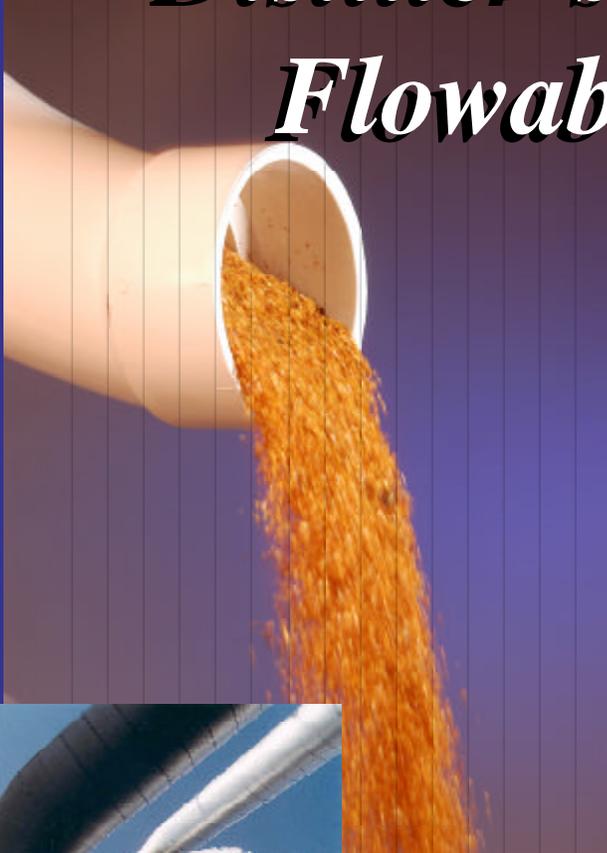


Distiller's Dried Grains Flowability Report

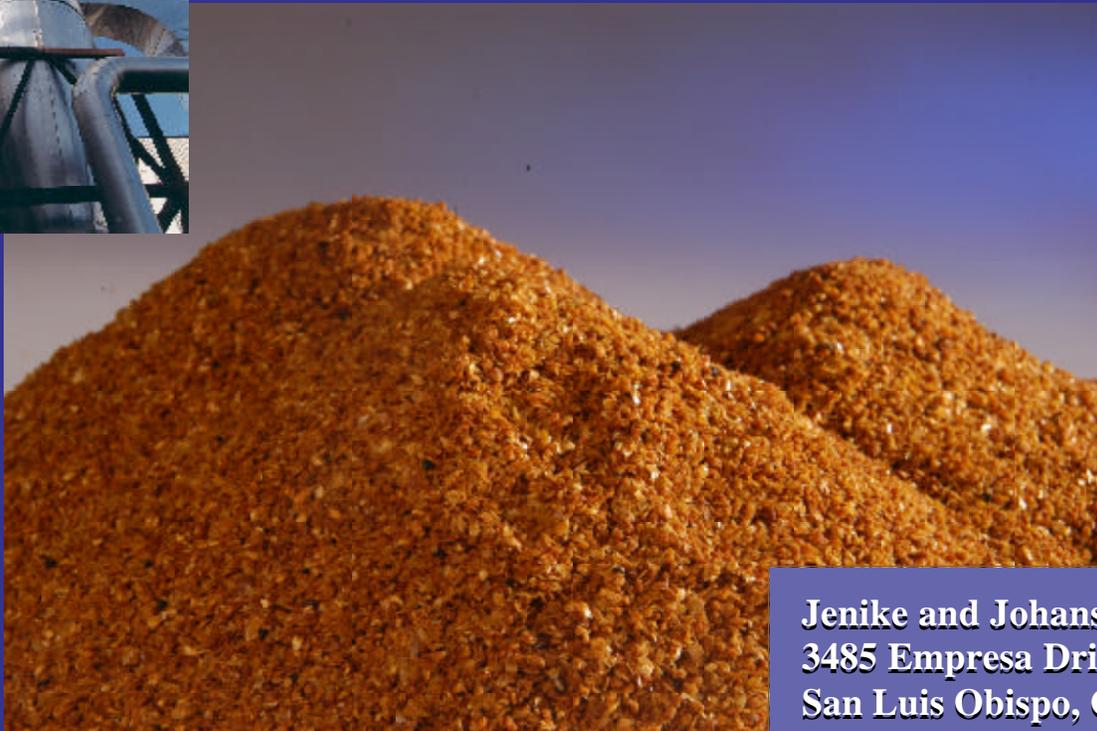


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AURI and Minnesota Corn Growers Distiller's Dried Grain Flowability Study Summary

Methods to Improve Flowability and Pelleting of Distiller's Dried Grains with Solubles (DDGS)

October 17, 2005

Goal of Project:

With the expansion of ethanol plants in Minnesota, the ethanol industry, Minnesota Corn Growers, Agricultural Utilization Research Institute (AURI), state colleges and universities receive multiple inquiries per year concerning the improvement of flowability characteristics and pelleting of distiller's dried grains with solubles (DDGS).

The focus of this study was to evaluate characteristics of corn DDGS that yielded satisfactory flowability. Testing was initiated to determine effects that physical size, oil content and moisture may have on the flowability properties associated with DDGS

There have been numerous articles published related to the problem of DDGS flowability. However, very few focus on solutions or opportunities to improve flowability through formulation, process changes or mechanical designs. Thousands of dollars have been lost in specific incidences where DDGS shipments were declined due to poor product flowability. Improvements to product flowability offers the potential for expanded markets due improved product flow when shipped via truck, rail, or during storage. Evaluating pelleting procedures to produce a quality pellet with improved durability can lead to new or expanded product potentials for marketing distillers dried grains with solubles.

Involved Organizations:

Appreciation is given to the Minnesota Corn Growers and the Agricultural Utilization Research Institute for co-funding the DDGS flowability study.

Information obtained benefits Minnesota ethanol plants involved in the sale and distribution of DDGS. Test results also provide valuable information to producers, manufacturers, and entrepreneurs involved with every-day handling of DDGS in which product flowability and pelleting are critical.

