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June, 2009

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An ASABE Meeting Presentation

Paper Number: 095567

Effect of Elusieve Fractionation on Physical Properties of Distillers Dried Grains with Solubles (DDGS)

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**Written for presentation at the
2009 ASABE Annual International Meeting
Sponsored by ASABE
Grand Sierra Resort and Casino
Reno, Nevada
June 21 – June 24, 2009**

Abstract. *The production of corn-based ethanol in the U.S. has been dramatically increasing in recent years, and consequently so has the quantity of coproduct materials generated from this processing sector. These streams are primarily utilized as livestock feed, and can partially offset the need for corn in feed rations. As the industry evolves, new technologies are being developed to improve the value proposition of the coproducts, especially distillers dried grains with solubles (DDGS). One of these is the Elusieve process, which combines sieving and air classification to accomplish fractionation of the DDGS into component-rich (i.e., protein, lipid, fiber) streams. Previous studies have discussed the efficacy of the nutrient concentrations achievable using this process, but no studies to date have examined the physical properties of these resulting streams. Thus the objective of this research was to examine physical properties of DDGS fractions produced by the Elusieve process. Using standard laboratory methods physical properties, including bulk density and angle of repose, were determined. Preliminary results from this study will be presented. The data generated during this study will be useful to both the ethanol and livestock industries, but further study is warranted in order to more fully characterize the differences in other physical properties of these coproduct streams..*

Keywords. Corn, DDGS, distillers grains, fractionation, Elusieve, physical properties.

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Introduction

As the ethanol industry continues to evolve, it is becoming important to explore all potential avenues for increasing profitability. One way to add value is to implement fractionation. Fractionation is the overarching term applied to the separation of substances into two or more component parts (Adapa et al., 2004). Fractionation processes allow for the subsequent utilization of the valuable chemical constituents within the parent material. Products such as protein, fiber, or oil may be utilized in various value-added products, such as industrial materials, pharmaceuticals, or even additional biofuels.

Fractionation of corn kernel components (i.e., prior to fermentation) is one option. “Quick Germ Processing” (Singh and Eckhoff, 1997) can be used to remove the germ portion of the raw corn kernels, and is a combination of dry and wet milling techniques. This involves equipment changes at the front end of a dry grind production process. Depending on the scale of the process, although the capital investment will be higher than for a traditional dry grind plant, it may reduce the overall manufacturing costs for ethanol production, because DDGS produced by the quick germ process will be lower in fat and higher in protein content.

Another opportunity to extract value is fractionation of DDGS components (i.e., post fermentation). For example, Wu and Stringfellow (1986) found that a simple screening method for fractionation of corn distillers dried grains (DDG) and distillers dried grains with solubles (DDGS) could result in finer fractions with substantially higher protein content, as well as lower fiber, compared to the initial materials. Yield and composition of the screened DDG fractions depended on the mesh size, from 20 through 80 mesh, and consisted of 14.5 to 49.3% protein, 9.1 to 9.5% fat, and 1.7 to 2.4% ash content (on a dry basis); screened DDGS, on the other hand, yielded protein contents from 19.8 to 36.8%, fat contents 7.4 to 23.0%, and ash contents of 2.0 to 6.5%.

In another study, Wu and Stringfellow (1982) fractionated corn distillers grains (CDG) and corn distillers grains with solubles (CDGS) with various moisture contents (5 to 31%). The materials were pin milled (9,000, 14,000 and 18,000 rpm) and screened on various sieves (sieve numbers 20 to 80). CDGS at 21% initial moisture was ground twice at 14,000 rpm and separated on a 50 mesh screen to produce a high protein fraction (43% protein content and 41% yield). The parent CDGS had 30% protein content. For CDG, the resultant fractions ranged in protein from 13 to 50%, and the optimal conditions were determined to be 21% initial moisture content and one pass through the mill at 14,000 rpm. The parent CDG, on the other hand, had 25% protein content. Corn protein appeared to attract fat, as the fractions having the highest protein fractions also had the highest fat. This relationship was noted both in the CDG and the CDGS. Fiber appeared to be the most resistant to grinding. Fractions with the largest particle size tended to have the highest fiber content.

