October, 2007

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An ASABE Section Meeting Presentation

Paper Number: RRV-07146

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Written for presentation at the
2007 CSBE/ASABE North Central Intersectional conference
Sponsored by ASABE
North Dakota State University
Fargo, North Dakota
12-13 October 2007

***Abstract*.** Distillers dried grains with solubles (DDGS) are an excellent feed ingredient for ruminant livestock and are used in monogastric rations as well. With the remarkable growth of the US fuel ethanol industry in the past decade, large quantities of distillers grains are now being produced. Flowability of DDGS has become a problem throughout the industry, as it is often restricted by caking and bridging during storage and transportation. As DDGS contains modest levels of corn oil (typically between 3 and 13% db), some studies are being directed at removing the fat from DDGS, to improve the marketability of DDGS by concentrating protein and thus making it more equivalent to other high-protein feeds that are typically used for swine and poultry diets. Additionally, the corn oil in DDGS is a ready source of oil for biodiesel production. This use for DDGS corn oil can increase the revenue of ethanol processing facilities, and help move them toward a greater diversity of biorefining products. Removing oil from DDGS will alter the chemical nature of these coproduct feed materials, and may also affect the physical properties as well. In fact, removal of the fat may improve flowability. The objective of this study was to examine and compare the physical (moisture, compressive modulus, and shear stress) and flow (Carr and Jenike) properties of regular and reduced fat (approximately 2% db) DDGS. The compressive modulus of reduced fat DDGS was higher than unmodified DDGS. On the other hand, the compressibility of reduced fat DDGS was less than regular DDGS. For regular DDGS, the flow function curve shifted towards the shear stress (σc) axis, which indicated slightly worse flowability. Overall, a reduction in the fat content did show some improvement in the flow properties, but many of these differences were not significant. As it appears that fat content is not the main driver for DDGS flowability problems, continued research should be pursued, including an examination of the effects of other chemical constituents, as well as particle morphology.

Keywords. DDGS, Reduced fat DDGS, Carr Indices, Jenike shear test, Physical properties, flowability

Introduction

It has been estimated that approximately 85% of all the energy consumed in the United States is from fossil fuel sources (USDOE, 2007), and it is expected that this will increase over the next several years. As fossil fuels are not renewable and are diminishing, the world is beginning to focus on producing and utilizing renewable energy sources. This is leading to a high demand for ethanol for use as motor fuel, and as a result, there has been a tremendous growth in the US ethanol industry in the past decade.

Corn is the predominant cash crop in the Midwest region of the US, and it is widely used for dry grind ethanol production. Distillers dried grains with solubles (DDGS) consists of the non-fermentable residues (i.e., protein, fiber, fat, and ash) from ethanol manufacture, and is thus the main coproduct resulting from this process. DDGS has been widely used for several years as a valuable source of energy, protein, water soluble vitamins, and minerals for livestock feeds. DDGS typically contains about 86 to 93% (db) dry matter, 26 to 34% (db) crude protein, and 3 to 13% (db) fat (Rosentrater and Muthukumarappan, 2006).

DDGS production is forecasted to be nearly 13 million tons in 2007, and it is expected to continue to grow as new ethanol plants come online (AAFC Bulletin, 2006). It is necessary to increase the use of distillers grains in domestic and international markets in order to maintain the viability of the ethanol industry. DDGS is usually transported by railcars, and the transportation cost can be high if the DDGS has to be transported out of Corn Belt states. During transportation, DDGS is subjected to various environmental conditions and it can become hardened (i.e. caking or bridging), which can lead to damage of the railcars while unloading (e.g., sledgehammers are often used on the cars to induce flow). The storability, and resulting flowability, of DDGS is currently very problematic (AURI and MCGA, 2005).