Testing

Testing of DDGS occurred in three areas: (1) analytical, (2) flow characteristics, and (3) pellet durability.

Five samples of DDGS were sourced. These samples included two control samples from two ethanol plants (Control #1 and Control #2) and three modified samples of DDGS. The modified samples included a de-oiled DDGS sample (Mod #3 de-oiled), a reduced syrup DDGS sample (Mod #4 reduced syrup), and a pelleted DDGS sample (Mod #5 pellet).

Analytical

Analytical work took place at the AURI analytical lab in Marshall, Minnesota. Testing focused on moisture, fat and protein content. These components are identified as the largest factors potentially affecting product flowability. Following are the results (Table 1).

Table 1.

LAB #	05-222		
SAMPLE TYPE	1) CONTROL		
<u>ASSAY</u>	<u>METHOD #</u>	<u>UNITS</u>	<u>ASSAY RESULTS</u>
MOISTURE	AOAC 950.46B	g/100g	13.98
PROTEIN	AOAC 981.10	g/100g	25.94
ASH	AOAC 923.03	g/100g	3.40
OIL	AOAC 991.36	g/100g	9.37
LAB #	05-223		
SAMPLE TYPE	2) CONTROL		
<u>ASSAY</u>	<u>METHOD #</u>	<u>UNITS</u>	<u>ASSAY RESULTS</u>
MOISTURE	AOAC 950.46B	g/100g	9.93
PROTEIN	AOAC 981.10	g/100g	26.80
ASH	AOAC 923.03	g/100g	3.38
OIL	AOAC 991.36	g/100g	8.44
LAB #	05-224		
SAMPLE TYPE	3) DE-OILED DDGS		
<u>ASSAY</u>	<u>METHOD #</u>	<u>UNITS</u>	<u>ASSAY RESULTS</u>
MOISTURE	AOAC 950.46B	g/100g	10.14
PROTEIN	AOAC 981.10	g/100g	31.34
ASH	AOAC 923.03	g/100g	6.21
OIL	AOAC 991.36	g/100g	2.30
LAB #	05-225		
SAMPLE TYPE	4) DDGS WITH REDUCED SYRUP		
<u>ASSAY</u>	<u>METHOD #</u>	<u>UNITS</u>	<u>ASSAY RESULTS</u>
MOISTURE	AOAC 950.46B	g/100g	14.12
PROTEIN	AOAC 981.10	g/100g	26.85
ASH	AOAC 923.03	g/100g	2.07
OIL	AOAC 991.36	g/100g	6.26
LAB #	05-226		
SAMPLE TYPE	5) DDGS PELLET		
<u>ASSAY</u>	<u>METHOD #</u>	<u>UNITS</u>	<u>ASSAY RESULTS</u>
MOISTURE	AOAC 950.46B	g/100g	11.67
PROTEIN	AOAC 981.10	g/100g	26.75
ASH	AOAC 923.03	g/100g	3.30
OIL	AOAC 991.36	g/100g	9.85

