# SELECTED ADDITIVES DID NOT IMPROVE FLOWABILITY OF DDGS IN COMMERCIAL SYSTEMS

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**ABSTRACT.** An experiment was conducted at a commercial, dry-grind ethanol plant to determine if selected additives would improve flowability of dried distillers grains with solubles (DDGS). Main treatment effects were moisture content of DDGS (9 vs. 12% w.b.) and anti-caking agent (ACA). The ACA treatments were: No additive (Control); a moisture migration control agent at 2.5 kg/metric ton (DMX-7); calcium carbonate at 2% w.b. (Calcium carbonate); or a clinoptilolite zeolite at 1.25% w.b. (Zeolite). The ACA were added at the desired level to about 2,275 kg of DDGS using a vertical-screw feed mixer. Batches of DDGS were weighed and loaded into one of eight compartments in an auger-equipped feed truck. After loading, the truck traveled 250 km, sat motionless for at least 60 h, and traveled 250 km back to the ethanol plant. Time required to unload each compartment was recorded. There were no significant interactions between moisture level and ACA for any response criteria. Mean moisture levels were 9% and 11.6% for low and high moisture treatments. Flow rate of DDGS at unloading was higher (P < 0.01) for the 9% compared with 12% moisture level (620 vs. 390 kg/min). Flow rates of DDGS at unloading were: 509 (Control), 441 (DMX-7), 512 (Calcium carbonate), and 558 (Zeolite) kg/min. None of the ACA created flow rates that differed significantly from Control. In conclusion, increasing moisture content from 9% to 11.6% clearly decreased flowability of DDGS.

Keywords. Flowability, DDGS, Angle of repose, Anti-caking agents

he Energy Policy Act passed by the U.S. Congress in 2005 mandates that 7.5 billion gallons of renewable fuels be included in gasoline sold in the United States by the year 2012 (U.S. Environmental Protection Agency, 2008). This mandate has supported the rapid expansion of ethanol production in the United States. Ethanol production in 2008 is projected to be nearly 8 billion gallons (Renewable Fuels Association, 2008). Dried distillers grains with solubles (DDGS) is an important co-product of ethanol production. About 7.2 kg of DDGS and 10.6 L of ethanol are produced from 25 kg of corn. Consequently, large quantities of DDGS are available for feeding livestock domestically and internationally. DDGS has many positive attributes for the feeding of livestock (University of Minnesota, 2008). Unfortunately, DDGS can have some undesirable handling characteristics related to poor flowability under certain conditions (Agricultural Utilization Research Institute (AURI) and Minn. Corn Growers Assoc., 2005; Bhadra et al., 2008). Reduced flowability, or the potential for reduced flowability of DDGS prevents the routine use of railcars for transport. Reduced flowability and bridging of DDGS in bulk storage containers and transport vehicles limits the use of DDGS for feeding

livestock and poultry. Livestock producers and feed mills do not want to deal with the inconvenience and expense of handling a feedstuff that does not flow through their feeding and milling systems. Consequently, some pork producers have used DDGS in the past but have discontinued their use of DDGS due to poor flowability (J. Goihl, President, Agri-Nutrition Services, personal communication).

Flow is defined as "the relative movement of a bulk of particles among neighboring particles or along the container wall surface" (Peleg, 1977). Many factors influence the flow of a bulk material (Peleg, 1977) and there is no one measurement that adequately describes flowability (Bhadra et al., 2008). Consequently, characterization of and methods to improve DDGS flowability under controlled conditions have been slow. The AURI and Minn. Corn Growers Assoc. (2005) studied a limited number of DDGS samples under laboratory conditions. They reported that relative humidity greater than 60% seemed to reduce flowability of DDGS which was likely due to the product's ability to adsorb moisture. Ganesan et al. (2007) demonstrated that DDGS does adsorb water during storage. While moisture in the environment and moisture content of DDGS likely influence flowability, many other factors have been suggested as possible controllers of flowability such as particle size, content of solubles, dryer temperature, moisture content at dryer exit, and others (Ganesan et al., 2008a,b,c).

Most interventions to improve flowability of DDGS have been limited to trial and error approaches within ethanol plants. These interventions relate to the completeness of fermentation, adjusting moisture content, and changing particle size, but have not been reported in the public domain. ILC Resources (Richard Bristol, Director of Nutrition and Technical Services, personal communication) investigated the utility of including 2% calcium carbonate in DDGS as a flowability agent. They reported a 6% to 12% reduction in the

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angle of repose determined in a laboratory setting when calcium carbonate was added to DDGS after drying. Determination of flowability under practical industry conditions was not attempted in their study. Similarly, Ganesan et al. (2008a) evaluated the addition of calcium carbonate to DDGS that contained varying concentrations of solubles and moisture in a laboratory setting. They reported reduced flowability of DDGS as concentration of solubles and moisture increased. Addition of calcium carbonate had no significant effect on flowability of DDGS. Because moisture and relative humidity seem to play an important role in flowability of DDGS, some have suggested use of zeolites and/or grain conditioners as a way of controlling moisture migration through DDGS. However, no controlled studies to evaluate this concept have been reported.

