AGRICULTURAL MATERIALS

Comparison of Standard Moisture Loss-on-Drying Methods for the Determination of Moisture Content of Corn Distillers Dried Grains with Solubles

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This study quantified the variability among 14 standard moisture loss-on-drying (gravimetric) methods for determination of the moisture content of corn distillers dried grains with solubles (DDGS). The methods were compared with the Karl Fischer (KF) titration method to determine their percent variation from the KF method. Additionally, the thermo-balance method using a halogen moisture analyzer that is routinely used in fuel ethanol plants was included in the methods investigated. Moisture contents by the loss-on-drying methods were significantly different for DDGS samples from three fuel ethanol plants. The percent deviation of the moisture loss-on-drying methods decreased with decrease in drying temperature and, to a lesser extent, drying time. This was attributed to an overestimation of moisture content in DDGS due to the release of volatiles at high temperatures. Our findings indicate that the various methods that have been used for moisture determination by moisture loss-on-drying will not give identical results and therefore, caution should be exercised when selecting a moisture loss-on-drying method for DDGS.

Distillers dried grains with solubles (DDGS) is a co-product produced in corn dry-grind processing for ethanol production. It is a rich source of energy and is primarily used as a livestock feed ingredient. DDGS is a granular bulk product and is as hygroscopic as its parent feedstock, corn. DDGS contains about 9–14% moisture (wet basis, w.b.), 26–34% crude protein, and 8–13% fat (1). The quality and shelf life depend on the physical and chemical stability of DDGS, which are mainly influenced by moisture content. Accurate moisture determination is critical because water adds to the weight and must be paid for when the product is bought and sold (2). Moisture content also plays an important role in safe storage, and works in feedstuffs as a diluent to energy, protein, minerals, and vitamins in diets for animals (3). Proper moisture content of feed is necessary for optimum intake and maximum animal performance (3). In addition, moisture determinations are used when converting all other nutrient contents to a dry matter basis; therefore, errors in moisture determination are incorporated into measurements of other nutrient concentrations and calculations based on those concentrations (3).

Chen (4) evaluated six standard methods of moisture determination for rice and found that adequate drying time and temperature could give precise indication. Samuelsson et al. (5) reported the influence of temperature during oven-drying on moisture content value of biomass due to loss of volatile compounds. A halogen type moisture analyzer was found to be accurate and fast in determining moisture of flour (6).

The lack of standardized testing procedures for determining nutrient content of DDGS, and even one of the most basic of these measurements, moisture content (MC), complicates comparison of results obtained from different laboratories. Different methods are followed for MC determination by different industries and institutions according to convenience and availability of instruments. Gravimetric methods standardized by AOAC INTERNATIONAL, the American Association of Cereal Chemists (AACC) International, the National Forage Testing Association (NFTA), and the American Society of Agricultural and Biological Engineers (ASABE) are currently used for the determination of moisture content of DDGS. In most ethanol plants, the faster gravimetric method, halogen moisture analyzers, are used for moisture content determination.

A round-robin study was recently conducted by the American Feed Industry Association (AFIA) sub-working group to evaluate four standard gravimetric methods for determining the moisture content of DDGS (7). Five samples from six ethanol plants (five fuel dry-grind ethanol plants and one beverage ethanol plant) were analyzed at 23 participating laboratories. Along with crude protein, fat, and fiber, four loss-on-drying (gravimetric) methods (AOAC **934.01**, AOAC **935.29**, NFTA 2.2.2.5, and AOAC **930.15**) for moisture content determination were compared with the Karl Fischer

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Table 1. Methods used for DDGS moisture determination