All five samples were selected for their variability, primarily in moisture and oil content. These samples represented variable assay results for testing flowability and subsequently determining contributing factors. Protein variability was not significant. A variation of moisture from 9.93% to 14.12% was noted. Oil contents of the samples submitted were most impressive, with oil content variability ranging from 2.3 % to 9.85%.

All tests were performed at room temperature (72 degrees F) unless otherwise noted.

Flowability

This evaluation was performed by Jenike & Johanson, Incorporated, a bulk solids science, engineering and design firm based in Westford, Massachusetts. Testing was conducted following defined protocol within funding limits. A summary of the DDGS testing and results follow:

Particle Size

Samples 1 through 4 were evaluated for particle size as an indicator to potentially effect flowability. The pelleted sample was eliminated from this test.

This test first removed any material over 10 mesh or 2mm for proper analysis. The DDGS Control #2 had the most *plus* 2mm product removed at 12%, this was followed by Control #1 having 7.5% removed followed by Mod #3 de-oil and Mod #4 reduced both having 3% removed.

After moving the largest particles, analysis indicated that the **Mod #4 reduced syrup sample had the smallest average particle size** followed by Control #2. Control #1 and Mod #3 de-oiled samples had about the same average particle size.

These are not surprising results since Control #1 DDGS was the source of the Modified #3 de-oiled sample. Control #2 DDGS was the source of the Modified #4 reduced Syrup sample.

Cohesive strength

This is a study of a materials performance as a result of consolidated pressure. This test looked at all five DDGS samples in a simulated continuous flow system and material flow properties after three days at rest.

Based on the results, **no arching or ratholing was present in any of the materials when handled on a continuous flow basis.** These results show that there was no minimum outlet dimensions required when handling DDGS on a continuous flow basis.

If these materials were allowed to rest in a bin for three days, Control #2 and Mod #5 Pellets had a small potential for ratholing. If these five materials were placed in a mass flow bin with a conical hopper and a six inch outlet, no ratholing would be expected after three days at rest. In a mass flow bin, the hopper is sufficiently steep and smooth to cause flowing of all the solids without stagnant regions whenever any solid is discharged. This test was limited to a 3 day rest period.

Compressibility

The second part of this test assessed compressibility and overpressure. Bulk density – pressure relationship is needed for calculating opening sizes, wall angles, and flow rates. This provides greater pressure exposure to materials from such activities as vibration or impact upon loading. Samples were exposed to 100% overpressure based on the materials bulk density. Average bulk densities from least to greatest of the materials are as follows:

DDGS Modified #3 De-Oiled	30.8 pcf
DDGS Modified #4 Reduced Syrup	33.1 pcf
DDGS Control #1	33.2 pcf
DDGS Control #2	34.05 pcf
DDGS Modified #5 Pellet	37.3 pcf

This test concluded that with 100% overpressure and **when** materials were stored at a rest period of three days, **Control #1 and Modified #3 De-oiled samples were not pressure sensitive and flowed freely.** The Control #2 sample was third followed by the Modified #5 Pellet sample. Both samples needed additional discharge area to begin product flow. Unassisted gravity discharge was not possible with Modified #4 Reduced Syrup sample.

