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Physico-chemical characteristics of wheat distiller's grain with solubles sourced from a Saskatchewan fuel ethanol plant

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ABSTRACT There is still very limited information on the physical and chemical properties of dried distiller's grain with solubles (DDGS) derived from wheat-based fuel ethanol production. As this co-product become increasingly more available in Western Canada, baseline information on its properties is essential in responding to problems posed by existing processing, handling, storage, and transportation systems. This paper presents initial results on selected physico-chemical characteristics of wheat DDGS obtained from a Saskatchewan ethanol plant. These include particle size and size distribution, bulk and particle densities, frictional, compression, and moisture sorption characteristics, all determined at varying moisture levels. Proximate analysis and gross energy determination were also carried out using standard laboratory methods.

Keywords: wheat DDGS, distiller's dried grain with solubles, physical properties, chemical composition.

INTRODUCTION With the significant expansion of the wheat-based fuel ethanol industry in western Canada in recent years, dried distiller's grain with solubles (DDGS), an important co-product of ethanol production, is increasingly becoming more and more available in the region. Recent estimates indicate that western Canada's ethanol plants collectively produce about 460,000 tonnes of wheat DDGS annually (University of Saskatchewan 2009). Since about one-third of the

total grain used in ethanol production ends up as distiller's grain (Rosentrater 2006a; US Grains Council 2007; McKinnon 2007), the sale of DDGS as an alternative animal feed ingredient contributes to the economic viability of ethanol plants (Belyea et al. 2004; Liu 2008; Ganesan et al 2008a). DDGS supply chains, however, are confronted with a number of challenges: a highly energy-intensive drying process (Murphy and Power 2008; Tang and Cenkowski 2001; Tang et al. 2005), inconsistent product quality (Belyea et al. 2004; Knott et al 2004; Ileleji et al. 2007; Liu 2008; Kingsly 2009; Clementson et al. 2009), poor flowability (Rosentrater 2006b; Ganesan et al 2007; Ganesan et al. 2008b), and high logistics cost (Rosentrater 2006b).

Baseline information on the physical and chemical properties of wheat DDGS, essential in addressing problems posed by existing processing, storage, and handling systems, is still very limited. A research project, which covers investigations on proximate composition, particle size and size distribution, bulk and particle densities, color, flowability and frictional properties, airflow resistance, and visco-elastic, thermal and moisture sorption characteristics, is currently underway. Thus, this study aims to contribute toward better understanding of the physico-chemical properties of wheat DDGS and their important implications during processing, storage and handling operations. This paper presents some of the research project's initial results.

MATERIALS AND METHODS DDGS samples were obtained from a fuel ethanol plant in south Saskatchewan in two production batches (Oct 2008 and Jun 2010). Samples were placed in tightly sealed bins and were stored in a 3°C walk-in cooler until these were used.

Moisture content Moisture content of DDGS samples was determined using AOAC Official Method 920.36 (AOAC, 2003a). For each production batch, samples at three moisture levels were generated. To achieve lower moisture levels than that of the materials obtained from the ethanol plant, DDGS samples were dried at 80°C. To adjust moisture to a higher level, DDGS was sprayed with an appropriate amount of water, thoroughly mixed, placed into sealed plastic bags, and stored for 24 h before use.

Bulk and particle density To determine bulk density, the sample was placed on a funnel and was allowed to freely flow into a 0.5 L steel cup (SWA951, Superior Scale Co. Ltd., Winnipeg, MB). The cup contents were leveled using a steel rod and weighed. Bulk density was calculated by dividing sample mass contained in the cup with the cup volume. Particle density was determined using a gas multipycnometer (QuantaChrome, Boynton Beach, FL).

Particle size and size distribution. Sieving was done in two stages. In the first stage, US sieve sizes 10, 12, 20, 30, 40, 50, and 60 were used while sieves with finer openings (US No. 70, 80, 100, 140, 200, 275) were used in the second stage. Each sieving lasted for 10 min using a Ro-Tap shaker, following ANSI/ASAE S319.4 - Method of Determining and Expressing Fineness of Feed Materials by Sieving (ASABE, 2008).