Sl. No.	Method/Standard	ID	Procedure and conditions	Materials prescribed for the standard
1	AOAC **930.15** (9); AACC 44-19 (10)	A	Drying ground samples (2 g) in air oven at 135°C for 2 h	Moisture in feed
2	AOAC **925.10** (11)	B	Drying ground samples (2 g) in air oven at 130°C for 1 h	Total solids and moisture in flour
3	AACC 44-31 (12)	C	Drying ground samples (2 g) in air oven at 103°C for 3 h	Moisture and volatile matter in soy flour
4	AOAC **945.32** (13)	D	Drying ground samples (2 g) in air oven at 103°C for 3 h	Moisture of brewer's grains
5	AOAC **945.15** (14); AACC 44-15A (15); ASAE S352 (16)	E	Drying unground samples (15 g) in air oven at 103°C for 72 h	Moisture in cereals; moisture in corn; moisture of grain and seeds
6	NFTA 2.2.2.5 (17)	F	Drying ground samples (2 g) in air oven at 105°C for 3 h	Dry matter
7	AOAC **934.01** (18)	G	Drying ground samples (2 g) in vacuum oven at 105°C at 100 mm Hg for 5 h	Moisture in feed
8	AOAC **925.09** (19); AACC 44-40 (20)	H	Drying ground samples (2 g) in vacuum oven at 100°C at 25 mm Hg for 5 h	Solids and moisture in flour
9	AOAC **934.01** (high molasses; 18)	I	Drying ground samples (2 g) in vacuum oven at 70°C at 50 mm Hg for 5 h	Moisture in feed with high molasses content
10	Thermo-balance	J	Drying ground samples (1 g) in halogen moisture analyzer at 105°C until the reading stabilizes	Dry matter content
11	Karl Fisher method, AOAC **2001.12** (21)	K	Ground samples (~25 mg)	Moisture content

(KF) method (7). The moisture contents of the 30 samples of DDGS that were analyzed by the lead laboratory ranged from 9.0 to 12.7%. The AFIA sub-working group concluded that the NFTA 2.2.2.5 (2 g DDGS dried at 105°C for 3 h) was the best gravimetric method of the four standard methods investigated for moisture determination of DDGS.

The round-robin study conducted by the AFIA (7) sub-working group was a comprehensive evaluation, and a first good step toward the development of standard methods for chemical composition determination of DDGS. However, the comprehensive list of other standard methods reported in the literature for DDGS moisture determination and measurement practices that are used in fuel ethanol plants need to be thoroughly evaluated as well. This study worked from the premise that the importance of accurate moisture determination is necessary as a first step toward standardizing chemical composition analyses. We expanded the list of methods investigated in the AFIA (7) sub-working group round-robin study to include other loss-on-drying methods that have been reported in the literature or that we know are being used by fuel ethanol plants for moisture content measurement of DDGS or similar products. Thus, the specific objective of this study was to quantify the variability between various standard testing methods for determination of moisture content of DDGS using gravimetric methods. A comparison of the gravimetric methods with the KF method was also conducted.

Materials and Methods

DDGS Sample Handling and Preparation

The DDGS samples used in this study were obtained from three fuel ethanol dry-grind corn processing plants (one "old" and two "new" generation plants located in Indiana). An "old" generation dry-grind fuel ethanol process plant, as defined in this paper, is one that was built in the early 1980s, compared with "new" generation fuel ethanol dry grind processing plants built after 1990. Samples collected from these plants by our lab in previous studies suggest that DDGS from these two generation plants vary in physical and chemical characteristics. In this study, the DDGS samples obtained from the old generation plant were darker in color and larger in particle size than samples from the new generation plants.

The samples collected from the plants were stored in a controlled environment (at 4°C) to avoid mold growth and quality deterioration. Because DDGS particles tend to segregate during handling (8), extra caution was taken during DDGS sample preparation to prevent particle segregation. Thus, prior to removing DDGS samples from the storage container, the bulk was thoroughly mixed manually in order to uniformly homogenize DDGS particles. Subsamples for the moisture content measurements were prepared by splitting each sample from a plant into three replications using a Boerner Divider from Seedburo Equipment Co. (Chicago, IL). The replicated samples were then ground in a Wiley mill from Thomas Scientific (Swedesboro, NJ) using a 1.0 mm

Table 2. Chemical composition of DDGS samples used for the study[a]

	Plant 1		Plant 2		Plant 3	
Constituents, %	Mean ± SE[b]	CV[c], %	Mean ± SE	CV, %	Mean ± SE	CV, %
Crude protein	24.72 ± 0.12^{d}	0.47	27.76 ± 0.08^{e}	0.27	26.69 ± 0.14^{f}	0.14
Crude fat	11.38 ± 0.14^{d}	1.21	12.62 ± 0.06^{e}	0.48	10.80 ± 0.16^{f}	0.16
Crude fiber	5.76 ± 0.08^{d}	1.30	5.88 ± 0.10^{d}	1.70	5.28 ± 0.06^{e}	0.06
Ash	4.15 ± 0.02^{d}	0.48	4.35 ± 0.02^{e}	0.35	4.00 ± 0.15^{d}	0.15
Total reducing sugars	5.32 ± 0.13^{d}	2.44	2.82 ± 0.05^{e}	1.88	5.38 ± 0.27^{d}	0.27

[a] $n = 3$.