Research results reported in the scientific literature have focused on studies conducted under tightly-controlled conditions within laboratories. No studies have been reported that evaluate flowability of DDGS in commercial ethanol plants operating at typical production rates. Understanding the efficacy of interventions to improve DDGS flowability under such "real-world," commercial conditions would provide great value to the ethanol and feed milling industries. Consequently, we designed a study to determine if the addition of selected anti-caking agents is effective in improving flowability of DDGS under practical commercial conditions. Our secondary objective was to identify physical and/or chemical characteristics of DDGS produced in a commercial ethanol plant that might be related to flowability of DDGS.

## **MATERIALS AND METHODS**

This experiment was conducted at a dry-grind ethanol plant (BushMills Ethanol Inc., Atwater, Minn.) constructed in 2005. Experimental treatments were replicated on four separate days beginning on 1 September and ending on 27 October 2006.

### MOISTURE LEVEL AND ANTI-CAKING AGENTS

Treatments were imposed in a  $2 \times 4$  factorial arrangement (eight total treatments). The main treatment effects were moisture content of DDGS (9% vs. 12% w.b.) and type of anti-caking agent (ACA). The moisture treatments were selected to represent DDGS that was expected to flow readily (9%) and DDGS that was expected to present poor flowability (12%). The selected moisture levels are within ranges reported by Spiehs et al. (2002; 9.8% to 12.8% w.b.) and the University of Minnesota (2008; 7.6% to 13.8% w.b.) for DDGS samples collected from commercial ethanol plants. The ACA treatments were: 1. control; 2. a grain conditioner purported to control moisture migration (2.5 kg/metric ton, DMX-7, Delst, Inc., Temecula, Calif.); 3. calcium carbonate (2% Unical-P, ILC Resources, Inc., Des Moines, Iowa); and 4. a clinoptilolite zeolite (1.25% St. Cloud Zeolite, St. Cloud Mining Co., Winston, N. Mex.). The control was standard DDGS produced in the plant on a selected day with no ACA added. The ACA's were incorporated at the desired level to DDGS containing 9% or 12% moisture from the plant's stockpile.

During the night shift prior to our arrival at 0900 h, the ethanol plant produced DDGS containing 9% or 12% (w.b.)

moisture and placed it in two separate stockpiles. Stockpiles were housed in the plant's warehouse and all experimental work was completed in the warehouse. At about 1000 h, we began applying ACA treatments to the DDGS. About 2.275 kg of DDGS was augered into a New Holland portable on-farm grinder mixer (Model 358) by-passing the grinding hammers and the appropriate ACA was added. This mixer was equipped with a single vertical screw in the mixing hopper and an electronic scale. Solid ACA's (calcium carbonate, zeolite) were added to DDGS through the hand-add hopper and the hopper was flushed with DDGS. The liquid ACA (DMX-7) was sprayed on DDGS with a garden hand sprayer as it exited the top of the vertical mixing screw. The DDGS and ACA were allowed to mix for 3 min after all the DDGS was added. Treated and control lots of DDGS were augered into one of eight individual compartments in an auger-equipped feed truck. Weight of each lot at loading was recorded. Anti-caking agents were applied to one moisture level of DDGS before switching to the other moisture level. Order of selecting moisture level and application of ACA's was random. Environmental temperature and relative humidity outside the warehouse was recorded every 10 min during the period that the truck was being loaded (about 4 to 6 h). Temperature of each lot of DDGS was recorded immediately after being placed in the truck.

Once the truck was loaded on Friday afternoon, it traveled 250 km and sat motionless for about 60 hours over the weekend. On Monday morning, the truck traveled 250 km back to the ethanol plant where it was unloaded back into the warehouse. Speed of the unload augers was held constant for each compartment and across unloading days by operating the truck engine at a constant rpm. Time required to unload each compartment was recorded and flow rate (kg/minute) for DDGS in each compartment was calculated. The operator assigned a subjective flowability score (scale: 1 = free flowing; 10 = completely bridged to each compartment based on the number of interventions (pokes, prods, blows to side of compartment) required to unload the compartment. The same truck and operator were used on four different days (4 loads total) which provided 32 truck compartments (4 loads  $\times$  8 compartments/load). Before the start of the experiment, each of the eight truck compartments used for the experiment were loaded with 2,275 kg of DDGS containing the same moisture content and no ACA's. The truck was immediately moved from the load-out area to the warehouse and unloaded as described above. This provided a baseline unload rate of DDGS from each compartment under ideal conditions. This baseline rate was used to correct flow rate of experimental DDGS from each compartment to adjust for inherent differences in the truck which were unrelated to treatments.

At the time of loading, a sample of DDGS was collected. Six samples (about 1.6 kg) of each lot were collected from throughout the lot as the DDGS fell into the feed truck compartment. Weight recorded for each lot loaded into the truck was adjusted to account for the amount of sample collected. Samples were thoroughly mixed by hand. Both drained and poured angle of repose measurements (McGlinchey, 2005) were recorded on each composite sample at the time of loading in a modified Hele-Shaw cell. To measure drained angle of repose, about 8 kg of DDGS was placed in the top compartment of the modified Hele-Shaw



Figure 1. Modified Hele-Shaw cell used to measure drained angle of repose (indicated by angle labeled "A") and poured angle of repose (indicated by angle labeled "B"). See text for details of measurements.

cell (fig. 1) as it sat on a level surface. The trap door covering the opening in the floor of the top compartment was removed and the DDGS was allowed to drain into the bottom compartment. The angles labeled "A" were measured, averaged, and recorded as the drained angle of repose. To measure the poured angle of repose, the angles labeled "B" were measured and a mean value calculated.

