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Effect of Moisture content and Soluble levels on the Physical and Chemical Properties of DDGS

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Abstract. *Over the past few decades, researches concerning handling and storage characteristics of bulk solids have been conducted to a large extent. Distillers Dried Grains with Solubles (DDGS) are a bulk material that has been widely used as a protein source for ruminants and non-ruminants for more than two decades. Distillers' grains are energy dense as they contain high fat levels. With the exponential growth of the fuel ethanol industry in the past several years, significant quantities of distiller's grains are now being produced. To effectively utilize these feeds in the domestic market, however, these coproduct streams are increasingly being transported greater distances, and must be stored until final use. DDGS flow is often problematic as it can become restricted by caking and bridging which occurs during transportation and storage. This issue probably results from storage moisture, temperature, relative humidity, particle size, time, or temperature variations. The objective of this phase of the study is to examine the effect of various moisture content levels (10, 15, 20, 25, 30% db) on the resulting physical and chemical properties of DDGS with varying soluble percentages (10, 15, 20, 25% db).*

Keywords. Bridging, Caking, DDGS, Granular Powders, Moisture, RH, Storage, Temperature.

Introduction

The production of ethanol from corn grain is a continually evolving process as new discoveries that can make the process more efficient and cost-effective are made and applied. The actual production of ethanol requires only the carbohydrate portion of the grain; the other materials, including protein, fiber, oil, are superfluous to the process. Distillers' dried grains (DDG), distillers' dried solubles (DDS) or a combination product, distillers' dried grains with solubles (DDGS), are the by products produced from ethanol dry grind processing. Presently very little DDS are produced. In most plants, the solubles are added back to the grain or cake. Distillers dried solubles (DDS) is also termed as syrup. DDS is a brown, free flowing to semi-solid liquid and because of its nutritive composition and high palatability, it can be a valuable addition to many livestock rations, especially those requiring high nutrient density or those diets which include ingredients which animals may find less acceptable such as poorer quality roughages. The Distillers' Dried Solubles fraction has the highest concentration of nutrients when compared to DDG and DDGS. It is a rich source of vitamins, and is the lowest in fiber and highest in fat, yielding a digestible energy (DE) value that is approximately 91% of that found in corn. Distillers' dried grain with solubles (DDGS) has been renowned as a valuable source of energy, protein, water soluble vitamins and minerals for animal feeds. Distillers' grains are energy dense as they contain high fat (10-12%) content. With the tremendous growth of the U.S. ethanol industry in the past decade, significant quantities of distiller's grains are now produced. Currently, almost 5.0 million tons of distillers dried grains are created in domestic dry grind ethanol production, and this number is expected to increase to 9 million tons by the year 2006. Moreover, exports of DDGS to Europe, a major outlet for these feed materials, might decrease in coming years. As a consequence, it is important to increase the use of distillers' grains in domestic market. To achieve this, however, they will have to be economically hauled to greater distances, and will have to be stored in bins and silos until use; sometimes this may be a relatively long time. The storability and resulting flowability of DDGS is currently becoming very problematic.

Furthermore, marketing of distillers' grain products is hampered by inconsistencies in physical properties, both within a single plant over time, as well as between plants. Quantification of physical properties is important, because DDGS storage and flow behavior depends on the physical properties of DDGS, as well as external conditions such as temperature, relative humidity, and compression. At times, small differences in moisture content, particle size, storage time, temperature, and even relative humidity can cause caking / bridging of granular materials. This results in a great disparity in flowability. The flowability of DDGS and their flow behavior under various pressures, temperatures and humidities are important in handling operations such as storage in silos or hoppers, transportation and packaging. As there is currently no information available regarding the handling and flow behavior of DDGS, there is a critical need for this data.

Flowability is defined as the ability of granular solids and powders to flow. Flow behavior is multidimensional in nature, and it depends on many physical characteristics. Because of this, no single test can quantify a product's flowability. Flowability, in fact, is not a natural material property at all. It is the consequence of the combination of material physical properties that influence material flow, environmental conditions, and the equipment used for handling, storing, and processing the material (Prescott and

Barnum, 2000). Some of the factors that affect flowability are moisture content, humidity, temperature, pressure, fat, particle size, and addition of flow agents.