[b] SE = Standard error.

[c] CV = Coefficient of variation.

$^{d-f}$Percent moisture content followed by the same letters are not significantly different ($P \geq 0.05$).

sieve. For the KF method, samples were ground using a 0.5 mm sieve. The grinding time was less than a minute to avoid moisture loss from heat generation in the grinding chamber. The samples were then weighed into tarred moisture dishes before being placed in desiccators until moisture determination. Moisture content of the prepared samples was measured on the same day as sample preparation.

Moisture Content Methods Investigated

From the official methods of AOAC, AACC, NFTA, and ASABE (previously ASAE-American Society of Agricultural Engineers; 9–20), 14 loss-on-drying (gravimetric) methods specified for moisture content determination of grains, oilseeds, animal feed, and plant tissue were identified from various standards methods for moisture content determination for our study (Table 1). Of these 14 standard gravimetric methods, nine were distinctly different from each other, while four were common to two or more of the standard methods and, as a result, were not performed separately. Another gravimetric method included in this study used a thermo-balance (HB43 halogen moisture analyzer, Mettler Toledo Inc., Columbus, OH). Thermo-balances are routinely used in ethanol plants for rapid determination of moisture content of DDGS during product drying.

In order to determine the accuracy of the loss-on-drying methods for moisture content determination of DDGS, the KF titration method according to AOAC Method **2001.12** (21) was used to determine the moisture content of the samples. KF

Table 3. Moisture content of DDGS by the various standard methods used[a]

	Plant 1		Plant 2		Plant 3	
ID	Mean ± SE[b]	CV[c], %	Mean ± SE	CV, %	Mean ± SE	CV, %
A	14.76 ± 0.42^{d}	2.86	14.46 ± 0.22^{d}	1.53	13.07 ± 0.19^{d}	1.45
B	11.76 ± 0.23^{e}	1.91	12.53 ± 0.10^{e}	0.80	11.27 ± 0.03^{f}	0.25
C	11.66 ± 0.17^{e}	1.48	12.43 ± 0.07^{e}	0.54	11.28 ± 0.05^{f}	0.48
D	9.92 ± 0.06^{f}	0.58	10.71 ± 0.01^{g}	0.05	11.75 ± 0.11^{e}	0.91
E	13.42 ± 0.18^{h}	1.31	13.35 ± 0.15^{h}	1.09	12.31 ± 0.12^{h}	0.95
F	9.53 ± 0.13^{f}	1.35	11.49 ± 0.10^{f}	0.91	10.63 ± 0.11^{g}	1.02
G	8.68 ± 0.3^{g}	3.44	10.49 ± 0.08^{g}	0.73	10.32 ± 0.06^{i}	0.56
H	8.51 ± 0.28^{g}	3.27	10.39 ± 0.09^{g}	0.90	10.15 ± 0.07^{i}	0.72
I	6.52 ± 0.06^{i}	0.81	9.18 ± 0.05^{i}	0.56	9.57 ± 0.08^{j}	0.85
J	6.18 ± 0.33^{i}	5.38	6.65 ± 0.08^{j}	1.22	8.72 ± 0.09^{k}	0.99
K	6.59 ± 0.15^{i}	2.28	6.63 ± 0.14^{j}	2.09	8.76 ± 0.07^{k}	0.81

[a] $n = 3$.

[b] SE = Standard error.

[c] CV = Coefficient of variation.

$^{d-k}$Percent moisture content followed by the same letters are not significantly different ($P \geq 0.05$).

