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A Comparison of Measurement Procedures Used to Determine the Bulk Density of Distillers Dried Grains with Solubles (DDGS)

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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 co-product, distillers dried grains with soluble (DDGS) generated from this processing sector. DDGS is sold as a commodity for animal feed and like corn; its bulk physical and chemical properties are quality attributes normally used in trading by merchandisers. Consistency in product quality have been cause for concern in the industry and the lack of standard methods for physical and chemical property determination does potentially confound the issue, especially when results of attributes measured by different methods have not been evaluated for method parity. This research examined bulk density measurements of DDGS, using DDGS from six commercial ethanol plants (three from Indiana, and three from South Dakota) and conducted in two different labs. The effects of sample handling, measuring container filling method, filling height and measuring container size were examined. Variations due to each of these factors were quantified, it was determined that filling height and measuring container caused significant difference in the bulk density measured. As a guide for the standard method of determining bulk density for DDGS, we recommend the combination of a short drop height, using a 1 L measuring cup, with the sample obtained using a Boerner divider.

Keywords. *Bulk density measurement, DDGS, physical property.*

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Introduction

Distillers dried grains with solubles (DDGS) is the co-product of ethanol production from corn by the dry grind process. The marketability of DDGS has significant implications for the success of the ethanol industry. However, this product cannot be fairly traded because of inconsistencies in its physical property especially bulk density. Rosentrater (2006) showed that the bulk density of DDGS produced at six ethanol plants in South Dakota ranged from 391 to 496 kg/m³. Bhadra et al. (2009) found ranges of 490 to 590 kg/m³ from five plants in South Dakota and in another study of DDGS from sixty-nine sources in 2004 and 2005, it was found that the bulk density ranged from 365 to 561 kg/m³ (US Grains Council, 2008). These variations present economic challenges with regards to shipping the product (Ileleji and Rosentrater, 2008). Some variation could be caused by differences in process conditions, as pointed out by Kingsly et al. (2009). They showed that by varying the solubles content of a particular plant the bulk density changed from 420.47 – 458.05 kg/m³.

While bulk density of DDGS can inherently vary due to varying process variables, particle segregation during handling of DDGS could also cause bulk density variations with the same bulk. This is because on a particle level, DDGS bulk consists of a heterogeneous mixture of granular particles that vary in morphology and particle size (Ileleji et al., 2007). Thus, sample handling during physical property characterization must be done with care to avoid inducing errors due to inappropriate sampling methods for bulk solids. Hence it is critical to have an established procedure for determining basic physical properties such as bulk density. Manley et al. (2009) investigated a similar question for determining bulk density of wheat samples, using various equipment and procedures.

Currently there is no standard procedure for the bulk density measurement of DDGS. The ASTM International Standard ASTM C357 (Standard test method for bulk density of granular refractory materials) is proposed for grain material; however this method applies for materials in the size range of US sieve 4 (4.75 mm) to 12 (1.70 mm) while DDGS bulk has a wider particle size distribution and much smaller particles. This study evaluated some current practices used for bulk density measurement of DDGS, identified sources of variability, and ascertained the best procedure for DDGS bulk density measurement.

Materials and Methods

Bulk Density Measurement and Analysis

DDGS was obtained from six (6) ethanol plants, three (3) plants in South Dakota (SD 1, SD 2 and SD 3) and three (3) plants from Indiana (PU 1, PU 2 and PU 3). The DDGS from each plant was collected in individual five (5) gallon containers that were marked to clearly identify their sources. The bulk density of DDGS was assessed using a Seedburo filling hopper and stand (Seedburo Co), which consists of a brass hopper mounted in a tripod (figure 1). DDGS was poured into the hopper and the hopper was centered over the measuring cup. The hopper valve was opened quickly and DDGS was allowed to flow freely into the measuring cup. After the cup was filled, the excess material was leveled off with gentle zigzag strokes with a standard Seedburo striking stick. The measuring cup filled with DDGS was then weighed and the mass of DDGS in the measuring cup was determined by subtracting the mass of the measuring cup. The bulk density of DDGS was then calculated by using the expression:

Bulk density, ρ = mass of DDGS in measuring cup/ volume of measuring cup.