Amid all factors affecting product storage, moisture is a key, as it influences microbial growth. Most organic granular materials are hygroscopic in nature, so they gain or lose moisture when they are exposed to various humidity conditions. This leads to physical and chemical changes in the material itself. Moisture sorption is coupled with increased cohesiveness, chiefly because of interparticle liquid bridge formation. Moisture content is a significant variable that affects the cohesive strength and arching ability of bulk materials (Johanson, 1978). As the moisture content of a powder increases, the adhesion (Craik and Miller, 1958) and cohesion (Moreyra and Peleg, 1981) tend to increase. Even a small change in moisture content affects the frictional properties (wall friction angle, internal angle of friction) of material significantly (Marinelli and Carson, 1992). Physical properties are highly dependent on the material's moisture content, and that each material behaves differently. For example, the angle of friction of zinc ore at 18% moisture was found to be 32°. But when the moisture was increased to 23%, the friction angle reached 56°. This showed that the material adhered strongly to the steel bin and would undoubtedly have caused a severe build up problem (Johanson, 1978). Duffy and Puri (1994), studied the flowability parameters of confectionary sugar and detergent at two moisture contents (0.3% and 3.3% for sugar; 1.4% and 4.4% for detergent). As the moisture content increased, the internal angle of friction of both the sugar and detergent decreased. This is totally different from Johanson's (1978) case. Also the unconfined yield strength of sugar increased seven fold as the moisture increased by 3%. It has also been found that with an increase in moisture, bulk density of granular solids decreases and the compressibility increases (Moreyra and Peleg, 1981; Yan and Barbosa, 1997). Moisture at higher levels can possibly alter the surface properties of particles to such an extent that the adherence pattern may also be modified (Hollenbach et al, 1983).

Hence the objective of this study was to examine the effects of various moisture content levels on the physical and chemical characteristics of DDGS with varying soluble percentages.

Materials and Methods:

Determination of Moisture Content:

Distillers' dried grains (DDG) and syrup were obtained from Dakota ethanol plant, located in Wentworth, South Dakota. For determining the moisture content, DDG was first ground finely. A sample size of two grams were taken and placed in an aluminum dish and placed in an oven at 135°C for 2 hrs (AACC 44-19). Triplicates were done for each sample. At the end of heating period, the samples were transferred to desiccators. The dishes were weighed once they reach the room temperature. Moisture content (%db) of the samples was calculated using the following formula:

$$MC (\%db) = \frac{W_{ini} - W_{Fin}}{W_{Fin}} \times 100 \quad (1)$$

where: W_{ini} is the initial weight of the samples before placing in oven; W_{Fin} is the final weight of the samples after drying. The moisture content of syrup was also determined in the same way.

Calibration of Soluble Percentage:

Generally solubles refer to substance suspended or dissolved in syrup. In our study we had taken into account the dissolved matter as solubles and suspended matter as insoluble. Solubles are the portion of syrup that passes through a filter of 2.5 μm or smaller and insolubles are the portion retained on the filter. The procured DDG and syrup had about 10 and 70 % db of solubles. The soluble percentage of syrup differs from batch to batch of productions. To determine the soluble percentage, 0.5 grams of DDG was taken in centrifuge tube with three fourth volume of water. It was then centrifuged in International Centrifuge (International Equipment & Co., Needham Hts., MA) at 1700 rpm for 10 min. After centrifuging, it was filtered in a funnel and conical flask set up using Whatman 42 filter paper which has a pore size of 2.5 μm . Once the filtration was done, both the conical flask and the filter paper were put in an oven at 135°C for 2 hrs (AACC 44-19) to evaporate all the moisture present in the samples. The increase in flask weight represented the total solubles. All these were done in triplicates. The soluble percentage was calculated as follows

