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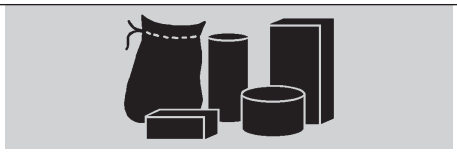

Nutrient variability for distillers grains plus solubles and dry matter determination of ethanol by-products[1]

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ABSTRACT

Three experiments were conducted to evaluate nutrient content and DM determination methods of dry milling by-products. In Exp. 1, nutrient composition was determined for wet distillers grains plus solubles (WDGS) and modified distillers grains plus solubles (MDGS) from 6 ethanol plants with 10 samples collected per day, across 5 d, and sampling was repeated over 4 separate months. Mean composition was 31.0% CP, 11.9% fat, 0.84% P, and 0.77% S (DM basis). Coefficients of variation for DM content were greater for some plants than others, and variation occurred within and across days. Variability was small for CP and P, whereas fat differed among ethanol plants. Large variation in means and CV were observed for S in period 1, but variation subsequently decreased. Coefficients of variation for S were similar for samples collected within the same day and across days. In Exp. 2, samples of WDGS, MDGS, Dakota Bran Cake, and distillers solubles were used to determine DM content by drying samples at 105°C for 3, 8, and 24 h and 60°C for 24 and 48 h, vacuum oven drying, toluene distillation, and Karl Fischer titration. Compared with toluene distillation, drying at 105°C resulted in less DM ($P \leq 0.10$) and vacuum drying and Karl Fischer titration resulted in greater DM ($P < 0.01$). In Exp. 3, additional WDGS, MDGS, and wet grains with no solubles were used to determine DM with oven drying at 60°C for 48 h, oven drying at 105°C for 3 h, or toluene distillation. Drying at 60°C for 48 h was similar to toluene distillation ($P \geq 0.60$).

Key words: distillers grains, dry matter, laboratory method, nutrient composition, variation

INTRODUCTION

Although wet distillers grains plus solubles (**WDGS**) has become a common feedstuff in the livestock industry, there is concern about its nutrient composition and consistency (Babcock et al., 2008). Three nutrients commonly measured in WDGS are DM, fat, and S. Price paid for WDGS on a DM basis may be problematic if the DM content is less than expected or is incorrectly determined. If large amounts of high-fat WDGS are fed, then cattle intakes may decrease if dietary fat is greater than 8% DM (Vander Pol et al., 2009). The NRC (1996) suggested the maximum tolerable S level was 0.40% for potential occurrence of polioencephalomalacia, thus making S in WDGS important if it is high or variable. Little research has been reported on nutrient variability with WDGS.

Methods to determine the DM of feeds are widely used in the agriculture industry. Given the variation in moisture, understanding these methods is of particular importance when considering wet ethanol by-products (50 to 70% moisture). Dry matter content of feeds is typically defined as the material remaining after heating the sample in an oven for a fixed period of time, with the calculated loss of weight assumed to be water. This method is used most commonly because it is rapid and inexpensive. However, Mo and Tjornhom (1978) determined volatile organic substances are also lost and additional side reactions may occur for wet, fermented forages during the oven-drying process. Toluene distillation offers an alternative method to determine

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DM content of feed through direct but separate removal of moisture (Brahmakshatriya and Donker, 1971). However, no published research exists for comparing DM methods in wet by-products. Our objectives were to determine nutrient composition plus variability for WDGS from several ethanol plants across many days and to compare drying methods to toluene distillation for determining DM of wet by-products.

MATERIALS AND METHODS

Experiment 1

Six ethanol plants in Nebraska agreed to sample distillers grains plus solubles. Four plants produced WDGS and 2 plants produced modified distillers grains plus solubles (**MDGS**), but the samples will be generally referred to as DGS to maintain confidentiality. A collected sample represented a semitruck load of DGS a cattle producer would receive. Samples were collected from 4 to 5 locations in the DGS pile to be loaded on a semitruck or from the loader that filled the truck. These samples were combined and mixed thoroughly, and a 250- to 500-g subsample was collected and placed into a plastic, air-tight bag and frozen. Ten samples were taken across a day for 5 consecutive days, with 50 total samples during the week. This was repeated over 4 sampling months (periods) throughout a year, totaling 200 samples per ethanol plant and 1,200 samples in the data set. Samples were shipped frozen overnight following the sampling period to the University of Nebraska–Lincoln ruminant nutrition laboratory for analysis.