Color. Color of the samples was determined using the HunterLab spectrocolorimeter (Hunter Associates Laboratory Inc, Reston, VA) and was expressed in terms of the CIE L, a, and b parameters.

Frictional properties. The angle of repose, coefficient of static friction, and coefficients of internal and external friction were determined. The angle of repose was determined using the side-emptying box and the top-filling box devices while the coefficient of static friction of the sample was evaluated in three contact surfaces (wood, concrete and steel) using the tilting table.

In determining the coefficients of internal and external friction, the Wykeham Farrance shear box apparatus (Wykeham Farrance International Ltd., Slough, U.K.) was used. In determining the internal friction friction coefficient, shear stress was measured at five normal loads (200, 400, 600,

800, 1000 N). For the external friction coefficient, normal loads 100, 200, 300, 400 and 500 N and a steel surface were used.

Compressibility. The compression characteristics of wheat DDGS samples were determined using a single pelleting unit (6.35mm diameter and 135.34mm length), heated at 50, 70 and 90°C to simulate the pelleting conditions in commercial mills (Shankar et al. 2009). A plunger of the same diameter, which is attached to the Instron Model 1011 testing machine (Instron Corp., Canton, MA), was used to compress the 1 g samples at 1000, 2500 and 4000 N. Crosshead speed was set at 50mm/min (Shankar et al. 2009).

Experimental data were fitted to four models to describe wheat DDGS volume changes during compression: Cooper and Eaton (Mani et al. 2004; Emami and Tabil 2007; Shaw 2008), Kawakita and Ludde (Emami and Tabil 2007; Shaw 2008), Walker (Tabil and Sokhansanj 1997; Emami and Tabil 2007; Shaw 2008) and Jones' equations (Tabil and Sokhansanj 1997; Mani et al. 2004; Shaw 2008). The most suitable model was chosen using coefficient of multiple determination (R^2) and mean square error (MSE) as criteria.

Moisture sorption characteristics. Sorption isotherm characteristics of wheat DDGS at two temperature (3°C, 23°C) and five relative humidity (RH) levels (50, 60, 70, 80 and 90%) were determined using the static gravimetric method. Airtight cylindrical chambers, as described by Dadgar (2005), were used to hold the samples during the duration of the study. Each chamber contained 4 petri dishes, holding about 0.75 g of thinly spread samples. Saturated salt solutions (magnesium nitrate, potassium iodide, sodium chloride, potassium bromide, barium chloride, and potassium nitrate) were used to maintain the RH level inside these chambers. To prevent microbial growth during the experiment, crystalline thymol (Ganesan et al. 2008c) were placed near the samples in each of these chambers. Sample weight, RH and temperature of each of the chambers were regularly monitored until the sample weight reached equilibrium. Duplicate runs were made for each of the RH-temperature treatment combinations.

Experimental data were fitted to four moisture sorption models laid out in ASABE Standard D245.5: Moisture relationships of plant-based agricultural products (ASABE 2005): Modified Henderson, Modified Chung-Pfost, Modified Halsey, Modified Oswin, and the Guggenheim-Anderson-deBoer (GAB) equations. The most suitable model was chosen using R^2 and MSE as evaluation criteria.

Chemical composition. The proximate composition of wheat DDGS was determined using standard procedures: protein (AOAC 2003b), mineral matter (AOAC 2003c), fat (AOAC 2003d), neutral detergent fibre (NDF) (Van Soest et al. 1991), and acid detergent fiber (AOAC 2003e). AAFCO 0728, a lamb starter feed sample, was used as control during all the proximate analysis runs.

