Effects of condensed distillers solubles and drying temperature on the physico-chemical characteristics of laboratory-prepared wheat distillers grain with solubles

Maria Rosario P. Mosqueda[1], Lope G. Tabil[1*], Kasiviswanathan Muthukumarappan[2]

(1. *Department of Chemical and Biological Engineering, University of Saskatchewan, Saskatoon, SK S7N 5A9, Canada*;
2. *Department of Agricultural and Biosystems Engineering, South Dakota State University, Brookings, SD 57007, USA*)

Abstract: Samples of wheat distillers grain with solubles were prepared at 15%, 30%, and 45% condensed distillers solubles (CDS) and dried under 40°C, 80°C, and 120°C to examine the effects of CDS level and drying temperature on their chemical, physical, flow, compression, thermal, and frictional properties. As CDS level increased, protein and ash contents increased while fat and fiber contents decreased. Fat and acid detergent fiber contents were also markedly affected by drying temperature. While CDS level, drying temperature, and their interaction significantly affected a number of the physical properties, results suggest that CDS level had a stronger influence. Samples with high CDS level, for example, were significantly finer, denser, less flowable, and less dispersible than those with lower CDS. These samples also had significantly higher thermal diffusivity and coefficient of internal friction and produced pellets with higher failure stresses than those with lower CDS. Their pellet density increased with CDS level and was also significantly affected by drying temperature. Further, the samples were classified as fairly flowable and floodable and their compression characteristics were adequately described by the Kawakita-Ludde model.

Keywords: condensed distillers solubles (CDS), chemical composition, distillers dried grain with solubles (DDGS), physical properties, wheat distillers grain
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1 Introduction

Product variability is one of the challenges that currently confront wheat distillers dried grain with solubles (DDGS) production in western Canada[1]. Nuez-Ortin[2], for example, found significant nutrient variability in wheat DDGS samples obtained from two Saskatchewan plants and stressed the importance of product consistency not only in formulating more accurate feed rations but also in improving the market prospects of wheat DDGS. Blending of condensed distillers solubles (CDS) and wet distillers grain (WDG)

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Biographies: Maria Rosario P Mosqueda, PhD candidate, 57 Campus Drive, Saskatoon, SK S7N 5A9, Canada. Email: charie.mosqueda@usask.ca. **Kasiviswanathan Muthukumarappan**, Distinguished Professor and Graduate Coordinator; Email: Kas.Muthukum@sdstate.edu.
***Corresponding author: Lope G Tabil,** PhD, Professor, Department of Chemical and Biological Engineering, University of Saskatchewan, 57 Campus Drive, Saskatoon, SK S7N 5A9 Canada. Research interests: Pelleting and Binding Characteristics of Alfalfa. process involved in feed and forage processing, physical properties of agricultural materials and postharvest technology of agricultural crops. The areas of research in which he works and maintains interest include bioprocess engineering, value-added engineering and postharvest handling of crops. He has also conducted applied research and development projects related to value-added processing of agricultural products including storage, drying and cooling, and physical properties of agricultural and biological materials. Dr. Tabil continues to work and has interest in projects such as agricultural waste and processing byproduct utilization and the processing of agricultural materials into industrial products. Email: lope.tabil@usask.ca.

has been identified as among the major causes of variation in the physico-chemical characteristics in both corn and wheat DDGS. While a number of laboratory[3-8] and plant[9-10] scale investigations had studied its effect on the physico-chemical characteristics of corn DDGS, there is still very limited information on how CDS:WDG blending proportion affects wheat DDGS properties.

In a laboratory-scale investigation, Ganesan et al.[3] reported that soluble level and moisture content had significant effects on protein content, color, aerated and packed bulk densities, compressibility, dispersibility, flowability, and floodability of corn DDGS. As soluble level increased, protein content, and the color parameter L decreased while the color parameter increased. They also reported significant differences in particle size as soluble level was varied but they did not observe distinct trends. In their study of the flow properties of laboratory-scale produced corn DDGS using the Jenike shear tester, they also reported significant soluble and moisture content effects but did not observe any definitive trend[4]. These two studies defined "solubles" as the "non-water portion of CDS that passed through a filter media". Bhadra et al.[5-8] also investigated the effect of CDS level (10%, 15%, 25% w.b.), drying temperatures (100°C, 200°C, 300°C), and cooling temperatures (-12°C, 25°C, 35°C) on the drying kinetics, glass transition temperature, and on physical and flow characteristics of corn DDGS produced in the laboratory-scale. They reported significant main and interaction effects on most of the physical and flow properties[5]. Compared to CDS level, drying temperature was seen to be the more important factor that affected drying rate[7] and flowability[5,7]. Higher drying temperature increased drying rate and resulted to better flowability. Cooling temperature levels did not produce significant differences on five important flow properties[5].

Kingsly et al.[9] and Clementson and Ileleji[10] also conducted plant-scale investigations on this subject. Both these corn DDGS studies reported increase in particle size, bulk density, and particle density as CDS level was increased. They attributed the increase in particle size to the agglomeration of smaller particles with CDS acting as a binding agent[9,10]. Kingsly et al.[9] further reported that as CDS level increased, the color parameter, fat, ash, sugars, and glycerol content, increased while the color parameter L, crude protein, acid detergent fiber (ADF), and neutral detergent fiber (NDF), on the other hand, decreased. Amino acids lysine, methionine, threonine and tryptophan also decreased, indicating higher concentration of amino acids in the WDG than in the CDS component[9]. Clementson and Ileleji[10] also reported that particle porosity, pore volume and effective bulk porosity decreased as CDS level increased[10]. They did not find significant correlation between CDS level and the shape functions.

Inherent compositional differences between corn and wheat kernels and subsequently between their corresponding CDS and WDG fractions (Table 1) necessitate separate investigations for wheat DDGS. Corn-based CDS, for example, had lower protein and fiber but higher ash and fat content compared to the WDG fraction. In contrast, wheat-based thin stillage, from which the CDS component is derived, showed higher protein and ash but lower fat content compared to the WDG fraction[11,12]. These compositional differences could translate to differences in physico-chemical characteristics. The overall objective of this study, therefore, was to examine the effect of CDS:WDG blending proportion on the physico-chemical characteristics of laboratory-prepared wheat DDGS.