Analyses for DM, CP, fat, P, and S content were conducted in duplicate. If the CV was greater than 5%, then the analysis was repeated and the new results were used. Based on results from Exp. 1 and 2, DM analysis was conducted using a 60°C oven for 48 h because this method is statistically similar to toluene distillation. After drying, samples were ground through a 1-mm screen (Wiley Mill, Thomas Scientific, Swedesboro, NJ) before nutrient analysis. Crude protein was calculated from percent nitrogen using a LECO nitrogen analyzer (LECO Corp., St. Joseph, MI; AOAC, 1999; method 990.03). Phosphorus and S were determined by wet ashing with nitric and perchloric acids and analyzed colorimetrically (AOAC, 1999; methods 968.08, 965.17; Tinsdale et al., 1985). Fat was determined by extraction with petroleum ether under pressure in filter bags (AOCS, 1998; method Am 5–04). Fat, P, and S analyses were performed at a commercial laboratory (Ward Laboratories Inc., Kearney, NE).

Data were summarized by day, ethanol plant, and sampling period to compare mean nutrient values. Coefficients of variation were calculated to evaluate variability within day, across day, and within plants. A CV was calculated each day (10 samples/d) within each ethanol plant and sampling period. These 5 CV per ethanol plant and period were then averaged, and this CV value will be expressed as "within-day variation." Average nutrient content was calculated per day. These daily averages (5 d) within each period and ethanol plant were used to calculate a CV, which will be expressed as "across-day variation." Statistical analysis on the within-day variation CV within period for each nutrient was conducted using the Proc Mixed procedure of SAS (Version 8.02, SAS Inst. Inc., Cary, NC), which used the within-day CV from each day as the experimental unit. This procedure was used to evaluate average ethanol plant nutrient composition by using average daily nutrient composition as the experimental unit. Probabilities less than or equal to 0.05 were considered significant.

Experiment 2

Four different types of high-moisture, by-product feeds were used to evaluate drying methods for determining DM content. These feed samples included WDGS (31–35% DM; Abengoa Bioenergy, York, NE), MDGS (42–48% DM; Husker Ag, Plainview, NE), Dakota Bran Cake (**Dbran**, 50–54% DM; POET Nutrition, Sioux Falls, SD), and distillers solubles (**DS**, 25–35% DM; Abengoa Bioenergy). Random grab samples were obtained from the piles (representing one semitruck load) of wet by-products that were being fed to cattle at the University of Nebraska–Lincoln Agricultural Research and Development Center research feedlot near Mead, Nebraska. These samples were mixed together (totaling 2.5 kg) and subsampled for each analysis of DM.

Methods for determining DM included drying samples in a 60°C forced air oven for 24 or 48 h, drying samples in a 105°C forced air oven for 3, 8, or 24 h, using a vacuum oven, toluene distillation, and Karl Fischer titration. The 105°C and 60°C oven methods were conducted by weighing 5 g as-is sample into dry aluminum pans (8 replications). Weights were recorded on the same samples at 3, 8, and 24 h for the 105°C oven and at 24 and 48 h for a different set of samples in the 60°C oven. A vacuum oven analysis (AOAC, 1999; method 934.01) was conducted on each sample type (3 replicates). Samples were weighed (5 g as-is) into preweighed moisture tins and placed on a vacuum oven tray. Trays were placed in a 70°C vacuum oven, the door was sealed, and the vacuum was applied at 50 mmHg. After 4 h, the vacuum was turned off, and the tins were removed from the tray, allowed to cool in a dessicator, and then weighed. In addition, a Karl Fischer titration (AOAC, 1999; method 2001.12; Thiex and Van Erem, 2002) was conducted in duplicate on all samples. Toluene distillation (AOAC, 1999; method 925.04) was conducted in duplicate on every sample. A 25-g as-is sample was weighed into a 250-mL Pyrex round-bottom flask, and toluene was added to cover the sample. Toluene was rinsed down the sides of the condenser into the collection trap, and the trap was filled until toluene ran over into the flask. Heat was provided to the flask so the toluene would boil within 10 min, at which point the 90-min reflux began. Moisture measurements