Gross energy. About 0.5 to 0.7 g of the samples were compressed into pellets. Gross heat of combustion of these pelleted samples was determined using the Parr 1281 Oxygen Bomb Calorimeter (Parr Instrument Co., Moline, IL). One gram Parr benzoic acid pellets, which were standardized for bomb calorimetry, were also used as control.

RESULTS AND DISCUSSION Table 1 shows the some of the chemical composition of wheat DDGS samples sourced from two production batches. Protein, ash, fat, and fibre contents of the two samples were significantly different at the 0.05 significance level. The Jun 2010 samples had significantly lower protein but higher fibre, ash, and fat contents compared to the Oct 2008 samples. These differences could be attributed to inherent variations in the raw materials, such as the wheat grains used, as well as variations in the processing conditions. Variations in the blending proportions of wet distiller's grain and condensed distiller's solubles could result to composition variations in the resulting DDGS. The chemical composition of the condensed distiller's solubles (CDS) is quite different from that of the wet distiller's grain (WDG. The latter, for example, has

higher fibre and fat content. The initial proximate composition values obtained during this study (Table 1) were close to the values reported by Widyarante and Zijlstra (2007). Gross energy values were not significantly different between the two production batches.

Table 1. Chemical characteristics of wheat DDGS sourced from a south Saskatchewan fuel ethanol plant in two production batches. Protein, fat, ash, fibre, and gross energy values are presented on dry matter basis, and are expressed in the form, arithmetic mean (standard deviation). These values were derived from duplicate runs.

Production Month	% Moisture, wet basis	% Protein	% Fat	% Ash	% Fibre		Gross energy, kJ/kg
					ADF	NDF	
Oct 2008	12.9	45.14 (0.52)	3.37 (0.05)	6.41 (0.02)	21.10 (0.11)	42.74 (0.88)	21,379 (35)
June 2010	13.3	38.81 (0.14)	4.89 (0.18)	7.10 (0.01)	17.45 (0.27)	46.55 (0.80)	21,426 (9)

Figure 1 shows the particle size distribution of samples drawn from the two production batch sources (Oct 2008 and Jun 2010). It can be observed that the October 2008 sample were coarser compared to the June 2010 sample. This size variation is shown in succeeding tables (Tables 2 and 3) as well. Particle size differences could be attributed to variations in the blending proportions of CDS and WDG. Kingsly et al (2009) indicated that increasing the CDS level in the blend could increase the particle size because it acts like a binding agent, increasing inter-particle affinity and inducing particle agglomeration of particles during drying.

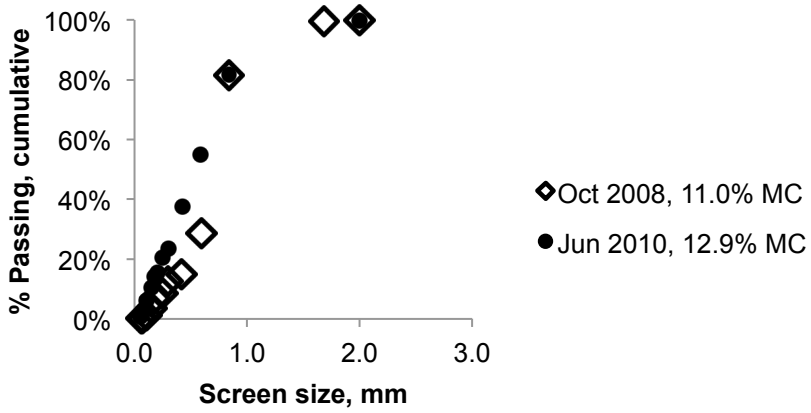

Figure 1 Particle size distribution of wheat DDGS samples obtained from a Saskatchewan ethanol plant in two production batches

Tables 2 and 3 shows some of the physical properties of the wheat DDGS samples obtained from an October 2008 production batch of a Saskatchewan ethanol plant. Variations in bulk and particle densities between the two samples could be attributed to variations in chemical composition as well as variations in the particle size distribution.

