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The effect of process variables during drying on the physical and chemical characteristics of corn dried distillers grains with solubles (DDGS) – Plant scale experiments

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ABSTRACT

Corn dried distillers grains with solubles (DDGS) are highly valued as an animal feed for its nutrient content. The amount of wet distillers grains (WDG) and condensed distillers solubles (CDS) blended together during drying affects nutritive value and physical characteristics of DDGS. Effect of changing the ratio of WDG and CDS, and recycled DDGS during drying on particle size, particle size distribution, particle and bulk densities, color, chemical composition, and amino acid content was studied. Moisture content and particle size of DDGS decreased with decreasing amount of CDS added. About 80% of the particles were within a narrow size range (<1500 μm). Bulk density and tapped density of samples produced with different CDS content ranged from 420.5 to 458.1 and 498.8 to 544.3 kg/m^3, respectively. True density decreased with reduction in CDS added. As the CDS content reduced, DDGS became lighter in color. Insoluble fiber contents (protein and insoluble fiber) and amino acids increased while fat, total soluble sugars and glycerol decreased as the CDS content added to WDG reduced. The correlation coefficient of individual chemical components with CDS was above 0.90. Results from this study will be helpful in predicting the physical and nutritive property changes due to variable ratios of blending CDS to WDG during the drying process.

1. Introduction

Processing of ethanol from corn is mainly classified into three types namely: wet milling, dry milling (dry fractionation) and dry grinding processes. In the US, primary production of ethanol is by the dry grinding process (Westcott, 2007). Production of ethanol by the dry grinding process results in a primary co-product called dried distillers grains with solubles (DDGS). It is produced by blending and drying the non-fermentable residues in corn after fermentation of the starch, i.e., wet distillers grains (WDG) and condensed distillers solubles (CDS). After the fermented beer is distilled, the whole stillage containing the non-fermentable portions of corn grain is centrifuged to separate insoluble solids from liquids (thin stillage). The thin stillage is further condensed by removing water using evaporators to syrup known as condensed distillers solubles (CDS) which has about 35–40% solids content. The insoluble solids at about 65–70% moisture (wet basis) known as wet distillers grains (WDG) is mixed with CDS and dried in rotary drum dryers to produce DDGS. The operational parameters of rotary dryers are usually controlled to result in a stable product within the moisture range of 10–13%. Due to the presence of high nutritive components such as protein, fat, minerals, vitamins and starch, DDGS is highly valued as a feed supplement. For the ethanol industry, shelf-life and other factors influencing transportation logistics of DDGS such as product flow and caking in storage and transport vessels are important to its marketability and economic viability.

The physical and chemical properties of DDGS are normally the measure used to indicate the identity of a particular DDGS product. The physical properties of DDGS such as true and bulk densities, particle size and particle size distribution affect how much of the product can be shipped in a given volume (Ileleji and Rosentrater, 2008). Large variations in physical properties have been reported by Shurson (2005), Rosentrater (2006) and Ileleji et al. (2007). Chemical properties such as the proximate analysis (moisture, protein, fat, fiber and ash contents) are normally used as market value indicators of DDGS and directly impact its price. Similar to physical properties, variations in chemical properties of DDGS have also been reported in the industry (Shurson, 2005; Spiehs et al., 2002; Belyea et al., 2004; Clementson et al., 2009). A change in physical and chemical properties alters the flow and storage behavior of this bulk granular material. Also, it can give important information

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about product stability under any environmental conditions. The changes in physical and chemical constituents have been shown to influence the EMC–ERH relationship, which does have an effect on bulk behavior and can lead to liquid bridging, caking and mold growth (Kingsly and Ileleji, 2009). The change in quality of DDGS has a direct impact on economics of ethanol production (Singh et al., 2001) and its feed value.

While it is well known in the industry that inconsistency in the physical and chemical properties of DDGS supplied is one of the major market barriers to using this product as a livestock feed, no detailed study under real plant operating conditions has been published to elucidate the effect of process changes during drying on DDGS physical and chemical properties, especially as it relates to both bulk handling characteristics and its end use as a livestock feed. Change in process variables, namely WDG and CDS feed stream ratio, and the amount of recycled DDGS product affects the physical and chemical characteristics (Rosentrater and Muthukumarappan, 2006; Ileleji et al., 2007; Ileleji and Rosentrater, 2008) of the final product. Apart from DDGS variations from plant-to-plant, batch-to-batch variations have been reported by Belyea et al. (1998) and Shurson (2005) as well.