Table 1 Comparative chemical composition of corn and wheat grain, wet distillers grain (WDG), thin stillage, and condensed distillers solubles (CDS), % dry matter basis

Sample	Crude protein	Ash	Crude fat	NDF[a]	ADF[b]
Corn-based					
Grain[2]	10.13	1.73	4.59	14.47	3.66
WDG[13,14]	34.4-35.0	2.0-2.35	10.9-14.0	39.0	11.9-17.2
CDS[13,15]	22.4-23.0	6.6-11.6	21.6-34.4	3.6-4.9	2.92
Wheat-based					
Grain[2]	14.28	2.12	1.91	17.22	3.68
WDG[12]	26.4	2.7	6.6	74.9	24.1
Thin stillage[11,16]	45.7-48.5	8.0-8.3	9.63-13.6	34.0-34.5	3.4-4.0
Wet distillers solubles[17]	32.6	8.9	5.7	2.3	

Note: [a]NDF means neutral detergent fiber; [b]ADF means acid detergent fiber.

2 Materials and methods

CDS and WDG were obtained from a south Saskatchewan fuel ethanol plant, placed in tightly sealed bins, and stored in a -16℃ freezer. Five hundred grams to one kilogram batches of wet distiller's grain with solubles (WDGS) were prepared by mixing thawed CDS and WDG at three ratios, by mass: 15:85, 30:70, and 45:55 using a 6-speed Toastmaster hand mixer (model Toastmaster Inc., China) for 15 to 30 minutes at medium speed. These blends are subsequently referred to in this paper as 15%, 30%, and 45% CDS, respectively. Prepared WDGS were placed in sealed plastic bags and stored in the -16°C freezer until these were used.

2.1 Drying the WDGS

Bags of frozen WDGS were thawed overnight and were placed in the sample preparation room for equilibration with room temperature (22-24°C) prior to the thin layer drying runs. Samples were dried using the forced air convection method until moisture content reached 8% (w.b.). The drying system consisted of an air conditioning unit equipped with humidity and temperature sensors, a vane-axial circulating fan, a drying chamber with three wires, scale-mounted trays, and a duct system. Kashaninejad and Tabil[18] and Kashaninejad et al.[19] provided a detailed description of the drying system. Drying air temperature was set at three levels (40°C, 80°C, 120°C) while air velocity and relative humidity were set at 0.7-0.8 m/s and below 8%, respectively. The temperature, relative humidity, air velocity and weight monitoring devices of the drying system were connected to a computer installed with LabView 8.2 (National Instruments, Austin, TX) software for data capture. Although the temperature levels used in this study were considerably lower than those employed in ethanol plants because of laboratory equipment constraints, a previous study had indicated that protein quality changes were already detected at these levels[20]. Thus, changes in other physico-chemical characteristics could also be discernible at these levels.

2.2 Chemical composition

The chemical composition of both wet and dried samples was determined. Wet samples (WDGS, CDS, and WDG) were freeze-dried using the Labconco FreeZone Freeze Dry System (Labconco Corp., Kansas City, MO) prior to the proximate analysis runs. All dried samples were ground using a Thomas-Wiley knife mill (Thomas Scientific, Swedesboro, NJ) equipped with a 1.0 mm screen.

Moisture content was determined using the AOAC Official Method 920.36[21]. Crude protein was estimated using the Kjeldahl method, AOAC Official Method 984.13[22]. Crude fat was determined using the Goldfisch fat extractor (Labconco Corporation, Kansas City, MO), following the AOAC Official Method 920.39[23], with anhydrous diethyl ether as extraction solvent. Crude ash was determined using AOAC Official Method 942.05[24], while ADF and NDF were estimated through AOAC Official Method 973.18[25] and through the method laid out by van Soest et al.[26], respectively. A lamb starter feed sample (AAFCO 0728) was used as a check sample. The proximate analysis was conducted in duplicate runs.

2.3 Physical properties

Dried samples were passed through a Thomas-Wiley mill (Thomas Scientific, Swedesboro, NJ), in batches of 150-200 g for two minutes to generate the bulk material. The mill was equipped with a 2.68 mm screen, a size about four times larger than the observed geometric mean diameter of commercial wheat DDGS samples. Density, particle size and size distribution, flow, compression, frictional, and thermal properties of forced-air convection-dried samples were determined. Property measurement was conducted in duplicate runs, unless otherwise stated. Moisture content of samples ranged about 8%-9% (w.b.).

(1) Particle size, density, and color. Sieve sizes of 12, 20, 30, 40, 50, 60, 70, 80, 100, 140, 200, and 270 and a Ro-tap sieve shaker (model RX-29, Tyler Manufacturing, Mentor, OH) were used for particle size analysis, following ANSI/ASAE S319.4[27]. The calculated geometric mean diameter was used to represent particle size in this study.

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). Bulk porosity (ε), expressed as a percentage, was determined as a function of bulk (ρ_b) and particle densities (ρ_p) using Equation (1) below.

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \quad (1)$$

(2) Flow properties. The Carr flowability and floodability indices were determined by measuring seven properties using a Hosokawa Micron Powder Tester PT-R (Hokosawa Micron Corp., Osaka, Japan). Each property measurement was assigned an index value, ranging from 0 to 25, depending on the point score classification system developed by Carr[28]. The flowability index, which ranged from 0 (very, very poor flowability) to 100 (excellent flowability), was derived from the sum of the individual index values of compressibility, angle of repose, angle of spatula, and uniformity coefficient. The floodability index, which also ranged from 0 (will not flood) to 100 (very floodable), was derived in a similar manner, using the index values of flowability, angle of fall, angle of difference, and dispersibility. Detailed description of each flow property and its measurement was presented by Carr[28], Hokosawa Micron Corp.[29], and Ganesan et al.[3]. The Hosokawa Micron Powder Tester had been used in determining the flow properties of corn DDGS[3,4,7] and in other materials[29] with average particle sizes larger than wheat DDGS.

(3) Thermal properties. Thermal conductivity and thermal diffusivity of dried samples at ambient room condition were determined using a KD 2 Thermal Properties Analyzer (Decagon Devices, Inc., Pullman, WA). Distilled water and 15% agar solution were used as check samples. Bulk density was determined immediately before the thermal properties measurement, using the same procedure previously outlined. At low bulk densities, the volume of pores is greater, thus, the thermal conductivity would be lower because air has very low thermal conductivity. At higher bulk densities, porosity is decreased, thus, resulting in higher thermal conductivities.