Figure 1: Seedburo bulk density measurement assembly.

For comparative analysis the bulk density was measured at two laboratories (Agricultural and Biological Engineering, Purdue University, West Lafayette, IN and USDA-ARS North Central Agricultural Research Laboratory, Brookings, SD). At each laboratory three variables were changed when assessing the bulk density: (1) drop height- the distance between outlet valve of the funnel and the base of the measuring cup, (2) measuring cup size and (3) sample reduction method – the method used to reduce the bulk quantity of DDGS in the buckets to an appropriate amount for the tests. The drop height was varied from an upper level (highest point the hopper can be mounted on the tripod) to a lower level (the lowest point the hopper can be mounted on the tripod). Drop heights of 13.9 and 17.2 cm were used. Measuring cups with volumes of 1 L and 1/2 L were used. Two sampling methods were used to provide the samples poured into the hopper: a) obtaining sample directly from the holding bucket, and b) using a Boerner sample divider to split the samples into desired proportions. This provided a 2^3 factorial experiment (Table 1); each treatment combination was replicated three times in each laboratory (that is, eight (8) treatment combinations in triplicate), using DDGS from each plant.

Table 1. Experimental design used in the study (replicated three times at each laboratory).

Sample	Treatment	Sampling Method	Cup Size	Drop Height
PU1	C1	Scooped	1/2 L	Lower Position
	C2			Upper Position
	C3		1 L	Lower Position
	C4			Upper Position
	C5	Divided	1/2 L	Lower Position
	C6			Upper Position
	C7		1 L	Lower Position
	C8			Upper Position
PU2	C1	Scooped	1/2 L	Lower Position
	C2			Upper Position
	C3		1 L	Lower Position
	C4			Upper Position
	C5	Divided	1/2 L	Lower Position
	C6			Upper Position
	C7		1 L	Lower Position
	C8			Upper Position
PU3	C1	Scooped	1/2 L	Lower Position
	C2			Upper Position
	C3		1 L	Lower Position
	C4			Upper Position
	C5	Divided	1/2 L	Lower Position
	C6			Upper Position
	C7		1 L	Lower Position
	C8			Upper Position
SD1	C1	Scooped	1/2 L	Lower Position
	C2			Upper Position
	C3		1 L	Lower Position
	C4			Upper Position
	C5	Divided	1/2 L	Lower Position
	C6			Upper Position
	C7		1 L	Lower Position
	C8			Upper Position
SD2	C1	Scooped	1/2 L	Lower Position
	C2			Upper Position
	C3		1 L	Lower Position
	C4			Upper Position
	C5	Divided	1/2 L	Lower Position
	C6			Upper Position
	C7		1 L	Lower Position
	C8			Upper Position
SD3	C1	Scooped	1/2 L	Lower Position
	C2			Upper Position
	C3		1 L	Lower Position
	C4			Upper Position
	C5	Divided	1/2 L	Lower Position
	C6			Upper Position
	C7		1 L	Lower Position
	C8			Upper Position

With DDGS from six (6) ethanol plants used, a total of one hundred and forty-four (144) tests in each laboratory (or 288 measurements in all) were conducted. Statistical analysis was conducted on the data collected using the SAS v9.1 (SAS Institute, Cary, NC) PROC GLM analysis of variance (ANOVA) procedure, using a Type I error rate of 0.05. ANOVA was used to determine whether there were significant differences in the bulk density of DDGS due to the main effects (drop height, measuring cup size and sample reduction method) and also whether there was any interaction of these effects. The results of each laboratory were analyzed independently for trends of handling characteristics. Furthermore, statistical evaluation was done using The R Project for Statistical Computing (<http://www.r-project.org>; Department of Statistics and Mathematics, Wien University, Wien, Austria) to perform randomized permutation tests and determine the best suited treatment combination for measuring the bulk density of DDGS.

