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Weitao Zhang
Iowa State University, wtzhang1@iastate.edu

Kurt A. Rosentrater
Iowa State University, karosent@iastate.edu

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Abstract
With the rapid development of the ethanol industry, various research on distillers dried grains with solubles (DDGS) as a main co-product from the ethanol industry has been done in recent years. However, related research about basic properties to DDGS lacks of comprehensiveness. This study examined 18 samples from 10 plants in Midwest area and utilized standard laboratory methods to measure a series of properties. Final results showed moisture content of 8.69% (w.b.), water activity of 0.55, angle of repose of 48.04 Âº, geometric mean diameter (d_{gw}) of 0.74 mm, geometric standard deviation (S_{gw}) of 1.72 mm, loose bulk density of 483.9 kg/m^3, packed bulk density of 568.5 kg/m^3, Hunter L of 56.71, Hunter a of 13.85, Hunter b of 46.51, shear strength of 0.0324 kg/cm^2. This study represents another step toward a complete baseline understanding of DDGS.

Keywords
Biofuels, Coproduct, Distillers dried grains with solubles, DDGS, Physical properties

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Some properties of evolving distillers dried grains with solubles (DDGS) in 2012

Weitao Zhang, Graduate Research Assistant

Department of Agricultural and Biosystems Engineering, Iowa State University,

3122 NSRIC Building, Ames, IA 50011, wtzhang1@iastate.edu.

Kurt A. Rosentrater, Ph.D.

Department of Agricultural and Biosystems Engineering, Iowa State University,

3167 NSRIC Building, Ames, IA 50011, karosent@iastate.edu.

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Keywords. *Biofuels, Coproduct, Distillers dried grains with solubles, DDGS, Physical properties*

Introduction

With pressure from shortage of fossil fuels, bioethanol as a fuel additive is gradually utilized to reach the demand for fuel (Schnepf and Yacobucci, 2013). Conversion corn to ethanol is the most efficient method in the US ethanol industry, and has grown rapidly in recent years. In 2011 United States fuel ethanol production was the top producer in the world (RFA, 2012), which reached 13.9 billion U.S. liquid gallons (52.6 billion liters). According to Rosentrater (2006), more than 95% US fuel ethanol plants are used corn as a major raw material to produce ethanol.

In the corn-based fuel manufacturing, bioethanol, distillers dried grains with solubles (DDGS) (or other co-products), and carbon dioxide are three main products. Among all products from bioethanol industry, DDGS is an important ingredient, which is directly related to sustainability of dry grind plants, and is sold at a varying market price (US$85–140/ton) (Liu, 2008).

Common physical properties of DDGS include particle size, loose bulk density, packed bulk density, and angle of repose; these influence how much of the product can be stored in a given volume (Ileleji et al., 2008). In addition, moisture content, water activity and shear strength also affect the storability and material milling properties of DDGS. However, large variations in physical properties have been reported by different research groups over the years. (Shurson, 2005; Rosentrater, 2006; Ileleji et al., 2007).

Particle size distribution is a very important property, as it affects other properties. By using a series of six selected sieves (Nos. 8, 12, 18, 35, 60, and 100), Liu (2008) measured surface color and moisture, protein, oil, ash and starch in both original samples and sieved fractions. That research indicated that there was a great variation in composition and color among DDGS from different plants. It may be feasible to fractionate DDGS for compositional enrichment based on particle size, which could be a vital addition to quality of DDGS. Clementson and Ileleji (2012) utilized three samples to measure morphological and chemical characteristics of DDGS produced by mixing three levels of condensed distillers soluble (CDS) with wet distillers grains and drying according to official methods (AOAC, 2002). Results showed that pore volume, particle porosity and effective bulk porosity decreased when CDS level increased. Furthermore, they observed that heterogeneity and particle segregation could cause sampling errors, and as a consequence nutrient and bulk density variability.

Another key property is bulk density. Bulk density directly affects the cost for shipping of DDGS (Ileleji et al. 2008). Clementson and Ileleji (2010) designed a simulated apparatus to investigate the bulk density variability of DDGS during filling of railcar hoppers, and found that there was a significant difference between the initial and final measures of bulk density and particle size as the hoppers were emptied in both mass and funnel flow patterns, which was caused by particle size variations.

