b (1.10)
Uniformity (-)	2.26a (0.13)	2.13b (0.01)	1.86c (0.01)	2.34a (0.11)	2.05b (0.20)	1.85c (0.01)
Total Flow Index (-)	75.86b (3.15)	74.83b (1.06)	79.40a (1.26)	78.178a (2.17)	75.25c (1.64)	76.66b (1.72)
Angle of Fall (°)	41.27a (2.17)	39.49a (1.93)	38.90a (2.00)	40.96a (1.94)	41.00a (1.16)	37.69b (1.15)
Angle of Difference (°)	3.19b (0.07)	5.37ba (0.41)	7.46a (0.32)	1.89c (0.55)	4.69b (0.52)	9.43a (0.07)
Dispersibility (%)	39.30b (5.32)	52.38a (4.73)	47.88a (4.62)	36.81c (4.84)	64.07a (3.70)	38.66b (3.62)
Total Flood Index (-)	63.83c (1.32)	69.33a (1.26)	67.17b (2.17)	62.67c (2.07)	69.83a (3.15)	67.83b (3.02)
δ (°)	34.44a (0.72)	36.22a (0.97)	36.56a (0.56)	39.00a (0.41)	33.55b (0.31)	34.66b (0.27)
Φ (°)	19.66b (1.07)	24.79a (1.02)	23.67a (1.32)	24.22a (1.75)	21.55a (1.89)	22.33a (2.52)
σ_c (kPa)	21.57a (0.65)	17.98b (0.57)	18.98b (0.41)	18.63b (0.72)	20.33a (0.81)	19.56a (0.32)
σ_1 (kPa)	40.21b (2.13)	47.24a (2.13)	36.87c (1.72)	42.00a (1.42)	42.54a (1.37)	39.77b (1.26)
Jenike Flow Function Index (-)	1.87c (0.11)	2.68a (0.02)	2.18b (0.31)	2.58a (0.25)	2.09b (0.71)	2.04b (0.81)
Jenike Compressibility (1/cm)	13.82ab (3.71)	9.54b (4.51)	16.30a (5.31)	12.05ba (4.07)	9.85b (3.17)	17.76a (4.02)
Hunter L (-)	48.63a (2.71)	46.43b (1.35)	43.87c (1.42)	45.51a (3.15)	47.53b (2.73)	45.89c (1.17)
Hunter a (-)	9.99a (1.71)	10.05a (2.17)	9.35b (2.34)	9.74a (3.17)	9.63a (3.07)	10.01b (2.71)
Hunter b (-)	23.47a (2.10)	21.93a (3.12)	20.61c (2.17)	21.91a (3.02)	22.38b (3.17)	21.69c (1.36)
a_w (-)	0.68a (3.51)	0.44b (3.15)	0.28c (2.00)	0.40c (1.71)	0.47b (3.02)	0.52a (1.71)
Thermal Conductivity (W/(m)(°C))	0.06b (0.01)	0.07a (0.01)	0.07b (0.02)	0.06b (0.02)	0.06ab (0.10)	0.07a (0.04)
Thermal Resistivity ((m)(°C)/W)	16.54a (0.02)	15.06b (0.07)	15.98a (0.03)	17.12a (0.02)	15.36b (0.03)	15.08b (0.12)
Thermal Diffusivity (mm ² /s)	0.17a (0.01)	0.15b (0.00)	0.15b (0.01)	0.17a (0.01)	0.15b (0.02)	0.15b (0.00)
Geometric Mean Diameter (mm)	0.73b (0.01)	0.78b (0.51)	0.99a (0.42)	0.91a (0.21)	0.75b (0.21)	0.83ab (0.31)
Porosity (-)	0.58a (2.13)	0.47b (3.17)	0.47b (1.51)	0.54a (2.17)	0.51b (2.07)	0.46c (1.91)
PDI (%)	9.3a (2.11)	7.61b (1.45)	7.21b (3.02)	8.33a (0.89)	7.59b (1.02)	8.19a (1.50)

* Values with differing letters within a given row for a given independent variable are significantly different ($p < 0.05$, LSD); values in parentheses indicate ± 1 standard deviation; CDS is condensed distillers solubles (% wb); δ is effective angle of internal friction (°); Φ is angle of internal friction (°); σ_c is unconfined yield strength (kPa); σ_1 is major consolidation stress (kPa); a_w is water activity (-); PDI is protein dispersibility index (%).

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Table 2: Interaction effects (p-values) of drying temperature and CDS levels on resulting flow and physical properties of DDGS.*

Properties	Temperature (°C)	CDS (% wb)	CDS*Temp
Angle of Repose (°)	<0.0001	<0.0001	0.0003
Hausner Ratio (-)	0.0004	0.0344	0.2664
Carr Compressibility (%)	0.0003	0.033	0.2766
Angle of Spatula (°)	<0.0001	<0.0001	<0.0001
Uniformity (-)	<0.0001	<0.0001	<0.0001
Total Flow Index (-)	<0.0001	<0.0001	<0.0001
Angle of Fall (°)	<0.0001	<0.0001	<0.0001
Angle of Difference (°)	<0.0001	<0.0001	<0.0001
Dispersibility (%)	<0.0001	<0.0001	<0.0001
Total Flood Index (-)	<0.0001	<0.0001	<0.0001
δ (°)	0.0362	<0.0001	<0.0001
Φ (°)	0.0032	0.1519	0.0175
σ_c (kPa)	<0.0001	0.003	0.0042
σ_1 (kPa)	<0.0001	0.0002	<0.0001
Jenike Flow Function Index (-)	<0.0001	<0.0001	<0.0001
Jenike Compressibility (1/cm)	<0.0001	<0.0001	<0.0001
Hunter L (-)	<0.0001	<0.0001	<0.0001
Hunter a (-)	0.0012	0.1158	0.0019
Hunter b (-)	<0.0001	0.0871	0.0052
a_w (-)	<0.0001	<0.0001	<0.0001
Thermal Conductivity (W/((m)(°C))	0.0008	0.0221	0.0358
Thermal Resistivity ((m)(°C)/W)	<0.0001	<0.0001	<0.0001
Thermal Diffusivity (mm ² /s)	<0.0001	<0.0001	0.004
Geometric Mean Diameter (mm)	<0.0001	<0.0001	<0.0001
Porosity (-)	<0.0001	<0.0001	0.0176
PDI (%)	<0.0001	0.0006	0.0064

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* Values in the table are the p-values determined by PROC GLM using SAS, p<0.05; CDS is condensed distillers solubles (% wb); δ is effective angle of internal friction (°); Φ is angle of internal friction (°); σ_c is unconfined yield strength (kPa); σ_1 is major consolidation stress (kPa); a_w is water activity (-); PDI is protein dispersibility index (%).