Air aspiration can be used to remove fiber from DDG and DDGS. Singh et al. (2002) obtained samples from two types of industries: corn-based dry grind ethanol plants from the Midwest, and beverage alcohol plants using corn (80%), rye (10%), and malted barley (10%) as feedstocks. An aspiration technique, which entailed placing a sample of DDGS on a 20 mesh (840 micron) screen and aspirating with an air jet at a pressure of 2.8 atm, was able to separate the pericarp from the germ fraction, though not very effectively. Phytosterol compounds are usually concentrated in fiber and cell wall tissues, which ultimately amass in DDGS. The total phytosterol content of the original DDGS samples ranged from 292.97 to 342.95 mg/100g (for the corn DDGS) and 236.50 to 246.12 mg/100g (for the beverage DDGS). The percentage of total phytosterols that were able to be recovered in the aspirated corn DDGS samples were 28.2 to 34.9%, while aspirated beverage DDGS had 28.4 to 37.4% recovery. Overall, the aspirated

DDGS samples from both industries contained 25.68 to 28.91% protein, 39.34 to 51.79% NDF, and 13.08 to 17.91% ADF.

Further, Srinivasan et al. (2005) developed a process (“Elusieve” process) using the combination of elutriation (air classification or aspiration) and sieving to separate fiber from DDGS. In the Elusieve process, DDGS is sieved into four size fractions and the three larger size fractions are subjected to air classification (Srinivasan et al., 2009) (Figure 1). The smallest size fraction, “pan” DDGS, representing nearly 35% of the original DDGS had reduced fiber and increased protein content (by 5 to 6% points) compared to the original DDGS. The remaining three size fractions were subjected to air classification to yield heavier and lighter fractions. The lighter fractions had higher fiber (NDF) and lower protein and fat contents than heavier fractions. The heavier fractions from air classification of the three larger size fractions are combined together to produce the “big” DDGS product that has nearly same protein content, but lower NDF content, than the original DDGS. The lighter fractions from air classification of the three larger size fractions are combined together to produce the “Elusieve Fiber” product that has higher NDF and lower protein and fat contents than the original DDGS (Figure 1).

Although the Elusieve process was successful in concentrating nutrient streams from DDGS, little information is currently available regarding the physical characteristics of the resulting streams. Thus the objectives of this research were to examine physical and flowability properties of products resulting from Elusieve fractionation of DDGS.

Materials and Methods

Corn-based DDGS (which had been produced at a fuel ethanol plant in the Midwest) was collected from a feed mill in Prairie, MS. Elusieve processing was carried out at the pilot plant facility at Mississippi State University (Srinivasan et al 2009). DDGS (188.4 kg) was gravity fed from a hopper to a sifter (Model 484, Gump, Savannah, GA) at 0.25 kg/s (1 ton/h) (Figure 1). The sifter had a sieving area of 1.8 m² (19 ft²) per deck and consisted of three decks for stack sieving was used to produce four size fractions (Srinivasan et al 2009). The screens used in this study had opening sizes of 1041 µm, 680 µm and 470 µm. The large, medium and small size fractions were each air classified using commercial multi-aspirators (Model VJ8X6, Kice, Wichita, KS) to result in 24.6, 38.4 and 15.5% lighter fraction yields, respectively. Fiber product, pan DDGS and big DDGS comprised 17.8, 29.5 and 52.7% by weight of original DDGS.

The original DDGS (Figure 2), the pan DDGS ($D < 470 \mu\text{m}$), and the big DDGS ($D > 470 \mu\text{m}$) streams were then stored at room temperature ($24 \pm 1^\circ\text{C}$) in sealed plastic containers. All physical property determinations, except moisture content, were conducted at room temperature. Each property was studied using a completely randomized design.

The streams were subjected to chemical analysis to examine the effects of Elusieve processing on the resulting nutrient contents. Protein, fiber, lipid, and ash contents were determined following official Methods 990.03, 978.10, 920.39, 920.48, respectively (AOAC, 2003). Carbohydrate content was determined by difference. Moisture was determined using a laboratory oven at 105°C for 3 h. All chemical properties were measured in duplicate ($n = 2$) for each sample.

Extensive physical and flowability analyses were also conducted. The geometric mean diameter and geometric standard deviation of the particles were calculated following ASAE/ANSI standard S319.3 (2004) using a Rotap sieve analyzer (Mentor, OH). Color was measured using a spectrophotocolorimeter (LabScan XE, Hunter Associates Laboratory, Reston, VA) using the L-a-b opposable color scales (Hunter Associates Laboratory, 2002). Angle of repose, loose bulk density, packed bulk density, Hausner Ratio, and uniformity were

determined through Carr index testing (Carr, 1965), using a powder characteristics tester (Model PTR, Hosokawa Micron Powder systems, Summit, NJ), following the procedures described by ASTM D6393 (1999). All physical and flowability properties were determined using three replicates ($n = 3$) for each sample.

