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Color and compositional characteristics of wheat distiller's grain with solubles prepared from varying WDG:CDS blends and dried under three methods

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ABSTRACT This paper presents the initial results of an ongoing study aimed at assessing the effect of the different drying parameters and CDS levels on handling and nutritive quality of wheat DDGS, a co-product of fuel ethanol production primarily utilized as animal feed ingredient. Wheat distiller's grain with solubles, prepared at 15, 30, and 45% condensed distiller's solubles (CDS) levels, were dried using forced air, microwave, and microwave convection methods. Proximate composition analyses were conducted using AOAC official methods. Lysine content was also determined using the Waters AccQ•Tag method. Color was determined using a HunterLab spectrocolorimeter. Increasing the CDS levels increased protein and ash content but decreased fibre and fat content. No significant difference in the chemical composition was observed under the microwave and microwave convection methods. Under the forced convection method, there was a significant difference in ADF content across the temperature settings. Average lysine content of 30% and 45% CDS samples dried under this method showed negative linear correlations with drying air temperature, and with the color parameters a and b, as well as positive correlations with the color parameter L (lightness).

Keywords: wheat DDGS, distiller's dried grain with solubles, drying characteristics, wet distillers grain, condensed distillers solubles, physical properties, chemical composition

INTRODUCTION An energy-intensive drying process and inconsistent product quality are among two of the challenges currently confronting the supply of distiller's dried grain with solubles (DDGS), which is primarily utilized as an alternative animal feed ingredient.

While drying is an essential operation in DDGS production, conventional heated air drying is time consuming and requires high operating temperatures (Tang and Cenkowski 2001; Tang et al. 2005), accounting about one-third of the energy consumption of ethanol plants (Murphy and Power 2008). The excessively high temperatures used during the process also negatively impact the product's nutritional quality, particularly the availability and digestibility of lysine, an essential amino acid in animal diet (Ergul et al. 2003; Batal and Dale 2006; Fastinger et al. 2006; Widyaratne and Zijlstra 2007; Lan et al. 2008; Świątkiewicz and Koreleski 2008). This highlights not only the need for an energy-saving drying technology but also one that can preserve the nutritional quality of this alternative animal feed ingredient.

Further, inconsistent DDGS quality also hampers its marketing and use as an animal feed ingredient. Clementson et al (2009), for example, indicated that animal nutritionists are reluctant to use corn DDGS as an alternative feed ingredient because of nutrient composition variation across sources. Several studies on corn DDGS observed significant product variability between production batches in an ethanol plant and between ethanol plants in the United States (Knott et al. 2004; Rosentrater 2006; Bhadra et al. 2007; Liu 2008). The blending proportions of CDS and wet distiller's grain (WDG) used during DDGS production had been identified as one of the possible causes for its physical and chemical composition variability. In corn DDGS, Kingsly et al (2010) reported that incorporating higher levels of CDS result to: (1) a darker product since more reducing sugars are available to react with proteins (Maillard reaction) during the drying process; (2) increased particle size because CDS acts like a binding agent and induces particle agglomeration; and (3) decreased protein content but increased mineral content.

Considering these, a research project focusing on wheat DDGS has been conducted, whose main objective was to examine the effect of alternative drying methods and variation in the CDS:WDG blending proportions on its physico-chemical characteristics. The use of microwave-assisted methods was also investigated because studies showed these could offer shorten drying times without significant loss of product quality with proper selection of process variables (Chung and Furutan 1989; Sosyal 2004; Du et al 2005). This paper presents the initial results from this ongoing study.

MATERIALS AND METHODS CDS and WDG materials were obtained from a south Saskatchewan fuel ethanol plant in Feb 2010. They were placed in tightly sealed bins, and stored in a -16°C freezer until these were used. Batches of wet distiller's grain with solubles (WDGS) were prepared from these materials at 15, 30, 45% CDS levels (by mass). Each batch (500 or 1000 g) was prepared by mixing the WDG and CDS using a Toastmaster 6-speed Hand Mixer (model 1776CAN, Toastmaster Inc., China) for a set time period (30 or 60 min, depending on the total mass used). Unused WDGS were also stored in the freezer using plastic bags.