(4) Compression characteristics. An Instron Model 3366 testing machine (Instron, Norwood, MA), fitted with 10 kN load cell and a 6.30 mm plunger, was used to compress 1 g samples placed inside a cylindrical die (6.35 mm diameter and 135.34 mm length) that was constantly heated at 90°C. Crosshead speed was set at 30 mm/min. When the preset compressive load of 4 400 N was reached, the plunger remained in its place for 1 min before ejecting the pellet. The test was conducted in five replicates. Force, displacement, and time data were directly logged into the computer. Experimental data generated were fitted to two models: Jones (Equation (2))[30,31] and Kawakita and Ludde (Equation (3))[31]. The most suitable model was chosen using R^2 and MSE as criteria.

$$\ln\rho = m \ln P + n \quad (2)$$

$$\frac{P}{C} = \frac{1}{df} + \frac{P}{d} \quad (3)$$

where, $C = \frac{V_0 - V}{V_0}$; ρ is bulk density; P is the applied compressive pressure; m and n are the model parameters; C is the degree of volume reduction; V_0 is the volume of compact at zero pressure; V is volume of compact at pressure P (MPa); d and f are the model parameters. Model parameter d is equal to the initial porosity of the sample while the reciprocal of the model parameter f (f^{-1}) is related to failure stress (MPa) of the pelleted sample.

Pellet density was determined by measuring the mass, length, and diameter of the resulting pellet immediately after it was extruded from the die. Specific energy consumption during compression and extrusion was estimated by calculating the area under the corresponding force-displacement curve using the trapezoidal rule and dividing it by the pellet mass.

(5) Frictional properties. The coefficient of internal friction and cohesion were determined using the Wykeham Farrance shear box apparatus (Wykeham Farrance International Ltd., Slough, U.K.), as described in Mani et al.[30] and Emami and Tabil[31]. Maximum shear stress was determined at four normal loads (200,

400, 600, and 800 N)[31]. Values for the angle of internal friction and cohesion were estimated through regression analysis of experimental data using Equation (3)[32], where τ is the shear stress (Pa), σ is the normal stress (Pa), θ is the angle of internal friction and C_0 is the cohesion (Pa).

$$\tau = \tan\theta\sigma + C_0 \quad (4)$$

2.4 Statistical analysis

The general linear model univariate procedure in SPSS 14.0 for Windows (SPSS Inc., Chicago, IL) was used to test the effect of CDS level and drying temperature. Tukey's test was used to further evaluate statistically significant main effects and interactions. Linear regression analysis was conducted to determine the relationship between CDS level and the chemical constituents of freeze-dried DDGS samples. All tests were conducted at the 0.05 significance level.

3 Results and discussion

3.1 Chemical composition

3.1.1 Freeze-dried samples

Table 2 shows the proximate composition of freeze-dried wheat WDG, CDS, and DDGS samples at varying CDS level. The CDS component had higher crude protein and ash content but lower fat and fiber content compared to WDG. Ojowi et al.[11,12] also reported similar trends, wherein thin stillage, from where the CDS component was derived, had higher crude protein and lower NDF and ADF than the WDG fraction (Table 1). Differences in the CDS and WDG composition are attributed to differences in the composition of the wheat kernel components that comprised these two fractions. The bran, which is high in fiber, predominantly comprised the WDG fraction while endosperm and germ particles made up the CDS fraction. The chemical constitution of wheat DDGS was not similar to those reported for corn DDGS[3,9,10,13] due to the basic compositional differences between corn and wheat grains and between their respective CDS and WDG fractions (Table 1).

The laboratory-prepared DDGS samples with 15%, 30%, and 45% CDS exhibited the expected trends based on the CDS and WDG composition. As CDS level in the blend increased, crude protein and ash content increased while fat, NDF, and ADF content decreased (Table 3). The corresponding coefficients of determination (R^2) reflected in Table 3 indicate that the 85%-98% of the variability in the chemical constituents of freeze-dried samples can be explained by CDS level.

Table 2 also shows the chemical composition of the plant-sourced wheat DDGS sample. The sample had higher protein and ash but lower fat and NDF content than the laboratory-prepared samples. This suggests that the plant-sourced sample was produced at a higher CDS level than what was used in the laboratory-prepared samples. The sample was obtained from the same ethanol plant on the same production date as the CDS and WDG samples.

Table 2 Chemical composition of freeze-dried, wheat-based wet distillers grain (WDG), condensed distillers solubles (CDS), and distillers dried grain with solubles (DDGS) samples. Values in parentheses represent standard deviation (N = 2).

Sample[2]	Moisture/%, w.b.		Dry matter[3] /%				
	Initial	Freeze-dried	Protein	Ash	Fat	NDF	ADF
WDG	66.82 (0.24)	16.24 (0.29)	28.84 (0.60)[a1]	2.90 (0.02)[a]	6.71 (0.04)[a]	77.53 0.24)[a]	18.39 (0.39)[a]
CDS	75.52 (0.26)	18.98 (1.93)	45.82 (0.39)[b]	9.30 (0.02)[b]	2.21 (0.04)[b]	18.14 0.29)[b]	6.66 (0.08)[b]
DDGS							
15% CDS	67.71 (0.26)	6.81 (0.31)	29.02 (0.24)[a]	3.56 (0.03)[a]	5.72 (0.13)[a]	60.45 (0.38)[a]	18.61 (0.03)[a]
30% CDS	69.60 (0.40)	8.08 (0.23)	30.70 (0.11)[b]	4.05 (0.01)[b]	5.59 (0.04)[a,b]	55.00 (0.03)[b]	17.48 (0.13)[b]
45% CDS	71.44 (1.04)	11.14 (0.25)	33.74 (0.00)[c]	4.88 (0.02)[c]	5.35 (0.01)[b]	51.62 (0.16)[c]	15.58 (0.45)[c]
Plant-sourced	13.32 (0.23)	-	38.81 (0.14)	7.10 (0.01)	4.89 (0.18)	46.55 (0.80)	17.45 (0.27)

Note: [1] Values with the same superscript letter, within each sample category, are not significantly different at the 0.05 level;

[2] Except for the plant-sourced wheat DDGS sample, all other samples were freeze-dried prior to proximate analysis;

[3] NDF means neutral detergent fiber; ADF means acid detergent fiber.

Table 3 Significant linear relationships (P = 0.05) between selected chemical constituents and condensed distillers solubles (CDS) in wheat distillers dried grain with solubles (DDGS). Samples were produced at 15%, 30%, and 45% CDS and dried under freeze drying and forced air convection methods. Values inside the parentheses are coefficients of determination (R^2).