In addition, drying distillers wet grains (DWG) and CDS to DDGS affects nutritive value and physical characteristics. Kingsly et al. (2011) tested four properties as affected by the drying process, including particle size, particle size distribution, particle bulk density and color, all of which are.

Through some research has been done to study the properties of DDGS, production processes have been changing in recent years, and oil is now commonly removed. In order to understand the changes in the DDGS industry, new baseline data about these properties should be established, because they are essential for design of equipment, processing facilities, storage and material handling systems (Rosentrater, 2011). Thus the objective of this study was to investigate basic properties of contemporary DDGS, including moisture content, water activity, angle of repose, particle size, loose bulk density, packed bulk density, color and shear strength, from ten dry grind corn ethanol facilities in the Midwest U.S.

Materials and Methods

Materials

Sixteen DDGS samples were supplied by ten dry grind corn ethanol facilities located in the Midwest US, and labeled as 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10, to ensure anonymity. All samples were collected during the fall of 2011 and spring of 2012 (i.e., three unique samples per plant from two plants, two unique samples per plant from two plants, then one unique sample per plant from six plants), and were stored at room temperature (24±1 ºC) in sealed plastic storage bags. All properties were measured at room temperature (except moisture content) and studied with a completely randomized design.

Methods

Moisture content was determined following the standard Forage Analysis Procedure (NFTA, 2002), using a forced-convection laboratory oven (Thermo OGH & OMH180, Scientific Heratherm, Langenselbold, Germany) at 105 ºC for 3 h. Water activity was measured with a calibrated water activity meter (AquaLab series 3 TE, Decagon Devices, Pullman, Washington, USA). Angle of repose was measured by allowing DDGS to fall onto a 15.5 cm x 15.5 cm square plate in a Helle Shaw cell following the method described by Mohesenin (1980), and angle was measured by ImageJ software. Particle size was measured according to ANSI/ASAE S319.3 (ASABE, 2004), using U.S. sieve nos. 6 (3.36 mm), 8 (2.38 mm), 10 (2.00 mm), 14 (1.680 mm), 16 (1.19 mm), 20 (0.841 mm), 30 (0.595 mm), 40 (0.420 mm), 50 (0.297 mm), 70 (0.210 mm), Pan (0.044 mm). From the weight of DDGS collected on each sieve, the geometric mean diameter (d_{gw}) and the geometric standard deviation (S_{gw}) were calculated according to the standard. Bulk density of DDGS was measured using a filling hopper, stand, and 1 L cup (Seedburo 151, Seedburo Equipment Co, Chicago, IL, USA) with the method designed by USDA (1999). Color was measured using a spectrocolorimeter (LabScan XE 16807, Hunter Associates Laboratory, Reston, VA, USA), with the L-a-b opposable color scales (Hunter Associates Laboratory, Reston, VA, USA) (HAL, 2002). Shear strength was tested by a torvane shear device (26-2261, ELE International, Loveland, CO, USA) following the procedures described by Goossens (2004) and Zimbone et al. (1996).

Data treatment and statistical analysis

All collected data were analyzed with Microsoft Excel v. 2010 (Microsoft Corp, Redmond, WA), and SAS Enterprise 4.3 (SAS Institute, Cary, NC) software. Summary statistics, t-test (to test for differences within the processing plant), and ANOVA (to test for differences among processing plants) were tested for each property to determine whether significant differences existed, using a Type I (α) error rate of 0.05; if so, post-hoc LSD tests were conducted using a 95% confidence level to determine where those differences occurred.

Results and Discussion

Table 1 summaries the measured properties of the DDGS in this study, including minimum, maximum, mean values and standard deviations for each property, both for each individual plant and overall. Statistically significant differences were found from samples of the same plant, and among samples from different plants. Results show large variations in most properties, which are similar to other prior studies

(Shurson, 2005; Ileleji et al., 2007; Rosentrater et al., 2006).