Ganesan et al. (2008) studied the effect of moisture content and soluble level on density, protein, fat and Carr indices of DDGS in lab-scale experiments, but the procedures used in preparing DDGS at the various moisture contents investigated differed from DDGS production methods in fuel ethanol plants. In an earlier study, bench-scale production of DDGS that closely mimicked DDGS drying in a rotary drum dryer similar to plant conditions was conducted by Ileleji et al. (unpublished data). Their study showed that process variables, primarily the levels of CDS blended with WDG, affected the physical and chemical properties of DDGS. However, the effect of CDS blended with WDG during drying on the physical and chemical properties of DDGS have not been verified and quantified under real plant conditions. Additionally, it is not fully understood how variable ratios of CDS blended with WDG affect DDGS feed value with respect to the primary limiting amino acids in feed (methionine, lysine, threonine and tryptophan) and mineral levels (calcium, phosphorus, potassium and sodium). Therefore, the primary objective of this study was to investigate the effect of blending variable levels of CDS with WDG during drying on the physical and chemical variability in DDGS produced under real plant operating conditions.

2. Methods

2.1. Process conditions for the plant-scale production of DDGS

Drying of DDGS in this study took place in a "new generation" 416 million liters (110 million gallons per year) fuel ethanol plant commissioned in 2007 in Indiana. The two-stage drying process consisted of two rotary drum dryers in series to dry the product efficiently (Fig. 1). The two process variables changed during plant-scale production were WDG to CDS ratio and the addition of recycled DDGS into both dryers. The team of researchers from Purdue University first studied the plant's DDGS production process conditions for several weeks beforehand in order to identify the process conditions to be investigated in such a way as to minimize plant downtime after running the tests, as well as maintain the recommended safe operating conditions of the rotary drum dryers. The process conditions were varied by adjusting the total input quantity of CDS in Dryer I and II without changing the WDG input. The CDS ratio was varied by adjusting the flow rates from the maximum possible of 212 L/min (the plant's normal operating condition) to zero CDS. The amount of recycled DDGS could not be determined due to the non-availability of proprietary engineering information. The setting used was obtained from the computer process control screen which was based on the recycle screw conveyor speed operated at 60% of the maximum speed. The following process conditions were evaluated in the order described below:

1. 212 L/min (56 gals/min) CDS and 60% of maximum recycle conveyor speed (Batch 1)
2. 106 L/min (28 gals/min) CDS and 60% of maximum recycle conveyor speed (Batch 2)
3. 0 L/min (0 gals/min) CDS and 60% of maximum recycle conveyor speed (Batch 3)
4. 106 L/min (28 gals/min) CDS and 0% of maximum recycle conveyor speed (Batch 4)

Converting the flow rates of CDS added to percent of CDS in the DDGS, about 7.39% (percent volumetric flow basis) CDS was added for the total amount of WDG and CDS in both dryers combined for Batch 1. The CDS percent was lowered to 3.69% for Batch 2 and to zero for Batch 3. No recycled DDGS were added back in Batch 4. Note that the recycled DDGS into both dryers was not taken into consideration in the above percentage estimates because the total recycled amount was not known.

Experiments were started with the first process conditions, Batch 1, and the operating parameters of the rotary dryers were changed in increments until the temperature set point was achieved to maintain safe operating conditions. After the set point temperature and CDS rate were achieved, the dryer was allowed to run for 15 min to ensure steady-state conditions. DDGS were produced at these conditions to obtain piles of about 9100 kg of product discharged to the flat storage cooling pad at the plant after which the process conditions were changed to the next one in the following sequence: Batch 2, Batch 3 and Batch 4. To change

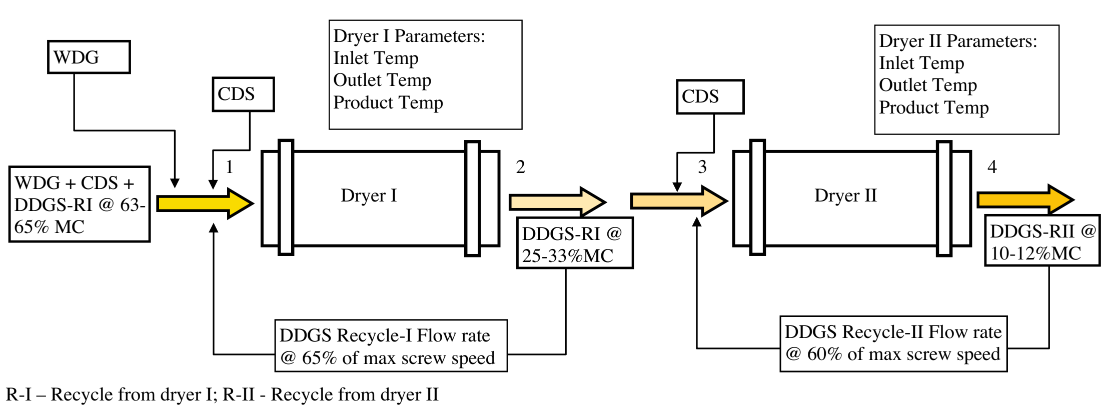

Fig. 1. Product flow of DDGS through rotary dryers in series.