For each property, formal statistical data analysis was conducted using Microsoft Excel v.2003 (Microsoft Corp., Redmond, WA) and SAS software v.8 (SAS Institute, Cary, NC). A general linear models (GLM) procedure was tested for each property in order to determine if significant differences existed between samples using a Type I (α) error rate of 0.05, and if so, then a Least Significant Difference (LSD) test was conducted using a 95% confidence level to determine where those differences occurred. Additionally, Pearson product-moment linear correlations were quantified among all measured variables.

Results and Discussion

The composition of the original DDGS (Table 1) had protein, lipid, ash, carbohydrate, ADF, and NDF contents of 33.00, 7.95, 4.70, 54.35, 12.40, and 37.80% (dry basis), respectively. Due to the Elusieve fractionation process, these components in the resulting big DDGS were changed by, respectively, 3.48, -8.81, 0.00, -0.83, 6.45, and 8.60%. In the pan DDGS, however, they were altered by -12.88, 11.95, -6.38, 6.62, 7.66, and 22.88%, respectively. The composition of the resulting “fiber” stream is provided in Table 2. As shown, compared to the original DDGS, the “fiber’s” protein and lipid contents were reduced by 23.6 and 17.0%, respectively, whereas the ash and NDF were increased by 4.0 and 11.9%, respectively. Thus, the resulting compositional changes due to Elusieve fractionation were reflective of the results of Srinivasan et al. (2009). So it appears that, for the DDGS used in this study, the Elusieve process was successful in altering the composition of the resulting streams vis-à-vis the original DDGS material.

As observed in Figure 2, the appearance of the original DDGS was slightly different compared to the big DDGS and the pan DDGS. The original DDGS appeared to have a greater size dispersion compared to either fraction, whereas the pan and big DDGS each appeared to have more uniform particle sizes (which should be the case due to the fractionation which occurred via the Elusieve process). Additionally, the pan and big DDGS each appeared to have a more consistent color as well (although the pan appeared more so than the big). The original DDGS had fairly high standard deviation for all color parameters (Hunter L, a, and b), although the big DDGS had relatively high variability in the Hunter L, while the pan DDGS had fairly high variability for the Hunter L and b parameters. Moreover, the pan DDGS was both lighter and yellower than either the original DDGS or the big DDGS.

Regarding the physical properties (Table 3) of the original DDGS, the moisture content was 13.28% (db), the geometric mean diameter was 0.52 mm, the geometric standard deviation was 2.05 mm, the Hunter L value was 44.85, the a value was 10.49, and the b value was 21.02. The values of these physical properties were similar to those reported previously by Bhadra et al. (2009) and Rosentrater (2006) for other commercial DDGS samples. Most of these properties were altered by the fractionation process. For example, in the big DDGS, the geometric mean diameter, geometric standard deviation, moisture content, L, a, and b values were changed by -43.71, 26.42, 1.82, 0.55, -3.88, and 0.13%, respectively. The pan DDGS, on the other hand, exhibited changes of 47.74, -43.76, -5.31, -8.78, -7.59, and -11.97%, respectively. It was anticipated *a priori* that the fractionation would alter the geometric mean diameter of the resulting product streams, as the Elusieve process is based, in part, on size segregation. The subsequent changes in color, however, were ancillary.

Flowability properties also exhibited a few changes (Table 3). The original DDGS had an angle of repose of 41.40 ($^{\circ}$), a loose bulk density of 0.53 (g/cm³), a packed bulk density of 0.57 (g/cm³), a Hausner Ratio of 1.07 (-), and a uniformity of 8.10 (-). The values of these flowability parameters were similar to those reported previously by Bhadra et al. (2009) for other commercial DDGS samples. Again, the fractionation altered these properties as well. Angle of repose, loose bulk density, packed bulk density, Hausner Ratio, and uniformity were changed by 0.08, 0.13, 0.82, 0.71, and -49.38%, respectively in the big DDGS. The pan DDGS, however, exhibited changes of -3.06, 0.38, -1.71, -2.08, and 39.51%, respectively. While the angle of repose and loose bulk density were not significantly altered (at $\alpha = 0.05$) by the fractionation process, the packed bulk density appeared to increase slightly as the particle size decreased. Thus, the Hausner Ratio consequently increased as particle size decreased, which was to be expected, because smaller particles often exhibit greater packing ability. Additionally, as the particle size decreased, a significant decrease in the uniformity value was observed, which indicated that the smaller particles had a lower dispersion in size, and thus were more uniform. It appears that the flowability of all three products was essentially similar, because the angle of repose and Hausner Ratio were only slightly different among the three materials. It should be noted, however, that because the uniformity value decreased as particle size decreased (because the particles were more uniform), it appears that the pan DDGS may potentially have the best flowability. Follow-up experiments should be conducted to verify this supposition.