As shown in Table 1, these samples ranged in moisture content from 6.66 to 10.48 % (w.b. - wet basis), with a mean of 8.69%. After converting to dry basis, the results ranged from 7.13% to 11.71% (d.b. – dry basis), with a mean of 9.52%. According to the results, these DDGS samples were well suited for storage because the lowest limit of moisture content to most microbial growth in corn and related products is 13.5 % (d.b. – dry basis) (Beauchat, 1981). In addition, the moisture content data in this study are generally between the results of Rosentrater (2006) and Bhadra et al. (2009), and very similar to Kingsly et al. (2010) and Spiehs et al. (2002). The reasons for these differences probably are caused by the method of producing DDGS at the ethanol plants.

Overall, DDGS in this study had a low water activity, which ranged from 0.46 to 0.61. Water activity is a measure of the energy status of the water in a system, and it directly affects the activity of microbes. Prezant et al. (2007) has shown that most bacteria are adapted for growing in an environment with a water activity of 0.9, mold is adapted to between 0.7 and 0.8, yeast is adapted more than 0.7, and very little microbial growth can occur if the water activity is below 0.65. Thus, water activity results are related to moisture content, and should be limiting to microbe growth. The samples in this study have a low water activity, which means a small probability of spoilage problems, DDGS should still be stored in bulk cautiously, in case of potential moisture migration from the environment, especially during the shipping. These results are very similar to those found in previous work (Rosentrater 2006).

Angle of repose ranged from 35.48^o to 82.87^o, with a mean of 48.04^o (Figure 1 and Table 1). According to the LSD analysis, the results have an obvious separation into two types of behaviors: a low value of about 40^o (including plant 1, 2, 3, 4, 5, 6 and 7); the other had a high value of about 75^o (including 8, 9 and 10). The results of the former were similar to Bhadra et al. (2006) and a little higher than Rosentrater (2006). The reason for the high value in the latter group may be influenced by particle size, composition of the DDGS particles, and the drying and cooling conditions, especially when sugar and fat molecules on the surface reach glass transition temperature, which affects the surface frictional properties such as stickiness and cohesion (Liu et al., 2011; Rosentrater, 2006).

Overall, geometric mean diameter (d_{gw}, mm) had a range from 0.34 to 1.28 mm, with a mean of 0.74 mm (Table 1). According to the LSD analysis, the results had an obvious separation into three types: the first group includes Plant 1 and 2, which had high values similar to the results of Clementson et al. (2009); the second group included Plant 3, 4, 5, 6 and 7, which had a mean value about 0.65, which was similar to the results of Liu (2008); the third group included Plant 8, 9 and 10, which had a low value, about 0.4, similar to Bhadra et al. (2012). Geometric standard deviation (S_{gw}, mm) ranged from 1.47 to 2.14 mm, with a mean of 1.72 mm (Table 1), which is very similar to the results of U.S. Grains (2008), and higher than Bhadra et al. (2009), Clementson et al. (2009) and Liu (2008). All these results show large variations in particle size distribution due to different plants.

Loose bulk density ranged from 439.8 kg/m^3 to 570.6 kg/m^3 , with a mean of 483.9 kg/m^3 (Table 1), which is similar to the results of Bhadra et al. (2009), and a little lower than Clementson et al. (2009) and Liu (2008). Packed bulk density ranged from 476.4 kg/m^3 to 666.6 kg/m^3 , with a mean of 568.5 kg/m^3 (Table 1). According to the LSD analysis, most samples from different plants were significantly different from each other, which means that there is a large variation across the different plants instead of bulk density.

The DDGS color values in this study are shown in Table 1 as well. The range of Hunter – L (white-black axis) ranged from 51.77 to 61.29 with a mean of 56.70; the range of Hunter – a (red-green axis) was from 12.25 to 15.91, with mean of 13.85; the range of Hunter – b (blue-yellow axis) was from 41.63 to 51.60, with mean of 46.51. All these value were significantly higher than Rosentrater (2006) and Bhadra et al. (2007); Hunter – b was nearly 100% higher, which means more yellow and possibly better nutrient quality (Goihl, 1993 and Ergul et al., 2003). According to the LSD, most plants were significant different from each other, except the relationships among Plant 8, 9 and 10.