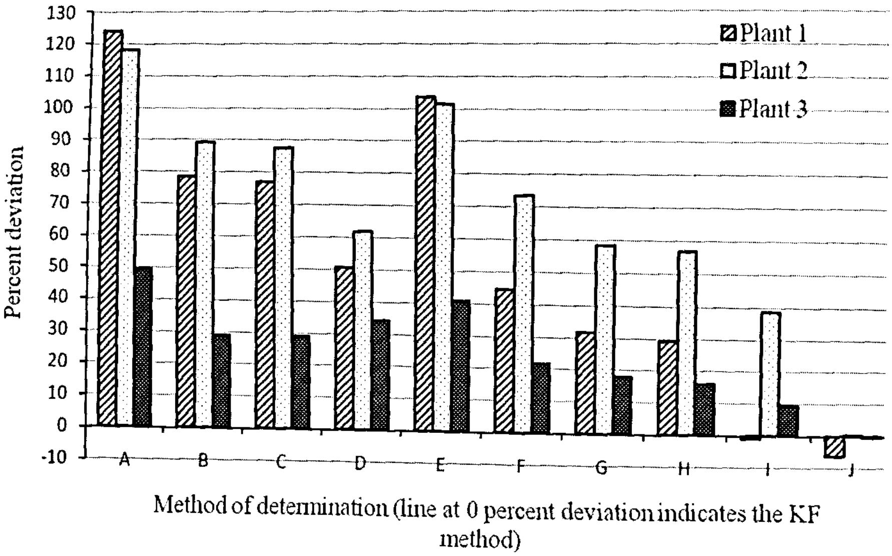

Figure 1. Percent deviation of moisture content by loss-on-drying (gravimetric) methods compared with KF titration method.

is a direct method for moisture determination using a specific chemical reaction (22).

Moisture Content Determination by Gravimetric Methods

The aluminum dishes (with lids) used for moisture determination were prepared according to the procedure specified in the methods. For the air-oven methods, the oven was preheated to the specified temperature before placing the samples inside. For vacuum-oven methods, the temperature and vacuum pressure of the oven were calibrated at least 2 h before moisture content determination. After drying, the aluminum dishes were closed and kept in a desiccator (about 30 min) for cooling to ambient temperature. For moisture content calculations, sample weights before and after experiments were recorded using a digital balance of 0.0001 g accuracy (AB 104-S, Mettler-Toledo, Inc.).

The method of moisture determination using a thermo-balance involved drying about 1 g of ground sample at 105°C in an electrically heated chamber while the unit automatically weighs moisture loss from the sample in the process. Heating stops when the weight of the sample is stable for 30 s. The time taken for a measurement usually ranges from 4.5 to 5.3 min. The KF method and procedure used by Thiex and Van Erem (23) were followed in this study. The KF apparatus used was a new Brinkmann Instrument, Inc. 795 KFP Titrino with homogenizer (Westbury, NY). Standard procedure given by the equipment manufacturer was used to calibrate the KF apparatus before conducting the experiments according to AOAC Method **2001.12** (21). The laboratory standard error of the apparatus was 0.03%. Moisture content determinations were conducted in triplicate for subsamples from the three ethanol plants. All moisture contents were reported on a wet basis.

Chemical Composition Analysis of DDGS Samples

Three replicate subsamples from each DDGS sample group (from old and new generation plants) weighing about 40 g per replicate were analyzed for crude protein, crude fat, crude fiber, ash, and total reducing sugars in an external laboratory using AOAC *Official Methods*^{SM} for proximate analyses. The AOAC methods used were **984.13** for crude protein, **920.39** for crude fat, **978.10** for crude fiber, and **942.05** for ash content; HPLC was used to analyze for total reducing sugars.