Even though anecdotal knowledge regarding flowability is common in the industry (e.g., flow problems may be caused by moisture content, fat level, temperature, humidity, etc.), it is often incomplete and proprietary in nature. From studies of other granular materials, it is probable that flowability problems may arise from a number of synergistically-interacting factors, including product moisture, fat content, particle size distribution, storage temperature, relative humidity, time, compaction pressure distributions within the product mass, vibrations during transport, and/or variations in levels of these factors throughout the storage process (Craik and Miller, 1958; Fitzpatrick et al., 2004a, 2004b; Johansson, 1978, Moreyra and Peleg, 1981; Teunou et al., 1999). Only a few formal studies, however, have been directed towards studying the effect of these conditions on the resulting flow properties of DDGS (Ganesan et al., 2005, 2006, and 2007).

On the other hand, studies are beginning to focus on extracting oil out of the DDGS to utilize it for bio-diesel production (GS AgriFuels, 2006). This will change not only the chemical nature of the DDGS, but also may impact the physical properties as well. Thus, it is expected that the flowability of DDGS may be altered (and hopefully improved), which in turn could improve the marketability of DDGS domestically as well as internationally. Also, extracting oil out of DDGS could reduce the energy used for drying DDGS, and the ethanol producers would benefit with another revenue stream.

General physical properties have been quantified for unmodified DDGS (Rosentrater, 2006), and for DDGS that has had the oil removed via solvent extraction (Saunders and Rosentrater, 2007). To date, however, there has been no comparison between these two products to determine if indeed there is a difference in flowability. Hence the objective of this study was to compare the physical and flow properties of regular and reduced fat DDGS.

Materials and Methods

Sample Collection

The unmodified distillers dried grains with solubles (DDGS) and reduced fat DDGS (produced by a proprietary solvent extraction process) were obtained from a commercial dry grind ethanol plant in eastern South Dakota, and were stored in sealed plastic buckets at room temperature (25oC ± 1oC) until needed for experimentation. Using standard AOAC methods, it was determined that the DDGS had a protein content of 27.6% (db), fiber content of 11.1% (db), fat content of 9.3% (db), ash content of 4.2% (db), and other carbohydrates of 47.8% (db). The deoiled DDGS, on the other hand, had a protein content of 33.3% (db), fiber content of 9.7% (db), fat content of 2.1% (db), ash content of 4.8% (db), and other carbohydrates of 50.1% (db).

Physical Properties

Physical properties included moisture content, mechanical compression behavior, and behavior during shear. The moisture content was determined using moisture measurement method S352.2 (ASAE, 2003). Three replicates were measured for each type of DDGS. Following methods similar to Van Pelt (2003) and Stroshine (2001), the mechanical behavior of the samples was measured with an Instron compression tester (Model 5564, Instron Corporation, Canton, MA), using a 1 kN load cell. Ten replicates from both types of DDGS were analyzed. A 101.6 mm diameter by 101.6 mm high cylindrical receiving vessel was filled with sample, and the plunger (98.09 mm diameter) was lowered at a constant rate of 0.1 mm/min. During testing, the force applied to the plunger was measured, as was progressive travel distance; thus stress (MPa) and strain (%) were determined over time via the compression tester's computer control software. During testing of each sample, at least 4000 individual stress-strain data points were collected. Additionally, compressive modulus was determined for each sample. The data were then analyzed using TableCurve 2D v 5.0 (SYSTAT Software Inc., Richmond, CA) software to determine regression parameters that best described the nonlinear behavior.

After compression testing, each of the 10 consolidated samples from each type of DDGS was then subjected to shear resistance testing using a Torvane shear device (Model 26-2261, ELE International, Loveland, CO) following the procedures described by Goossens (2004) and Zimbone et al. (1996), which are commonly used in soil mechanics.

Carr Indices

Carr (1965) described a number of standard procedures that permit the evaluation of flow characteristics, or indices, of granular materials. A Hosokawa powder characteristics tester (Model PTR, Hosokawa Micron Powder Systems, Summit, NJ) was used to measure the Carr Indices of the DDGS samples following these procedures as well as method D6393 (ASTM, 1999). Carr Indices included angle of repose (AoR), compressibility, angle of spatula (AoS), uniformity, flowability index, angle of fall (AoF), angle of difference (AoD), dispersibility, and floodability index. Five replicates were measured for each type of DDGS, for each parameter measured. Detailed test procedures and further explanations of these indices can be found in Ganesan et al. (2006). A brief description of each follows.