Chemical constituent	Freeze-dried samples		Forced air convection samples	
Protein	0.16CDS + 26.44	(0.97)	0.12CDS + 28.52	(0.95)
Ash	0.04CDS + 2.84	(0.98)	0.05CDS + 2.48	(0.98)
Fat	- 0.01CDS + 5.92	(0.85)	-0.03CDS + 6.79	(0.47)
Neutral detergent fiber	-0.29CDS + 64.52	(0.98)	-0.38CDS + 68.16	(0.80)
Acid detergent fiber	-0.10CDS + 20.26	(0.96)	-0.10CDS + 20.98	(0.56)

3.1.2 Forced air convection-dried samples

Table 4 shows the chemical constituents of forced air convection-dried samples. The chemical constituents followed similar trends exhibited by the freeze-dried samples with respect to CDS level (Table 3). The protein and ash content increased while fat, NDF, and ADF decreased as CDS level increased. While the R^2 values for protein, ash and NDF content of these samples were comparable to those found in freeze-dried samples, their fat and ADF contents, however showed much lower R^2 values (Table 3). This indicates that other factors aside from CDS level had affected fat and ADF content.

Table 4 Proximate composition[1] of forced air convection-dried wheat distillers dried grain with solubles (DDGS) samples with varying condensed distillers solubles (CDS) level. Values in parentheses represent standard deviation (N = 2).

CDS, mass/%	Drying temperature/℃	Moisture, dry basis /%	Dry matter[2] /%				
			Protein	Ash	Fat	NDF	ADF
15	40	7.78 (0.18)	30.3 (0.3)^{aA}	3.3 (0.0)^{aA}	5.9 (0.0)^{aA}	65.1 (0.1)^{aA}	18.9 (0.3)^{aA}
	80	8.84 (0.03)	30.0 (0.1)^{aA}	3.4 (0.0)^{bA}	6.9 (0.1)^{bA}	61.1 (0.0)^{bA}	18.8 (0.0)^{aA}
	120	8.91 (0.02)	30.5 (0.2)^{cA}	3.2 (0.0)^{aA}	6.1 (0.1)^{aA}	62.9 (0.4)^{aA,bA}	20.7 (0.3)^{cA}
30	40	9.15 (0.19)	31.7 (0.2)^{aB}	4.2 (0.0)^{aB}	6.3 (0.0)^{aB}	57.2 (1.3)^{aB}	16.4 (0.2)^{aB}
	80	8.62 (0.05)	32.0 (0.2)^{aB}	3.9 (0.0)^{bB}	6.5 (0.0)^{aB}	51.9 (2.1)^{bB}	18.0 (0.0)^{bB}
	120	9.60 (0.08)	32.3 (0.2)^{cB}	4.0 (0.1)^{bB}	5.6 (0.0)^{cB}	58.0 (0.2)^{aB}	19.4 (0.2)^{cB}
45	40	9.65 (0.02)	33.4 (0.1)^{aC}	4.9 (0.0)^{aC}	5.7 (0.2)^{aC,aA}	50.9 (0.4)^{aC}	15.3 (0.5)^{aC}
	80	7.81 (0.00)	33.6 (0.1)^{aC}	4.9 (0.0)^{aC}	5.2 (0.0)^{bC}	49.9 (0.9)^{aC,bB}	16.1 (0.4)^{bC}
	120	7.89 (0.04)	34.3(0.1)^{cC}	4.9 (0.0)^{aC}	5.3 (0.1)^{bC}	54.2 (0.7)^{cC}	18.0 (0.2)^{cC}

Note: [1] Tukey's test at 5% significance level for the same CDS level at varying drying temperatures (a, b, c) and for the same drying temperature at varying CDS levels (A,B,C). Values followed by the same set of letters are not significantly different; [2] NDF means neutral detergent fiber; ADF means acid detergent fiber.

Fat and ADF content showed significant difference due to CDS level, drying air temperature, and their interaction (Table 4). In terms of fat content, 15% CDS samples dried under 80°C showed significantly higher fat content than those dried under 40°C and 120°C. Among 30% CDS samples, those dried under 40°C and 80°C had significantly higher fat content than those dried under 120°C. In samples with 45% CDS, those dried under 40°C contained significantly higher fat content than those dried under 80°C and 120°C. With respect to ADF content, samples at all CDS levels that were dried under 120°C showed significantly higher ADF than those dried under 40°C and 80°C. Those with 15% and 45% CDS also showed significantly higher ADF content when these were dried under 80°C than under 40°C.

Samples dried under 120°C showed lower fat and higher ADF content compared to those dried under lower temperatures. Lower fat content of the 120°C-dried samples is attributed to losses due to lipid oxidation because of elevated drying temperature. Increase in ADF under higher drying air temperature is attributed to the formation of artifact lignin as a result of Maillard reaction[33]. The effect of drying air temperature on ADF content was further highlighted when it was incorporated into the original ADF-CDS linear model (Table 3). The R^2 value of the model improved from

0.56 to 0.94 when drying air temperature was added. These increases in ADF indicate that protein quality may have been affected because of Maillard reaction, although changes in protein content were not as markedly manifested as those seen in fat content.

Overall, the level of CDS incorporation significantly affected the chemical composition of wheat DDGS. As CDS level increased, protein and ash content increased while fat and fiber content decreased, regardless of the drying methods employed. The influence of drying temperature on chemical composition was markedly manifested in the samples' fat and ADF content. At the same CDS level, drying under 120°C produced samples with significantly lower fat and higher ADF content than drying under lower temperatures. These results highlight the importance of selecting the appropriate WDG:CDS blending proportion and drying condition that will maximize its nutritive value, and thus, its market potential as an alternative feed ingredient.

3.2 Physical properties

3.2.1 Particle size and size distribution

Table 5 and Figure 1 present the average particle size and the size distribution of laboratory-prepared wheat DDGS samples, respectively, while Table 6 shows the results of the corresponding analysis of variance. Average particle size ranged from 0.52 mm to 0.79 mm (Table 5). While CDS level, drying air temperature, and their interaction significantly affected particle size, the CDS level-associated sums of squares, which comprised 74% of the total sums of squares (Table 6), suggested the stronger influence of CDS level. This is further illustrated in Figure 1, where samples with 45% CDS tended to have finer particles than those with 15% CDS. Decrease in particle size with increased CDS level is attributed to the increased presence of finer, endosperm- and germ-derived solids and decreased amount of the fibrous, bran-related particles. The effect of CDS level on particle size was still discernible even though these samples were ground after drying to generate the bulk material. The use of a screen size that was four times larger than the geometric mean diameter of the plant-sourced sample had helped curb the adverse effect of grinding.