Table 1
Drying process parameters during the plant scale trials.

Dryer	Process parameters	Batch 1	Batch 2	Batch 3	Batch 4
	Recycle screw speed, % of maximum	60	60	60	0
Dryer I	WDG, L/min	2566.1	2567.3	2569.9	2567.6
	CDS, L/min	45.4	22.7	0.0	22.7
	Inlet air temp., °C	488.4	492.3	479.3	418.7
	Outlet air temp., °C	104.8	106.1	106.3	108.7
	Product temp., °C	93.4	94.3	93.3	94.4
Dryer II	CDS, L/min	166.6	83.3	0.0	83.3
	Inlet air temp., °C	499.1	377.3	273.3	415.2
	Outlet air temp., °C	108.3	111.1	107.4	114.6
	Product temp., °C	99.7	102.0	100.9	100.1

the operating parameters from one set of conditions to the next, CDS inflow and inlet air temperature were gradually increased or decreased to avoid any instability in the process system. Inlet and outlet air temperature and product temperature in both dryers were continuously monitored and controlled to the dryer's safe operating temperature by an experienced plant operator. Inlet air temperature was controlled according to the amount of CDS added to avoid a fire hazard by keeping the dryer below the safe operating temperature threshold of 593 °C (1100 °F). In order to decrease the percent of CDS, the inlet air temperature was reduced to maintain the dryer air temperature below the threshold level. The process parameters evaluated and the resulting dryer conditions are illustrated in Fig. 1 and given in Table 1.

During the initial period of the process runs, the CDS input was reduced while keeping the recycle conveyor speed at 60% of the maximum to produce Batches 1, 2 and 3. After the conditions stabilized at 0 L/min of CDS inflow condition at 60% of recycle (Batch 3), CDS inflow was increased to 106 L/min and the recycle conveyor speed was reduced to 0% (Batch 4). Product temperature was maintained at about 93.3–94.4 °C in Dryer I and 99.7 – 102 °C in Dryer II during entire drying process.

On exiting Dryer II, DDGS were conveyed with screw conveyors to the outside concrete pad. The batch treatments produced where discharged onto the concrete pad in four piles of about 9100 kg per pile per batch. The piles were left to sit for about 48 h, after which they were bagged in 1000 kg heavy-duty bulk bags and shipped for storage at Purdue University, West Lafayette, IN, USA. Samples used for the physical and chemical characterization in this study were collected from various locations of the piles using best sampling practices for granular bulk solids (Clementson et al., 2009) on the same day they were produced, and shipped in plastic containers to a laboratory at Purdue University. The product was allowed to cool down in the containers in the lab before further analyses were conducted.

2.2. Measurement of physical properties

Moisture content of DDGS in both dryers was constantly monitored (at 20 min intervals) in order to control the drying process and in order to know the stability of the process at the set conditions. DDGS samples were collected from both dryers through built-in sample ports. Moisture content during drying was measured by drying 3 g sub-samples at 140 °C in a thermal balance (Mettler Toledo, OH, USA). All moisture content results reported were on a wet basis (w.b.).

Particle size distribution influences physical properties of DDGS (Ileleji and Rosentrater, 2008) and reduction in particle size usually increases the bulk and particle density (Ileleji and Rosentrater, 2008; Zhou et al., 2008). Particle size and particle size distribution were determined using ASABE *Standard* S 319.4 (ASABE, 2008). This involved sieving about 100 g of sample charge through a nest of sieves with sizes ranging from US sieve No. 4 (size of opening: 4.75 mm) to sieve No. 270 (size of opening: 0.053 mm). The set of sieves was then vibrated in a Ro-Tap shaker (RX-29, Tyler Inc., Mentor, OH, USA) for 10 min, after which DDGS retained in each sieve were weighed. The geometric mean diameter (d_{gw}), geometric standard deviation (S_{gw}) and cumulative size distribution was calculated using the procedure specified in the standard.

The bulk density of DDGS was determined using a standard Winchester cup setup with hopper, funnel, and leveling rod (Seedburo Equipment Co., Chicago, IL, USA) used for grain bulk density determination. Bulk density was calculated from the weight and volume of materials filled in a cup of known volume (5.5×10^{-4} m^3).

To determine the tapped bulk density, the sample was filled in the same cup of known volume and vibrated at a rate 900 vibrations per minute for 10 min using a Fisher–Wheeler sieve shaker (Model 5, Fisher Scientific Co., Pittsburg, PA). From the consolidated volume and weight, the tapped density was calculated.

The true density was determined using a gas multipycnometer (Quantachrome Corporation, Boynton Beach, FL, USA) and the small cell (2.74×10^{-5} m^3) provided with the equipment. The cell was filled to about 70% of its volume with the test material as instructed in the equipment operations manual.