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Table 3: Treatment combination effects of drying temperature and CDS levels on resulting flow and physical properties of DDGS.*

Properties	Temperature (°C)								
	100			200			300		
	CDS (% wb)			CDS (% wb)			CDS (% wb)		
	10	15	20	10	15	20	10	15	20
Angle of Repose (°)	42.06c (0.42)	44.71b (0.61)	46.61a (0.51)	42.02c (0.31)	44.94b (0.25)	47.62a (0.75)	44.55c (0.44)	47.43a (0.37)	47.14b (0.31)
Hausner Ratio (-)	1.13a (0.02)	1.19a (0.07)	1.20a (0.00)	1.11a (0.17)	1.15a (1.21)	1.18a (0.05)	1.09a (1.03)	1.07a (1.71)	1.09a (1.21)
Carr Compressibility (%)	11.28a (3.21)	15.85a (2.71)	17.00a (1.17)	9.39b (3.12)	13.55ab (1.91)	15.45a (1.71)	8.67a (2.56)	7.120a (1.71)	8.16a (2.01)
Angle of Spatula (°)	55.86a (1.07)	53.50b (1.21)	52.49c (1.11)	46.26c (0.92)	50.18a (2.17)	49.56b (1.31)	48.51c (0.91)	49.20b (1.07)	50.05a (1.03)
Uniformity (-)	2.53a (0.01)	2.00b (0.21)	1.85c (0.01)	2.51a (0.31)	2.31a (0.02)	1.95c (0.01)	2.000a (0.03)	1.840b (0.21)	1.75c (0.01)
Total Flow Index (-)	77.83a (4.12)	75.25b (2.17)	74.50c (3.17)	78.50a (2.72)	74.50b (3.51)	71.50c (2.16)	78.20a (1.50)	79.00a (1.42)	81.00a (1.37)
Angle of Fall (°)	39.87c (1.41)	40.75b (1.37)	43.18a (1.21)	39.36a (1.06)	39.750a (1.21)	39.36a (1.03)	43.66a (0.04)	42.50a (0.51)	30.53b (0.72)
Angle of Difference (°)	2.19b (0.07)	3.96a (0.09)	3.43ab (1.02)	2.66c (0.06)	5.193b (1.21)	8.26a (1.03)	0.84c (0.04)	4.93b (0.51)	16.60a (0.72)
Dispersibility (%)	41.70b (3.71)	46.07a (4.02)	30.13c (5.72)	34.16c (4.62)	73.40a (3.12)	49.57b (2.17)	34.59b (5.07)	72.75a (4.31)	36.29b (3.76)
Total Flood Index (-)	61.00b (1.71)	70.00a (2.07)	60.50c (1.51)	65.50c (1.32)	70.00b (1.40)	72.50a (0.91)	61.50c (2.16)	69.50b (1.73)	70.50a (1.51)
δ (°)	37.66a (0.45)	33.33b (0.32)	32.33b (0.21)	34.33b (0.74)	35.33b (0.51)	39.00a (0.71)	45.00a (0.64)	32.00b (0.81)	32.66b (1.02)
Φ (°)	17.33b (1.01)	21.66a (1.07)	20.00ab (1.21)	27.00a (1.31)	22.33b (0.75)	25.00a (0.51)	28.33a (1.72)	20.66a (2.01)	22.00a (2.31)
σ _c (kPa)	21.94a (0.41)	21.68a (0.21)	21.09a (0.23)	15.67b (0.71)	19.93a (0.50)	18.32a (1.51)	18.27a (1.72)	19.39a (1.63)	19.28a (1.54)
σ ₁ (kPa)	37.68b (1.71)	42.13a (1.52)	40.81a (1.31)	53.22a (0.72)	45.91b (0.56)	42.59c (1.15)	35.11b (1.72)	39.58a (1.63)	35.91b (1.54)
Jenike Flow Function Index (-)	1.72b (0.11)	1.94a (0.02)	1.94a (0.00)	3.39a (0.31)	2.30b (0.71)	2.33b (0.31)	2.64a (0.43)	2.03b (0.500)	1.86c (0.37)
Jenike Compressibility (1/cm)	12.57b (4.12)	7.10c (3.17)	21.79a (2.71)	2.19b (5.16)	12.82a (5.01)	13.61a (3.74)	21.39a (3.02)	9.64c (1.21)	17.87b (1.94)
Hunter L (-)	46.22c (2.17)	51.72a (1.23)	47.94b (2.13)	47.24a (1.93)	46.67a (1.16)	45.38a (1.53)	43.07b (1.62)	44.18a (2.10)	44.36a (0.77)
Hunter a (-)	9.48b (2.01)	9.80ba (1.71)	10.68a (1.51)	10.59a (1.010)	9.51b (0.09)	10.03ba (0.91)	9.13a (0.72)	9.59a (0.77)	9.32a (0.73)
Hunter b (-)	22.94ab (2.13)	24.73a (1.71)	22.73b (1.63)	22.66a (1.63)	21.68ab (2.07)	21.45b (0.17)	20.15b (0.89)	20.75a (1.31)	20.91a (0.74)
a _w (-)	0.64c (0.71)	0.68b (0.43)	0.71a (0.06)	0.33c (0.31)	0.46b (0.21)	0.54a (0.01)	0.23c (1.20)	0.28b (1.03)	0.32a (0.17)
Thermal Conductivity (W/(m)(°C))	0.06b (0.03)	0.06b (0.31)	0.07a (0.01)	0.07a (0.02)	0.07a (0.04)	0.07a (0.05)	0.06a (0.03)	0.07a (0.01)	0.066a (0.00)
Thermal Resistivity ((m)(°C)/W)	17.86a (0.01)	16.73b (0.01)	15.03c (0.07)	15.30a (0.06)	14.60b (0.64)	15.26a (0.05)	18.20a (0.04)	14.76b (0.06)	14.96b (0.51)
Thermal Diffusivity (mm ² /s)	0.18a (0.01)	0.173a (0.34)	0.16b (0.07)	0.16a (0.06)	0.143a (0.17)	0.15a (0.12)	0.17a (0.51)	0.14b (0.42)	0.14b (0.31)
Geometric Mean Diameter (mm)	0.69b (0.13)	0.67c (0.71)	0.82a (0.53)	0.72c (0.07)	0.79b (0.06)	0.83a (0.51)	1.31a (0.41)	0.81c (0.31)	0.85b (0.27)
Porosity (-)	0.61a (1.37)	0.57b (2.01)	0.55b (3.14)	0.49a (1.42)	0.49a (2.15)	0.43b (2.14)	0.53a (1.71)	0.47b (1.62)	0.42c (1.06)
PDI (%)	9.56a (0.71)	9.34a (1.45)	8.98a (3.51)	8.10a (1.03)	6.85b (3.12)	7.88a (4.02)	7.33a (3.140)	6.59ab (0.89)	7.71a (0.93)

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* Values with differing letters within a given row are significantly different (p<0.05, LSD) across all treatment combinations; values in parentheses indicate ±1 standard deviation; bold numbers indicate values for which there were no significant differences amongst the CDS levels for a given drying temperature level. CDS is condensed distillers solubles (% wb); δ is effective angle of internal friction (°); Φ is angle of internal friction (°); σ_c is unconfined yield strength (kPa); σ₁ is major consolidation stress (kPa); a_w is water activity (-); PDI is protein dispersibility index (%).

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Table 4: Pearson linear correlation coefficients (r) between resulting flow and physical properties for DDGS prepared using varying drying temperature and CDS combinations. Only significant (p < 0.05) correlations are listed*

Variable combinations	r	p-value
Thermal Conductivity × Temperature	-0.936	<0.0001
Angle of Difference × Angle of Fall	-0.889	<0.0001
Angle of Repose × Uniformity	-0.881	<0.0001
σ_1 × Jenike Compressibility	-0.857	<0.0001
Temperature × a_w	-0.820	<0.0001
Jenike Compressibility × Angle of Spatula	-0.796	<0.0001
Temperature × Hunter a	-0.792	<0.0001
CDS × Uniformity	-0.736	<0.0001
Thermal Diffusivity × Thermal Resistivity	-0.716	<0.0001
Temperature × Angle of Spatula	-0.705	<0.0001
Thermal Conductivity × Carr Compressibility	0.705	<0.0001
Thermal Conductivity × Hunter a	0.721	<0.0001
a_w × Thermal Conductivity	0.736	<0.0001
PDI × Geometric Mean Diameter	0.743	<0.0001
Thermal Conductivity × Angle of Spatula	0.796	<0.0001
Thermal Diffusivity × Geometric Mean Diameter	0.828	<0.0001
Hunter a × a_w	0.852	<0.0001
σ_1 × Angle of Spatula	0.867	<0.0001
Hausner Ratio × Carr Compressibility	0.998	<0.0001

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* CDS is condensed distillers solubles level (%; wb); σ_1 is major consolidation stress (kPa); a_w is water activity (-); PDI is protein dispersibility index (%).