***Drying the WDGS*:** Prior to the thin layer drying runs, bags of frozen WDGS were thawed overnight and were placed in the sample preparation room for equilibration with room temperature (22C). About 100 g of WDGS were used for each drying run and the sample was dried until its moisture reached between 8 – 12% (wet basis). Three drying methods were used: forced air convection, microwave, and microwave-convection.

For forced air drying, an air recirculating drying system was used, which consists of an air conditioning unit equipped with humidity and temperature sensors, a vane-axial circulating fan, a drying chamber with three wire, scale-mounted trays, and a duct system. The temperature, humidity, velocity and weight measuring devices were connected to a computer equipped with LabView 8.2 (National Instruments, Austin, TX) software for easy data capture. Drying air temperature was set at three levels (40, 80, 120°C) while air velocity and relative humidity were set at 0.7 – 0.8 m/s and below 8%, respectively.

A Panasonic® microwave/convection oven (model NNC980W, Panasonic Canada, Ltd., Missisauga, ON) was used, with four power levels (40%, 60%, 80%, and 100% microwave power) for microwave and four combination settings (130°C-30% power, 150°C-30% power, 160°C-30% power, and 190°C-30% power) (Matshushita Electric Industrial Co., 2000) for microwave-convection drying. The

Moisture content of both wet and dried samples was determined using the AOAC Official Method 920.36 (AOAC, 2003).

Proximate composition: Wet samples (WDGS, CDS, and WDG) were freeze dried using the Labconco Freeze Dry System/Free Zone (Labconco Corporation, Kansas City, MO). Freeze-dried samples, those generated from the three drying methods described above, and DDGS samples directly sourced from the ethanol plant were ground using a Thomas-Wiley mill (Thomas Scientific, Swedesboro, NJ) equipped with a 1 mm screen in preparation for chemical composition analysis. The proximate composition of DDGS samples directly obtained from the ethanol plant was determined for comparison purposes.

Crude protein was estimated using the Kjeldahl method, AOAC Official Method 984.13 (AOAC, 2003). Fat was determined using the Goldfisch fat extractor (Labconco Corporation, Kansas City, MO), following the AOAC Official Method 920.39 (AOAC, 2003) with anhydrous diethyl ether as extraction solvent. Mineral matter was determined using AOAC Official Method 942.05 (AOAC, 2003), while acid detergent fibre and neutral detergent fiber were estimated through AOAC Official Method 973.18 (AOAC, 2003) and through the method laid out by Van Soest et al (1991), respectively. A lamb starter feed sample (AAFCO 0728) was used as control during the proximate analysis runs.

Total starch, using the Megazyme total starch assay procedure - amyloglucosidase/α-amylase method (Megazyme International Ltd., Wicklow, Ireland), acid detergent- and neutral detergent insoluble crude protein, using the Kjeldahl method, are yet to be conducted.

Lysine content determination: Freeze-dried samples and the DDGS samples generated from the three drying methods were sent to the laboratory of MCN BioProducts, Inc. (Saskatoon, SK, Canada) for amino acid analysis using the Waters AccQ•Tag method (D. Culbert, senior research assistant, personal communication, 13 January 2010). The method involves hydrolysis and dilution, derivatization of amino acids, and subsequent separation and analysis using an HPLC system (Waters Corp. 1996). AccQ•Fluor™ derivatization buffer and AccQ•Fluor reagent (Waters Corporation, Milford, MA) were used during the derivatization step (Waters Corp. 1996). Two duplicate tests were made from each sample and the average lysine content was reported.

Color parameters: Color of the ground samples was determined using the HunterLab spectrocolorimeter (Hunter Associates Laboratory Inc, Reston, VA) and was expressed in terms of the L, a, and b parameters. Color was determined before the lysine and proximate composition runs were conducted.

RESULTS AND DISCUSSION Figure 1 shows the proximate composition of CDS and WDG, the two components making up wheat DDGS. The CDS sample showed a higher protein and ash content while the WDG had higher fibre and fat content. Thus, increasing the CDS level during blending would increase protein and mineral content and decrease fibre and fat content of the resulting WDGS. This trend was shown in the WDGS samples that were produced at 15, 30, 45%

CDS levels and were freeze-dried. Figure 2 shows that the protein and ash content of the freeze-dried samples increased as the CDS level was increased. Fibre content, on the other hand, decreased with increasing CDS levels.