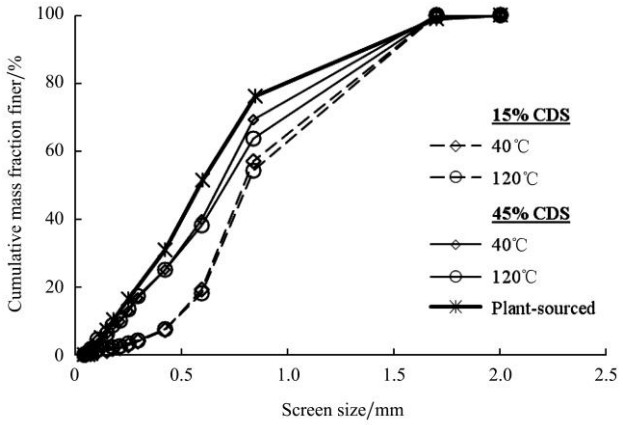

Figure 1 Average particle size distribution of plant-sourced (9%, w.b.) and laboratory-prepared (8%-9%, w.b.) wheat distillers dried grain with solubles (DDGS) samples (N = 2)

Figure 1 also shows the particle size distribution of the plant-sourced DDGS sample with moisture content similar to those of the laboratory-prepared samples. The plant sample has finer particles compared to the laboratory-prepared ones, further supporting the observation that increased CDS level in the blend tended to produce finer particles. It was previously suggested that the plant-sourced sample may have been produced at a CDS level higher than 45% (by mass), considering its chemical composition relative to what was found in the CDS and WDG fractions (Table 2). This sample was obtained from an ethanol plant that employed a ring dryer for DDGS production. Under this drying system, the WDGS is dispersed and conveyed through the dryer in a hot air stream at high speed. Thus, incidence of particle agglomeration was not a concern compared to rotary drum drying. Plant-scale studies[9,10] on corn DDGS indicated that particle size increased with CDS level due to agglomeration during rotary drum drying.

3.2.2 Density and porosity

Like particle size, CDS level, drying air temperature, and their factorial interaction had significant effect on bulk density values (Tables 5 and 6). The sums of squares associated with CDS level comprised about 93% of the total variance (Table 6), once again suggesting the dominant influence of CDS level. Under each drying air temperature, samples with 45% CDS had significantly higher bulk density values than those with 15% and 30% CDS. Similarly, those with 30% CDS had significantly higher bulk density than those with 15% CDS.

Increased CDS level in the blend translates to increased presence of finer but heavier solids and decreased amount of the larger, more fibrous particles. These finer particles would easily move through and fill the inter-particle spaces, consequently leading to a heavier bulk mass and lower porosity. In corn DDGS, Ganesan et al.[3] reported that solubles level, along with moisture content, had significant effect on aerated and tapped bulk densities but did not observe a specific trend. Particle density also increased with CDS level but was not significantly affected by drying air temperature.

Table 5 Structural properties[1] of wheat distillers dried grains solubles (DDGS) samples, produced at varying condensed distillers solubles (CDS) level and dried under forced air convection method. Values in parentheses represent standard deviation (*N* = 2).

CDS, mass/%	Drying temperature/℃	Moisture, w.b./%	Particle size/mm	Bulk density/kg m^{-3}	Particle density/kg m^{-3}	Porosity/%
15	40	8.32 (0.05)	0.79 (0.00)^{aA}	317 (2)^{aA}	1346 (8)^{aA}	76.4
	80	9.23 (0.41)	0.78 (0.02)^{aA}	347 (1)^{bA}	1336 (5)^{aA}	74.0
	120	8.14 (0.22)	0.79 (0.02)^{aA}	333 (2)^{cA}	1315 (1)^{aA}	74.7
30	40	8.93 (1.55)	0.76 (0.01)^{aB,aA}	381 (5)^{aB}	1337 (2)^{aA}	71.5
	80	8.80 (0.22)	0.67 (0.02)^{bB}	404 (7)^{bB}	1340 (6)^{aA}	69.8
	120	8.97 (0.06)	0.55 (0.02)^{cB}	388 (2)^{aB}	1338 (3)^{aA}	71.0
45	40	8.75 (0.31)	0.58 (0.02)^{aC}	422 (7)^{aC}	1359 (1)^{aC}	68.9
	80	8.32 (0.06)	0.52 (0.00)^{bC}	440 (2)^{bC}	1365 (1)^{aC}	69.2
	120	8.51 (0.05)	0.60 (0.02)^{cC}	420 (2)^{aC}	1357 (1)^{aC}	69.0

Note: [1] Tukey's test at 5% significance level for the same CDS level at varying drying temperature (a, b, c) and for the same drying temperature at varying CDS level (A, B, C). Values followed by the same set of letters are not significantly different.

Table 6 Analysis of variance (ANOVA) results for selected physical properties of wheat distillers dried grain with solubles (DDGS). The samples with 15%, 30%, and 45% condensed distillers solubles (CDS) were dried under 40, 80, and 120°C forced-air convection

Property	Total sums of squares, distribution/%				P-values		
	CDS level	Drying temperature (T)	CDS × T	Residual	CDS level	T	CDS × T
Structural properties							
Particle size	72.6	6.5	19.9	1.0	0.000	0.000	0.000
Bulk density	92.7	6.1	5.2	0.6	0.000	0.000	0.025
Particle density	61.8	11.4	16.2	10.7	0.000	0.038	0.059
Flow properties							
Compressibility	61.6	14.8	18.6	5.1	0.000	0.002	0.004
Angle of repose	47.7	1.4	22.0	28.9	0.013	0.808	0.231
Ave. angle of spatula	57.4	22.0	11.1	9.5	0.000	0.005	0.104
Uniformity coefficient	81.8	1.6	16.2	0.4	0.000	0.000	0.000
Angle of fall	5.6	8.1	60.0	26.3	0.422	0.298	0.020
Angle of difference	29.1	12.5	32.1	26.3	0.035	0.173	0.096
Dispersibility	71.5	6.4	20.2	1.9	0.000	0.001	0.000
Overall flowability index	58.6	21.8	11.3	8.3	0.000	0.003	0.075
Overall floodability index	18.1	14.7	23.8	43.3	0.268	0.208	0.361
Compression characteristics							
Pellet density	50.4	12.0	6.6	31.0	0.000	0.003	0.129
Specific compression energy	19.8	12.8	25.3	42.1	0.001	0.008	0.002
Specific extrusion energy	5.0	12.7	26.1	56.1	0.219	0.025	0.007
Kawakita-Ludde model d	53.8	15.4	7.7	23.1	0.000	0.000	0.052
Kawakita-Ludde model f^{-1}	50.7	12.6	10.3	26.4	0.000	0.001	0.016
Thermal properties							
Conductivity	20.6	11.8	14.7	52.9	0.228	0.405	0.656
Diffusivity	53.1	5.4	19.0	22.4	0.004	0.377	0.193
Frictional properties							
Coefficient of internal friction	52.6	0.0	42.1	5.3	0.001	0.850	0.001
Cohesion	25.5	11.7	47.2	15.6	0.054	0.078	0.015

3.2.3 Flow properties

Table 7 shows the various flow property measurements made from the wheat DDGS samples. The overall flowability index, which is the sum of the component indices of compressibility, angle of repose, average angle of spatula, and uniformity coefficient[28], ranged from 70.5 to 77.5. With this value range, the wheat DDGS samples were classified as having a "fair" degree of flowability[28]. Materials under this category would sometimes require vibration to assure flow[28]. In general, the overall flowability index was significantly affected by CDS level and drying air temperature, although the stronger influence of CDS was still suggested by the associated sums of squares results (Table 6). Samples with higher CDS level had lower flowability. Those dried under 120°C also had significantly lower flowability index than those dried under 40°C and 80°C.