Table 5: Prediction models for selected dependent variables developed by response surface regression.*

Dependent Variable	z	AOR (-)	HR (-)	Jenike Flow Function Index (-)	ζ (-)	Total Flow Index/Total Flood Index (-)	Total Flow Index/Jenike Flow Function Index (-)
Independent Variable	x	Temperature	Temperature	Temperature	Temperature	Temperature	Temperature
	y	CDS	CDS	CDS	CDS	CDS	CDS
Prediction Equation		$z=a+bx^3+c/y$	$z^{-1}=a+bx^3+c/y$	$z^{-1}=a+b/\ln x+c/x^{0.5}+d\ln y+e/\ln y$	$z=a+b/x+cy^3$	$z=(a+bx+cx^2+d\ln y)/(1+ex+fx^2+g\ln y+h(\ln y)^2)$	$z=a+b\ln x+c/y+d(\ln x)^2+e/y^2+f(\ln x)/y$
	R ²	0.881	0.601	0.804	0.643	0.920	0.943
Model Performance	Adjusted R ²	0.865	0.549	0.757	0.596	0.884	0.926
	F statistic	88.733	18.091	22.512	21.610	31.096	69.793
	Standard error	0.760	0.037	0.242	0.142	0.031	1.858
Model Parameters	a	5.05E+01	7.89E-01	2.60E+01	-9.37E-03	-9.73E-01	8.38E+02
	b	7.39E-08	2.63E-09	-1.77E+02	5.85E+01	-2.98E-03	-3.36E+02
	c	-8.52E+01	8.08E-01	1.76E+02	2.44E-05	6.17E-06	1.92E+03
	d			-8.16E-01		3.76E-01	3.51E+01
	e			-6.77E+00		-3.44E-03	1.08E+02
	f					7.53E-06	-3.93E+02
	g					-9.81E-01	
	h					2.38E-01	
Figure No.		7	8	9	10	11	12

* AoR is angle of repose (°); HR is Hausner Ratio (-); CDS is condensed distillers solubles level (% wb); ζ is an empirical flowability indicator defined as = $(C_d/\text{Dispensibility}) * (\delta/\Phi)$ (Ganesan et al., 2007b). Each equation is plotted in the denoted figure.

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Table 6. Partial Least Squares (PLS) regression results for Jenike Flow Function Index (-) as a multivariate function of all other flow and physical properties (excluding the Jenike properties).

Predictor Variables	Parameter Estimates
Constant	9.6106
Angle of Repose	-0.0817
Hausner Ratio	0.2675
Compressibility	0.0036
Angle of Spatula	-0.1203
Uniformity	0.2858
Total Flow Index	-0.0100
Angle of Fall	0.0145
Angle of Difference	-0.0272
Dispersibility	-0.0115
Total Flood Index	0.0301
Water Activity	-0.2967
Thermal Conductivity	12.4430
Thermal Diffusivity	-0.9390
Geometric Mean Diameter	0.0266
Porosity	-0.4575
R ²	0.90
p-Value	0.0001
F Statistic	100.39
PLS Components Required	2

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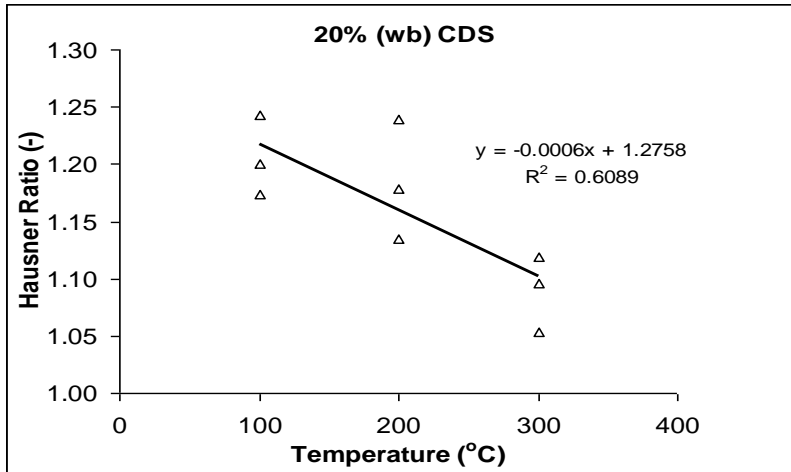
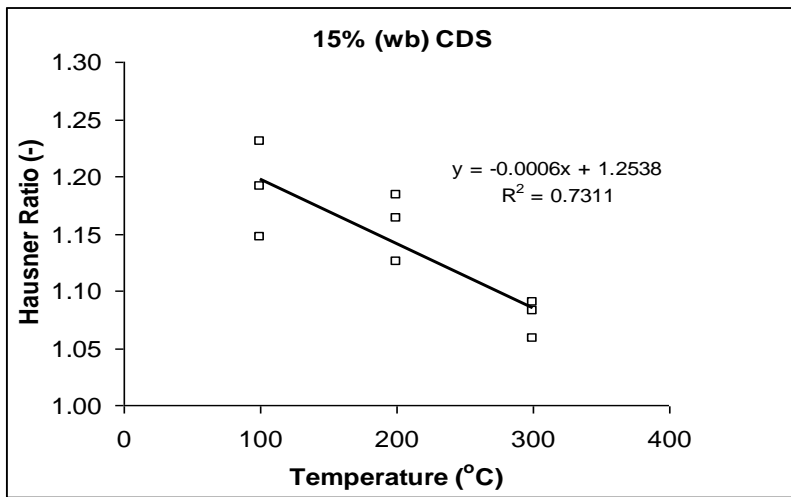
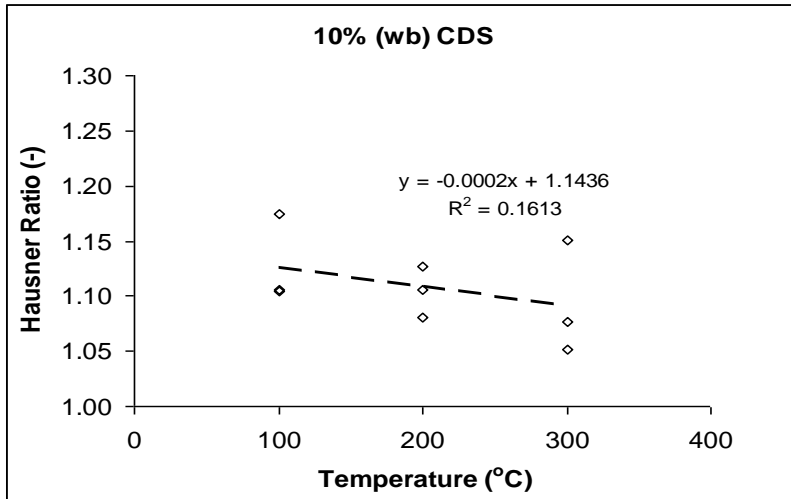


Figure 1: Relationships between Hausner Ratio and drying temperature according to CDS level.