$$\text{Soluble \% (db)} = \frac{W_{fl}}{W_{fl} + W_{fp}} \times 100 \quad (2)$$

where: W_{fl} is the weight of the dried matter in the flask (g); W_{fp} is the weight of the dried matter in the filter paper (g). To prepare DDGS with certain soluble percentage, first the proportion of DDG and syrup to be mixed has to be calculated. For determining that, different amounts of syrup from 0.0g to 1.75 g in the interval of 0.25 g were added to 0.5g of DDG. After mixing, the prepared samples were centrifuged, filtered and dried as said before. The soluble percentages were calculated using equation (2). Also the same procedure was followed for determining the soluble percentage in syrup. A graph (Figure 1) was plotted between amounts of syrup vs. soluble percentage. From the plot, an empirical formula (Eqn 3) has been developed to calculate the soluble percentage of DDG, syrup mixture.

$$Y = 15.468 X + 13.046 \quad (3)$$

From equation (3), the amount of DDG and syrup to be mixed to get DDGS with four soluble percentages (10, 15, 20 & 25% db) were calculated. Soluble percentage in syrup varies greatly from batch to batch production. So for every batch the soluble percentage has to be calculated. Then the samples were prepared by blending both DDG and syrup in “Hobart Blender” for 10 min. After blending, the mixture was kept in refrigerator at about 5°C for a day, so that there will be an even distribution of solubles and moisture content through out the mixture.

Preparation of DDGS with various moisture content levels:

After preparing DDGS with different soluble percentages, the moisture content of the mixture was calculated using the formula:

$$\text{Mixture MC (\% wb)} = \frac{(W_D \times M_D) + (W_S \times M_S)}{W_T} \quad (4)$$

where: W_D is weight of DDG (g); W_S is weight of syrup (g); M_D is moisture content of DDG (% wb); M_S is the moisture content of syrup (% wb); W_T is the sum of the weight of

both DDG and syrup (g). From equation (4), the amount of water and dry matter in the mixture was calculated. For preparing the DDGS with certain moisture content, either water has to be added to the mixture or the mixture has to be dried. To determine that, the following formula has been used,

$$W_M = (MC_T \times W_{DM}) + W_{DM} \quad (5)$$

where: W_M is final weight of the mixture (g); MC_T is target moisture content of the mixture (%db); W_{DM} is weight of the dry matter in the mixture (g). If W_M is greater than W_T , then $(W_M - W_T)$ gram of water was added to the mixture. If W_M is not greater than W_T , then the mixture was spread in an aluminum plate and dried in an oven at 100°C until it reaches the target weight (W_M). By this way, DDGS with five moisture content levels (10, 15, 20, 25 & 30%) were prepared. Around 3 kg of samples were prepared for each combination of soluble and moisture content. The prepared samples were stored at 4°C to prevent the moisture loss. Few hours before the experiment, the samples were kept out so that it would reach the room temperature.

Physical Properties

- Color (L^* , a^* , b^*) values were measured using Minolta chromameter. Five replicates were done on each sample.
- Particle size distribution was measured by sieve analysis method (ASAE S319.3) using Tyler sieve analyzer (Model RX-29). Five replicates were done on each sample.

Flow Property Measurement by Carr Indices Test:

The handling of DDGS is difficult without knowledge of how they flow in bins and hoppers. Carr (1965) described a number of standard procedures that permit the evaluation of flow characteristics of any granular materials. Hosokawa powder characteristic Tester (Model PTR, Hosokawa Micron Powder systems, NJ) (Figure 2) was used for measuring the flow properties of DDGS with various soluble percentages and moisture content levels, which follows the procedure described by Carr (ASTM D 6393). The properties measured in powder tester are angle of repose, angle of fall, angle of difference, aerated bulk density, packed bulk density, compressibility, angle of spatula, uniformity and dispersibility.

Chemical Properties:

For chemical analysis, the moisture content of DDGS was not taken into account (i.e.) DDGS with four soluble percentage was ground and bone dried before analysis.

- Fat content of DDGS samples were measured using Gold Fisch extraction method (AACC 30-20)
- Protein content was measured using Flash EA 1112 N/Protein Analyzer, which is based on dynamic flash combustion method (AACC 46-30)

Triplicates were done on each sample.