Compressibility Index (CI) and Hausner Ratio (HR) were calculated using the following equations from the bulk and tapped densities used by Zhou et al. (2008) for corn stover bulk particles:

$$CI = \frac{(\rho_{TD} - \rho_{BD})}{\rho_{TD}} \times 100 \quad (1)$$

where CI = Compressibility Index (%), ρ_{BD} = bulk density (kg/m^3) and ρ_{TD} = tapped density (kg/m^3)

$$HR = \frac{\rho_{TD}}{\rho_{BD}} \quad (2)$$

where HR = Hausner Ratio (dimensionless).

Color, as *L*, *a* and *b* values, was measured using a HunterLab colorimeter (10°/D 65 Color Flex, Reston, VA, USA). In the Hunter scale, 'L' measures lightness and varies from 100 for white to zero for black. The chromaticity value '*a*' measures redness when positive, gray when zero, and greenness when negative. The '*b*' value measures yellowness when positive, gray when zero, and blueness when negative. The colorimeter was calibrated with standard black and white calibration tiles provided with the instrument before measuring the color of DDGS. Three replications of samples per batch were used for the colorimetric measurements.

2.3. Chemical composition analysis

Three replicate sub-samples from each DDGS batch weighing about 40 g per replicate were analyzed for crude protein, crude fat, crude fiber, ash, total reducing sugars, glycerol, amino acids and minerals by an external lab using AOAC Official Methods (AOAC, 2000). The methods used for the properties determined were AOAC 934.01 for moisture, AOAC 984.13 for crude protein, AOAC 920.39 for crude fat, AOAC 978.10 for crude fiber, and AOAC 942.05 for ash content. The amino acid profiles were determined by AOAC 982.3. The mineral content was determined for composition of calcium, phosphorus, sodium and sulfur using Atomic Absorption Spectroscopy.

2.4. Statistical analysis

Statistical analysis was conducted using PROC GLM using SAS v 9.1 (SAS Institute, Cary, NC) to determine means and Tukey's

Honestly Significant Difference test was used for comparison between treatments ($\alpha = 0.05$) based on ANOVA. PROC CORR was used to find the correlation coefficient (Pearson) between the levels of CDS added and chemical components of DDGS, and among chemical components of DDGS.

3. Results and discussion

3.1. DDGS product and process conditions during drying

Table 2 shows the moisture content profiles of DDGS from all four process conditions that were monitored at 0, 20 and 40 min intervals from samples collected at the outlet of Dryer I and II during the initial period of process change prior to steady-state. The amount of CDS and recycled DDGS added in Dryer I and II had an effect on the moisture content at the dryer outlets. While the percent moisture content (% MC) of DDGS exiting Dryer I were between 32% and 36% for Batches 1, 2 and 3 with decreasing amount of CDS from 212 L/min to 0 L/min, respectively, the % MC of DDGS exiting Dryer II sharply decreased from around 10% after steady-stated at 212 L/min CDS to about 5.4% at 0% L/min CDS. For the same drying temperature conditions, the final end point moisture of DDGS exiting Dryer II and the residence time of product in the drum reduced as the CDS amount was reduced. This was expected because reducing the CDS level in the mixture reduced its initial MC. When the DDGS recycled was reduced to 0%, that is, when no DDGS were recycled back to the drum dryer, the MC of DDGS from both dryers increased considerably. This explains the reason for the practice of recycling dried DDGS in the ethanol industry because it increases the drying efficiency of rotary drum dryers. The moisture increase for Batch 3 sampled at 40 min interval was much higher than was expected, 14.6% from 5.42%. The most likely cause might have been that DDGS samples were taken after changing the drying process variable to Batch 4 which caused % MC to increase. The final MC of DDGS monitored for Batch 4 with no DDGS recycled during drying was 17.6%; quite a bit higher than is normally acceptable for long-distance shipping and long-term storage.

The two rotary drum dryers operated at very high inlet drying air temperatures (415–499 °C) which reduced toward the outlet (105–111 °C) as the drying air became saturated with moisture (Table 1). Inlet drying air temperatures were reduced correspondingly with reduced levels of CDS and amount of recycled product.

3.2. The effect of process conditions on DDGS physical properties

The physical characteristics of DDGS are presented in Table 3. For DDGS with the same amount of recycled product added during drying but different amounts of CDS, the MC was significantly different at 0.05 probability level and decreased with decreasing CDS amount. Having no recycled product during drying sharply increased the final product MC. Because of the high MC of Batch 4, the DDGS product was expected to cake up in transport vessels over time and thus would be unacceptable for long-distance transport. While this result clearly underscores the importance of the recycled stream to attaining a low target MC, there is a need for optimizing the system using drying process modeling in order to understand the drying kinetics of DDGS in rotary drum and other dryer types.