Clearly, Control #1 and Modified #3 samples outperformed the three other samples. The only similarity between the two samples is the average particle size. When comparing to the other samples, moisture and oil content do not appear to have had an effect on product flow with overpressure. Product density also appeared to have no effect.

Wall friction

Friction between the bulk solid and the sloping walls of a storage hopper is required for determining flow patterns (funnel flow vs. mass flow). This test evaluated all five samples on hoppers lined with two materials; they were 304 #2B Stainless Steel and Mild Carbon Steel. These materials were tested for continuous flow and three day storage at rest.

The following results are averages from both types of steel and looking at the continuous flow and three day at rest study. Refer to the Jenike & Johanson final report for specific angles. Following are the results from best to worst:

1. DDGS Mod #3 De-oiled
2. DDGS Control #1
3. DDGS Mod #5 Pellet
4. DDGS Control #2
5. DDGS Mod #4 reduced syrup

Clearly the easiest flowing material evaluated for wall friction was the DDGS Modified #3 De-oiled. This product allowed for the GREATEST ANGLE FROM VERTICAL especially on a stainless steel sheet. The remaining samples required steep angles (from vertical) to maintain product flow on a sidewall of a bin. Three of the samples would not flow after three days storage at rest on specific wall surfaces. (Refer to the Jenike and Johanson summary report.)

Once again, it can be assumed average particle size has an effect on flow properties, as this is the only physical characteristic that can be attributed to assist flowability. Reduced oil content may be valuable in aiding product flow. It is assumed the oil content would need to be less than 3%.

Permeability

This test is a way to measure the mass flow capabilities of a solid when it contains fine particles and is deaerated. This test also gives an indication to how bulk solids 'settle' in a bin. These properties can often limit discharge rates independent of discharge methods or design.

This test evaluated the two control samples. If a mass flow cone with a 1.0 foot diameter outlet were used with a cylinder containing a 20 foot effective head of fully deaerated material, the discharge rates would be approximately 216 tons per hour for Control 1, and 198 tons per hour for control 2. This slight difference is not surprising after seeing the correlation between product flow and fine particles in a material. **Control 1 had larger particle sizes on average which would cause the material to deaerate less**, thus increasing flow rate due to permeability.

Effect of humidity

A sorption/desorption test was conducted on DDGS Control 1 to evaluate the effect humidity has on product flowability. Product was dried at 90 degrees Fahrenheit and to .04% humidity. The relative humidity (RH) was raised from near zero to 95% RH in increments of 5% RH in a controlled humidity atmosphere at a constant 90 degrees F. Product sorbent ability was measured and the process was replicated in reverse order.

The most noticed difference was at 60% RH; the adsorbed moisture only comprised 10% of the materials mass. **Anything over 60% RH rapidly caused the product to adsorb moisture.** At 95% RH this control sample had 73% of its dry mass as moisture.

This information is beneficial when handling DDGS in humid or wet conditions.

Dilatometry

This test is typically used to determine if there is a *softening point* for materials within a range of temperatures. Softening of a bulk material to specific temperatures would have a direct effect on product flowability. The test was conducted on DDGS Control 1. A sample was place in a controlled 60% RH and exposed to 68 degrees to 176 degrees F. **There was no softening point found for this sample in these temperatures ranges.** Product is unlikely to cake due to those temperatures at 60% RH. It may be possible to

have a caking problem if exposed to those same temperatures with relative humidity over 60% RH due to the products high sorbent ability at high humidity.

Product softening did not appear to be influenced by particle size or oil.

There was a large amount of dilation that was noticed. This would indicate the likelihood of **SIGNIFICANT PACKING** if constrained in a container. This could be one of the factors contributing to flowability problems associated with DDGS.