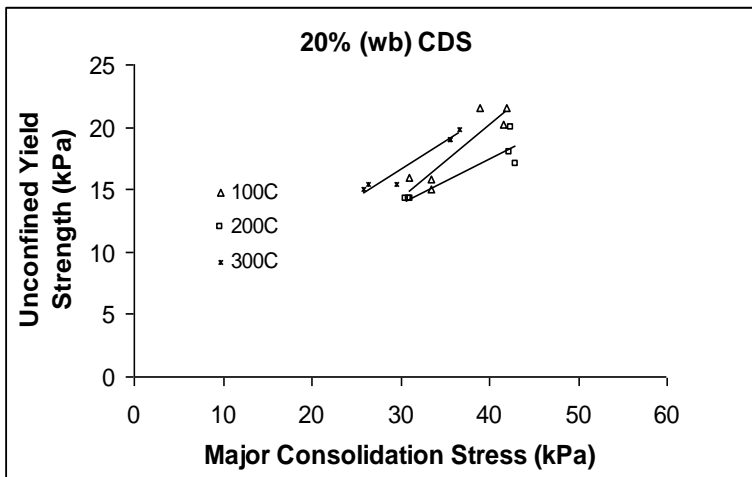
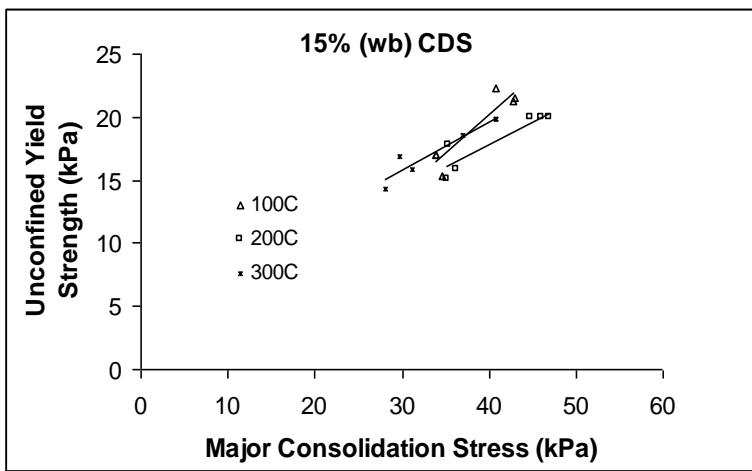
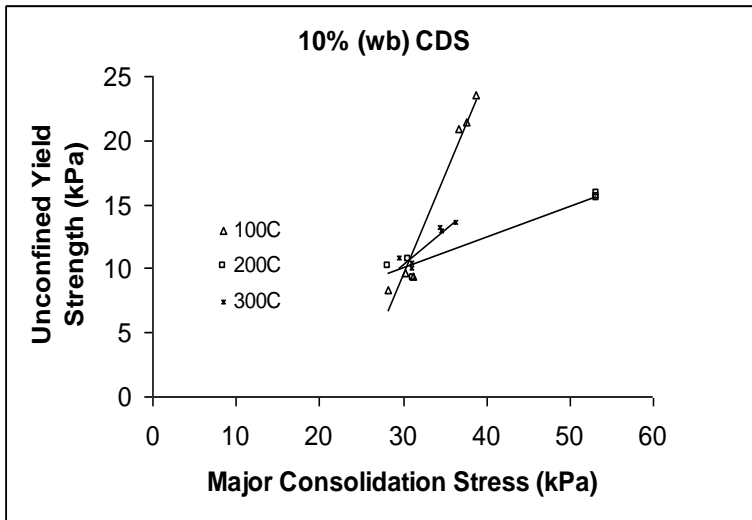


Figure 2: Relationships between unconfined yield strength (σ_c), major consolidation stress (σ_1), (which are known as Jenike Flow Function curves), and drying temperature according to CDS level.

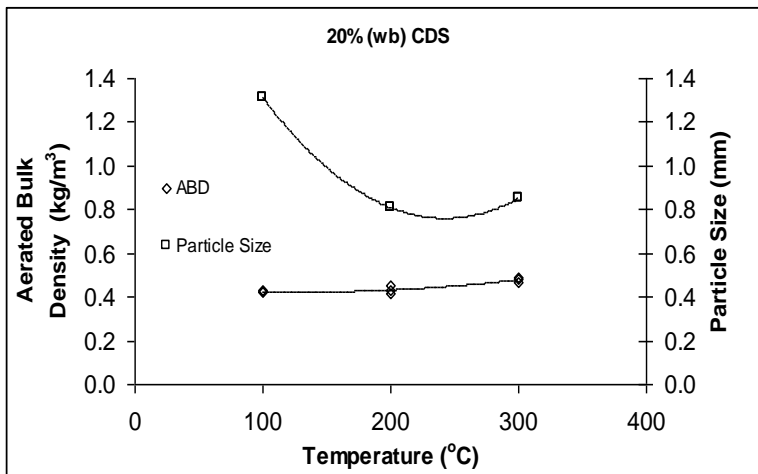
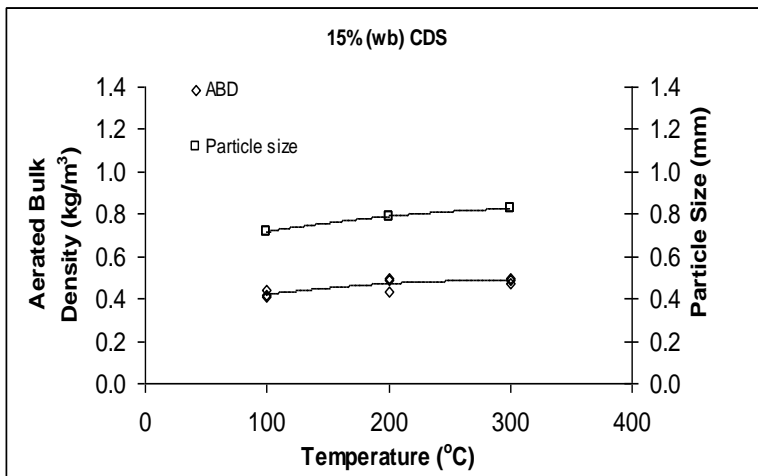
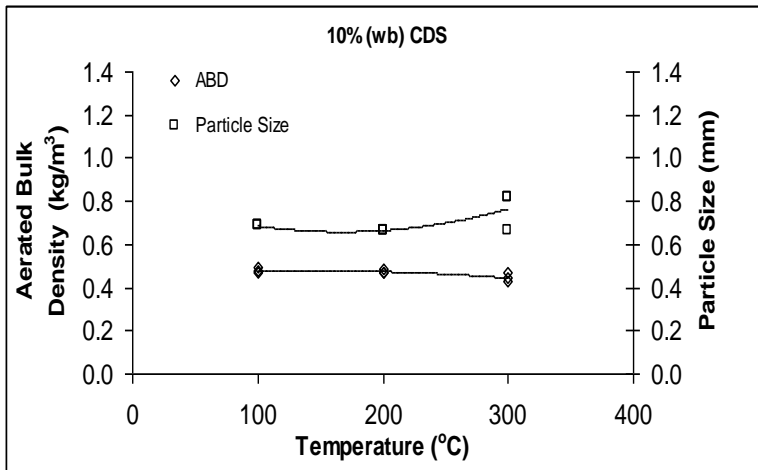


Figure 3: Relationships between aerated bulk density (ABD), particle size, and drying temperature according to CDS level. Particle size is defined as geometric mean diameter (d_{gw}).