Table 7 Flow properties[1] of wheat distillers dried grain with solubles (DDGS) samples, produced from varying condensed distillers solubles (CDS) level and dried under forced air convection method

CDS, mass/%	Drying temperature /°C	Moisture /%, w.b.	Compressibility /%	Angle of repose/(°)	Ave angle of spatula/(°)	Uniformity coefficient	Angle of fall/(°)	Angle of difference/(°)	Dispersibility/%	Overall index	
										Flowability	Floodability
15	40	8.49 (0.04)	15.0 (0.3)^{aA}	44.4 (2.0)^{aA}	41.5 (3.1)^{aA}	2.5 (0.0)^{aA}	36.9 (2.6)^{aA}	7.6 (0.6)^{aA}	29.4 (0.9)^{aA}	77.0 (0.0)^{aA}	66.1 (1.2)^{aA}
	80	9.40 (0.24)	15.3 (0.0)^{aA,bA}	41.5 (1.4)^{aA,bA}	43.4 (0.3)^{aA,bA}	2.3 (0.0)^{bA}	34.4 (0.7)^{aA,bA}	7.2 (0.8)^{aA,bA}	25.7 (0.6)^{bA}	77.5 (0.7)^{aA}	64.1 (1.2)^{aA}
	120	8.00 (0.20)	16.1 (0.7)^{aA}	43.7 (2.1)^{aA,cA}	49.8 (0.1)^{cA,bA}	2.9 (0.0)^{cA}	34.9 (2.4)^{aA,cA}	8.8 (4.5)^{aA,cA}	20.7 (2.0)^{cA}	73.3 (0.4)^{cA}	65.3 (3.2)^{aA}
30	40	8.37 (0.05)	15.2 (0.6)^{aB,aA}	45.8 (0.8)^{aB}	49.8 (1.6)^{aB}	3.3 (0.0)^{aB}	37.4 (0.2)^{aB,aA}	8.5 (0.6)^{aB}	23.1 (1.4)^{aB}	73.5 (0.0)^{aB}	65.8 (1.1)^{aA}
	80	8.86 (0.38)	15.9 (0.7)^{aB,bA}	46.6 (1.7)^{aB,bB}	51.5 (1.8)^{aB,bB}	3.6 (0.1)^{bB}	37.4 (2.1)^{aB,bA}	9.2 (0.4)^{aB,bB}	17.7 (0.4)^{bB}	72.3 (1.8)^{aB,bB}	62.8 (0.4)^{aA}
	120	8.51 (0.31)	18.5 (0.6)^{cB}	46.3 (1.6)^{aB,cB}	51.2 (0.1)^{cB,bB}	2.9 (0.0)^{cB,cA}	32.4 (0.1)^{cB,cA}	13.9 (1.4)^{aB,cB}	15.4 (1.4)^{cB,bB}	70.5 (2.1)^{cB}	66.5 (2.1)^{aA}
45	40	8.54 (0.02)	18.0 (0.4)^{aC}	43.7 (0.6)^{aC,aA,aB}	49.9 (3.0)^{aC,aB}	4.2 (0.1)^{aC}	32.5 (0.9)^{aC}	11.3 (0.4)^{aC,aA,aB}	10.0 (1.1)^{aC}	73.0 (0.0)^{aC,aB}	63.8 (1.8)^{aA}
	80	7.89 (0.10)	19.4 (0.4)^{aC}	45.6 (0.1)^{aC,bA,bB}	51.5 (0.9)^{aC,bC,bB}	3.8 (0.1)^{bC}	35.4 (0.9)^{aC,bA}	10.3 (0.8)^{aC,bA,bB}	13.6 (1.0)^{bC}	71.3 (0.4)^{aC,bB}	64.0 (1.4)^{aA}
	120	8.45 (0.19)	18.2 (0.4)^{aC,cB}	45.2 (0.7)^{aC,cA,cB}	53.6 (1.0)^{cC,bC,cB}	3.7 (0.1)^{cC}	35.9 (1.1)^{aC,cA}	9.4 (0.4)^{aC,cA,cB}	15.4 (0.8)^{cC,bC,cB}	71.8 (0.4)^{cC,cB}	62.8 (0.4)^{aA}

Note: [1] Tukey's test at 5% significance level for the same CDS level at varying drying temperatures (a, b, c) and for the same drying temperature at varying CDS levels (A,B,C). Values followed by the same set of letters are not significantly different.

The two flowability index component properties, compressibility and uniformity coefficient, were significantly affected by CDS level, drying air temperature, and their interaction (Tables 6 and 7). Similar to density and size, the ANOVA sums of squares associated with these two properties (Table 6) also suggest a stronger influence of CDS level. Samples with higher CDS level tended to be more compressible and more differentiated in size than those with lower CDS level. Increasing the CDS level in the blend increased the presence of finer solids and decreased the amount of fibrous particles, thus, leading to slightly more differentiated particle sizes.

Aside from easily penetrating the void spaces in the bulk material, which translates to higher bulk density and higher compressibility, these finer particles also promote increased inter-granular friction because of the greater surface area of contact. This is seen in the angle of repose and average angle of spatula values. Samples with 30% CDS had significantly higher angles of repose than those with 15% CDS. Similarly, those with higher CDS (30%, 45%) level had significantly higher average angle of spatula compared to those with lower CDS (15%). The angle of repose was significantly affected by CDS level while the angle of spatula was affected by variations in both CDS level and drying air temperature. The 120°C-dried sample had significantly higher angle of spatula than the 40°C-dried sample.