Shear strength ranged from 0.022 kg/cm^2 to 0.050 kg/cm^2, with a mean of 0.032 kg/cm^2, which is similar to the data of Ganesan et al. (2007) and Ganesan et al. (2009). According to the LSD, there were no significant differences in most samples, except Plant 1 which means that most samples had similar shear strength.

Conclusions

The goal of this research was to provide baseline property data for typical DDGS from Midwest from USA in 2011 and 2012. After experimental test, this study got the data of DDGS properties and compared with other researcher's results, which included moisture content, water activity, angle of repose, geometric mean diameter (d_{gw}), geometric standard deviation (S_{gw}), loose bulk density, packed bulk density, color content, shear strength. This research supplies up to date engineering data which is key to storing and handling DDGS, designing and utilizing equipment, and producing co-products from DDGS. Future work will focus on examining correlations between physical and chemical properties and explore the reasons why the differences occur in different samples.

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Table 1. Properties of distillers dried grains with solubles (DDGS). [a]

Property	Processing Plant	Number of Observations	Minimum	Maximum	Mean	Standard Deviation
Moisture Content (%, wb)	Overall	48	6.66	10.48	8.69	1.13
	1	9	7.72	8.90	8.37 bc	0.38
	2	9	6.66	7.21	6.99 a	0.20
	3	6	9.82	10.48	10.18 g	0.28
	4	6	7.70	10.32	9.63 fg	0.98
	5	3	8.16	8.86	8.61 cd	0.39
	6	3	9.01	9.63	9.33 def	0.31
	7	3	8.95	9.80	9.36 def	0.43
	8	3	8.34	9.60	8.90 ce	0.64
	9	3	9.04	9.60	9.27 def	0.29
	10	3	7.35	8.04	7.78 b	0.38
Water activity (-)	Overall	48	0.46	0.61	0.55	0.05
	1	9	0.54	0.56	0.55 a	0.01
	2	9	0.46	0.48	0.47 b	0.01
	3	6	0.59	0.60	0.60 c	0.01
	4	6	0.59	0.60	0.59 c	0.00
	5	3	0.53	0.53	0.53 d	0.00
	6	3	0.58	0.59	0.59 e	0.01
	7	3	0.58	0.58	0.58 ef	0.00
	8	3	0.57	0.58	0.58 f	0.00
	9	3	0.6	0.61	0.60 g	0.01
	10	3	0.56	0.56	0.56 h	0.00
Angle of Repose (º)	Overall	48	35.48	82.87	48.04	13.32
	1	9	38.44	44.54	42.03 ab	1.56
	2	9	37.89	43.42	41.31 b	1.20
	3	6	35.48	44.23	41.09 b	2.33
	4	6	41.32	47.91	43.92 a	2.05
	5	3	39.14	42.09	40.76 b	1.31
	6	3	39.52	42.97	41.14 b	1.23
	7	3	40.30	43.78	41.47 ab	1.38
	8	3	70.74	82.87	76.90 c	5.40
	9	3	65.32	81.78	73.06 c	5.91
	10	3	71.63	80.12	75.20 cd	2.95
Geometric mean diameter (d_{gw}, mm)	Overall	48	0.34	1.28	0.74	0.27
	1	9	0.74	0.92	0.82 a	0.06