Particle size and its distribution affect granular solids flowability (Barbosa-Canovas et al., 2005) and cause segregation during handling. Particle segregation has been shown to occur in a DDGS bulk with a large particle size distribution during gravity-driven discharge (Ileleji et al., 2007). For livestock feed, which DDGS is primarily used for, it influences feed digestibility (Wondra et al., 1995). The particle size for all DDGS batches expressed as the geometric mean diameter (d_{wg}) is shown in Table 3. While there was no significant difference in particle size (PS) between DDGS with 212 L/min (Batch 1) and 106 L/min (Batch 2), the PS of DDGS with no CDS (Batch 3) was significantly lower than the former two mentioned batches. The PS of DDGS with the recycled product (Batch 2) was significantly higher than the PS of DDGS with no recycled product, but having the same CDS level (Batch 4). It appears that the amount of CDS added during drying had the most influence on the resulting DDGS product particle size. Increasing the amount of CDS increased particle size by increasing the inter-particle affinity and inducing agglomeration of particles during drying (Ileleji et al., 2007; Ileleji and Rosentrater, 2008). This phenomenon was also observed in the bench-scale tests by Ileleji et al. (unpublished data).

The cumulative particle size distribution of DDGS is presented in Fig. 2 and there was very little difference between all four DDGS batches. The majority of particles for all batches were within a narrow size range (80% < 1500 μm).

CDS content significantly influenced the bulk density of DDGS and the bulk density significantly varied among the batches with different CDS levels (Table 3). The bulk density decreased with the reduction in CDS content added during the drying process. The bulk density also increased with increasing particle size and

Table 2
Moisture content profile of DDGS monitored during drying process.[a]

Operating conditions	Sampling time interval, min	Moisture content, MC, % wet basis	
		Dryer I	Dryer II
Batch 1	0	35.9	12.7
	20	36.2	13.5
	40	34.1	9.61
Batch 2	0	33.3	9.42
	20	32.8	9.98
	40	32.5	6.46
Batch 3	0	32.7	5.51
	20	34.0	5.42
	40	36.1	14.6
Batch 4	0	44.6	23.6
	20	42.9	19.5
	40	41.9	17.6

[a] Not replicated. Measured till the process stabilizes at stated operating condition.

Table 3
Physical properties of DDGS.

Properties	Batch 1	Batch 2	Batch 3	Batch 4
Moisture content, % w.b.	10.56 (0.13)^a	7.91 (0.29)^b	6.45 (0.05)^c	16.57 (0.16)^d
Geometric mean diameter (d_{gw}), mm	1.006 (0.02)^a	0.985 (0.02)^a	0.874 (0.01)^b	0.850 (0.01)^b
Geometric standard deviation (S_{gw}), mm	0.489 (0.001)^a	0.477 (0.006)^b	0.474 (0.004)^{ab}	0.466 (0.010)^b
Bulk density, kg/m^3	458.05 (2.49)^a	427.70 (2.73)^b	420.47 (1.01)^c	417.74 (3.80)^c
Tapped density, kg/m^3	544.29 (8.42)^a	509.98 (9.26)^b	498.82 (6.37)^c	509.50 (7.40)^b
True density, kg/m^3	1290.47 (3.34)^a	1287.13 (2.71)^b	1280.91 (2.50)^c	1269.13 (2.18)^d
Compressibility Index,%	15.83 (1.00)^a	16.11 (1.60)^a	16.25 (0.88)^a	17.47 (0.69)^b
Hausner Ratio	1.188 (0.01)^a	1.192 (0.02)^a	1.194 (0.01)^a	1.211 (0.01)^b

$n = 3$, values in parenthesis are standard deviation; same superscript letter within the same row indicates no significant difference ($P \geqslant 0.05$).

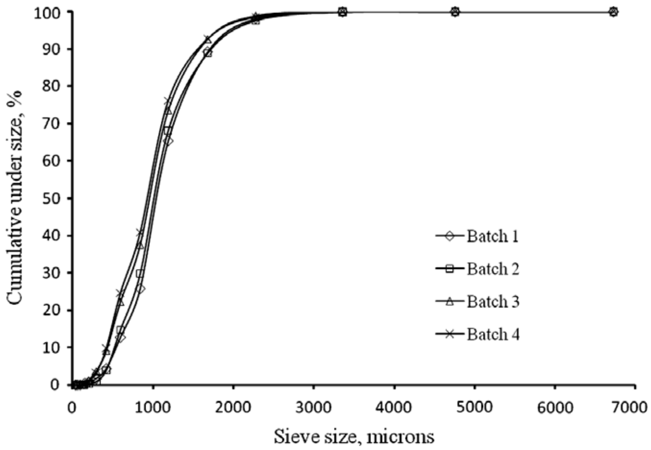

Fig. 2. Particle size distribution of DDGS products.

followed the same increasing trend of particle density with increasing CDS level observed in bench-scale process studies by Ileleji et al. (unpublished data). The increase in bulk density can be explained by the formation of denser agglomerates of DDGS caused by increasing the amount of CDS which acts as a binder gluing fine particles together into denser spherical pellets (Ileleji and Rosentrater, 2008; Ileleji et al., unpublished data). At the same CDS input rate, DDGS produced with recycling had higher bulk density than DDGS produced without recycling (i.e., Batch 2 vs. Batch 4).