A subsample of each composite sample was placed in a sealed plastic bag and frozen at -20°C for subsequent analysis. Each sample was analyzed for moisture (AOAC Method 935.29), nutrient content [protein (AOAC Method 990.03), calcium (AOAC Method 985.01), phosphorus (AOAC Method 985.01), crude fiber (AOAC Method Ba6a-05), crude fat (AOAC Method 945.16), ash (AOAC Method 942.05)], particle size (ASAE Standard S319), bulk density, residual sugars (AOAC Method 982.14), color (Hunter Labs methodology), and angle of repose. Drained angle of repose as described above was measured on DDGS samples after storage of DDGS samples for a minimum of 4 weeks. In addition, poured angle of repose was measured in a Hele-Shaw cell and by the Carr method with the following modifications on all DDGS samples after storage. Poured angle of repose determined by the Carr method used a plastic funnel and no vibration. These characteristics of each DDGS sample were related to the measure of flowability recorded as flow rate at truck unloading.

The PROC MIXED procedure of SAS (SAS Institute Inc., 2002, Cary, N.C.) was used to determine the effects of replicate day, and the effects of moisture level and ACA's on flowability. The statistical model to determine the effects of replicate day included day as a fixed effect. Since only one compartment was available in the truck for each combination of moisture level and ACA treatments, replicate day and treatments were confounded. The statistical model to determine the effects of treatments included: moisture level, ACA, and the moisture level by ACA interaction as fixed effects with replicate day as a random effect. Where necessary, treatment means were separated by Tukey's test protected to control the Type I error rate of multiple comparisons. Statistically significant differences were assumed when P < 0.05.

#### **DDGS CHARACTERISTICS AND FLOWABILITY**

To satisfy our secondary objective, we conducted two independent analyses to identify physical and/or chemical characteristics of DDGS that might predict flowability. The first analysis included a stepwise linear regression procedure (SAS Institute Inc., 2002, Cary, N.C.). In this analysis, flow rate of DDGS (kg/min) from the feed truck was the dependent variable and temperature and moisture content of DDGS at loading and unloading; particle size; acid detergent fiber concentration; neutral detergent fiber concentration; ash content; bulk density; Hunter L\*, a\*, and b\* color scores; ambient temperature and humidity at the experiment site; flowability agent; and concentration of residual sugars were offered as independent variables. To enter the regression model, independent variables needed to be significant at P < 0.15. Independent variables that were significant at P < 0.10after additional variables entered the model remained in the final model.

The second analysis relied on a classification and regression tree (CART) procedure (Systat 12, Systat Software Inc., 2007, Chicago, Ill.) to identify DDGS characteristics that might predict flowability. Flow rate of DDGS from the feed truck was the dependent variable. The same independent variables offered to stepwise regression were also used as independent variables in the CART analysis. The flow rate of DDGS from the feed truck was predicted using a least squares fitting method with a minimum split index value of 0.05 and a minimum improvement in proportional reduction of error equal to 0.05.

## **RESULTS AND DISCUSSION**

MOISTURE LEVEL AND ANTI-CAKING AGENTS

As described above, this experiment was conducted on four separate days beginning 1 September 2006. The same truck was used on each day to control variation in unloading rate that would likely occur among different trucks. The necessity to standardize the truck used limited our ability to replicate treatment combinations (2 moisture levels by 4 ACA treatments yields 8 treatment combinations) within each experimental day since the feed truck contained only eight compartments. Consequently, experimental treatments and day are confounded which limits our ability to determine any interactive effects of ambient environmental conditions and flowability treatments.

Environmental conditions and DDGS production conditions for each replicate day are presented in table 1. Logistical considerations with the feed truck owner and the ethanol plant dictated that replicate days be spaced at least 14 days apart. Obviously, there were differences in environmental temperature and humidity recorded just outside the warehouse where the experiment was conducted. However, addition rate of condensed distillers solubles (CDS) to the DDG before drying and dryer temperatures were relatively consistent across replicate days. This experiment was conducted during normal operations of the ethanol plant with only one intervention by the research team (different moisture levels). Consequently, addition rate of CDS and dryer temperatures were the conditions prevailing during the manufacture of DDGS used for this study and are offered for descriptive purposes.

Table 1.	Production	conditions on	replicate da	ivs of the ex	periment (Mean	± Standard	error of the mean)
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	Day						
Item	9/1/06	9/15/06	9/29/06	10/27/06			
Outdoor temperature (°C) <sup>[a]</sup>	$23.8 \pm 1.68$	$27.8 \pm 0.92$	19.9 ± 2.85	12.9 ± 3.13			
Outdoor humidity (%) <sup>[a]</sup>	$67.1 \pm 8.84$	$34.2 \pm 4.10$	$42.1 \pm 10.84$	$42.5 \pm 15.0$			
Condensed distillers solubles addition to DDG (l/min) <sup>[b]</sup>	189	174	193	193			
Dryer temperature ranges (°C) <sup>[b]</sup> :							
Entry	427 - 455	420 - 463	454 - 460	316 - 464			
Drop box	94 - 97	93 - 97	100 - 104	103 - 107			

<sup>[a]</sup> Mean of readings recorded every 10 min while the truck was being loaded (about 4 to 6 h).

<sup>[b]</sup> One observation per day collected from process control software at the ethanol plant.