Statistical Analysis of Data

One-way analysis of variance (ANOVA) using PROC GLM procedure in SAS v9.1 (SAS Institute, Cary, NC) was used to determine whether the mean differences between the moisture content determined by the various standard gravimetric methods were statistically significant. Tukey's Studentized Range test was used for comparison of the moisture content among the gravimetric methods at the 0.05 probability level. The CV, % within replications of a method was used to evaluate the method repeatability. The KF method was used as the bench-mark for evaluating the accuracy of the gravimetric methods in measuring the moisture content of DDGS. The gravimetric method(s) with the least percent deviation from the KF method was considered the most suitable for moisture determination of DDGS. The percent deviation from the KF method was calculated using the following equation:

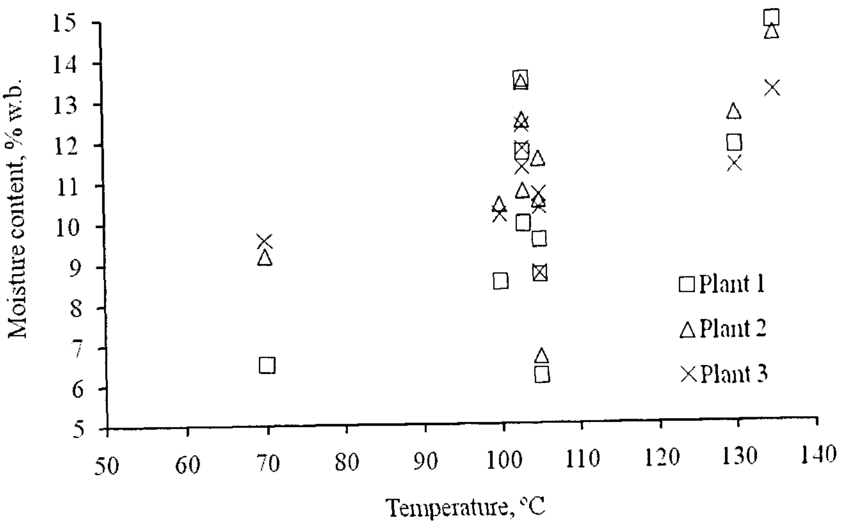

Figure 2. Correlation between moisture content and temperature of drying.

$$\text{Percent deviation} = \frac{MC_G - MC_{KF}}{MC_{KF}} \times 100 \quad (1)$$

where MC_{KF} and MC_G are the moisture content determined by KF method and gravimetric methods, respectively.

Results and Discussion

Chemical Composition of Samples Used for the Study

The chemical compositions of the samples from the three dry-grind fuel ethanol plants are presented in Table 2. Plant 1 was an old generation plant, while Plants 2 and 3 were new generation plants built within the last 4 years. The chemical composition of DDGS samples from all three plants (crude protein, crude fat, crude fiber, ash, and total reducing sugars) was significantly different from one another. DDGS from the new generation plants had higher crude protein contents than DDGS from the old generation plant. The total reducing sugars were significantly higher in samples from Plants 1 and 3 than samples from Plant 2. In a recent study by the authors (24), the total reducing sugars were found to correlate with the amount of condensed distillers solubles (CDS) blended with wet distillers grains during drying to produce

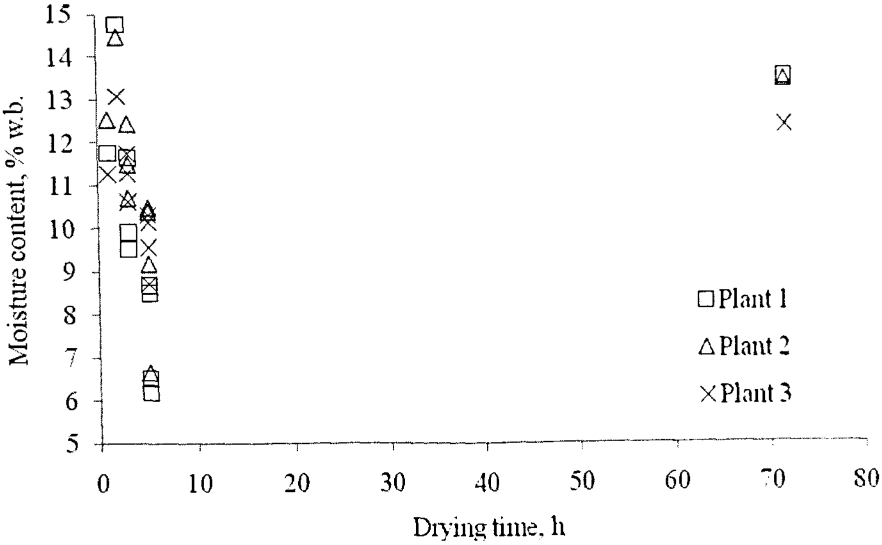

Figure 3. Correlation between moisture content and duration of drying.