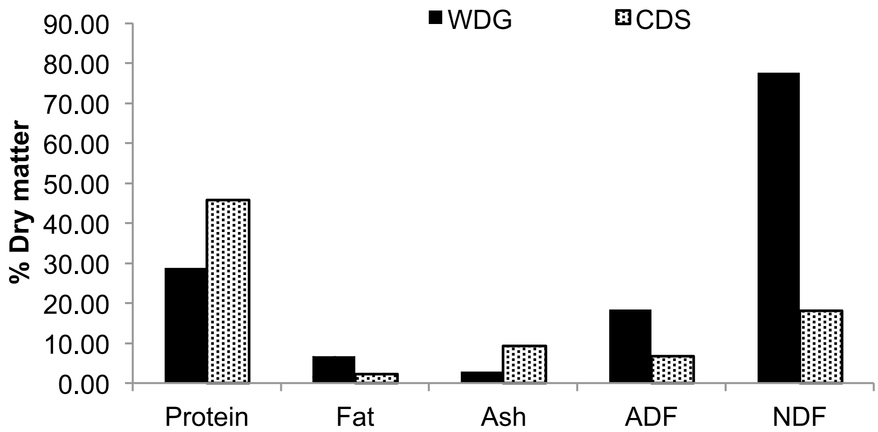

Figure 1 Proximate composition of wet distiller's grain (WDG) and condensed distiller's solubles (CDS) obtained from a Feb 2010 production batch of a south Saskatchewan wheat-based fuel ethanol plant. Analyses were done in duplicate runs.

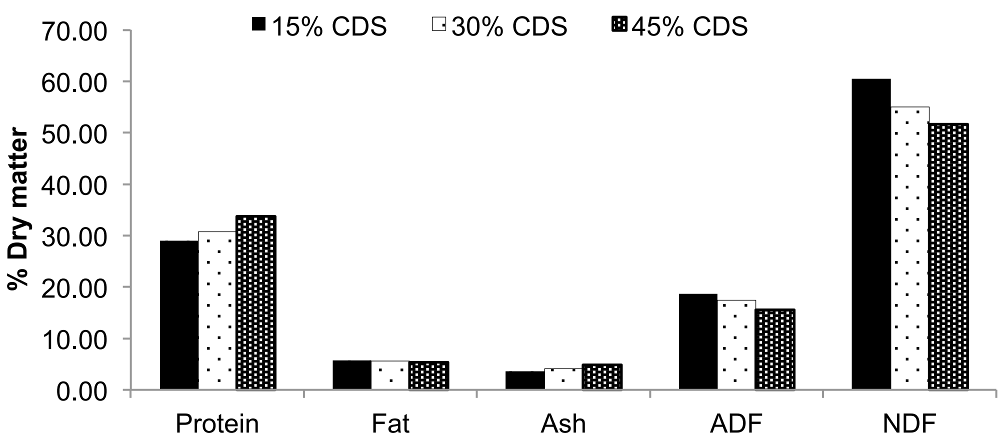

Figure 2 Proximate composition of laboratory-blended, freeze-dried DDGS produced with 15, 30 and 45% CDS levels. Samples were obtained from a Feb 2010 production batch of a south Saskatchewan wheat-based fuel ethanol plant. Analyses were done in duplicate runs.

These observations are quite different from those reported for corn DDGS. Kingsly et al (2010), for example, reported that increasing CDS level during blending with WDG resulted to reduced protein, ADF, and NDF content while ash and fat increased. The proximate composition of corn CDS and WDG, as reported by Kingsly et al (2010), is reflected in Figure 3 for comparison.

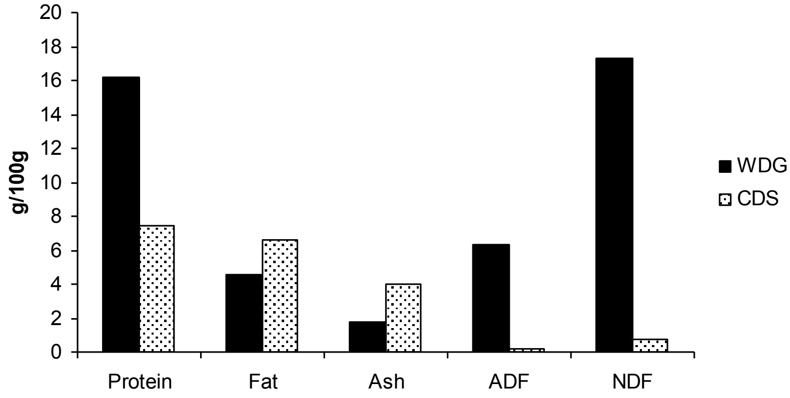

Figure 3 Proximate composition of corn wet distiller's grain (WDG) and condensed distiller's solubles (CDS), as reported by Kingsly (2010).