The differences in environmental temperature apparently influenced temperature of the DDGS at loading (table 2). Moisture content (w.b.) of the DDGS averaged between the 9% and 12% treatments was not significantly different across replicate days at loading. There was no significant drying of the DDGS while it sat in the feed truck during the 60 h between loading and unloading because the moisture content at loading and unloading was very similar. There were no statistically significant differences in flow rate of DDGS across replicate days.

We observed no significant interactions between moisture level of the DDGS and anti-caking agents for any of the response criteria measured in this experiment. This suggests that the response to anti-caking agents was similar regardless of the moisture content of the DDGS. Consequently, we will present only main effect means and no interaction means. Temperature of DDGS at loading was slightly higher for 9% DDGS compared with 12% DDGS; however, at unloading temperature was not different (table 3). As designed, the 9% DDGS contained significantly less moisture than the 12% DDGS. The production staff at the ethanol plant effectively controlled the moisture content of the DDGS and provided product that allowed true evaluation of the treatments

imposed in this study. Particle size of DDGS was smaller (P < 0.01) for the 12% DDGS compared with the 9% DDGS. This difference was unexpected and may have been a chance occurrence. One may speculate that quality and grind of the incoming corn influenced particle size of the resulting DDGS. However, Rausch et al. (2005) reported little correlation between particle size categories in ground corn and the resulting DDGS. Particle size of the DDGS in the present study was lower than that reported by Rausch et al. (2005) and Bhadra et al. (2008) but similar to that reported by Liu (2008). Unfortunately, these authors did not directly relate particle size to flowability of DDGS. Ganesan et al. (2005) stated that small changes in particle size will result in a significant difference in flowability of bulk materials. In a preliminary analysis of our data, particle size did not explain a meaningful proportion of the variation in flow rate when it was used as a covariate in the statistical analysis. This suggests that factors other than particle size, probably moisture level, were responsible for the reduced flow rate of the 12% DDGS. Flow rate and flowability score were clearly poorer (P < 0.01) for 12% DDGS compared with 9% DDGS. These results are supported by the recent work of Bhadra et (2008) in which flowability index of DDGS al.

Day							
Item	9/1/06	9/15/06	9/29/06	10/27/06	PSE <sup>[a]</sup>	P <	
No. of samples	8	8	8	8			
DDGS temperature (°C) at <sup>[b]</sup>							
Loading	$32.5 \pm 0.81^{a}$	$32.0 \pm 0.72^{a}$	$27.4 \pm 0.52^{b}$	$23.7 \pm 0.30^{\circ}$	0.62	0.001	
Unloading	$23.9 \pm 0.54^{x}$	$26.4 \pm 0.56^{\text{y}}$	$24.3 \pm 0.28^{x}$	$18.1 \pm 0.40^{z}$	0.46	0.001	
DDGS moisture (% w.b.) at[c]							
Loading	$10.4 \pm 0.58$	$9.8 \pm 0.63$	$10.8 \pm 0.37$	$10.4 \pm 0.42$	0.51	0.60	
Unloading	$10.4 \pm 0.61$	$9.6 \pm 0.67$	$10.8 \pm 0.34$	$10.4 \pm 0.39$	0.52	0.43	
Particle size (µ)	$632 \pm 20.0$	$584 \pm 14.4^{d}$	$636 \pm 23.5$	$668 \pm 23.9^{e}$	20.8	0.06	
Flow rate (kg/min) <sup>[d]</sup>	$481 \pm 34.4$	577 ± 73.2	$404 \pm 28.2$	$558 \pm 61.0$	52.6	0.10	
Flowability score <sup>[e]</sup>		$6.25 \pm 0.99$	$6.50 \pm 0.68$	$3.75 \pm 0.75$	0.82	0.06	

Table 2. Characteristics and flow rate of DDGS used on replicate days of the experiment (Mean ± Standard error of the mean).

[a] Pooled standard error =  $\sqrt{(\text{Mean squared error/8})}$ 

[b] Loading = Recorded in lots of DDGS immediately after feed truck compartments were loaded; Unloading = Recorded in lots of DDGS immediately after feed truck was unloaded 60 h after treatments were imposed.

[c] Loading = Determined in DDGS samples collected while lots were being loaded in feed truck; Unloading = Determined in DDGS samples collected immediately after feed truck was unloaded 60 h after treatments were imposed.

<sup>[d]</sup> Rate of DDGS unloading from transport truck.

[e] Subjective score assigned by truck operator. Scale: 1 = Free flowing, 10 = Badly bridged.

<sup>abc</sup> Means with different superscripts within line differ (P < 0.001) among the replicate days.

xyz Means with different superscripts within line differ (P < 0.02) among the replicate days.

de Means with different superscripts within line differ (P < 0.05) among replicate days.