By examining bivariate plots of the data (Figure 3), it appears that several potential linear relationships were present in the data (linear regressions are also indicated in the figure). This was corroborated by linear correlation analysis (Table 4). As particle size (as quantified by geometric mean diameter) increased, the moisture content, angle of repose, packed bulk density, Hausner Ratio, protein, ash, and color values decreased, whereas uniformity, lipid, carbohydrate, and NDF increased. These relationships were expected *a priori*, because of previous work with the Elusieve process, and thus it was not surprising that these variables were functions of particle size. The goal of the Elusieve process is to concentrate nutrient streams according to particles, so compositional changes were anticipated. Figure 3 clearly shows the changes in compositional properties according to particle size, which was the result of the fractionation. Additionally, because the particle size was unique between products, it was not a surprise that the physical properties were altered as well. Packed bulk density was related to most of the physical, flow, and compositional properties, as was Hausner Ratio (by definition). Undoubtedly, the alteration in the physical and flow properties were due, in large measure, to the size and shape of the particles (Duran, 2000; McGlinchey, 2005; Mehta, 2007) in the resulting big and pan fractions. Future research should examine additional flowability parameters (such as dispersibility, flow and flood indices, angle of internal friction, Jenike flow function, etc.) for these three product streams to further test whether there are differences in flowability or not.

Conclusions

In this study, the Elusieve process successfully altered the composition of the DDGS; it also slightly altered the physical and flowability properties of the resulting fractions, primarily because of differences in particle size and shape. Overall, however, it appears that the physical and flowability properties were fairly similar between the original DDGS and the subsequent fractions, and thus flowability in an industrial setting may not really be different. As the industry grows, it will become increasingly important to find novel uses for DDGS in animal feeds, industrial materials, and even possibly human foods. Fractionation may be a key to accomplishing this, thus enhancing the profitability of the dry grind ethanol production process

as the industry evolves, which is important in light of the rapidly increasing production of ethanol from corn.

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Table 1. Effects of Elusieve process on the resulting chemical properties of the DDGS (% db). ¹

Property	Big		Original		Pan	
	Mean	St Dev	Mean	St Dev	Mean	St Dev
Protein	31.85 a	1.06	33.00 a	0.99	37.25 b	0.21
Lipid	8.65 a	0.07	7.95 b	0.07	7.00 c	0.01
Ash	4.70 a	0.01	4.70 a	0.01	5.00 b	0.01
Carbohydrate	54.80 a	1.13	54.35 a	0.92	50.75 b	0.21
ADF	11.60 a	0.71	12.40 b	0.57	11.45 a	0.07
NDF	34.55 a	0.49	37.80 b	0.14	29.15 c	0.21

¹ Means for a given property followed by different letters indicate significant differences (p<0.05, LSD) due to product stream.

Table 2. Resulting chemical properties of the “fiber” stream (% db, unless noted otherwise). ¹

Property	Mean	St Dev
Moisture (% wb)	13.0	0.15
Protein	25.2	1.24
Lipid	6.6	0.98
Ash	4.9	0.34
Carbohydrate	63.3	--
NDF	42.3	0.85

¹ “Fiber” is a composite of the small, medium, and large fiber streams (Figure 1) resulting from the Elusieve process.