Figure 4 shows the proximate composition of ethanol plant-generated wheat DDGS, highlighting the variation between the two samples. This variation could be attributed to a number of factors, including the raw materials used, variations in the processing conditions, and the level of CDS incorporation. Comparing the proximate composition in Figure 4 with that of Figure 2 seems to indicate that the ethanol plant could be incorporating CDS at higher levels than what was done in the study.

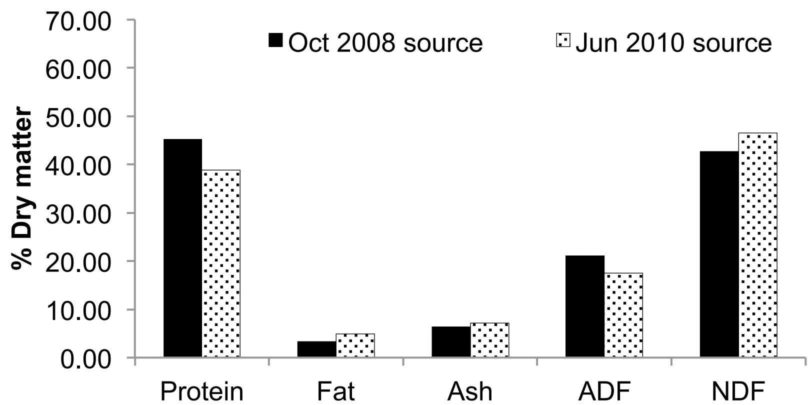

Figure 4 Proximate composition of DDGS directly sourced from an Oct 2008 and a Jun 2010 production batches of a south Saskatchewan wheat-based fuel ethanol plant. Analyses were done in duplicate runs.

Figures 5 and 6 summarize the proximate composition of wheat DDGS samples that were blended at varying CDS levels and dried under forced convection, microwave, and microwave convection methods.

In general, the values of the different chemical constituents (protein, fat, ash, NDF, ADF) were significantly different ($p<0.05$) across the CDS levels, under the three drying methods investigated. Protein (Figure 5a) and ash content (Figure 5b), for example, increased with increasing CDS levels, regardless of the drying method. Also, higher fat content tend to be found in the 15% CDS blends (Figure 5c). NDF (Figure 5d) showed decreasing values as CDS level was increased. ADF (Figure 6a) also showed a similar trend. These are attributed to differences in the chemical composition of CDS and WDG, the two components making up wheat DDGS.