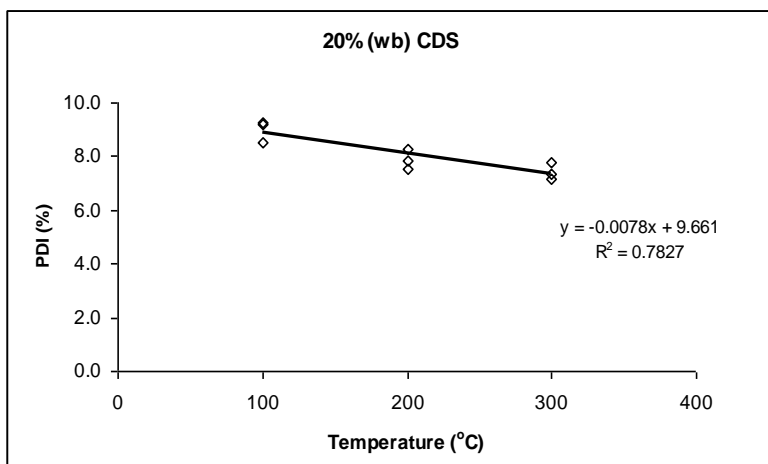
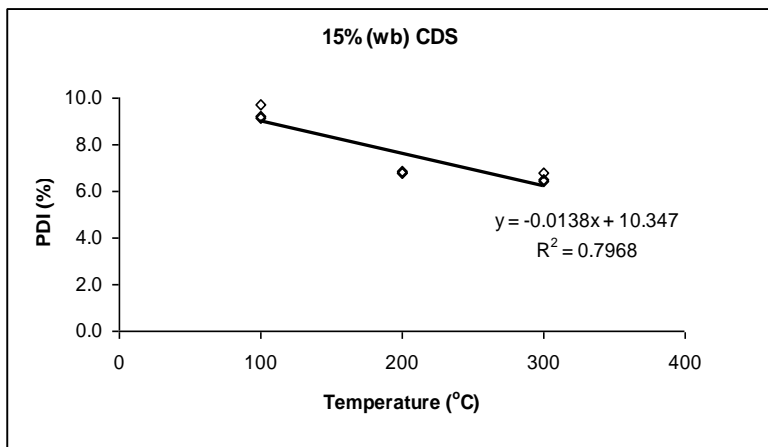
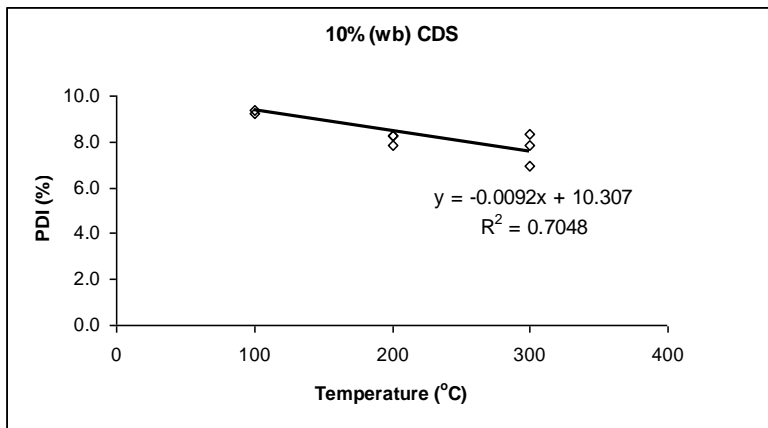


Figure 4: Relationships between protein dispersibility index (PDI) and drying temperature according to CDS level.

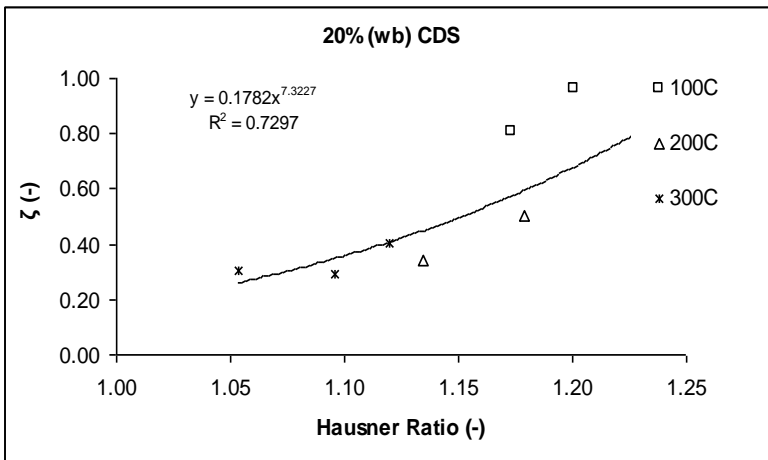
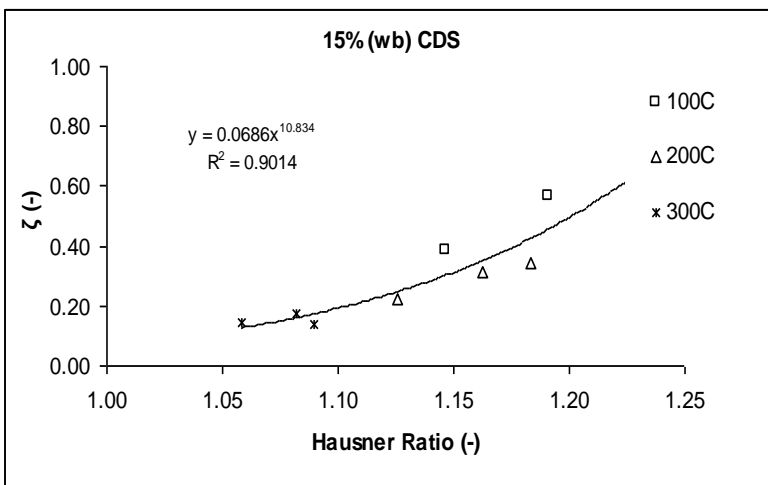
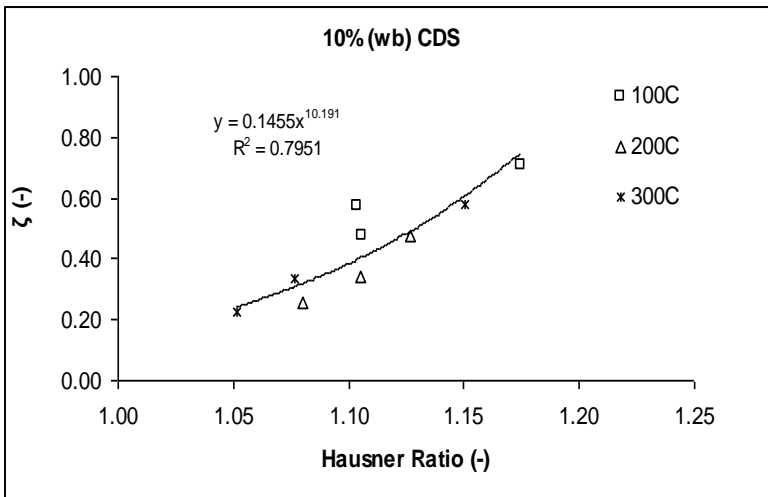


Figure 5: Validation of empirical flowability model (from Ganesan et al., 2007b) by fitting power law regression equations according to CDS level; ζ is defined as $(C_c/\text{Dispersibility}) * (\delta/\Phi)$.