Table 3. Effect of moisture level on flowabili	y of DDGS (Mean ± S	Standard error of the mean).
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	Targeted Moist		P <			
Item	9%	12%	PSE <sup>[a]</sup>	Moisture	Moisture × Anti-caking Agent	
No. of samples	16	16				
DDGS temperature (°C) at <sup>[b]</sup>						
Loading	29.6 <u>+</u> 1.24	28.2 <u>+</u> 0.69	0.63	0.05	0.85	
Unloading	23.4 <u>+</u> 0.81	23.0 <u>+</u> 0.89	0.42	0.38	0.55	
DDGS moisture (% w.b.) at <sup>[c]</sup>						
Loading	9.0 <u>+</u> 0.18	11.6 <u>+</u> 0.07	0.15	0.01	0.81	
Unloading	9.0 <u>+</u> 0.22	11.6 <u>+</u> 0.08	0.18	0.01	0.96	
Particle size (µ)	677 <u>+</u> 12.7	583 <u>+</u> 8.1	10.7	0.01	0.25	
Flow rate (kg/min) <sup>[d]</sup>	620 <u>+</u> 36.1	390 <u>+</u> 11.5	26.9	0.01	0.43	
Flowability score <sup>[e]</sup>	3.7 <u>+</u> 0.46	7.3 <u>+</u> 0.43	0.31	0.01	0.94	
Drained angle of repose (degrees)						
Day of loading	57.7 <u>+</u> 0.77	65.7 <u>+</u> 1.04	0.80	0.01	0.11	
After storage	64.6 <u>+</u> 1.17	67.6 <u>+</u> 0.59	0.96	0.01	0.63	
Poured angle of repose after storage (degrees) <sup>[f]</sup>						
Carr method	40.9 <u>+</u> 0.24	42.0 <u>+</u> 0.29	0.28	0.01	0.04	
Modified Hele-Shaw	38.2 <u>+</u> 0.30	37.7 <u>+</u> 0.22	0.35	0.20	0.47	

[a] Pooled standard error =  $\sqrt{(\text{Mean squared error/8})}$ .

[b] Loading = Recorded in lots of DDGS immediately after feed truck compartments were loaded; Unloading = Recorded in lots of DDGS immediately after feed truck was unloaded 60 h after treatments were imposed.

[c] Loading = Determined in DDGS samples collected while lots were being loaded in feed truck; Unloading = Determined in DDGS samples collected immediately after feed truck was unloaded 60 h after treatments were imposed.

<sup>[d]</sup> Rate of DDGS unloading from transport truck.

<sup>[e]</sup> Subjective score assigned by truck operator. Scale: 1 = Free flowing, 10 = Badly bridged. n = 12.

<sup>[f]</sup> Measurements were recorded after samples were stored in a freezer for a minimum of 4 weeks.

was greatest in a DDGS sample with 4.61% moisture (d.b.) while flowability index was lowest for a DDGS sample with 8.08% moisture (d.b.). Reduced flowability of 12% DDGS in the present study was confirmed by a significantly higher drained angle of repose measured on the day of loading and after storage of DDGS samples in a freezer at -20°C for a minimum of one month. Similarly, the Carr poured angle of repose determined after a storage period of at least 4 weeks was greater for 12% DDGS compared with 9% DDGS. Interestingly, the magnitude of difference in angle of repose measurements determined after storage was much less than the difference recorded on the day of loading. Angle of repose measured after storage requires the caked sample to be broken apart before conducting the test. Breaking the caked sample improves flowability, particularly of a poorly flowing sample, and masked differences between samples. This is an important factor to consider when evaluating data on flowability of stored samples. Flowability determinations made after DDGS samples have been stored likely are not reflective of DDGS flowability before storage and are not useful in determining the effectiveness of interventions to improve flowability. Usually, flowability of DDGS before storing the sample is of most interest in commercial situations.

Anti-caking agents had no effect on temperature at loading or moisture content of DDGS at loading or unloading (table 4). The control DDGS tended to be cooler than DMX-7-treated DDGS on the day of unloading; otherwise, temperature of DDGS at unloading was similar. Particle size of DDGS was not different among the ACA treatments tested in this experiment. None of the ACA's significantly altered the flow rate of DDGS compared to the control treatment which used no ACA. The flow rate of DDGS treated with DMX-7 was significantly lower than that of Zeolite-treated DDGS but neither of these were different than using no additive (Control). The drained angle of repose determined on the day of loading was significantly higher (worse) for DMX-7-treated DDGS compared with all other treatments. However, there were no differences among ACA treatments in any of the angle of repose measurements recorded after storage. Angle of repose measurements recorded after storage were of limited value in assessing flowability of DDGS under commercial conditions.

#### **DDGS CHARACTERISTICS AND FLOWABILITY**

We used stepwise linear regression to identify the characteristics of DDGS that were most predictive of flowability as measured by truck unloading rate. In this statistical procedure, we used the observed truck unloading rate (flow rate) of each DDGS sample as the dependent variable and the other measured characteristics of each respective sample as the independent variables in the regression analysis. The stepwise procedure used all 32 observations of flow rate and selects the measured characteristic(s) that is the most effective predictor of the observed flow rate. The utility of predictions can only be assessed with knowledge of the data on which the prediction is based. The mean and range for each independent trait included in the stepwise regression is presented in table 5. Each independent variable was included as a potential predictor because it may have an influence on flowability. Moisture content of DDGS can have an influence on flowability (table 3) so it was included in the analysis. Some researchers (Ganesan et al., 2005) suggested that particle size of bulk solids and powders has important influences on flowability. Bulk density was included as a potential predictor because of its interrelationship with particle size, particle shape, and cohesive forces (Peleg, 1977; Ganesan