Under the Carr classification system[28], the 45% CDS samples' angle of repose and average angle of spatula (Table 7) were considered on the borderline between the free-flowing and non-free flowing material. Their compressibility index (18.0%-19.4%) was also near the borderline (20%-21%). In terms of the uniformity coefficient, the samples were classified as having "excellent" flowability.

With respect to the overall floodability index, the values ranged from 62.8 to 66.5, classifying the wheat

DDGS samples under the "floodable" category[28]. Materials under this category require the use of a rotary seal to prevent flushing[28]. This means, for example, that in designing feed systems, adequate measures should be installed to ensure that these materials can be metered out under control.

The evaluation of the floodability index involved flowability index, angle of fall, angle of difference, and dispersibility. Dispersibility showed similar trends with flowability index with respect to the effect of CDS level. Samples with higher CDS level, which have higher bulk density values, were significantly less dispersible than those with lower CDS level. Although drying air temperature and its interaction with CDS level also significantly affected dispersibility, the associated sums of squares (Table 6) still suggests a stronger influence of CDS level.

3.2.4 Compression characteristics

Table 8 shows the pellet density, the specific energy consumption during the compression and extrusion processes, and the parameters of the Kawakita-Ludde model, which adequately described the compression characteristics of wheat DDGS samples.

Table 8 Compression characteristics[1] of wheat distillers dried grain with solubles (DDGS), produced at varying condensed distillers solubles (CDS) level and dried under forced air convection method. Values in parentheses represent standard deviation (N = 5)

CDS, mass/%	Drying temperature/°C	Moisture/%, w.b.	Pellet density /kg m^{-3}	Specific energy consumption		Kawakita-Ludde model parameters[2]			
				Compression	extrusion /M Jt^{-1}	d	f^{-1}/MPa	R^2	MSE
15	40	8.83 (0.26)	1168 (11)^{aA}	10.63 (0.39)^{aA}	0.30 (0.02)^{aA}	0.73 (0.00)^{aA}	0.49 (0.04)^{aA}	0.967	0.000
	80	9.60 (0.28)	1175 (7)^{aA,bA}	10.31 (0.16)^{aA,bA}	0.45 (0.06)^{bA}	0.70 (0.00)^{bA}	0.53 (0.07)^{aA}	0.970	0.000
	120	8.04 (0.11)	1162 (13)^{cA,bA}	14.99 (0.66)^{cA}	0.23 (0.02)^{aA,cA,aC}	0.75 (0.00)^{aA}	1.46 (0.12)^{cA}	0.994	0.000
30	40	9.01 (0.41)	1206 (12)^{aB}	12.71 (2.69)^{aB,aA}	0.31 (0.14)^{aB,aA}	0.70 (0.02)^{aB}	1.01 (0.48)^{aB,aA}	0.983	0.000
	80	9.03 (0.12)	1199 (16)^{aB,bB}	12.80 (2.70)^{aB,bA}	0.31 (0.15)^{aB,bB}	0.68 (0.02)^{bB}	1.42 (0.76)^{aB}	0.985	0.000
	120	8.96 (0.14)	1187 (12)^{cB,bB}	12.12 (0.08)^{aB}	0.20 (0.08)^{aB,cA}	0.69 (0.02)^{aB,cB}	1.20 (0.48)^{aB}	0.987	0.000
45	40	8.98 (0.07)	1210 (13)^{aC,aB}	12.71 (1.76)^{aC,aA}	0.32 (0.06)^{aC,aA}	0.69 (0.01)^{aC}	1.55 (0.43)^{aC,aB}	0.981	0.000
	80	8.66 (0.27)	1190 (7)^{aC,bC,bB}	15.47 (1.32)^{bC}	0.27 (0.02)^{aC,bB}	0.68 (0.01)^{bC}	2.38 (0.41)^{bC}	0.989	0.000
	120	8.74 (0.02)	1187 (11)^{cC,bC,cB}	14.98 (1.12)^{cC,bC,cA}	0.34 (0.08)^{cC}	0.70 (0.01)^{aC,cB}	2.24 (0.37)^{bC}	0.984	0.000

Note: [1] Tukey's test at 5% significance level for the same CDS level at varying drying temperature (a, b, c) and for the same drying temperature at varying CDS level (A, B, C). Values followed by the same set of letters are not significantly different; [2] The Kawakita-Ludde model parameters d and f^{-1} relate to initial porosity and failure stress, respectively.

Pellet density approached to about 86%-90% of particle density (Table 5) and was significantly affected by CDS level and drying air temperature (Table 6). Samples with 30% and 45% CDS had significantly higher pellet density than those with 15% CDS. Samples dried under 40°C and 80°C had significantly higher pellet density than those dried under 120°C. The ANOVA sums of squares associated with CDS level, however, comprised about 49% of the total observed variation (Table 5), suggesting a relatively strong influence of CDS level.

In terms of specific energy expenditure, about 9.9-17.2 MJ/t was consumed during compression, representing about 95%-99% of the total specific energy expenditure. The rest of the energy was consumed for extruding the pellet from the cylindrical die. While specific energy consumption during both processes was affected by one or both main factors and the CDS level × drying temperature interaction (Table 6), no consistent pattern was observed. Sizeable residual sums of squares vis-à-vis the total sums of squares (Table 6) derived from both compression and extrusion specific energy consumption data sets suggest that there could still be other factors that may have contributed to the observed variation.

The Kawakita-Ludde model adequately described the compression characteristics of wheat DDGS samples. Values of the model parameter d, which represent initial porosity[30,31] of the sample, were 95%-101% of the bulk porosity values (Table 5) and were significantly affected by CDS level and drying air temperature. Similar to what was observed in the density-derived bulk porosity

values (Table 5), samples with higher CDS level had significantly lower *d* values than those with lower CDS. Those dried under 40°C and 120°C also showed significantly higher *d* values than those dried under 80°C. On the other hand, the model parameter f^1, which relates to failure stress, was significantly affected by CDS level, drying air temperature, and their interaction. Samples with 30% and 45% CDS consistently showed significantly higher f^1 values compared to those with l5% CDS across the temperature levels. This indicates that pellets with higher CDS level would be harder to break than those with lower CDS. The 120°C-dried samples also had significantly higher failure stress than the 40°C-dried samples.

Increased pellet density and failure stress of samples with increased CDS could be attributed to the increased amount of finer particles in the blend as well as to the chemical composition. With increased CDS level, there is increased presence of finer particles that would easily fill the voids between larger particles during compression. These finer particles also have greater surface area for contact, thus, enhancing binding. Higher CDS level also translates to higher protein and ash but lower fat and fiber content. Heat applied during densification, as well as those resulting from friction, may have also altered the state of the chemical constituents and could promote better binding characteristics. For example, protein could have been denatured, and combined with the sugars present, positively affecting the strength of the pellets, as a result of Maillard reaction[34].