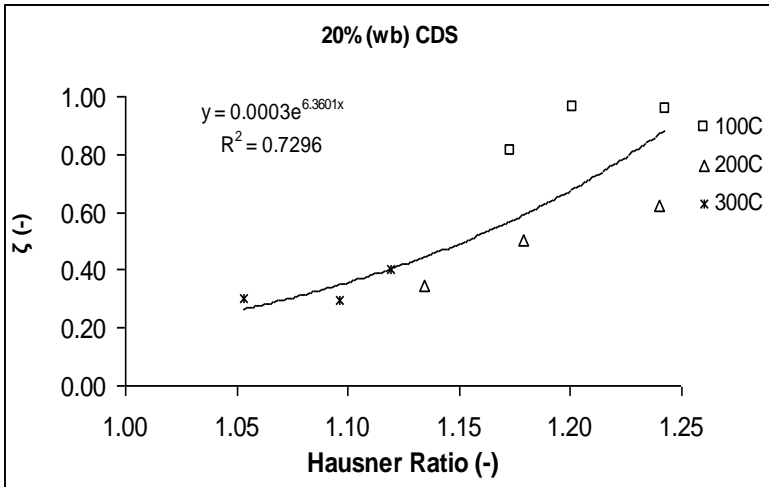
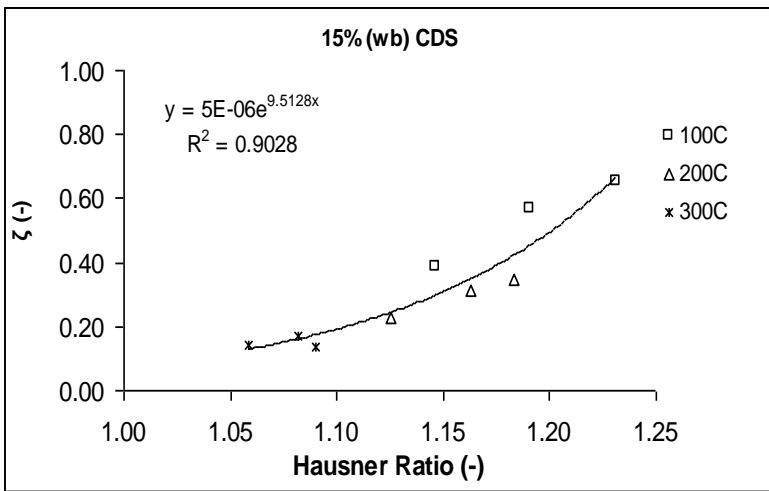
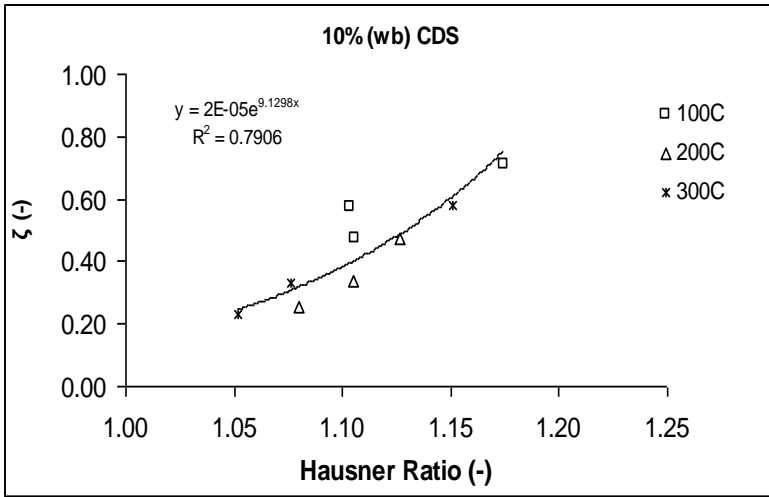


Figure 6: Validation of empirical flowability model (from Ganesan et al., 2007b) by fitting exponential regression equation, according to CDS level; ζ is defined as $(C_c/\text{Dispersibility}) * (\delta/\Phi)$.

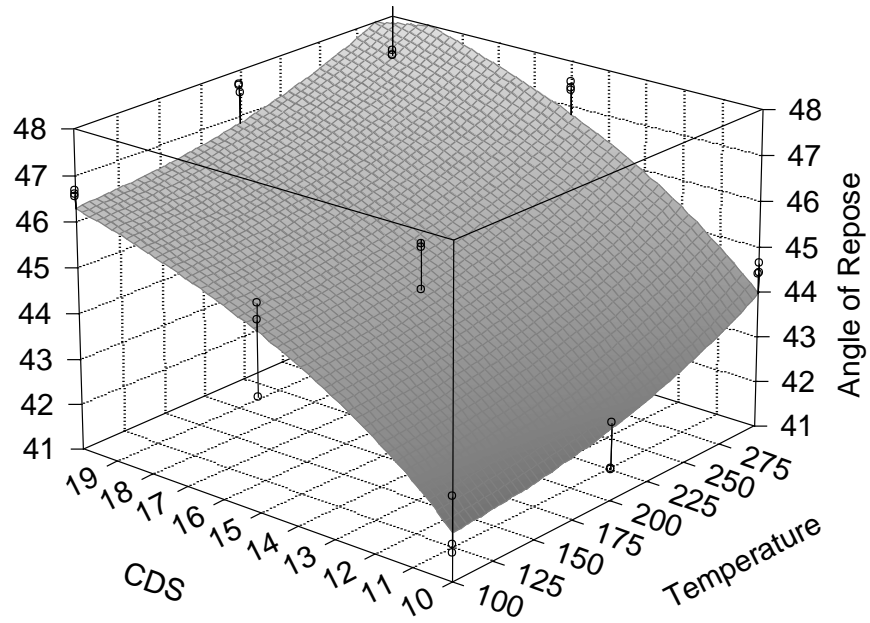


Figure 7: Surface plot showing the relationship between drying temperature (°C), CDS level (% wb), and angle of repose (°).

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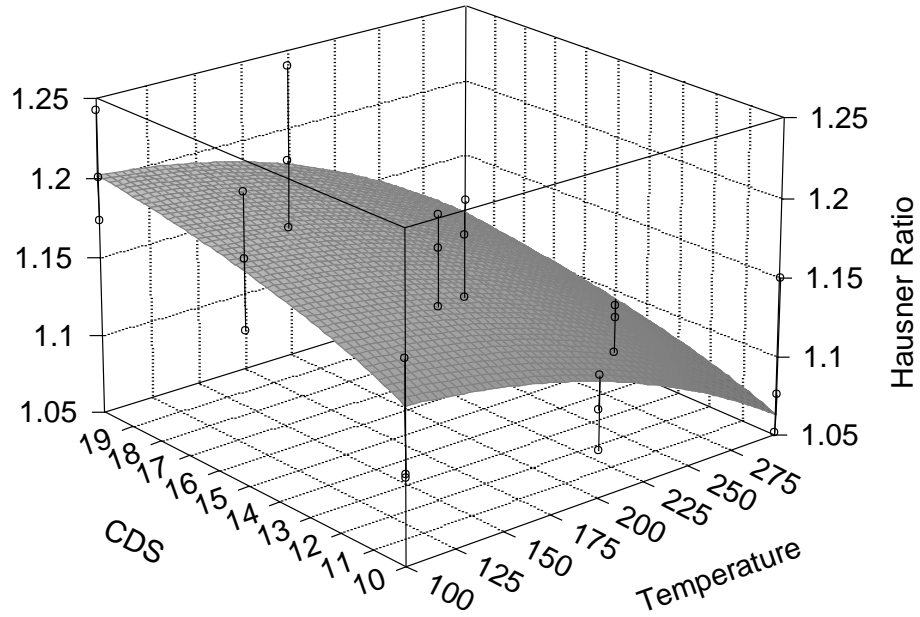


Figure 8: Surface plot showing the relationship between drying temperature (°C), CDS level (% wb), and Hausner Ratio (-).