Pellet durability

Pelleting of DDGS offers many opportunities for densification and marketing opportunities. AURI's Co-Products Lab has been very active in pelleting several DDGS samples utilizing different size pellet dies as well as combinations with other products. AURI scientifically documents the parameters to achieve improved pellet quality.

Pellet information pertaining to performance and production economics is critical to entering new markets. Five samples were pelleted on a California Pellet Mill, "Master" model, 40 HP. Pelleting was conducted to achieve optimum performance of DDGS pellets. The dies used for pelleting along with die compression ratio and use of steam is documented in all samples. After pelleting, samples were allowed to cool for 24 hours. Durability testing was conducted using the Kansas State Pellet Durability test, which is an accepted standard in the feed industry.

Initial pellet tests focused on the effect pellet die selection has on pellet quality. The first test conduct involved pelleting the DDGS Control #1 on a 5/32" diameter hole pellet die with a standard 8:1 compression. This is the compression ratio often used in the feed industry. This was followed by a pellet run using a 5/32" X 11.7:1 compression die. Compression ratio is determined by dividing the hole diameter by the effective thickness of the die (die depth). Following are the results (Table 2).

Table 2.

Material	Density	Steam Used	Die Size	Pellet Durability	Final Density
Control #1	26 lbs/Ft. ³	2% moisture	5/32" X 8:1	63%	29 lbs/Ft. ³
Control #1	26 lbs/Ft. ³	2% moisture	5/32" X 11.7:1	94%	34 lbs/Ft. ³

An obvious increase in pellet durability was achieved by using a greater compression die over dies similar to what is currently used in the feed industry. This also resulted in a greater bulk density. Pelleting on an 8:1 compression die with steam caused an increase of 64 degrees F in product temperature after pelleting compared to a temperature increase of 128 degrees F using the 11.7:1 compression die. Increased temperature is also a contributor to improved pellet durability.

The second set of pelleting tests evaluated how steam affects pellet durability when coupled with the reduction of syrup in the DDGS. It is understood that oil can not be compressed, so an increase in pellet durability was expected. Results follow (Table 3).

Table 3.

Material	Steam Used	Die Size	Pellet Durability
Control #1	1% moisture	5/32" X 11.7:1	81%
Mod #4 Reduced	1% moisture	5/32" X 11.7:1	74%
Mod #4 Reduced	No Steam	5/32" X 11.7:1	66%

A definite decrease in pellet durability was seen when adding 1% moisture through steam as compared to 2% moisture through steam as shown in Table 1. The greatest difference was seen in the product heat generation. Heat generated across the die was only a 66 degree F increase with 1% moisture compared to a 128 degree increase with 2% moisture addition through steam. This same effect was also noted in Table 3. The addition of steam to the Mod #4 sample resulted in an increase in pellet durability. With die selection being the same, steam is a major factor contributing to pellet durability after proper die selection.

The most surprising results were the lack of increased pellet durability using a reduced syrup DDGS as compared to the control. An increase was expected due to lower oil content; however testing did not support this premise.

Summary

Review of flowability characteristics of the DDGS samples indicates some favorable characteristics along with negative product performance by others. Regarding product flowability, it was surprising to see the lack of flowability improvement with pelleting. One constant outcome was the performance of the Modified #3 De-oiled sample. It clearly outperformed the other four samples when focusing on flowability. This was often followed by Control #1 which had substantially more oil, yet similar particle size. There was a difference in product moistures at testing time from AURI's samples vs. Jenike & Johanson. However, relationships between product moisture and flowability results were consistent.

Dilatometry and *sorbent ability* of materials may be the **largest contributing factors to product flow**. Both tests were conducted only on Control #1. Modified DDGS samples were not tested. Pelleting may have its greatest affect on a permeability test for identifying flow rate limits due to deaeration; however, this was not tested.

All ASTM standards, along with bin and outlet designs can be found in Jenike & Johanson **Flow Properties Test Report Dried Distiller's Grain**.