	2	9	1.14	1.28	1.19 b	0.05
	3	6	0.59	0.78	0.65 c	0.08
	4	6	0.64	0.75	0.71 c	0.05
	5	3	0.63	0.73	0.68 c	0.05
	6	3	0.60	0.73	0.65 c	0.07
	7	3	0.58	0.69	0.64 c	0.06
	8	3	0.37	0.38	0.37 de	0.01
	9	3	0.34	0.34	0.34 d	0.01
	10	3	0.43	0.46	0.45 e	0.02
Geometric standard deviation (S_{gw}, mm)	Overall	48	1.47	2.14	1.72	0.15
	1	9	1.74	1.84	1.79 a	0.03
	2	9	1.47	1.51	1.49 b	0.01
	3	6	1.66	1.79	1.72 cd	0.05
	4	6	1.66	1.75	1.72 cd	0.03
	5	3	1.66	1.78	1.73 acd	0.07
	6	3	1.65	1.84	1.76 ac	0.10
	7	3	1.70	1.88	1.76 ac	0.10
	8	3	1.80	1.90	1.85 e	0.05
	9	3	2.08	2.14	2.10 f	0.03
	10	3	1.65	1.71	1.67d	0.03
Loose Bulk Density (kg/m^3)	Overall	48	439.8	570.6	483.9	39.24
	1	9	543.4	570.6	555.5 a	11.20
	2	9	439.8	446.0	442.7 b	2.27
	3	6	465.8	469.6	467.6 c	1.30
	4	6	462.4	470.8	467.0 c	3.42
	5	3	479.2	482.8	480.9 d	1.80
	6	3	497.1	501.4	499.0 e	2.18
	7	3	443.4	447.9	445.0 b	2.49
	8	3	497.0	505.0	500.1 e	4.29
	9	3	478.9	481.4	480.2 d	1.25
	10	3	471.0	477.7	473.3 ed	3.81
Packed Bulk Density (kg/m^3)	Overall	48	476.4	666.6	568.5	58.35
	1	9	622.8	649.8	635.5 a	8.47
	2	9	476.4	506.2	491.1 b	8.96
	3	6	524.6	542.6	532.4 c	8.03
	4	6	546.8	559.2	554.2 d	5.20
	5	3	500.4	550.6	533.5 c	28.64
	6	3	569.6	574.0	571.2 e	2.43

	7	3	525.8	529.6	528.2 c	2.09
	8	3	654.2	666.6	661.0 f	6.29
	9	3	619.4	626.0	622.5 a	3.31
	10	3	615.8	632.0	626.4 a	9.19
Color - Hunter L (-)	Overall	80	61.29	51.77	56.71	2.57
	1	15	56.58	53.68	54.76 a	0.76
	2	15	56.18	53.81	55.22 ab	0.84
	3	10	54.23	51.77	53.23 c	0.82
	4	10	59.22	56.98	58.17 d	0.64
	5	5	61.07	59.98	60.42 f	0.44
	6	5	60.43	58.26	59.39 e	0.92
	7	5	61.29	59.49	60.68 f	0.81
	8	5	59.81	59.49	58.96 de	0.99
	9	5	60.31	58.37	59.31 e	0.91
	10	5	56.06	55.45	55.79 b	0.23
Color - Hunter a (-)	Overall	80	15.91	12.25	13.85	0.92
	1	15	15.91	14.89	15.35 a	0.28
	2	15	13.95	13.09	13.45 bc	0.23
	3	10	13.43	12.88	13.18 d	0.21
	4	10	12.83	12.25	12.62 e	0.22
	5	5	15.12	14.63	14.89 f	0.19
	6	5	14.25	14.02	14.12 i	0.09
	7	5	13.50	13.16	13.30 bd	0.15
	8	5	13.64	13.16	13.59 cg	0.07
	9	5	14.01	13.49	13.78 gh	0.23
	10	5	14.52	13.62	13.92 hi	0.35
Color- Hunter b (-)	Overall	80	51.60	41.63	46.51	2.55
	1	15	49.55	47.59	48.24 a	0.56
	2	15	44.89	42.98	44.24 b	0.59
	3	10	43.07	41.63	42.28 c	0.46
	4	10	46.32	44.55	45.60 d	0.50
	5	5	51.60	50.55	51.11 e	0.38
	6	5	47.03	46.05	46.60 f	0.50
	7	5	50.39	48.75	49.74 g	0.60
	8	5	48.14	48.75	47.94 ah	0.25
	9	5	47.90	47.12	47.65 h	0.31
	10	5	49.16	47.57	48.01 ah	0.65
Shear Strength	Overall	32	0.022	0.050	0.032	0.01

(kg/cm^2)	1	6	0.040	0.050	0.045 a	0.01
	2	6	0.028	0.038	0.033 c	0.01
	3	4	0.026	0.034	0.030 bc	0.00
	4	4	0.024	0.032	0.028 c	0.01
	5	2	0.022	0.024	0.023 c	0.01
	6	2	0.022	0.026	0.024 bc	0.00
	7	2	0.032	0.036	0.034 bc	0.01
	8	2	0.030	0.032	0.031 b	0.00
	9	2	0.026	0.030	0.028 bc	0.01
	10	2	0.028	0.030	0.029 bc	0.00