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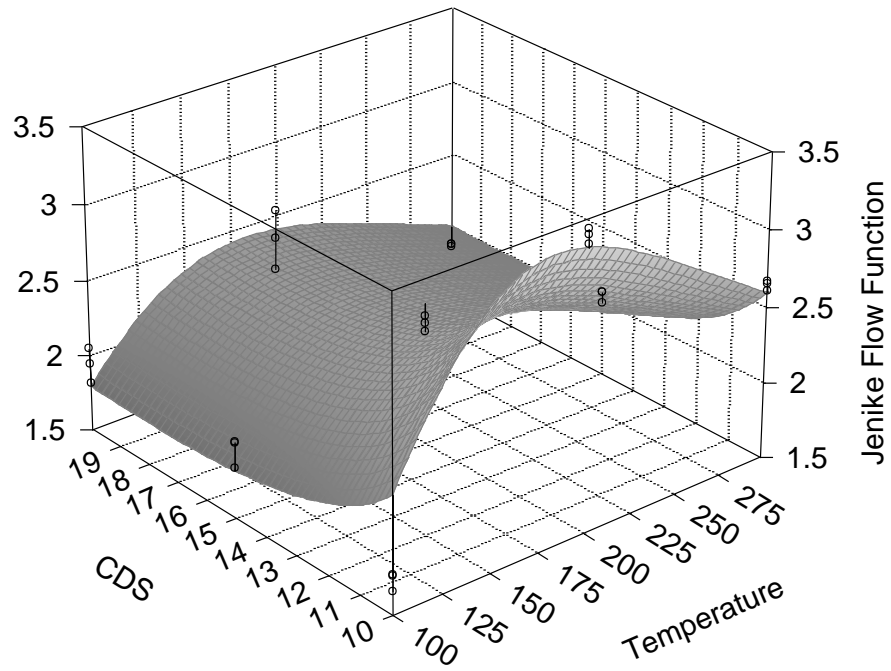


Figure 9: Surface plot showing the relationship between drying temperature (°C), CDS level (% wb), and Jenike Flow Function Index (-) [note that Jenike Flow Function Index is also known as Jenike Flow Index (-)].

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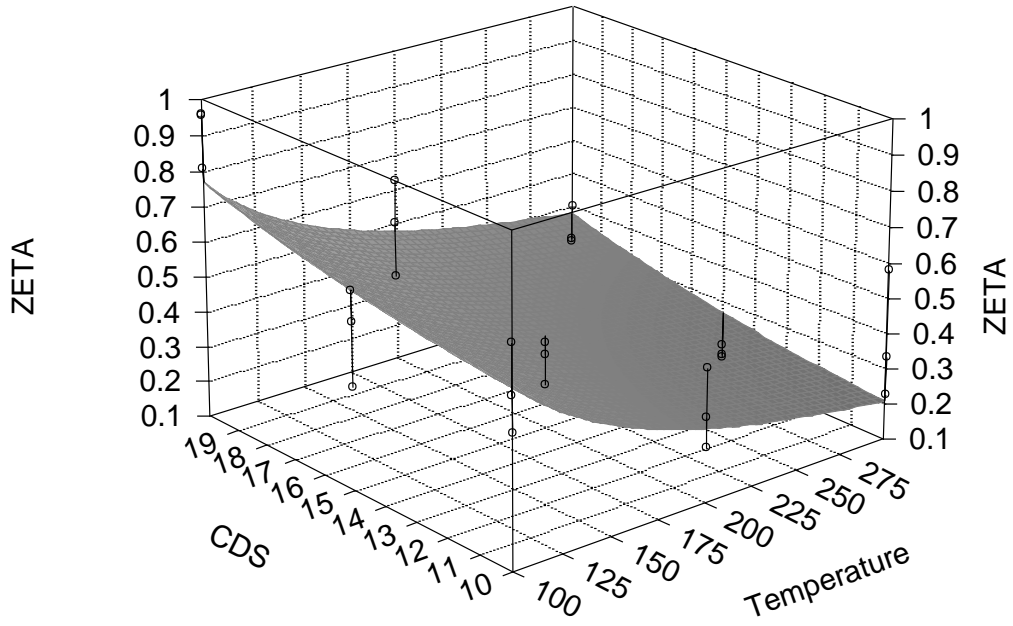


Figure 10: Surface plot showing the relationship between drying temperature (°C), CDS level (% wb), and zeta (ζ , -). Zeta is the empirical flowability indicator.

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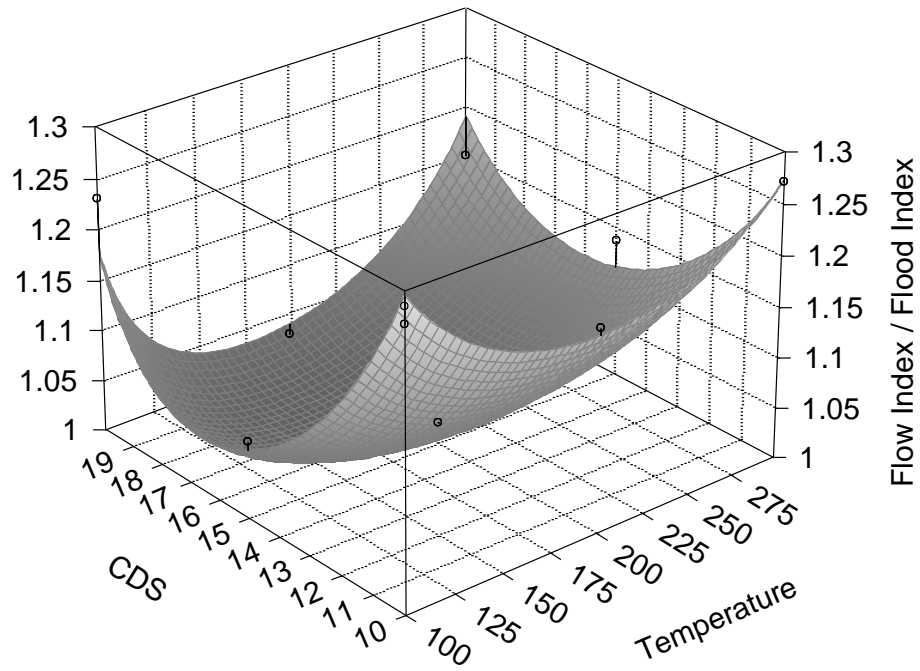


Figure 11: Surface plot showing the relationship between drying temperature (°C), CDS level (% wb), and the ratio of Total Flow Index/Total Flood Index.