The large variation in bulk density reported in the industry from batch to batch in the same plant and also from plant to plant (Rosentrater, 2006) might be explained by the variability in the amount of CDS added during DDGS production. In discussions with operation managers in ethanol plants, the amount of CDS added is based on the inventory of CDS produced; with the goal of minimizing CDS inventory. So when the inventory of CDS increases during production, the tendency is to add more than the normal levels and vice versa when inventory decreases. This haphazard approach causes variable CDS additions from batch to batch depending on the production situation. The negative impact of variable DDGS bulk density with respect to DDGS shipping was highlighted by Ileleji and Rosentrater (2008) and is a major cause of concern in the ethanol industry.

Change in process parameters affected the true density significantly (Table 3). True density decreased with reduction in CDS level. Increasing particle size with increasing CDS levels caused by particle agglomeration explains the trend observed with respect to the influence of CDS levels on true density.

Compressibility Index of DDGS produced in different trials ranged from 15.8% to 17.5%. These values were higher than those reported by Ganesan et al. (2006) for DDGS produced in lab-scale experiments using different soluble percentages and a different definition of DDGS than is used in the industry. In Ganesan et al. (2006), the percent of solubles was defined as the non-soluble portion of a coproduct stream that passed through a filter media which is different from our study and what is understood in the industry. Percent by weight (%w/w) solubles content in our study was the percent of CDS mixed with WDG which was metered at the plant on a volumetric flow rate (gal/min). The amount of DDGS product recycled during drying had a significant effect on the Compressibility Index of DDGS (Table 3). Hausner Ratio ranged from 1.19 to 1.21 indicating medium flowability of DDGS. No significant differences were seen for both the Compressibility Index and Hausner Ratio of DDGS samples with variable CDS level and similar amount of DDGS recycled (Batches 1, 2 and 3), except for the DDGS sample (Batch 4) with no recycled product. A note of caution here is that no one measure alone can be used as a flowability indicator for a product that is known to cake up and experience poor discharge from railcar hoppers. A better measure of flowability is a test that correctly simulates the loads and consolidation regimes, and the environmental conditions undergone by the bulk solid during transportation and storage.

The color value of DDGS is used to judge quality and to predict the digestible lysine content for poultry (Ergul et al., 2003) and pigs (Cromwell et al., 1993). The color values of DDGS produced using these trials are given in Table 4. As the CDS level was reduced from Batch 1 to Batch 3, *L* value increased indicating the color shifted towards a lighter color of DDGS. This was similar to the bench-scale experiments by Ileleji et al. (unpublished data) and those reported by Ganesan et al. (2008). It should be noted that the product temperatures for all four DDGS samples in this study were very close, and therefore temperature treatments on batches were similar. Likewise, the temperatures in the bench-scale studies of DDGS production at variable CDS levels by Ileleji et al. (2008) were quite lower (about 60 °C) and the same for all three CDS levels investigated. This suggests that for DDGS dried at the same temperature, the amount of CDS caused the difference in color. While CDS has a darker color than WDG and so darkening of DDGS is partly due to the addition of CDS, the Maillard reaction between sugars and protein in WDG and CDS during drying is most likely the primary cause of DDGS browning (Labuza and Baisier, 1992). Higher levels of residual sugars (total reducing sugars) in CDS increased the total residual sugars in DDGS with increasing CDS levels (see Table 5). This causes DDGS to progressively become darker with increasing CDS levels as was observed from Batch 3 with no CDS to Batch 1 with the highest level of CDS used in this study. Redness (*a* value; red–green axis) decreased as the CDS level was decreased. There was a trend in yellow color (*b* value; blue–yellow axis), but it was significantly different among DDGS batches. Additionally,

Table 4
Color of DDGS.

Color parameter	Batch 1	Batch 2	Batch 3	Batch 4
L	49.03 (0.35)[a]	51.11 (0.16)[b]	54.21 (0.31)[c]	53.41 (0.24)[d]
a	11.33 (0.06)[a]	10.61 (0.03)[b]	8.78 (0.13)[c]	10.56 (0.09)[b]
b	24.72 (0.14)[a]	25.31 (0.18)[b]	24.72 (0.09)[a]	26.50 (0.10)[c]

n = 3, values in parenthesis are standard deviation; same superscript letter within the same row indicates no significant difference ($P \geqslant 0.05$).

Table 5
Chemical properties (proximate analysis) of DDGS samples.