Physical Property and Moisture Content Measurement

A Boerner divider was used to obtain appropriate sample sizes of DDGS from each bulk sample for both physical property (particle size analysis, and true density) and moisture content measurement. A gas multipycnometer (Quantachrome Corporation, Boynton Beach, FL) was used to determine the true density. The true density also known as the absolute density of the particle is a measure of only the solid density, excluding all pores in the particle. The micro cell (1.0772 cm^3) was filled to about $7/10^{\text{th}}$ of its volume with DDGS and the measurement procedure followed as outlined in the standard protocol by the equipment manufacturer.

The particle size analysis was conducted using the standard procedure described in ANSI/ASAE S319.3 (ASAE *Standards* 2005). A stack of sieves orderly arranged from U.S. no. 4 (nominal sieve opening: 4.75 mm) through U.S. no. 270 (nominal sieve opening: 0.053 mm) was mounted onto a Ro-tap shaker (Model RX-29, W.S. Tyler Inc., Mentor, OH, USA.). A charge of about 100 g was placed on the top sieve and the

shaker operated for about ten (10) minutes. The weight of DDGS on each sieve was entered into a Microsoft Excel spreadsheet and the geometric mean diameter (d_{gw}) and the geometric standard deviation (S_{gw}) were calculated.

The moisture content of DDGS from each plant was assessed using the NFTA Method 2.1.4. This involved weighing a small sample of 2 g and drying in an air-oven for 3 hours at 105°C then determining the percent of weight lost after drying in an air-oven.

For each bulk sample; true density, moisture content and particle size analysis was measured in triplicate and the average for each sample calculated. Statistical analysis was conducted on these data collected using the SAS v9.1 (SAS Institute, Cary, NC) PROC GLM analysis of variance (ANOVA) procedure to determine whether they were significantly different, using a Type I error rate of 0.05.

Results and Discussion

Table 1 shows the particle size, true density and moisture content of the samples used in this study. There were significant differences in the geometric mean particle size of the DDGS samples used in this study. The PU 1 sample had the largest particle size (1861.75 μm) and widest particle size distribution (figure 2) while the PU 2 sample had the smallest particle size (679.81 μm). The range of particle size for the samples in this study was wider than values obtained by US Grains Council (2008) from DDGS of 16 ethanol plants in 2001, 35 in 2004 and 35 in 2005, and Kingsly et al. (2009); but it was less than the range of particle sizes studied by Clementson et al. (2009). The true density also showed significant difference amongst the samples. The true densities of the samples were within the range of values obtained by Ileleji et al. (2007) and greater than those obtained by Kingsly et al. (2009), they ranged from 1307.63 kg/m^3 (PU 1) to 1291.91 kg/m^3 (PU 2). Moisture content showed significant difference amongst

the samples, the moisture content ranged from 11.82% (PU 1) to 7.75% (SD 3) and generally was within the moisture content range at which DDGS are normally produced except for the SD 3 sample.

Table 2: Particle size, true density, and moisture content of DDGS samples.

Sample ID	Particle size (μm)		True Density (kg/m^3)		Moisture (w/w %)	
	d_{gw}^*	S_{gw}	Mean*	Std.	Mean*	Std.
PU 1	1861.75 a	1494.42	1307.63 a	1.57	11.82 a	0.55
PU 2	679.81 d	418.28	1291.91 c	2.21	9.28 b,c	0.11
PU 3	1043.31 b	456.67	1285.23 d	1.15	10.03 b	0.56
SD 1	830.24 c	430.19	1281.34 d	2.39	9.87 b	0.57
SD 2	921.37 c	419.10	1301.22 b	1.26	10.69 a,b	0.82
SD 3	1053.04 b	619.83	1292.16 c	1.09	7.75 c	0.82

* Means with same lower case letter are not significantly different at 0.05 probability level.