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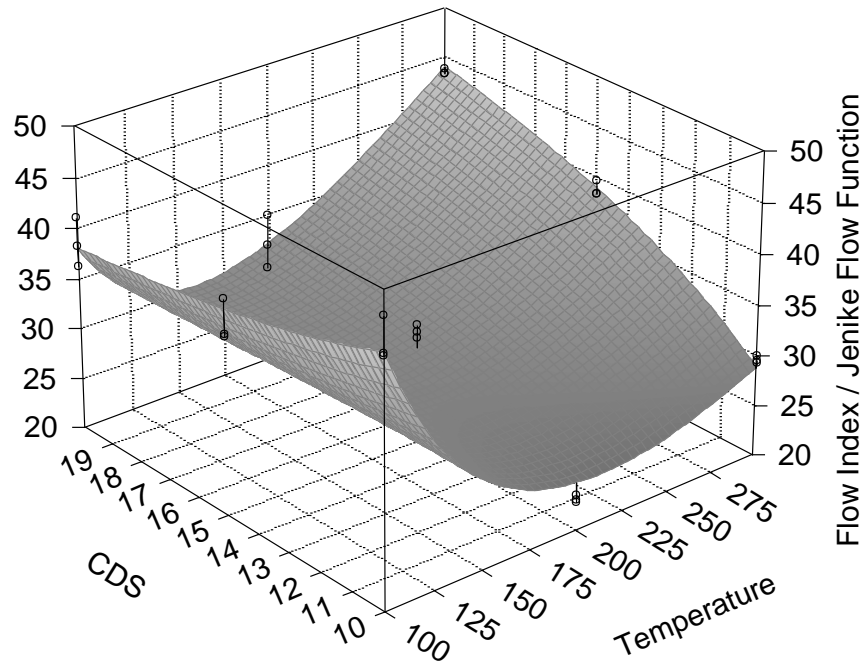
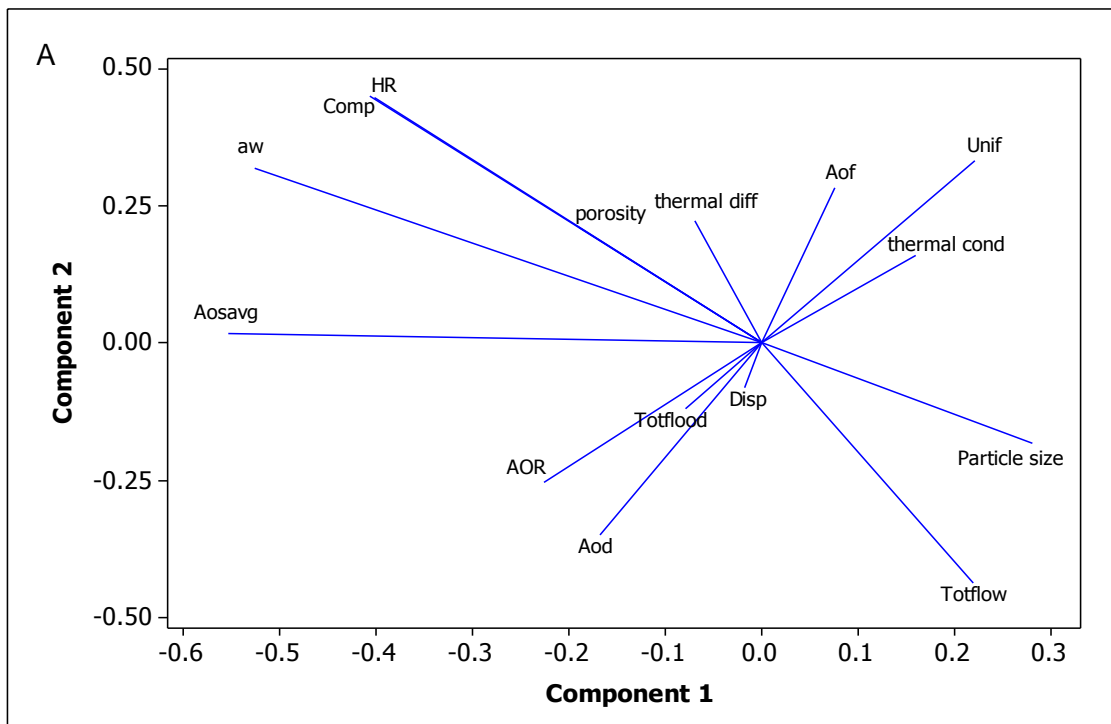
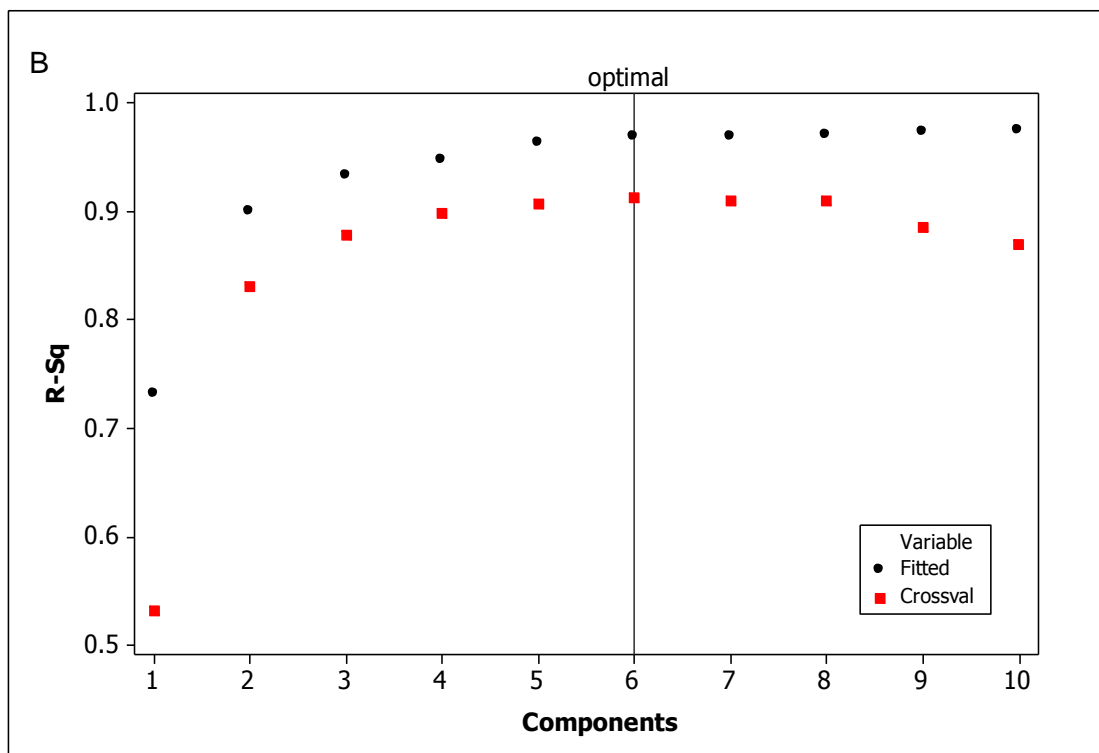


Figure 12: Surface plot showing the relationship between drying temperature (°C), CDS level (% wb), and the ratio of Total Flow Index/Jenike Flow Function Index [note that Jenike Flow Function Index is also known as Jenike Flow Index (-)].

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671 **Figure 13. Partial Least Square (PLS) regression results for Jenike Flow Function Index as a**
 672 **multivariate function of all flow and physical properties (excluding Jenike properties); (A) loading**
 673 **plot; (B) model selection plot for the PLS analysis. [“R-sq” is the coefficient of determination (R^2);**
 674 **“Fitted” indicates the fitted PLS regression line; “Cross val” is cross validation, which is a**

675 **multivariate procedure used to predict and validate the PLS regression curve by using alternative**
676 **data points from the entire data set and testing the line fit].**
677