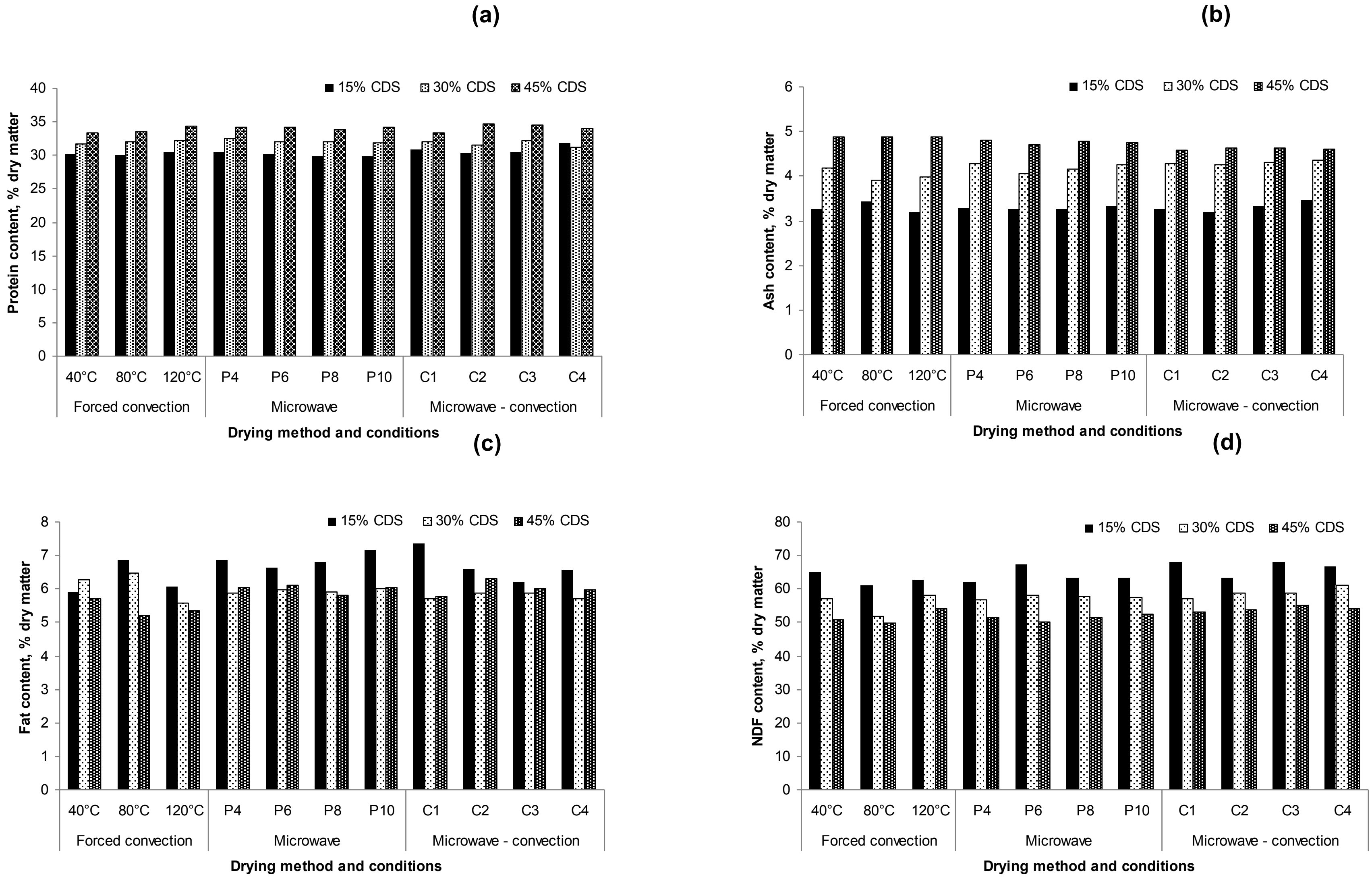

Figure 5. Protein (a), mineral (b), fat (c), and neutral detergent fibre (ADF) (d) content of wheat DDGS produced at varying CDS levels and dried under forced convection, microwave, and microwave convection methods. (Drying conditions P1-P4 and C1-C4 stand for the following: P4 – 40% power (420 W); P6 – 60% power (676 W); P8 – 80% power (701 W); P10 – 100% power (805 W); C1- 130°C, 30% power (303 W); C2-150°C, 30% power (316 W); C3 - 160°C,30% power (326 W); and C4-190°C, 30% power (332 W).) Duplicate runs were made for each test.

(a)