Table 4.	Effect o	f selected	l additives on	flowability (	of DDGS	(Mean ±	Standard	error of t	he mean)
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	Additives					P <
Item	Control	DMX-7	Calcium Carbonate	Zeolite	PSE <sup>[a]</sup>	Anti-caking Agent
No. of samples	8	8	8	8		
Inclusion rate		2.5 kg/metric ton	2.0% w.b.	1.25% w.b.		
DDGS temperature (°C) at <sup>[b]</sup>						
Loading	$28.5 \pm 1.40$	$29.0 \pm 1.60$	$29.0 \pm 1.37$	$29.2 \pm 1.56$	0.63	0.90
Unloading	$22.3 \pm 1.04$	$24.0 \pm 1.31$	22.8 ± 1.23	$23.6 \pm 1.30$	0.42	0.06
DDGS moisture (%) at <sup>[c]</sup>						
Loading	$10.3 \pm 0.56$	$10.6 \pm 0.49$	$10.2 \pm 0.50$	$10.3 \pm 0.55$	0.15	0.30
Unloading	$10.3 \pm 0.55$	$10.6 \pm 0.51$	$10.0 \pm 0.55$	$10.4 \pm 0.56$	0.18	0.22
Particle size (µ)	$636 \pm 28.4$	$640 \pm 22.9$	$621 \pm 23.6$	$623 \pm 18.1$	10.7	0.51
Flow rate (kg/min) <sup>[d]</sup>	$509 \pm 60.1^{\mathrm{ab}}$	$441 \pm 40.2^{a}$	$512 \pm 66.2^{ab}$	$558 \pm 56.1^{\mathrm{b}}$	26.9	0.05
Flowability score <sup>[e]</sup>	$6.0 \pm 0.81^{a}$	$6.5 \pm 0.91^{a}$	$5.5 \pm 0.97^{a}$	$4.0\pm0.89^{\rm b}$	0.31	0.01
Drained angle of repose (degrees):						
Day of loading	$61.0 \pm 1.25^{c}$	$65.1 \pm 2.43^{d}$	$60.4 \pm 2.03^{\circ}$	$60.3 \pm 1.62^{c}$	0.80	0.01
After storage	$66.7 \pm 1.54$	$67.2 \pm 1.28$	$64.4 \pm 1.48$	$66.1 \pm 1.38$	0.96	0.24
Poured angle of repose after storage (degrees) <sup>[f]</sup>						
Carr method	$41.0\pm0.54$	$41.7 \pm 0.44$	$41.8 \pm 0.45$	$41.4 \pm 0.25$	0.28	0.26
Modified Hele-Shaw	$37.7 \pm 0.23$	$38.1 \pm 0.34$	$37.6 \pm 0.53$	$38.3 \pm 0.36$	0.35	0.49

[a] Pooled standard error =  $\sqrt{(\text{Mean squared error/8})}$ .

[b] Loading = Recorded in lots of DDGS immediately after feed truck compartments were loaded; Unloading = Recorded in lots of DDGS immediately after feed truck was unloaded 60 h after treatments were imposed.

[c] Loading = Determined in DDGS samples collected while lots were being loaded in feed truck; Unloading = Determined in DDGS samples collected immediately after feed truck was unloaded 60 h after treatments were imposed.

<sup>[d]</sup> Rate of DDGS unloading from transport truck.

[e] Subjective score assigned by truck operator to six samples per treatment. Scale: 1 = Free flowing, 10 = Badly bridged.

[f] Measurements were recorded after samples were stored in a freezer for a minimum of 4 weeks.

<sup>ab</sup> Means with different superscripts within line differ (P < 0.05) among additives.

<sup>cd</sup> Means with different superscripts within line differ (P < 0.01) among additives.

Table 5. Range in characteristics of DDGS samples (n = 32) subjected to stepwise regression and CART analysis.

Trait	Average <sup>[a]</sup>	Minimum	Maximum
	I werager 1	winning	Maximum
DDGS temperature (°C)			
At loading	28.9	22.2	35.9
At unloading	23.2	15.7	28.2
DDGS moisture (%):			
At loading	10.35	7.93	12.16
At unloading	10.31	7.64	12.16
Particle size (µ)	630	522	772
Bulk density (g/cm <sup>3</sup> )	68.4	65.3	70.7
Crude fiber (%)	5.9	4.5	7.1
NDF (%)	28.0	24.8	31.4
ADF (%)	11.1	9.1	13.2
Ash (%)	5.5	4.2	7.1
Residual sugars (%) <sup>[b]</sup>	0.76	0.30	1.30
Hunter L	57.8	55.0	60.5
Hunter a	12.4	11.2	14.0
Hunter b	43.4	40.8	47.4
Ambient conditions at loading:			
Temperature (°C)	20.8	12.9	26.8
Humidity (%)	46.5	34.2	67.1

[a] Average represents 32 observations.