Angle of Repose (AoR)

AoR is defined as the angle between the horizontal and the slope of a heap of granular material dropped from a given elevation (6.8 cm – according to the standard). Angle of repose corresponds qualitatively to the flow properties of that material, and is a direct indication of the potential flowability (Carr, 1965).

Compressibility

The compressibility of a material can be computed by the following equation:

$$C = 100\ (P-A) / P \tag{1}$$

where C is the compressibility (%), P is packed bulk density (kg/cm^3), and A is aerated bulk density (kg/cm^3), both of which are measured as part of the Carr Indices. The greater the compressibility of a bulk solid, the less flowable it will be. Compressibility can therefore be used to indirectly assess properties such as uniformity in size and shape, deformability, surface area, cohesion and moisture content of the material (Carr, 1965).

Angle of Spatula (AoS)

AoS is a property which provides a relative angle of internal friction, or angle of rupture, for a granular material. A highly flowable material will have an obtuse angle of spatula. It is an indirect measurement of cohesion, surface area, size, shape, uniformity, fluidity, deformability, and porosity of the material (Carr, 1965).

Uniformity

The coefficient of uniformity is used with granular materials on which an effective surface cohesion cannot be measured. It is the ratio obtained between the width of a sieve opening that 60% of the sample will pass through and the width of a sieve opening that only 10% of the sample will pass through. The more uniform a mass of particles is in both shape and size, the more flowable it is likely to be. The uniformity coefficient is thus an indirect measurement of size, shape, and compressibility of the material (Carr, 1965).

Flowability Index

A composite evaluation of the flow characteristics of a granular material involves the use of the above four properties. After measuring the individual values above, each is assigned an index value according to Carr’s procedure. The sum of the individual indices (AoR, Compressibility, AoS, and Uniformity) constitutes the “flowability index” of the granular material. A maximum flowability index value of 100 indicates that material flowability is very good, while a low index indicates the material flowability is very bad. The flowability index also partly indicates the potential floodability of a bulk material. The higher the flowability index, the higher the risk that the material may flood (Carr, 1965).

Angle of Fall (AoF)

AoF is a new angle of repose formed when impaction or impulse energy is given to the sample. The more floodable the material is, the lower its original angle of repose and thus its angle of fall. The angle of fall is an indirect measure of fluidity, shape, size, uniformity, and cohesion (Carr, 1965).

Angle of Difference (AoD)

AoD is the difference between the angle of repose and the angle of fall. The larger the angle of difference, the greater the material's potential for flooding and fluidizing. It is an indirect measure of fluidity, surface area and cohesion (Carr, 1965).

Dispersibility

Dispersibility indicates the dusting and flushing characteristics of a material. Dispersibility and floodability are interrelated. The higher the dispersibility index of a material, the dustier and the more floodable it can be (Carr, 1965).

Floodability Index

The evaluation of the potential floodability of a material involves the use of the previously-determined flowability index, as well as the measured values of AoF, AoD, and dispersibility. The sum of these four parameters provides the bulk material’s “floodability index”. A maximum index value of 100 indicates that the chance of floodability is very high; whereas a low value indicates that the material should not flush.

Jenike Properties

Jenike (1964) was the first to apply soil mechanics techniques to measure the flow properties of granular materials. He developed a shear cell suitable for industrial powders. A Jenike shear tester (Model ST-5, Jenike and Johanson Co., Westford, MA) was used to measure the Jenike flow properties. The properties included unconfined yield strength, major consolidation stress, angle of internal friction, effective angle of internal friction, flow function, and compressibility. Five replicates were measured for each parameter for each type of DDGS. Detailed test procedures and property explanations can be found in Ganesan et al. (2007). A brief summary of each follows.

Unconfined Yield Strength, UYS, (σ_c)

UYS (σ_c) quantifies the compressive strength of the bulk solid (Pa) (Schulze, 2006). For example, when the stresses acting on an exposed surface are zero, the surface is the principal plane, and the major pressure within the solid is tangential to this surface. When this pressure causes yield, it is referred to as unconfined yield pressure. Unconfined yield strength is a very important factor that affects arching of bulk solids in silos, especially during unloading from hoppers (Jenike, 1964).