Table 3. Effects of Elusieve process on the resulting physical and flowability properties of the DDGS.¹

Property	Big		Original		Pan	
	Mean	St Dev	Mean	St Dev	Mean	St Dev
<u>Physical Properties</u>						
Geometric Mean Diameter (mm)	0.75 a	0.02	0.52 b	0.03	0.27 c	0.01
Geometric Standard Deviation (mm)	1.51 a	0.02	2.05 b	0.08	2.94 c	0.11
Moisture (% db)	13.04	0.19	13.28	0.86	13.98	0.22
L	44.60 a	0.39	44.85 a	1.07	48.79 b	1.28
a	10.90 a	0.15	10.49 b	0.30	11.29 a	0.09
b	20.99 a	0.14	21.02 a	0.52	23.53 b	0.49
<u>Flowability Properties</u>						
Angle of Repose (°)	41.37	0.80	41.40	0.60	42.67	2.15
Loose Bulk Density (g/cm ³)	0.53	0.01	0.53	0.01	0.53	0.01
Packed Bulk Density (g/cm ³)	0.56 a	0.01	0.57 a	0.01	0.58 b	0.01
Hausner Ratio (-)	1.06 a	0.01	1.07 ab	0.02	1.09 b	0.01
Uniformity (-)	12.10 a	0.01	8.10 b	0.01	4.90 c	0.01

¹ Means for a given property followed by different letters indicate significant differences (p<0.05, LSD) due to product stream.

Table 4. Results from Pearson linear correlation analysis on all measured variables.

	Geometric		MC (% db)	Angle of Repose (deg)	Loose Bulk Density (g/cm ³)	Packed Bulk Density (g/cm ³)	Hausner Ratio (-)	Uniformity (-)
	Geometric Mean Diameter (mm)	Standard Deviation (mm)						
Geometric Mean Diameter (mm)	1							
Geometric Standard Deviation (mm)	-0.9877	1						
MC (% db)	-0.6949	0.6930	1					
Angle of Repose (deg)	-0.4152	0.3834	0.6286	1				
Loose Bulk Density (g/cm ³)	0.0834	-0.0946	0.4286	-0.0757	1			
Packed Bulk Density (g/cm ³)	-0.8670	0.9090	0.5321	0.1918	-0.2730	1		
Hausner Ratio (-)	-0.6893	0.7255	0.1886	0.1830	-0.6961	0.8807	1	
Uniformity (-)	0.9929	-0.9733	-0.6453	-0.4019	0.1218	-0.8646	-0.7064	1
Protein (% db)	-0.9238	0.9493	0.5725	0.4161	-0.3223	0.9231	0.8476	-0.9073
Lipid (% db)	0.9920	-0.9894	-0.6516	-0.4359	0.1563	-0.8801	-0.7346	0.9874
Ash (% db)	-0.8757	0.9226	0.6632	0.4758	-0.1902	0.8485	0.7268	-0.8322
Carbohydrate (% db)	0.8760	-0.9126	-0.5386	-0.4047	0.3653	-0.9133	-0.8612	0.8534
ADF (% db)	0.1646	-0.2516	-0.4764	-0.3216	-0.2593	-0.1132	0.0430	0.0673
NDF (% db)	0.6354	-0.7183	-0.5592	-0.4374	0.2031	-0.6889	-0.6131	0.5656
L	-0.8234	0.8753	0.4401	0.2756	-0.2527	0.7897	0.7116	-0.7929
a	-0.4419	0.5260	0.1873	0.3203	-0.4133	0.4568	0.5423	-0.3867
b	-0.8400	0.8908	0.5102	0.3847	-0.2688	0.7999	0.7279	-0.8044

	Carbohydrate								
	Protein (% db)	Lipid (% db)	Ash (% db)	(% db)	ADF (% db)	NDF (% db)	L	a	b
Geometric Mean Diameter (mm)									
Geometric Standard Deviation (mm)									
MC (% db)									
Angle of Repose (deg)									
Loose Bulk Density (g/cm ³)									
Packed Bulk Density (g/cm ³)									
Hausner Ratio (-)									
Uniformity (-)									
Protein (% db)	1								
Lipid (% db)	-0.9507	1							
Ash (% db)	0.9584	-0.9052	1						
Carbohydrate (% db)	-0.9933	0.9092	-0.9602	1					
ADF (% db)	-0.2392	0.1911	-0.5031	0.2713	1				
NDF (% db)	-0.8194	0.6803	-0.9270	0.8602	0.6846	1			
L	0.9067	-0.8585	0.9205	-0.9058	-0.3646	-0.8382	1		
a	0.6556	-0.5185	0.7686	-0.6996	-0.6480	-0.8729	0.8073	1	
b	0.9321	-0.8776	0.9607	-0.9340	-0.4318	-0.8889	0.9893	0.8346	1

Figure 1. Schematic of the Elusieve process used to produce DDGS samples for this study.

Figure 2. a) Original DDGS; b) big DDGS; c) pan DDGS.

Figure 3. Scatterplot matrix indicates potential linear relationships between variables, as reflected by the correlation analysis.