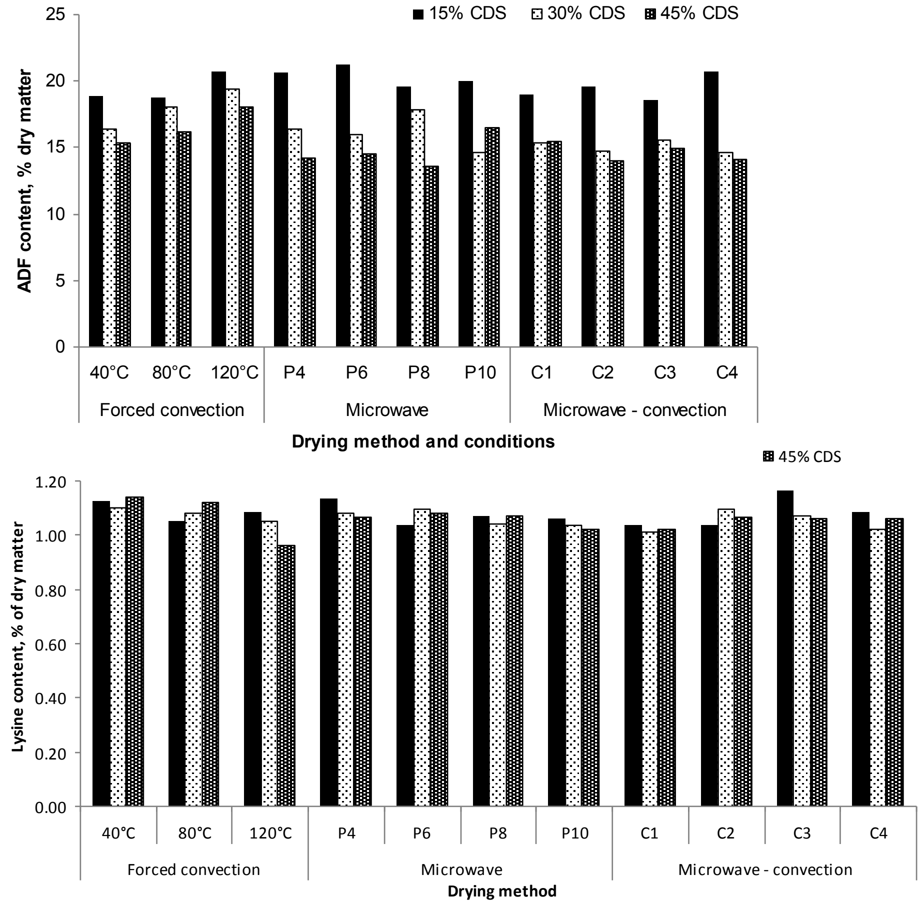

Figure 6. Neutral detergent fibre (a) and lysine (b) content of wheat DDGS produced at varying CDS levels and dried under forced convection, microwave, and microwave convection methods. (Drying conditions P1-P4 and C1-C4 stand for the following: P4 – 40% power (420 W); P6 – 60% power (676 W); P8 – 80% power (701 W); P10 – 100% power (805 W); C1- 130°C, 30% power (303 W); C2-150°C, 30% power (316 W); C3 - 160°C,30% power (326 W); and C4-190°C, 30% power (332 W).) Duplicate runs were made for each test.

Except for ADF under forced convection drying, there was no significant difference found in the values of the different chemical constituents with respect to the different drying conditions under each of the drying methods. However, it is worthwhile to point that some protein denaturation may have occurred during drying but may not have been reflected in the protein content measurement. Also, drying air temperature may affect the fat content of the samples. In Figure 5c, for example, fat content noticeably decreased in the 15% and 30% CDS samples when drying air temperature increased from 80°C to 120°C under the forced convection method.

Effect of CDS levels and drying conditions on lysine content. For the freeze-dried samples, lysine content of the 45% CDS sample was higher than at 15% and 30% CDS levels. This is attributed to the higher protein content of CDS compared to, as previously indicated, has higher protein content than WDG, thus, blending at higher CDS levels would expectedly produced WDGS with higher protein content.

Figure 6b shows the average lysine content of wheat DDGS produced by blending varying CDS levels and drying under forced convection, microwave, and microwave convection methods. Two-factor analysis of variance (without replication) showed that there is no significant difference in lysine content across CDS levels and across drying conditions under each method.

However, a close examination of the lysine content of samples dried under the forced convection method showed negative linear correlations with drying air temperature at 30% (r = -0.95) and 45% (r = -0.91) CDS levels. While negative linear correlations were found between microwave power and lysine content across the three CDS levels, these were also not significant at the 0.05 level.

Lysine and color correlation Under forced convection drying, lysine content of samples with 30% and 45% CDS content showed positive linear correlations of 0.76 and 0.99, respectively, with the color parameter L. At these CDS levels, higher lysine content tends to be found in lighter colored samples. For the 15% CDS samples, however, a negative linear correlation (r = -0.82) with L was observed. In terms of the color parameter *a*, the 30% and 45% CDS samples showed very good negative linear correlations (-0.90 and -0.999). As the samples became redder, lower lysine contents were observed. Negative linear correlations between lysine content and the color parameter *b* (-0.93, -0.54, and -0.84) were, respectively, observed in the 15%, 30%, and 45% CDS samples.