Major Consolidation Stress, MCS (σ_1)

A Mohr circle is the circle which passes through the steady state point (V, S) and is tangent to the Yield Locus (Jenike, 1964), where V is the normal load and S is the shear force applied during the shear test. The point of intersection of the Mohr circle with the normal stress axis determines the value of the MCS (Pa).

Angle of Internal Friction (ϕ)

The angle of internal friction (□, deg) is a measure of the inter-particle friction as a bulk solid starts to slide on itself at the onset of flow (Jenike, 1964). An increase in pressure generally increases the value of ϕ, but not always.

Effective Angle of Internal Friction (δ)

The effective angle of internal friction (□, deg) is the angle developed between the Effective Yield Locus and the applied normal load (V) (Jenike, 1964). The normal load is the load applied to the material vertically by adding the weights in the weight hanger. The effective angle of internal friction is thus the measure of interparticle kinematic friction which exists during steady flow. It generally varies between 30^{o} and 70^{o} for various bulk solids. Fine and dry solids tend to have low values of δ, while coarse and wet solids tend to have large values of δ. Effective angle of internal friction generally decreases with increasing pressure, particularly at low pressures (Jenike 1964).

Flow Function (F)

The relationship between UYS and MCS is called the flow function (F) of the material, and is calculated according to Eqn 2:

$$F = \frac{\sigma_1}{\sigma_c} \tag{1}$$

Flow function is a thus a dimensionless quantity. Jenike (1964) classified the flowability of solids according to flow function value, as depicted in Table 2.

Compressibility Testing

The bulk density (γ) of DDGS at various loads is determined. Then the compressibility (β) is determined from a semi-log plot of the normal load vs. the resulting bulk density, for a range of applied loads. The slope of the line is the compressibility. This is not, however, the same compressibility as that measured with the Carr Indices.

All the properties discussed are influenced not only by the physical nature of the particles themselves, but also by temperature, time of storage at rest, moisture, and chemical compositions. Although these effects were not investigated in this study, they would be fertile ground for follow-up studies.

Statistical Analysis

Statistical analyses on the collected data were performed using Proc GLM with a Type I error rate (α) of 0.05 to test for significant differences between the treatments with SAS software (SAS Institute, Cary, NC, USA).

Results and Discussion

Physical and flow property results obtained for both types of DDGS are provided in Table 1.

Physical Properties

An examination of the physical property results reveals that there was no significant difference in moisture content; for each type of DDGS moisture was close to 10% (db).

Shear strength/resistance of granular materials arises from frictional resistance when particles slide over each other as well as force absorption that occurs during solid-to-solid particle contact. The shear resistance results were not significantly different between the two types of DDGS; each had an average shear strength of 0.03 kg/cm^2. Thus it appears that the presence or absence of fat in the DDGS does not influence the propensity for formation of surface crusts during consolidation, and that flowability problems are more than just interface phenomena.

On the other hand, there were some differences in the uniaxial compression results. The compressive, or "apparent" Young's Modulus, was determined as the ratio of measured stress to strain. It thus defines the relationship between these two parameters. Since there was no actual mechanical yield point observed (as is common for biological products), the modulus was determined at the maximum stress point on each sample's curve. The compressive modulus of reduced fat DDGS was higher (by 28.2%) than the regular DDGS. In other words, the regular DDGS was more easily deformed. This shows that the reduced fat DDGS could take less stress for a given amount of deformation (i.e., had a stiffer matrix) compared to the regular DDGS. This may be due to the less fat content in deoiled DDGS, as fat plays a key role in particle binding which may lead to an increase in the compressibility of regular DDGS.

As the compression data appeared to show differences in response, each type of DDGS were then examined via non-linear regression modeling, in order to quantify compression behavior. Out of several thousand models that were examined with the software, the best-fit regression model was determined as:

$$y = a + b \cdot x^2 \tag{2}$$

The model intercept (a) values for both DDGS types were significantly different from each other (Table 1 and Figure 1). Also, the coefficient (b) value was significantly higher for reduced fat DDGS than the regular one. This further supports the supposition that the regular DDGS was more easily compressed compared to the low fat DDGS.