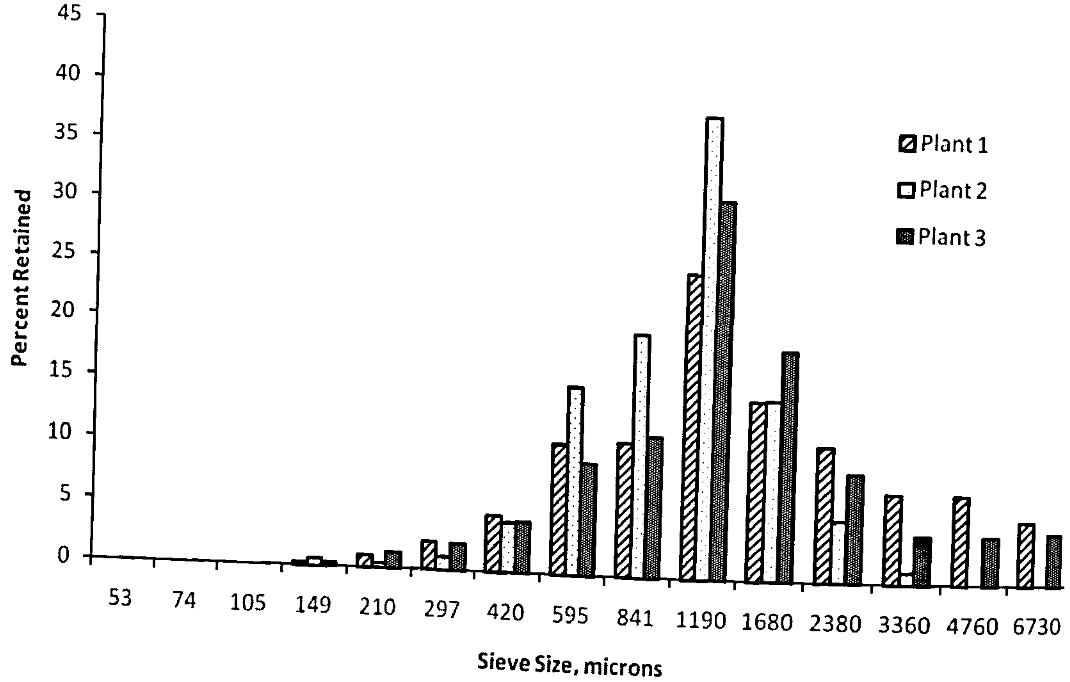

Figure 4. Particle size distribution of DDGS samples.

DDGS. Increased CDS levels increased the amount of total reducing sugars in DDGS.

Comparison of Moisture Loss-on-Drying (Gravimetric) Methods for Moisture Content Determination

Table 3 shows the data of the moisture content (w.b.) results for all the methods measured in this study for samples of DDGS from all three fuel ethanol plants. One-way ANOVA showed significant differences among all the standard gravimetric methods, the thermo-balance method, and the KF method used for moisture content determination. Among all the methods, the moisture content ranged from 6.18 to 14.76% for DDGS samples from Plant 1, 6.63 to 14.46% for samples from Plant 2, and 8.72 to 13.07% for samples from Plant 3 (Table 3). These very large ranges of moisture recorded among gravimetric methods and KF underscore the need for standardization of moisture content determination.

In general, the moisture contents determined by the various gravimetric methods using three different samples from three fuel ethanol plants followed a similar trend. Among the gravimetric methods, the highest moisture contents, 14.76, 14.46, and 13.07% for Plants 1–3, respectively, were obtained by AOAC **930.15**/AACC 49-19 (2 g ground sample dried at 135°C for 2 h) used for moisture determination in feed. Likewise, the thermo-balance method using 1 g of sample at 105°C in an electrically heated chamber (a method commonly used in fuel ethanol plants) gave the lowest moisture contents: 6.18, 6.65, and 8.72%, for samples from Plants 1–3, respectively.