Statistical Analysis

The flow characteristic test and chemical property analyses were replicated three times, while particle size distribution and color tests were replicated five times. The data were analyzed using PROC GLM of SAS software (SAS, Version 9.1, SAS Inst. Inc.,

Cary, NC, 1991) to find significant differences among the treatments. The statistical model employed for flow characteristic was:

$$Y_{ijk} = T_i + \alpha_{ij} + M_k + R_j + (T * M)_{ik} + \varepsilon_{ijk} \quad (6)$$

where: Y is the flow properties of DDGS, T is the effect of treatment, i.e. four soluble percentage, (i = 1, 2, 3, 4), R is the effect of replication, (j = 1, 2, 3), α_{ij} is interaction effect of treatment and replication (error-1), M is the effect of moisture content (k = 1, 2, 3, 4, 5), (T * M) is the effect of interaction of treatment and moisture content, and ε is the residual variation. The statistical method employed for chemical properties analysis was

$$Y_{ij} = T_i + R_j \quad (7)$$

where: Y is the chemical properties of DDGS, T is the effect of treatment, i.e. four soluble percentage (i = 1, 2, 3, 4), R is the effect of replication, (j = 1, 2, 3).

Results and Discussion

In our study, the physical and chemical properties of DDGS with four different soluble percentage (10, 15, 20, 25% db) and five moisture content levels (10, 15, 20, 25 & 30% db) were studied.

Color:

The color values ($P < 0.001$) were influenced by soluble as well as the interaction between soluble and moisture content (Table 1). The brightness (L^*) of DDGS, decreased with increase in soluble percentage and the redness (a^*) value increased with increase in soluble percentage. This is due to the addition of more syrup to DDG.

Flow properties

Carr Indices test (ASTM D 6393) was used to measure the flow properties of DDGS. Hosokawa powder tester consisted of eight measurement and two calculations to provide ten tests for Carr Indices. Each single test or a combination of several tests can be used to characterize the DDGS flow properties. The statistical analysis of the flow properties of DDGS are shown in Table 2. Soluble percentage, moisture content level and interaction between soluble and moisture content influenced aerated bulk density, packed bulk density, compressibility and uniformity values ($P < 0.001$) of DDGS. Angle of spatula values ($P < 0.01$) was influenced only by the soluble percentage of DDGS and dispersibility values were affected by soluble percentage ($P < 0.05$) and moisture content levels ($P < 0.001$). But there was no significant effect of solubles or moisture content on angle of repose, angle of fall and angle of difference values. Carr (1965) tested over 2800 dry material samples and created a point score (index) system for evaluating the flowability and floodability of dry solids. The point score system for flowability was divided into seven categories, ranging from 'excellent' to 'very very poor', while the floodability point score system was divided into five categories, ranging from very 'floodable' to 'would not flood'. Table 3 shows the summary of flow and flood indices for all combinations of solubles and moisture content levels. DDGS for all combination of solubles and moisture content, was not found be cohesive and are easy flow. There is no significant relationship between the DDGS flow properties and soluble and moisture content levels. This shows the difficulty in trying to compare the flowability of DDGS with different soluble and moisture content based on their physical properties. Increase in moisture content tends to make DDGS more cohesive, however above certain moisture content; the moisture may act as a lubricant and improve the flow.

Chemical properties:

Fat and protein contents of all DDGS (Table 4) were not similar ($P > 0.05$). There was no proper trend noticed for fat content of all DDGS. But it increased from 8.79% to 12.68% when the soluble percentage in DDGS was increased from 10% to 20%. The protein content was found to decrease with increase in the soluble percentage. Because of the lower protein content of the solubles, the amount of crude protein in DDGS declines slightly as more solubles are blended with the grains to produce DDGS. This may be due to the fact that the quality and composition of syrup (DDS) can be affected by a number of factors including the original substrate, the process used, and evaporation procedures. Nutritional properties of this product can vary greatly.