Figure 2: Particle size distribution of DDGS samples.

Table 2 shows the statistical results of the true density tests. This shows that there were significant differences between laboratories. This indicates that human factor must be considered

in the variability of DDGS. Also, there were significant differences in bulk density due to cup sizes and drop heights. This shows that the apparatus assembly and measuring cup used will contribute to variability of the bulk density measured. There were no significant difference in bulk density due to sampling method, this was not as expected, possibly because the bulk sample used was small compared to a pile and did not allow for sufficient segregation of DDGS as referred to by Ileleji et al. (2007) and Clementson et al. (2009). There were also some interactions of the variables examined that contributed to significant differences in the bulk density, most notably the interaction between sample, sample method and drop height. The premise here is that because of the heterogeneity of DDGS samples varying in morphology and physical properties such as particle size and shape, and true density; the emptying mechanism of the funnel will affect the filling of the measuring cup because of drop height variation.

The residuals from each treatment combination were used to obtain their respective variances (Table 3). Treatment combination C3; scooped method of funnel fill, 1 L measuring cup and lower drop height had the least variance. The randomized permutation tests were performed to ascertain the differences of variances. From these analyses it was shown (Table 4) that the variance of treatment combination C3 was significantly less than treatment combinations C2, C5 and C6 but was not significantly different from treatment combinations C1, C4, C7 and C8. From these analyses, it appears that treatment combination C3 would be a suitable combination for use as a protocol for measuring bulk density of DDGS, however with the scoop sampling method, it would be necessary to consider the prior handling history of the material which may not always be clear and encompass segregation of the bulk material. Hence the treatment combination C7 (sample splitting, 1 L measuring cup and shortest drop height) should be used.

Table 3: P-values for main and interaction effects determined by Analysis of Variance.

	Lab	Sample	Variables		Drop Height	Probability (p-value)
			Sample Method	Cup size		
Main effects and interactions	✓					<0.0001
		✓				<0.0001
			✓			0.5015
				✓		<0.0001
					✓	<0.0001
	✓	✓				<0.0001
	✓		✓			0.0007
	✓			✓		0.0024
	✓				✓	0.5253
		✓	✓			0.0005
		✓		✓		0.0016
		✓			✓	0.0503
			✓	✓		0.0462
			✓		✓	0.0513
				✓	✓	0.0013
	✓	✓	✓			0.0013
	✓	✓		✓		0.0557
	✓	✓			✓	0.2563
	✓		✓	✓		0.4937
	✓		✓		✓	0.2361
	✓			✓	✓	<0.0001
		✓	✓	✓		0.6179
		✓	✓		✓	0.0083
		✓		✓	✓	0.5850
			✓	✓	✓	0.1917
	✓	✓	✓	✓		0.2731
	✓	✓	✓		✓	0.0595
	✓		✓	✓	✓	0.0770
	✓	✓	✓	✓	✓	0.0027

Table 4: Treatment combinations and their associated variances.

Combination	Sample Method		Cup Size		Drop Height		Mean Variance
	Divided	Scooped	1 L	½ L	Lower	Upper	
C1		X		X	X		3.244
C2		X		X		X	9.961
C3		X	X		X		2.541
C4		X	X			X	4.609
C5	X			X	X		10.926
C6	X			X		X	10.775
C7	X		X		X		5.249
C8	X		X			X	4.158

Table 5: P-values for comparison of variances among treatment combinations.