Carr Indices

The angle of repose for regular DDGS was higher than that of the reduced fat DDGS. According to Carr classifications, materials with angle of repose less than 40^o should flow easily, but greater than 45^o probably would not flow well. The packed and aerated bulk densities were very similar between the two types of DDGS. But, the compressibility and angle of spatula of regular DDGS were significantly higher than reduced fat DDGS. Materials with compressibility values less than 25% are categorized as "good flowable materials", but greater than 25% are categorized as "less flowable materials" (Carr, 1965). In this study, both types of DDGS had compressibility less than 25%. This suggests that both types of DDGS should not have any flow problems. The degree of flowability for materials with an angle of spatula less than 60^o are ranked as "good", but greater than 60^o are ranked as "bad". The angle of spatula values ranged between 51.34^o to 54.76^o, which fall into the flowability category of "normal" to "not good". On the other hand, the uniformity, dispersibility, and flow index values of reduced fat DDGS were significantly higher than regular DDGS. The uniformity of both DDGS was less than 6, which falls in the flowability category of “very good”. According to Carr (1965), materials with dispersibility less than 20% have been categorized as “less floodable”, but greater than 20% as “highly floodable”. Our results show that the regular DDGS falls in the category of “less floodable” and reduced fat into “highly floodable”. This suggests that the reduced fat DDGS should flow and flush slightly more than regular DDGS. These results also indicate that both types of DDGS should not have any problems in flowability. All of these results have shown that reduced fat DDGS should flow slightly better than regular DDGS. We did not observe any difference in the values of angle of fall, angle of difference, or flood index between both types of DDGS either. The flow indices of regular and reduced fat DDGS were 80.10 and 80.70, which falls into the “fairly good” flowability category. This also suggests that both types of DDGS should not have any flow problems.

Jenike Properties

There were no significant differences observed in Jenike properties between both types of DDGS except for compressibility. Free flowing or non-cohesive materials practically show an unconfined yield strength value of 0, even at higher consolidations (Jenike, 1964). Even though the UYS values of both DDGS were not zero and did not show any statistically significant difference, the UYS of reduced fat DDGS was slightly less than regular DDGS. The maximum stress must prevail over the yield strength (i.e., $\sigma_1 > \sigma_c$) in order for an arch to break during hopper flow, and hence for ease of bulk material flow (Jenike, 1964). The MCS values of both DDGS were not statistically different from each other either, but the MCS value of reduced fat DDGS was slightly higher than regular DDGS. According to Jenike (1964), δ is equal to ϕ, (so $\delta/\phi=1$) for non-cohesive materials such as dry sand. As a result, the higher the δ of a material, the higher its probability to have flow problems, and vice-versa. Results show that the reduced fat DDGS had higher ϕ value and lower δ value than regular DDGS, but we did not observe any significant differences between both types of DDGS.

The flow functions and the flow index values obtained for both types of DDGS samples are presented in Figure 2 and Table 1, respectively. In the flow function figure, the line lying towards the bottom of graph (i.e., reduced fat DDGDS) represents easy flow, while more difficult flow is represented as the flow function moves towards the shear stress axis (i.e., regular DDGS). The flow function curve for regular DDGS appears to be shifted upwards in an anticlockwise direction towards the shear stress axis when compared to the reduced fat DDGS. This indicates that reduced fat DDGS was a relatively free-flowing material (compared to the regular DDGS) with the flow function curve located more towards the normal stress (σ_1) axis. But according to Jenike’s classification (Table 2), both types of DDGS fall into the category of “cohesive” material.

The plot of consolidating pressure vs. bulk density for both type DDGS is shown in Figure 3. The slope of the line gives the compressibility (Table 1) of DDGS. It was found that the compressibility of regular DDGS was higher than the reduced fat DDGS. This indicates that the regular DDGS is possibly more likely to have flow difficulty. This reflects the findings from the Instron and Carr testing as well.