In terms of color, L values closer to 100 are lighter compared to those which that are closer to 0. Positive values of a and b indicate redness and yellowness, respectively. Thus, as reflected in Table 2, the wheat DDGS samples in the study were on the darker side. Comparison between the two production batches could not be done since the color parameters for the Jun 2010 DDGS had not been determined yet. Ganesan et al (2008a) and Kingsly et al 2009 noted that Maillard reaction between sugars and proteins in WDG and CDS during the drying process could be the primary cause of DDGS darkening.The rate of Maillard reaction depends upon temperature, time, pH, water activity and chemical composition and variations in these parameters affect the final Maillard reaction outcome such as color and aroma compounds (Ames 1990; Mavromichalis 2001; Owusu-Apente 2004). Color has also been suggested in a number of corn DDGS studies (Fastinger et al. 2006; Batal and Dale 2006) to be a quick indicator of lysine content and digestibility.

Table 4 shows the static friction coefficients on various surfaces and angles of repose of the Oct 2008 wheat DDGS samples. Their values increased with increases in moisture content. Comparisons with samples from the Jun 2010 production batch have yet to be made.

Figure 2 shows the estimates of the coefficient of external friction of wheat DDGS at two moisture levels on steel surface. The coefficient of external friction, presented by the slope of the line in the figure, increased when sample moisture content was increased.

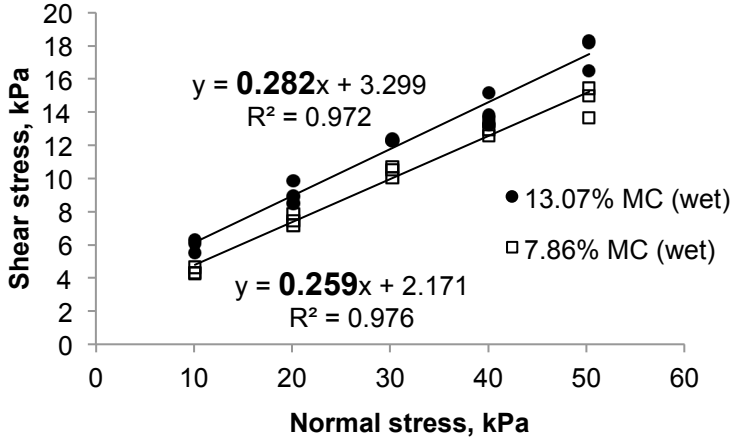

Figure 2 Coefficient of external friction of wheat DDGS on a steel surface. The friction coefficients (slope of the lines) are reflected in bold font

Figures 2 and 3 shows the moisture adsorption characteristics of wheat DDGS samples exposed to varying relative humidity levels at 3°C and 23°C, respectively. Moisture adsorption of the samples was higher in the low temperature environment compared to when they are exposed in the high temperature. The GAB model, shown below as Eq 1, adequately described the sorption characteristics of wheat DDGS, with the model parameters and statistical criteria shown in Table 5. The other four models did not fit the experimental data as well as the GAB model.

$$M = \frac{A*B*C*ERH}{(1-B*ERH)(1-B*ERH+B*C*ERH)} \quad (1)$$

where M = moisture content (dry basis), ERH = equilibrium moisture content (decimal), and A,B,C are constants of the model.

Table 2. Density, particle size and color characteristics of wheat DDGS sourced in Oct 2008 from a Saskatchewan fuel ethanol plant. Number in parenthesis is standard deviation (n = 9)

% Moisture content, wet basis	Bulk density, kg/m^3	Particle density, kg/m^3	% Porosity	Geometric mean diameter, mm	Color parameters		
					L	a	b
6.9	356.5 (3.6)	1211.1 (62.5)	70.5 (1.6)	0.59 (0.03)	32.68 (0.45)	8.83 (0.13)	13.44 (0.22)
11.0	358.2 (6.0)	1197.1 (65.6)	69.8 (1.8)	0.61 (0.03)	33.31 (0.57)	8.86 (0.34)	13.34 (0.05)
14.7	368.2 (9.4)	1233.6 (52.7)	70.1 (1.9)	0.62 (0.03)	32.51 (0.56)	9.04 (0.15)	13.48 (0.08)