Other than the negative linear correlations (-0.73, -0.93) between the color parameter *b* and lysine content for the respective 30% and 45% CDS microwave-dried samples, there was no other distinct pattern observed between lysine content and the color parameters in the microwave- and microwave-convection dried samples.

Effect of CDS levels and drying conditions on color. Initial results on color evaluation of freeze-dried samples showed that with increasing CDS level, the *L* color parameter tend to decrease whereas the *a* and *b* parameters tend to increase. Increasing the CDS level darkens the resulting blend, as CDS is darker (L = 45.7, a = 5.3, b = 16.9) than the WDG (L = 52, a = 3.2, b = 15.6).

Table 1 shows the various color parameters obtained from the dried, ground samples before these were sent for lysine analysis. Under forced convection drying, samples dried at 120°C, across the three CDS levels, tend to be darker compared to those dried at 40°C. The values of the color parameter *a* (redness), in particular, showed significant difference across the temperature settings. No significant difference was observed in the color parameters across the CDS levels at the temperature settings used.

Under the microwave-convection drying, there was no significant difference in the color parameters across the four combination settings but there was a significant difference observed across the CDS levels. Samples at higher CDS levels showed lower *L* and higher *a* and *b* values compared to those with lower CDS content.

There was also no significant difference observed in the color parameters *L* and *a* in the microwave-dried samples across microwave power and CDS levels. The values of the color parameter *b* (yellowness), however, showed significant difference across the two factors assessed.

SUMMARY Varying the CDS levels during blending with WDG affected the chemical composition and color characteristics of wheat DDGS. Increasing the CDS levels increased protein, and ash content while decreased fat and fibre content. It also increased the values of the color parameters a (redness) and b (yellowness) while it decreased the values of the L parameter (lightness).

While there were no significant differences observed in the compositional characteristics under the various conditions of each drying method, other than ADF content of forced convection-dried samples, some protein denaturation may have occurred. Also, fat content may also be affected with increasing drying air temperature.

Average lysine content of DDGS produced at 30% and 45% CDS levels under the forced convection method showed negative linear correlations with drying air temperature. Correlation coefficients were -0.95 and -0.91, respectively. No discernible trend was observed between the drying parameters and lysine content under the microwave- and microwave-convection methods. Further, at 30% and 45% CDS levels, lysine content was positively correlated with the color parameter *L* and negatively correlated with the color parameters *a* and *b*.

Lastly, 15%, 30% and 45% CDS levels used in this study were still lower compared to what was used in the Saskatchewan ethanol plant, where the DDGS, CDS and WDG samples were sourced. This was based on the comparative proximate composition of the laboratory produced DDGS and the DDGS sourced from the ethanol plant.

Table 1. Color parameters of ground DDGS samples prepared with varying CDS levels and dried using different methods.

Drying method	Nominal setting*	15% CDS			30% CDS			45% CDS		
		L	a	b	L	a	b	L	a	b
Forced convection	40	42.10	5.85	13.60	44.31	5.83	14.02	41.43	6.07	13.43
	80	45.88	5.85	14.70	45.14	5.83	14.36	41.46	6.21	13.85
	120	41.96	6.13	13.84	42.07	6.34	14.23	40.75	7.02	14.10
Microwave	P4	41.54	6.08	13.60	42.68	6.27	14.30	41.17	6.24	13.54
	P6	41.65	5.91	13.24	41.45	6.19	13.56	39.96	6.23	12.96
	P8	41.31	5.93	13.45	41.12	6.71	14.28	39.22	6.78	13.72
	P10	42.37	5.98	13.84	41.91	6.53	14.29	41.88	6.66	14.48
Microwave - convection	C1	41.80	5.87	13.40	42.47	6.45	14.60	40.72	6.32	13.50
	C2	41.91	5.91	13.54	42.40	6.37	14.01	40.60	6.71	13.85
	C3	41.95	6.08	13.48	42.14	6.62	14.36	41.81	6.23	13.62
	C4	41.46	6.11	13.90	42.71	6.35	14.59	39.49	6.37	13.22

*P4 – 40% power (420 W), P6 – 60% power (676 W), P8 – 80% power (701 W), P10 – 100% power (805 W)
C1- 130°C, 30% power (303 W), C2-150°C, 30% power (316 W), C3 - 160°C,30% power (326 W), C4-190°C, 30% power (332 W).

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