The deviation of the standard methods for moisture loss-on-drying from KF decreased with drying temperature (Figure 1). Also, deviation of moisture using loss-on-drying methods from the KF method was lower for samples from Plant 3, than for samples from Plants 1 and 2. The difference in sample chemistry (Table 2) of samples from the three plants does not give enough insight as to why the deviations seen would be different, nor does the particle size (Figure 4). Further investigation, including more samples, needs to be conducted in order to understand the causes of these differences. The recommendation of the most appropriate moisture loss-on-drying method based on the method that gave the least deviation from the KF method, as in the study by Thiex (7), was not the intent of this study because of the limited number of samples used, which was conducted by a single lab. Therefore, no conclusion of the accuracy of the moisture loss-on-drying methods compared with KF was drawn in this study.

The moisture content determined using various methods had good correlation with the temperature of drying (Figure 2). Higher temperature during drying leads to loss of volatiles, which might have resulted in higher moisture values at elevated drying temperatures. By comparing gravimetric methods (Table 1), it can be seen from the data (Table 3) that the moisture content of DDGS from all three plants determined by method ID ‘A’ at a higher temperature (135°C for 2 h) was higher than the moisture content of the same samples determined by method ID ‘B’ and ‘C’ at lower temperatures, 130°C for 1 h and 103°C for 3 h, respectively (Table 3). Likewise, the effect of temperature can also be seen by comparing method ID 'H' (100°C for 5 h) with 'I' (70°C for 5 h). The moisture content determined by the former (method ID 'H') was higher than the latter (method ID 'I'), which was lower in temperature. The influence of drying time on moisture determination can be seen by comparing method ID 'D' (103°C for 3 h) with method ID 'E' (103°C for 72 h; Table 3). Even though method ID 'E' was conducted with 15 g of unground DDGS compared with 2 g of ground DDGS for method ID 'D', the moisture determined by method ID 'E' was higher than ID 'D.' This was most likely due to the 72 h drying time, given that they both were dried at the same temperature (103°C). However, duration of drying was not correlated with the moisture content of DDGS (Figure 3), which implies that the moisture content values were more dependent on the temperature during the drying process. Therefore, temperature and, to a lesser extent, drying times are critical to accurate moisture measurement of DDGS using loss-on-drying methods.

The KF titration method results were 6.59, 6.63, and 6.00%, for Plants 1–3, respectively, and were significantly lower than all but one (thermo-balance method, ID 'J,' for Plant 1) of the loss-no-drying methods investigated in this study. The thermo-balance method also had the lowest percent variation, compared to the KF method for all three samples used (Figure 1). Of the standard loss-on-drying methods investigated, AOAC Method **934.01** using 2 g ground samples in a vacuum oven at 70°C at 50 mm Hg for 5 h (ID 'I,' Table 1) gave the lowest percent variation in comparison with the KF method. AOAC Method **934.01** method is recommended for use in feed with high molasses content. The particle size distribution of DDGS obtained from Plant 1 and Plant 3 were almost similar and slightly skewed to the right, indicating the presence of larger particles (Figure 4). Larger particles are formed mainly due to the agglomeration of particles by the addition of CDS during the drying process. Since most of the standard methods for moisture determination used ground samples (except for the method ID 'E' in Table 1), the effect of particle size on moisture content was not noticed in this study.

Conclusions

In this study, the primary goal was to quantify the variability among various moisture loss-on-drying methods that have been used for moisture determination of DDGS. It was observed that the moisture content variability among 11 distinct methods listed in 14 standard methods of moisture loss-on-drying was significantly different. Their variability can be linked primarily to the drying temperatures and, to a lesser extent, to the drying times. DDGS, being a byproduct of fermentation, contains a lot of volatiles, which, when dried at high temperatures, could be released along with moisture, thus exaggerating the product moisture in a moisture loss-on-drying measurement. Therefore, the use of the standard methods investigated for moisture loss-on-drying methods for moisture determination of DDGS will not give identical results and caution must be exercised when selecting an appropriate moisture loss-on-drying method. The recommendation of the NFTA 2.2.2.5 method (2 g ground sample dried at 105°C for 2 h) for DDGS (7) appears to be within the temperature range and drying time limiting volatile release, while ensuring most of the water in the sample is given off during drying.

Acknowledgments

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