<sup>[b]</sup> Sum of fructose, glucose, maltose, and sucrose.

et al., 2005) of bulk materials. Measures of fiber content (crude fiber, NDF, and ADF) were considered because bulk density of feed ingredients typically decreases as fiber content of the ingredient increases. Ash is a measure of the

mineral content of feed ingredients. Ground limestone (a mineral source) has been investigated as an ACA in DDGS previously (Ganesan et al., 2008a) and in the present experiment so we surmised that ash content of DDGS may influence flowability. Color of DDGS likely would not have a direct effect on flowability of DDGS. However, color can be an indicator of the amount of drying the DDGS was subjected to (Cromwell et al., 1993) and/or the amount of solubles included in the DDGS (Noll et al., 2007) which may influence flowability of the DDGS. In this experiment, moisture content at loading (P < 0.01) and Hunter b\* score (P < 0.05) were the two characteristics that predicted flow rate of DDGS [Flow rate (kg/min.) = 623 - 100\*Moisture content at loading (% w.b.) + 21\*Hunter b\*; r<sup>2</sup> = 0.74; P < 0.001]. Moisture content at loading was the most effective predictor of flow rate as it explained about 70% of the variation in truck unloading rate. For every increase in DDGS moisture at loading of 1%, unloading rate decreased by 100 kg/min. Anecdotal observations from truckers, feedmill managers, and pork producers suggest that flowability of DDGS declines with increased moisture content. Ganesan et al. (2008c) reported a general trend for declining flowability of DDGS with increasing moisture content. However, these investigators studied moisture levels ranging from 10% to 30%, which is far in excess of moisture levels typically found in commercial situations. Rosentrater (2006) found a significant negative correlation between moisture content of DDGS and angle of repose. But, moisture content only explained about 11% of the variation in angle of repose measurements in Rosentrater's experiment.

Hunter b\* score was positively related to flow rate; however, it only accounted for about 4% of the variation in flow rate. As Hunter b\* score increased, flow rate also increased. A positive Hunter b\* score indicates the sample has a yellow color while a negative score suggests a blue color. The Hunter b\* scores of samples in this experiment were all positive indicating that all samples were yellow in color. Noll et al. (2007) reported increased Hunter b\* values for DDGS with decreasing proportion of solubles added before drying. Possibly, the positive relationship between Hunter b\* and flow rate may be related to the amount of solubles included in the DDGS. While our regression analysis suggests that increasing yellowness elicits significant improvements in flow rate, the magnitude of those improvements are of little practical significance. None of the other measured characteristics of the DDGS samples collected in this experiment explained a meaningful proportion of the variation in flow rate.

The objectives of CART analysis are similar to stepwise linear regression in that the goal is to identify which characteristics of DDGS have the most influence on flow rate. However, the approach in this analysis is to consider all of the characteristics simultaneously and select the one characteristic that has the most influence on flow rate. Once this characteristic is selected, the analysis splits the data into two sub-groups to minimize the variation in the observed flow rate within each sub-group. Then these two sub-groups are evaluated, and the next most important characteristic with regard to flow rate is selected, and the sub-groups are again further divided. The tree is "grown" until the variation in flow rate cannot be reduced any further. In this analysis, Hunter L\* values less than 56.5 predicted a flow rate of 714 kg/min while higher L\* values predicted a lower flow rate. This result was puzzling and it was difficult to explain how lightness of color could influence flow rate of DDGS. The lower L\* indicated the DDGS was darker and this was associated with better flow rates. We speculated that the lower L\* value was a result of more extensive drying which caused browning reactions (Cromwell et al., 1993). This is supported by the highly significant positive correlation between Hunter L\* and moisture content of DDGS at loading (0.92) or unloading (0.89). This high correlation suggests that Hunter L\* score does not have a direct "cause and effect" relationship with flow rate of DDGS. Since Hunter L\* seemed to be a proxy variable for moisture content in our initial CART analysis, we eliminated Hunter L\* from consideration for subsequent CART analysis. These subsequent analyses revealed that moisture content at unloading was the characteristic determined to have the most influence on flow rate. If moisture content was less than 10.1%, flow rate averaged 657 kg/min, but flow rates of DDGS with greater than 10.1% moisture were only 400 kg/min. After moisture content, Hunter b\* score was the next characteristic selected. Similar to the regression analysis, a higher Hunter b\* score was indicative of greater flow rate. However, the influence of Hunter b\* score on flow rate was far less than that of moisture content. The CART analysis identified no other measured characteristics that had a meaningful influence on flow rate of DDGS in this study. Considering the stepwise regression and CART analyses together, it appears that moisture content of DDGS has an

overpowering influence on flowability of DDGS under the commercial conditions of this study.

On the surface, it would seem that use of ACA's could be a practical solution to many of the flowability problems with DDGS in the ethanol industry. However, we could not demonstrate positive effects of the ACA's we studied. Our results are consistent with those reported by Ganesan et al. (2008a) who reported the only other scientific evaluation of an additive (CaCO<sub>3</sub>) to improve flowability of DDGS. One cannot rule out the possibility that we selected the wrong ACA's or inappropriate inclusion rates of the additives selected. Given our findings that moisture level of DDGS has a highly significant influence on flowability, future research may need to focus on different additives that help mitigate the cohesive forces of moisture in DDGS. From an academic perspective, we would have liked to study several inclusion rates of each ACA. However, we used inclusion levels in this study that seemed to be at the upper end of the range for practical application within a commercial ethanol plant. A typical ethanol plant in the Midwestern United States produces about  $3.785 \times 10^8$  L of ethanol and about 260,000 metric tons of DDGS annually (Renewable Fuels Association, 2008). Assuming 354 days of operation annually, a typical ethanol plant produces 734 metric tons of DDGS daily. At the inclusion rates used in our experiment, 14.7 metric tons of calcium carbonate, 1,835 kg of the DMX-7 liquid, or 9.1 metric tons of zeolite would need to be handled daily in a typical ethanol plant. These quantities of additives seemed to be the upper limit of practicality. Higher inclusion rates would result in excessive cost and logistical handling challenges that would require plant modifications. Ethanol plants have some degree of control over flowability by controlling moisture content of DDGS. Our study clearly demonstrates that low moisture DDGS flows more easily than higher moisture DDGS. By increasing the amount of drying, ethanol plants can improve flowability of DDGS. However, this increased drying will increase costs to produce dry DDGS due to increased fuel costs and possibly decreased dryer throughput. Furthermore, harsh drying conditions resulting in dark color can decrease nutritional quality of DDGS (Cromwell et al., 1993; Stein et al., 2006) which may compromise the economic value of the DDGS. So, the target moisture content of the DDGS is selected by an ethanol plant after careful consideration of plant throughput, economics, and nutritional quality of the final product. To further complicate this decision, dry DDGS can adsorb moisture under certain environmental conditions (Ganesan et al., 2007).