3.2.5 Thermal properties

Table 9 presents the thermal properties of wheat DDGS at 23°C and at the specified bulk densities. Average thermal conductivity of wheat DDGS samples was not significantly different across CDS and drying air temperature levels (Table 6). Thermal diffusivity, on the other hand, was significantly affected by CDS level and its values ranged from 1.35×10^{-7} to 1.65×10^{-7} m^2/s. Samples with 15% and 30% CDS had significantly higher thermal diffusivity values than those with 45% CDS. This is attributed to differences in density and porosity. Since air has a much higher thermal diffusivity compared to that of water[35], more porous materials, like the 15% CDS samples, would have higher thermal diffusivity than the less porous, 45% CDS samples at the same moisture content.

Table 9 Thermal and frictional properties[1] of wheat distillers dried grain with solubles (DDGS), produced at varying condensed distillers solubles (CDS) level and dried under forced air convection method. Values in parentheses represent standard deviation (*N* = 2)

CDS, mass/%	Drying temperature/℃	Moisture /%, w.b.	Thermal properties (at 23°C)			Frictional properties[2]			
			Bulk density /kg m^{-3}	Conductivity /W m^{-1} °C^{-1}	Diffusivity (×10^{-7}) /m^2 s^{-1}	Coefficient of internal friction	Cohesion/kPa	Coefficient of determination/R^2	Standard error of estimate
15	40	8.83 (0.26)	330.6 (0.3)	0.05 (0.01)^{aA}	1.65 (0.01)^{aA}	0.63 (0.01)^{aA}	2.96 (0.52)^{aA}	0.999	0.38
	80	9.60 (0.28)	364.8 (0.6)	0.05 (0.00)^{aA}	1.55 (0.01)^{aA}	na	na		
	120	8.04 (0.11)	341.4 (3.1)	0.05 (0.01)^{aA}	1.60 (0.01)^{aA}	0.66 (0.02)^{cA}	2.86 (1.00)^{aA,cA}	0.999	0.53
30	40	9.01 (0.41)	397.3 (1.6)	0.05 (0.00)^{aA}	1.55 (0.01)^{aB,aA}	0.71 (0.01)^{aB}	2.74 (0.02)^{aB,aA}	0.996	1.09
	80	9.03 (0.12)	416.0 (0.3)	0.05 (0.00)^{aA}	1.45 (0.01)^{aB,aA}	na	na		
	120	8.96 (0.14)	389.6 (0.3)	0.05 (0.01)^{aA}	1.65 (0.01)^{aB,aA}	0.64 (0.01)^{cB,cA}	4.93 (0.18)^{cB}	0.997	0.95
45	40	8.98 (0.07)	427.6 (2.8)	0.05 (0.01)^{aA}	1.35 (0.01)^{aC}	0.69 (0.01)^{aC,aB}	3.15 (0.08)^{aC,aA}	0.997	1.03
	80	8.66 (0.27)	426.8 (0.8)	0.05 (0.00)^{aA}	1.45 (0.01)^{aC}	na	na		
	120	8.74 (0.02)	425.0 (1.1)	0.05 (0.00)^{aA}	1.40 (0.00)^{aC}	0.74 (0.00)^{cC}	2.79 (0.06)^{aC,cA}	0.999	0.52

Note: [1] Tukey's test at 5% significance level for the same CDS level at varying drying temperature (a, b, c) and for the same drying temperature at varying CDS level (A,B,C). Values followed by the same set of letters are not significantly different.

[2] na – data were not available.

3.2.6 Frictional properties

The coefficient of internal friction and cohesion values for wheat DDGS are also presented in Table 9. The friction coefficient ranged from 0.63 to 0.74, equivalent to about 32°-35° angle of internal friction, close to the values reported for chickpea[31] and wheat[36]

flours. It was significantly affected by CDS level, drying air temperature, and by the CDS level × drying temperature interaction, with the associated ANOVA sums of squares results suggesting a strong influence of CDS level (Table 6). The higher CDS samples, dried at 40°C and 120°C, presented significantly higher coefficient of friction than those with lower CDS. Decreased particle size with increased CDS level may have contributed to higher values of the friction coefficient because this provides greater surface area of contact.

Cohesion estimates ranged from 2.7 kPa to 4.9 kPa and were significantly affected by the CDS level × drying temperature interaction (Table 6). Only a few pairs of samples, however, showed significant difference. In samples dried under 120°C, those with 15% and 30% CDS, as well as those with 30% and 45% CDS, were significantly different. No consistent trend, however, was observed. Samples with 30% CDS and dried under 120°C showed significantly higher cohesion value than those dried under 40°C. The rest of the pairs were statistically similar.

3.2.7 Influence of drying air temperature

Although CDS level was seen as having a stronger influence on some of the physical properties investigated, there were consistent patterns observed with respect to the effect of drying temperature. The 40°C- and 120°C-dried samples, regardless of CDS level, showed significantly lower bulk density than those dried under 80°C. This was consistent with the trend of the Kawakita-Ludde parameter *d* values, where both 40°C- and 120°C-dried samples showed higher initial porosity than the 80°C sample. Between the 40°C- and 120°C-dried samples, the former had significantly higher uniformity coefficient, lower angle of spatula, higher flowability index, and higher pellet density.

Variability due to drying temperature could be attributed to the structural changes that occur during the drying process. Nowak and Lewicki[37] reported microstructure differences between convectively-dried and infrared-dried apple tissues and indicated that drying rate could be the probable cause of the microstructure differences. Higher drying rates, for example, by the higher drying temperature (120°C) used in the study, could result in larger shrinkage stresses and greater tissue damage[37]. Significant differences in the structural properties (such as bulk density and porosity) could, consequently, affect flow, compression, thermal, and frictional properties.

4 Conclusions

As CDS level in the blend was increased, protein and ash increased while fat and fiber decreased. Fat and ADF content were also markedly affected by drying air temperature. Compared to drying temperature, CDS level was seen as having a stronger influence on the most of the physical properties investigated. Wheat DDGS samples with higher CDS content were denser, smaller in size, less flowable, and less dispersible. These also had significantly lower thermal diffusivity, higher angle of internal friction, and produced pellets with higher density and failure stresses.

These results contribute toward better understanding of wheat DDGS variability, highlighting the importance of selecting the appropriate WDG:CDS blending proportion and drying conditions to maximize its nutritive value as an animal feed ingredient and improve the efficiency of related handling and processing operations.

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