Conclusions

Physical and flow properties of regular and reduced fat DDGS samples were measured using Instron, Torvane, Carr powder tester, and Jenike shear testers. There was no significant difference between both types of DDGS for many of the properties. But, differences were observed for compression behavior, Compressibility, Angle of Repose, Angle of Spatula, and Dispersibility. But, both types were ultimately classified as “cohesive” in nature. Overall, the study showed that the reduced fat DDGS had minimal flow property improvement when compared to regular, unmodified DDGS. Removing oil from DDGS did improve flowability slightly, but not substantially. Over a period of time, both types DDGS may be prone to caking/bridging problems. This suggests that the flow problems associated with DDGS may instead be due to other causes, such as chemical constituents, or even particle surface morphology (e.g., roughness, shape, and size). Future studies should investigate these properties of DDGS, as well as the effect of storage time and consolidation on the resulting flow properties.

Acknowledgements

The authors would like to thank the ethanol plant which contributed samples for the study, and the USDA-ARS for financial support.

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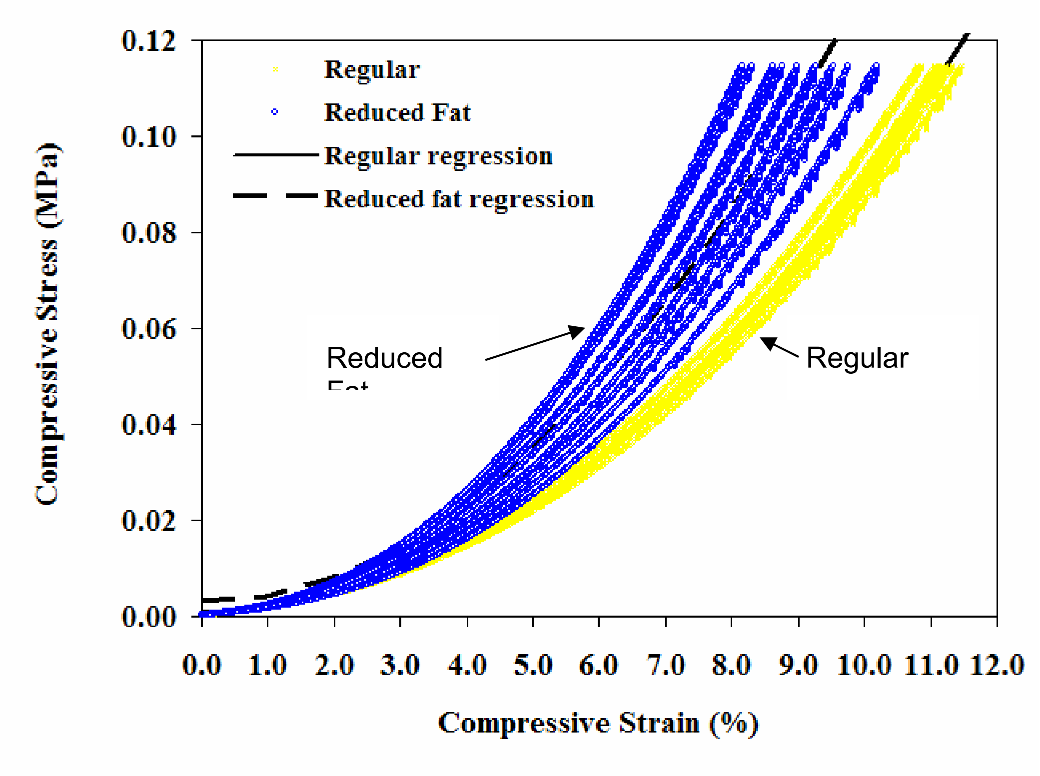

Figure 1. Compressive behavior of regular and reduced fat DDGS (actual data points and resulting regression lines) appear to show differences, both in terms of centrality as well as variability. As strain increases, resulting stress increases nonlinearly.