Table 3. Density, particle size and color characteristics of wheat DDGS sourced in June 2010 from a Saskatchewan fuel ethanol plant. Number in parenthesis is standard deviation (n = 6)

% Moisture content, wet basis	Bulk density, kg/m^3	Particle density, kg/m^3	% Porosity	Geometric mean diameter, mm
5.5	344.3 (2.3)	1297.6 (3.3)	73.5 (0.2)	
7.7	358.9 (2.5)	1296.1 (4.6)	72.3 (0.2)	
12.4	424.1 (4.7)	1345.8 (8.3)	68.5 (0.3)	0.45 (0.01)

Table 4. Friction properties of wheat DDGS sourced in Oct 2008 from a Saskatchewan fuel ethanol plant. Number in parenthesis is standard deviation (n = 9)

% Moisture content, wet basis	Coefficient of static friction				Angle of repose, degrees	
	Steel	Concrete	Wood, parallel to grain	Wood, perpendicular to grain	Side emptying	Top filling
6.9	0.33 (0.01)	0.49 (0.01)	0.59 (0.02)	0.62 (0.01)	38.4	33.9
11.0	0.44 (0.02)	0.51 (0.01)	0.59 (0.02)	0.63 (0.02)	40.7 (2.0)	33.2 (1.5)
14.7	0.49 (0.02)	0.54 (0.02)	0.61 (0.02)	0.64 (0.02)	46.7 (1.6)	34.7 (1.1)

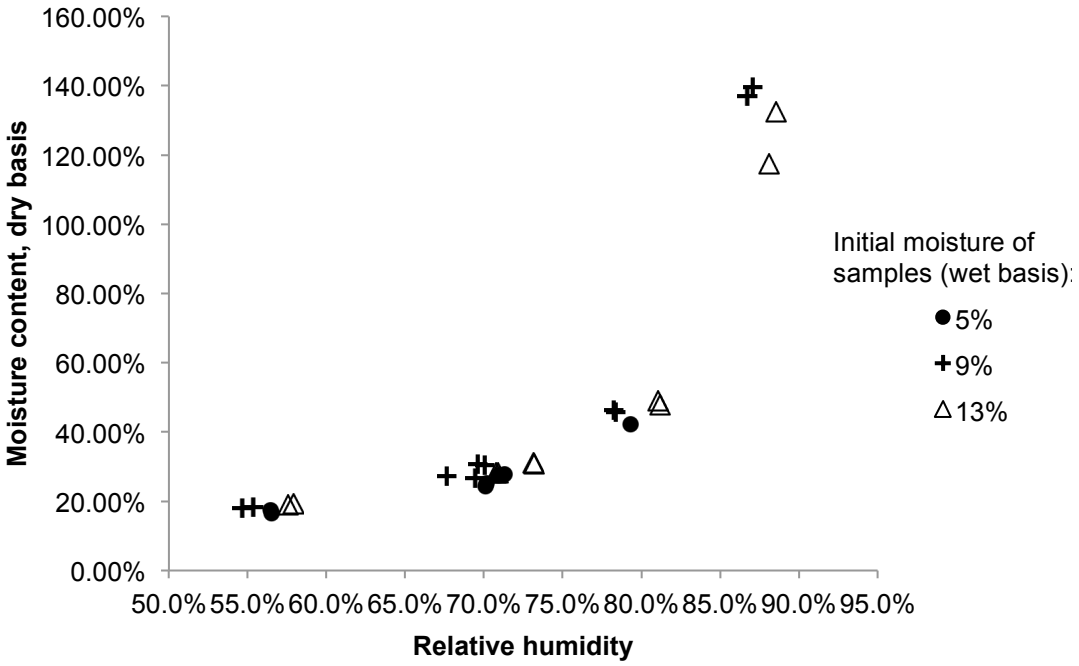

Figure 2. Moisture adsorption characteristics of wheat DDGS with varying levels of initial m
(5%, 9%, 13% wet basis) exposed to different relative humidity levels at 3°C.
90% relative humidity using samples with 5% initial moisture (wet basis) are still or