Chemical composition, g/100 g	Wet distillers grains (WDG)	Condensed distillers solubles (CDS)	Batch 1	Batch 2	Batch 3	Batch 4
Crude protein	16.22 (0.08)[a]	7.48 (0.03)[b]	26.69 (0.14)[c]	28.78 (0.03)[d]	32.33 (0.80)[e]	28.16 (0.45)[d]
Fat	4.56 (0.04)[a]	6.62 (0.20)[b]	10.80 (0.16)[c]	9.32 (0.19)[d]	7.76 (0.21)[e]	9.04 (0.12)[d]
Ash	1.74 (0.00)[a]	4.01 (0.01)[b]	4.00 (0.15)[b]	3.12 (0.05)[c]	2.04 (0.00)[d]	2.97 (0.02)[e]
Total reducing sugars	1.31 (0.06)[a]	6.53 (0.26)[b]	5.38 (0.27)[c]	3.97 (0.26)[d]	2.20 (0.12)[e]	3.71 (0.05)[d]
ADF	6.35 (0.28)[a]	0.16 (0.04)[b]	10.06 (0.27)[c]	12.48 (0.67)[d]	15.98 (1.05)[e]	10.68 (0.46)[c]
NDF	17.30 (0.12)[a]	0.71 (0.28)[b]	33.18 (0.62)[c]	39.96 (0.59)[d]	44.49 (2.34)[e]	34.56 (1.94)[c]
Glycerol	3.51 (0.10)[a]	7.41 (0.12)[b]	7.61 (0.27)[b]	6.01 (0.42)[c]	3.08 (0.07)[d]	5.85 (0.09)[c]

n = 3, values in parenthesis are standard deviation; same superscript letter within the same row indicates no significant difference ($P \geqslant 0.05$).

there was no significant change in the *a* and *b* values due to the change in recycled DDGS (Batches 2 and 4). Lighter colored DDGS have higher acceptance as feed for swine. Cromwell et al. (1993) reported that growth performance of pigs was poorer when darker colored DDGS were fed. So the factor affecting growth performance in DDGS might be in the liquid CDS stream which needs to be investigated further by animal feeding studies.

3.3. The effect of process conditions on DDGS chemical properties

Since the composition of WDG and CDS are substantially different (Kim et al., 2008), the ratio with which they were mixed during the production process significantly affected the chemical composition of final product, and thus could also decide the economic value as animal and poultry feed. Precise information about composition of DDGS is needed to calculate the nutrient content, total digestible nutrients and amino acid digestibility. Crude protein, acid detergent fiber (ADF) and neutral detergent fiber (NDF) of DDGS increased by reducing the CDS level while fat, ash, sugars and glycerol content decreased (Table 5). The same trend was reported by Noll et al. (2006), which was contrary to the findings reported by Ganesan et al. (2008). It was also evident from the high correlation of chemical components with CDS added (Table 8). Insoluble fiber content (protein and fiber) was rich in WDG and low in solubles (CDS), so at higher CDS levels insoluble fiber percent decreased (Belyea et al., 2004; Knott et al., 2004; Ganesan et al., 2008). Change in recycled DDGS had negligible effect on chemical composition. Each of the chemical components had a high correlation with the other components which may be due to the concentrating effect of starch disappearance (Table 8). Kingsly and Ileleji (2009) conducted moisture sorption studies with the samples prepared in this study and found that all four batches gave a different moisture sorption pattern. The implication of this fact is that variable CDS levels will cause DDGS bulk to exhibit different flow behaviors during transportation and storage. Thus, the effects of differences in physical and chemical properties are far more than is currently understood.

Amino acid content of DDGS is used to formulate diets for livestock. Variation in amino acid content can impact diet formulation, animal productivity and economic outcome (Belyea et al., 1998). Amino acid content of DDGS produced in different batches with variable CDS levels and recycled DDGS is given in Table 6. Amino acid content increased with the decrease in CDS addition and the amount of recycled DDGS had little significance. This correlates with the trends in protein content which was observed in the batches. The major limiting amino acids in animal feed, lysine, methionine, threonine and tryptophan decreased with decrease in CDS, which suggests that most of the amino acids are concentrated in the WDG fraction than in the CDS fraction. The amino acids of these protein components are mainly concentrated in WDG (Kim et al., 2008) and so the reduction in CDS content increased the availability of amino acids.