# **CONCLUSIONS**

The anti-caking agents and concentrations selected for this experiment provided little evidence for improved flowability of DDGS. Clearly, increasing moisture content of DDGS from 9% to over 11.5% significantly decreased flowability of DDGS. There appears to be a linear decrease in flowability as moisture content of DDGS increases. In this study, DDGS with moisture content below 10% displayed the best flowability. More extensive sampling of DDGS with a greater range in moisture concentrations will be necessary to identify the ideal moisture concentration for optimal flowability.

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## REFERENCES

- Agricultural Utilization Research Institute (AURI), and Minnesota Corn Growers Association. 2005. Distiller's Dried Grains Flowability Report. Waseca, Minn.
- Bhadra, R., K. A. Rosentrater, and K. Muthukumarappan. 2008. Understanding and modeling flowability of DDGS. ASABE Paper No. 083808. St. Joseph, Mich.: ASABE.
- Cromwell, G. L., K. L. Herkelman, and T. S. Stahly. 1993. Physical, chemical, and nutritional characteristics of distillers dried grains with solubles for chicks and pigs. J. Anim. Sci. 71(3): 679-686.
- Ganesan, V., K. A. Rosentrater, and K. Muthukumarappan. 2005. Flowability and handling characteristics of bulk solids and powders – A review. ASABE Paper No. 056023. St. Joseph, Mich.: ASABE.
- Ganesan, V., K. A. Rosentrater, and K. Muthukumarappan. 2007. Dynamic water adsorption characteristics of distillers dried grains with solubles (DDGS). *Cereal Chem.* 84(6): 548-555.
- Ganesan, V., K. Muthukumarappan, and K.A. Rosentrater. 2008a. Effect of flow agent addition on the physical properties of DDGS with varying moisture content and soluble levels. *Trans.* ASABE 51(2): 591-601.
- Ganesan, V., K. Muthukumarappan, and K. A. Rosentrater. 2008b. Effect of soluble and moisture content on the physical and chemical properties of DDGS. *Cereal Chem.* 85(4): 464-470.

- Ganesan, V., K. Muthukumarappan, and K. A. Rosentrater. 2008c. Flow properties of DDGS with varying soluble and moisture contents using jenike shear testing. Powder Tech., doi:10.1016/j.powtec.2008.02.003.
- Liu, K. 2008. Particle size distribution of distillers dried grains with solubles (DDGS) and relationships to compositional and color properties. *Bioresource Tech.* 99: 8421-8428.
- McGlinchey, D. 2005. Characterization of bulk solids. Oxford, U.K.: Blackwell Publishing.
- Noll, S., C. Parsons, and B. Walters. 2007. What's new since September 2005 in feeding distillers co-products to poultry. *Proc. MN Nutrition Conf.*, 149-154. St. Paul, Minn.: Univ. of Minnesota.
- Peleg, G. 1977. Flowability of food powders and methods for its evaluation A review. *J. Food Process Eng.* 1: 303-328.
- Rausch, K. D., R. L. Belyea, M. R. Ellersieck, V. Singh, D. B. Johnston, and M. E. Tumbleson. 2005. Particle size distributions of ground corn and DDGS from dry grind processing. *Trans. ASAE* 48(1):273-277.
- Renewable Fuels Association. 2008. Changing the Climate: Ethanol Industry Outlook 2008. Available at http://www.ethanolrfa.org/ objects/pdf/outlook/RFA\_Outlook\_2008.pdf. Accessed 29 September 2008.
- Rosentrater, K. A. 2006. Some physical properties of distillers dried grains with solubles (DDGS). *Applied Eng. Agric.* 22(4): 589-595.
- Spiehs, M. J., M. H. Whitney, and G. C. Shurson. 2002. Nutrient database for distiller's dried grains with solubles produced from new ethanol plants in Minnesota and South Dakota. J. Anim. Sci. 80(10): 2639-2645.
- Stein, H. H., M. L. Gibson, C. Pedersen, and M. G. Boersma. 2006. Amino acid and energy digestibility in ten samples of distillers dried grain with solubles fed to growing pigs. J. Anim. Sci. 84(4): 853-860.
- University of Minnesota. 2008. Available at www.ddgs.umn.edu. Accessed 4 September 2008.
- U. S. Environmental Protection Agency. 2008. Energy Policy Act of 2005. Title XV - Ethanol fuels, Subtitle A, Section 1501. Available at www.epa.gov/oust/fedlaws/publ\_109-058.pdf. Accessed 4 September 2008.