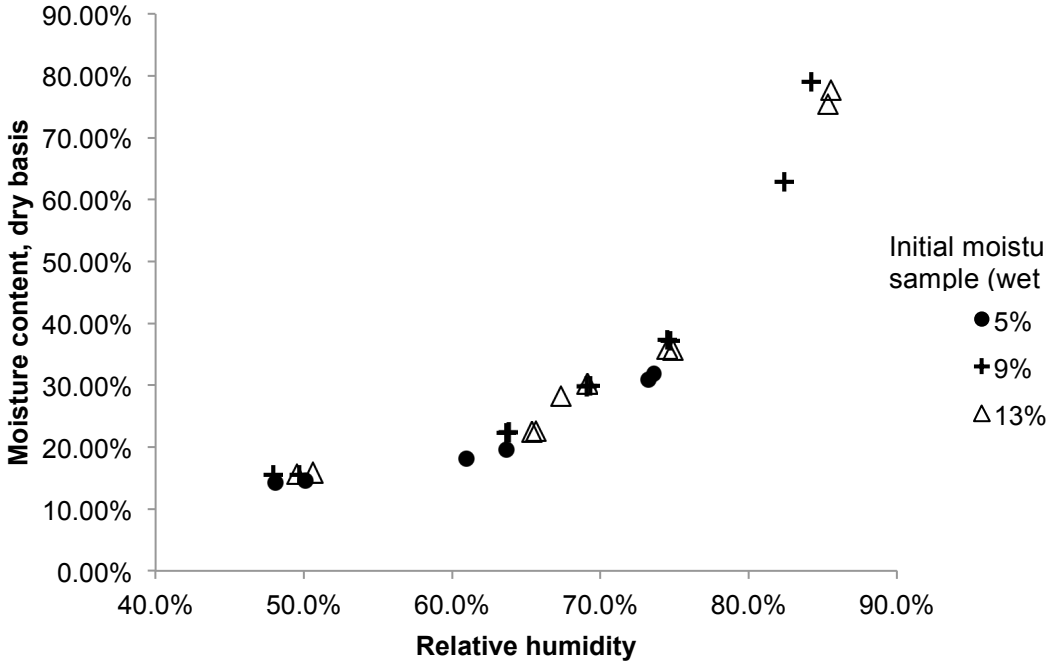

Figure 3. Moisture adsorption characteristics of wheat DDGS with varying levels of
moisture (5%, 9%, 13% wet basis) exposed to different relative humidity levels at 3°
at 90% relative humidity using samples with 5% initial moisture (wet basis) are still c

Table 5. GAB model parameters for wheat DDGS exposed to 3°C and 23°C environments at varying humidity levels

Temperature °C	Initial moisture of DDGS, % wet basis	Model parameters			R^2	MSE
		A	B	C		
3	9	0.065	1.096	-6.964	0.998	0.001
	13	0.058	1.079	-2.711	0.999	0.000
23	9	0.072	1.077	-40.582	0.997	0.000
	13	0.080	1.077	11.41	0.996	0.000

SUMMARY AND CONCLUSIONS This paper presented the initial results of an ongoing study aimed at investigating the physico-chemical characteristics of wheat DDGS and their practical industry implications in industry. Sourced from two production batches from a south Saskatchewan fuel ethanol plant, variations in chemical composition, bulk and particle density and particle size and size distribution were observed. Friction properties increased with moisture content. Moisture sorption characteristics were adequately described by the GAB model, with higher moisture adsorption observed in wheat DDGS exposed in a 3°C environment at various humidity levels compared to those in the 23°C environment.

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