The minerals determined in the batches, calcium, phosphorus, sodium and sulphur all decreased in amount as the levels of CDS decreased from Batch 1 (212 L/min) to Batch 3 (0 L/min) (Table 7). The minerals in Batches 2 and 4 having the same levels of CDS but with and without recycled DDGS (Batch 2 vs. Batch 4) were not significantly different as was observed for all the chemical components measured in this study, indicating that CDS level was the primary contributor to the differences in mineral levels among the batches. Three of the four minerals, phosphorus, sodium and sulphur have risks to the environment or livestock if they are over abundant in the feed. Phosphorus represents the most potential risk to the environment and hence its level in the diet should be accurate for optimum animal performance with minimum environmental impact (NRC, 2001). The levels in the batches ranged from 0.47% (Batch 3) with 0 L/min CDS to 0.78 (Batch 1) which was higher than the value in corn 0.3%. It appears that the opportunity in controlling the level of phosphorus in DDGS lies in the amount of CDS blended during drying. Sodium is an essential element, but if included at greater levels than needed by the bird, can lead to increased water consumption and wet litter or manure which can lead to additional bacterial growth increasing the susceptibility of a flock to intestinal infections (Applegate and Adeola, 2006). Sodium levels in DDGS can be controlled by the level of CDS added. The amount of sulphur in animal diets needs to be limited because polioencephalomalacia (PEM) in ruminants has been recognized as a consequence of excess sulphur intake (Kul et al., 2006). The sulphur levels in all the batches ranged from 0.54% to 0.66% which is more than the National Research Council (NRC) maximum tolerable levels (<0.30%). As with the other minerals, sulphur levels was higher in the CDS stream than in the WDG stream.

Table 6
Significant limiting amino acids of animal feeds in DDGS samples.

Amino acid, g/100 g	Wet distillers grains (WDG)*	Condensed distillers solubles (CDS)	Batch 1	Batch 2	Batch 3	Batch 4
Methionine	0.32[a]	0.13 (0.00)[b]	0.52 (0.02)[c]	0.55 (0.02)[cd]	0.62 (0.02)[e]	0.56 (0.01)[d]
Lysine	0.54[a]	0.35 (0.01)[b]	0.94 (0.03)[c]	0.96 (0.03)[cd]	1.08 (0.03)[e]	1.01 (0.02)[d]
Threonine	0.60[a]	0.25 (0.01)[b]	0.98 (0.04)[c]	1.01 (0.03)[c]	1.13 (0.02)[d]	1.01 (0.01)[c]
Tryptophan	0.17[a]	0.07 (0.01)[b]	0.19 (0.01)[ab]	0.20 (0.01)[b]	0.22 (0.02)[c]	0.20(0.01)[bc]

n = 3, values in parenthesis are standard deviation; same superscript letter within the same row indicates no significant difference ($P \geqslant 0.05$).
* Not replicated.

Table 7
Mineral contents in DDGS samples having feed and environmental limitations.

Minerals	Wet distillers grains (WDG)	Condensed distillers solubles (CDS)	Batch 1	Batch 2	Batch 3	Batch 4
Calcium (ppm)	100.0 (0.00)[a]	71.0 (4.00)[a]	519.3 (286.2)[b]	208.7 (24.7)[ab]	127.3 (7.4)[a]	166.7 (3.5)[a]
Phosphorus (%)	0.41 (0.01)[a]	0.28 (0.01)[b]	0.78 (0.03)[c]	0.65 (0.01)[d]	0.47 (0.01)[e]	0.62 (0.01)[d]
Sodium (ppm)	666.67 (57.74)[a]	471.7 (18.15)[b]	1142.0 (56.4)[c]	845.0 (9.5)[d]	411.7 (8.5)[b]	714.3 (11.2)[a]
Sulphur (%)	0.35 (0.00)[a]	0.19 (0.01)[b]	0.66 (0.03)[c]	0.60 (0.01)[d]	0.54 (0.01)[e]	0.60 (0.01)[d]

n = 3, values in parenthesis are standard deviation; same superscript letter within the same row indicates no significant difference ($P \geqslant 0.05$).

Table 8
Correlation between CDS and chemical components of DDGS.

	Protein	Fat	Fiber	Ash	Sugar	Glycerol
CDS[a]	−0.9464	0.9866	−0.9437	0.9923	0.9866	0.9752
Protein		−0.8985	0.9250	−0.9342	−0.9244	−0.9640
Fat			−0.9213	0.9813	0.9879	0.9678
Fiber				−0.9202	−0.9229	−0.9418
Ash					0.9884	0.9668
Sugar						0.9701

[a] CDS: condensed distillers solubles.

4. Conclusions

This study explained and quantified the variability in the physical and chemical properties of DDGS reported in the industry using a plant-scale study. It was shown that product variability of DDGS was primarily due to the levels of CDS added during the drying process. The solid (WDG) and liquid (CDS) streams after corn fermentation in ethanol production have different chemical properties as reported in this and other studies. Increasing CDS levels in the drying process progressively resulted in increasing darker colored DDGS with reduced levels of protein, ADF and NDF, while ash, oil, residual sugars and glycerol contents in DDGS increased. Additionally, the true and bulk density, particle size and particle size distribution increased with increasing CDS levels. The results from this study can be used in estimating the dietary limits for livestock feed based on the levels of CDS added and/or predicting trends in the physical and chemical properties of the DDGS during production. An obvious strategy for controlling the product consistency in DDGS lies in adding a consistent level of CDS to WDG during the drying process.

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