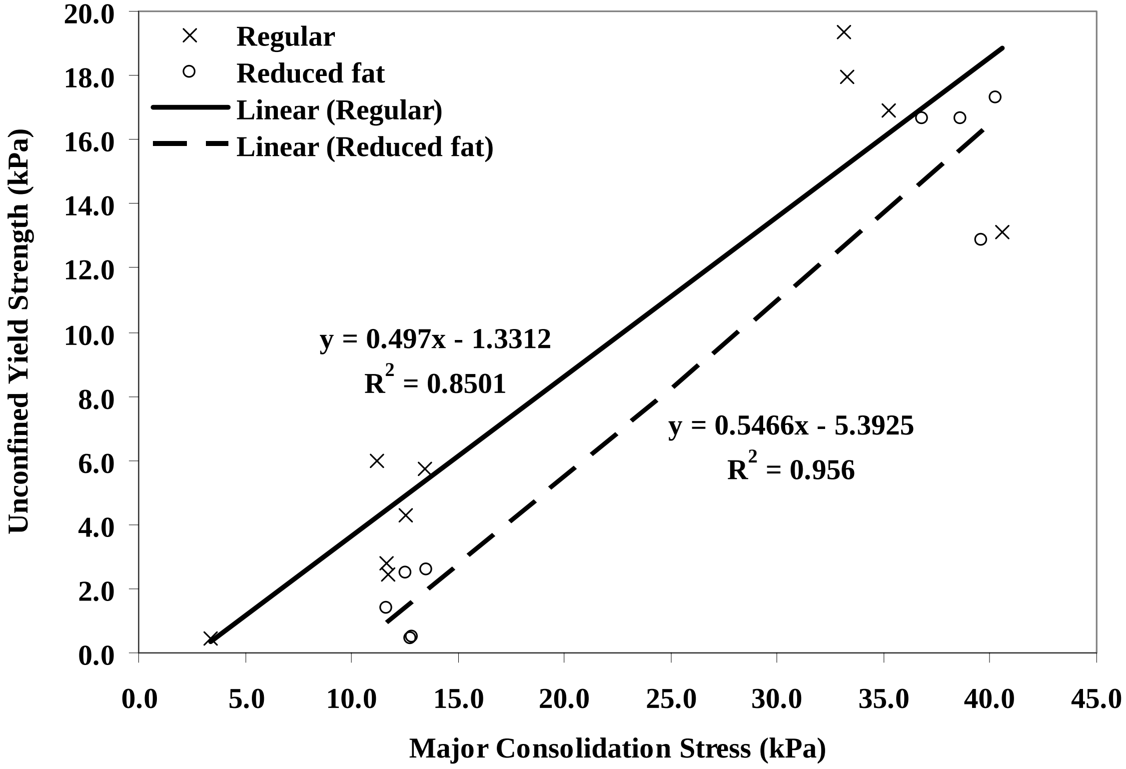

Figure 2. Flow function results for regular and reduced fat DDGS (actual data points and resulting regression lines) appear to show differences. As stress increases, yield strength increases linearly for both types of DDGS.