Conclusion:

To date, most of the research work has focused on chemical analysis of distillers' grains, but not on the related physical properties. To improve the domestic use of these coproduct feed materials, there is an urgent need to study their flow behavior as well as storage and handling characteristics. This can help optimize utilization of these materials, and thus improve process economics for ethanol producers, use by livestock feeders, and ultimately the real economy. Increase in soluble percentage and moisture content levels have not shown any flow crisis in DDGS. Soluble percentage had a significant effect on the color (L^* , a^* , b^*) values of DDGS. Color values were not influenced by the moisture content alone but the interaction between moisture and soluble showed a striking impact on it. Soluble percentage and moisture content had significant effect on aerated bulk density, packed bulk density, compressibility, uniformity and dispersibility of DDGS. Angle of spatula was influenced only by soluble percentage. There was no significant effect of soluble percentage and moisture content on angle of repose, angle of fall and angle of fall. Protein content decreased with increase in soluble percentage. Fat content didn't show any inclination towards the increase in soluble levels. On general, it seemed to be there is no flow problem in DDGS with increased soluble and moisture content levels. Consolidation of DDGS might make a big difference in the degree of flowability with various soluble and moisture content levels.

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Figure 1: Calibration curve for calculating soluble %

Table 1: Mean square and probability of color of DDGS with different soluble and moisture content levels

Source	df	L*	a*	b*
Soluble	3	601.26***	97.00***	516.22***
Error - 1	12	0.39	0.69	5.66
MC	4	1.42	0.90	42.51***
Soluble*MC	12	8.10***	2.76***	18.13***
Error - 2	64	0.85	0.74	4.97
R ²		0.97	0.88	0.86

*P < 0.05.

**P < 0.01.

***P < 0.001.

1 **Table 2: Mean square and probability of Flow properties of DDGS with different soluble and moisture content**
2 **levels**

Source	df	Angle of Repose	Aerated Bulk Density	Packed Bulk Density	Compressibility	Angle of Spatula	Uniformity	Angle of fall	Angle of Difference	Dispersibility
Soluble	3	1.08	0.007***	0.006***	7.68***	39.64**	0.42***	0.4	2.75	6.84*
Error - 1	6	2.87	0.00	0.00	0.28	7.99	0.01	1.50	0.74	0.72
MC	4	6.76	0.005***	0.002***	35.80***	13.68	0.52***	2.02	6.69	59.21***
Soluble*MC	12	3.54	0.000***	0.000***	2.23***	5.55	0.41***	2.75	8.58	2.85
Error - 2	32	2.10	0.00	0.00	0.41	7.98	0.01	3.19	5.17	1.83
R ²		0.59	0.99	0.98	0.94	0.53	0.96	0.35	0.48	0.84

3
4 *P < 0.05.

5 **P < 0.01.

6 ***P < 0.001.

Table 3: Summary of Mean values of Carr Indices test

MC (%db)	Soluble %							
	10		15		20		25	
	Flow Index*	Flood Index**	Flow Index*	Flood Index**	Flow Index*	Flood Index**	Flow Index*	Flood Index**
10	79.50	59.25	78.33	56.25	80.00	58.67	80.00	61.00
15	77.67	57.83	75.17	56.42	78.00	57.83	79.33	53.08
20	77.33	55.33	76.33	57.50	78.83	56.92	78.33	52.42
25	75.33	57.00	74	55.92	78.33	53.25	78.83	51.75
30	75.67	54.50	73.67	56.58	77.17	54.92	74.17	52.75

*70 -79 – Good

*80 - 89 – Fairly Good

**40 - 59 – Tends to Flush

Table 4: Average (n=3) fat and protein content of DDGS with four different soluble percentage

Parameter (% db)	Soluble %				SEM	LSD
	10	15	20	25		
Fat	8.79 ^b	7.53 ^b	12.68 ^a	11.77 ^a	1.7	2.6
Protein	30.54 ^a	30.16 ^a	27.23 ^b	26.02 ^c	0.26	1.019

^{a,b,c}Means within same row not sharing common superscript are different (P <0.05).
SEM = Standard error mean; LSD = Least significant difference.