[a] New values followed by the same letter within a given property are not significantly different among plants ($p < 0.05$)

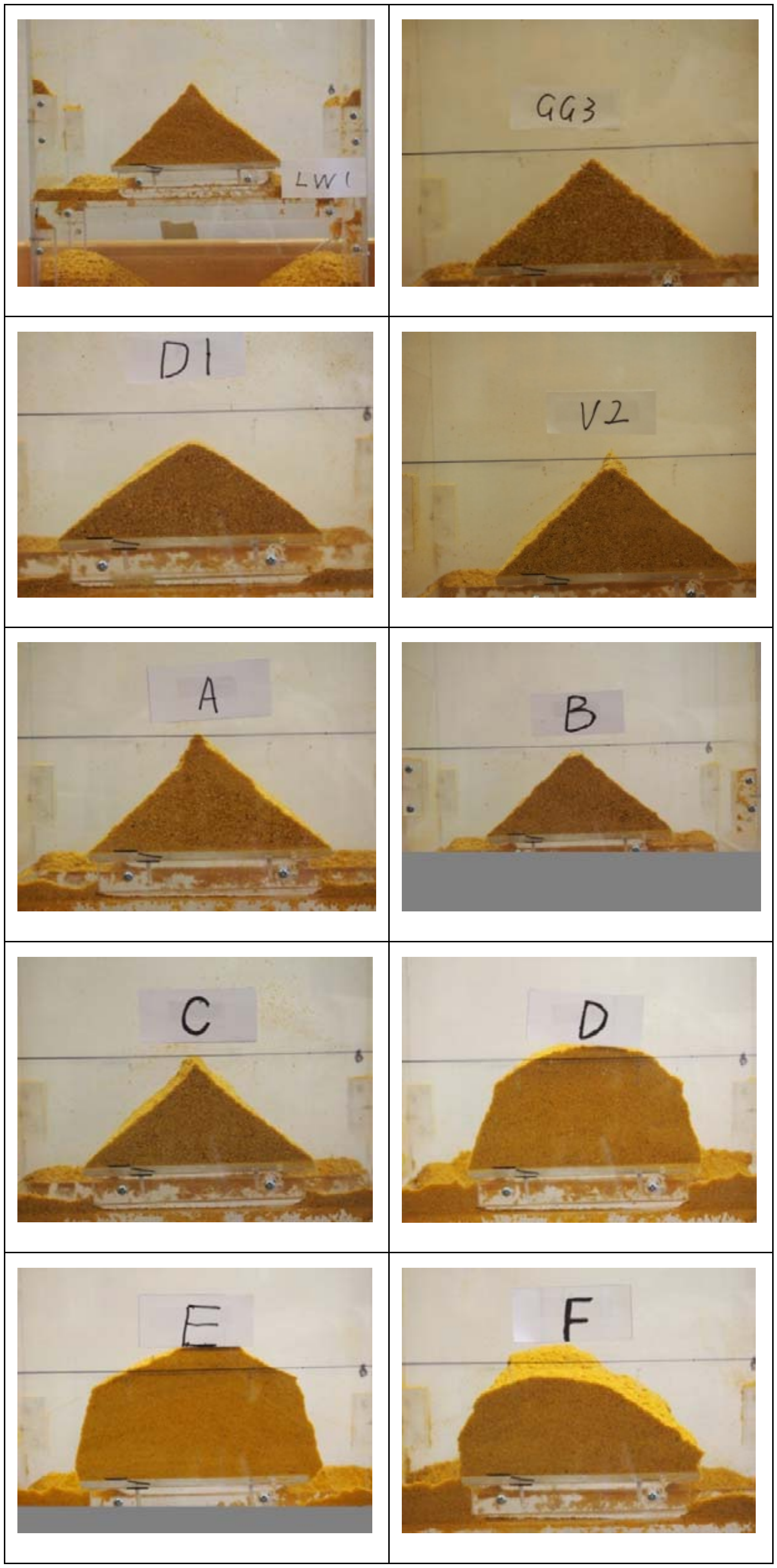

Figure 1: Angle of repose tests of distillers dried grains with solubles (DDGS) samples.