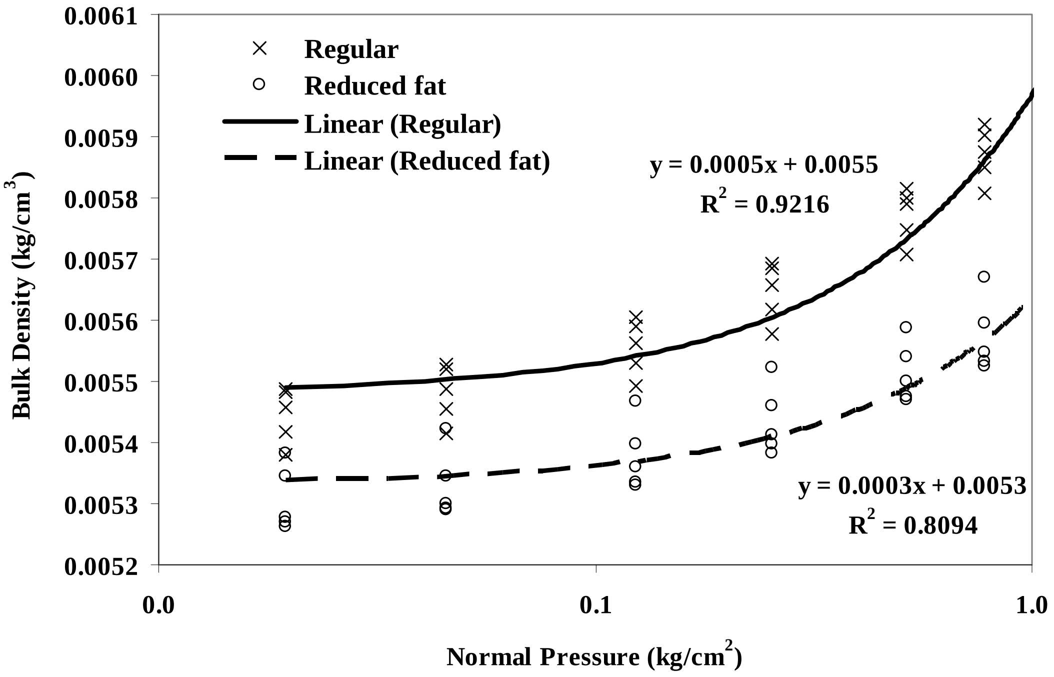

Figure 3. Compressibility of regular and reduced fat DDGS (actual data points and resulting regression lines) appear to show differences. As normal load increases, bulk density increases nonlinearly.

Table 1. Mean and standard error values for physical and flow properties of regular and reduced fat DDGS. +

	Regular			Reduced Fat		
Properties	n	Mean	SE	n	Mean	SE
Physical						
Moisture (% db)	3	10.06a	0.25	3	9.65a	0.05
Shear stress (kg/cm^2)	10	0.03a	0.00	10	0.03a	0.00
Compressive modulus (MPa)	10	1.95a	0.01	10	2.50b	0.06
Compressive regression parameters						
Intercept – a (MPa)	10	0.001a	<0.000	10	0.003b	<0.000
Coefficient – b (MPa)	10	0.0009a	<0.000	10	0.001b	<0.000
Carr						
AoR (deg)	5	42.94a	0.96	5	41.10b	0.91
ABD (g/cm^3)	5	0.56a	0.00	5	0.57b	0.00
PBD (g/cm^3)	5	0.57a	0.01	5	0.57a	0.00
Compressibility (%)	5	2.64a	0.44	5	0.80b	0.24
AoS (deg)	5	54.76a	0.53	5	51.34b	0.64
Uniformity (-)	5	2.0a	0.00	5	4.0b	0.00
AoF (deg)	5	36.68a	0.55	5	37.14a	0.80
AoD (deg)	5	6.26a	0.74	5	3.96a	1.01
Dispersibility (%)	5	15.28a	1.03	5	21.62b	1.90
Flow index (-)	5	80.10a	0.51	5	80.70b	0.44
Flood index (-)	5	58.60a	1.22	5	59.25a	1.43
Jenike						
UYS (kPa)	5	9.102a	2.19	5	7.669a	2.31
MCS (kPa)	5	24.05a	4.04	5	25.67a	4.35
□ (deg)	5	42.2a	1.88	5	46.0a	1.52
□ (deg)	5	52.10a	1.11	5	51.50a	0.43
Compressibility (1/cm)	5	0.0005a	0.00	5	0.0003b	0.00
Flowability index (-)	5	2.19a	0.31	5	2.48b	0.20
Classification	5	Cohesive	--	5	Cohesive	--

+ Means with similar letters for a given property are not significantly different at the α=0.05 level; AoR is Angle of Repose; ABD is Aerated Bulk Density; PBD is Packed Bulk Density; AoS is Angle of Spatula; AoF is Angle of Fall; AoD is Angle of Difference; UYS is Unconsolidated Yield Stress; MCS is Major Consolidating Stress.

Table 2. Classification of powder flowability according to flow function (following Jenike, 1964).

Flow Function (F)	Classification
F<1	No flow
1<F<2	Highly cohesive
2<F<4	Cohesive
4<F<10	Intermittent flow
10<F	Free flow