P(*C>C)	C1	C2	C3	C4	C5	C6	C7	C8
*C1	0.000	0.122	0.665	0.296	0.014	0.005	0.200	0.3542
*C2	0.892	0.000	0.960	0.829	0.464	0.488	0.767	0.864
*C3	0.370	0.049	0.000	0.091	0.001	0.000	0.0223	0.070
*C4	0.732	0.209	0.936	0.000	0.024	0.007	0.393	0.630
*C5	0.988	0.563	0.999	0.981	0.000	0.546	0.968	0.991
*C6	0.998	0.554	1.000	0.997	0.520	0.000	0.994	0.999
*C7	0.826	0.266	0.985	0.670	0.044	0.012	0.000	0.793
*C8	0.682	0.159	0.952	0.415	0.013	0.001	0.259	0.000

Conclusions

This study sought to evaluate the impact of the handling variables on the bulk density measurement of DDGS. The results show that there could be differences in bulk density measurement by performing the tests in different laboratories. We also determined that the drop height and measuring cup had a significant effect on the bulk density. This emphasize the need

of a standard protocol for DDGS bulk density measurement to minimize the variability of values reported and enhance the fair trade of DDGS product. Our analysis shows that the lowest variation of bulk density would be obtained from a combination of lower drop height and the 1 L measuring cup combined with the scoop method; however the scoop method will require consideration of the handling history of the material and may incorporate segregation in the bulk density measurement. Since there was no significant difference of the combination with scoop and divider method, we recommend the combination of the lower drop height, 1 L measuring cup and sample obtained by using a Boerner divider.

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References

- ASAE Standard. 2005. Method of determining and expressing fineness of feed materials by sieving. ASAE S319.3, American Society of Agricultural Engineers, St. Joseph, Michigan.
- ASTM International. 2003. ASTM F 1877-98, Standard practice for characterization of particles.
- ASTM International. 2007. ASTM C 357-07, Standard test method for bulk density of granular refractory materials.
- Bhadra, R., K. Muthukumarappan, and K. A. Rosentrater. 2009. Flowability properties of commercial distillers dried grains with solubles (DDGS). *Cereal Chemistry* 86(2): 170-180.
- Clementson, C.L., Ileleji, K.E., Stroshine, R.L., 2009. Particle segregation within a pile of bulk distillers dried grains with soluble (DDGS) and variability of nutrient content. *In Press Cereal Chemistry Journal*.

- Ileleji, K.E., Prakash, K.S., Stroshine, R. L., and Clementson, C.L. 2007. An investigation of particle segregation in corn processed dried distillers grains with solubles (DDGS) induced by three handling scenarios. *Bulk Solids and Powder Science and Technology* 2: 84-94.
- Ileleji, K.E. and K.A. Rosentrater. 2008. On the physical properties of dried distillers grains with solubles (DDGS). ASAE Paper No. 084576. St. Joseph, Mich.: ASAE.
- Kingsly, A.R.P., K.E. Ileleji, C.L. Clementson, A. Garcia, D.E. Maier, R.L. Stroshine and S. Radcliff. 2009. The effect of process variables on the physical and chemical characteristics of Corn Distillers Dried Grains with Solubles (DDGS) – Part II: Plant scale experiments. Submitted to *Bioresource Journal*.
- Manley, M., M. L. Engelbrecht, P. C. Williams, and M. Kidd. 2009. Assessment of variance in the measurement of hectolitre mass of wheat, using equipment from different grain producing and exporting countries. *Biosystems Engineering* (in press).
- National Forage Testing Association. NFTA Method 2.1.4, Dry matter by oven drying for 3 hrs at 105°C
- Rosentrater, K. A. 2006. Some physical properties of distillers dried grains with solubles (DDGS). *Applied Engineering in Agriculture* 22(4):589-595.
- U.S. Grain Council. 2008. Physical and chemical characteristics of DDGS. DDGS User Handbook. Retrieved 5/7, 2008, from <http://www.grains.org/galleries/DDGS%20User%20Handbook/08%20-%20Physical%20and%20Chemical%20Characteristics%20of%20DDGS.ERE%20revisions.pdf>.