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# Composition of corn dry-grind ethanol by-products: DDGS, wet cake, and thin stillage

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#### Abstract

DDGS and wet distillers' grains are the major co-products of the dry grind ethanol facilities. As they are mainly used as animal feed, a typical compositional analysis of the DDGS and wet distillers' grains mainly focuses on defining the feedstock's nutritional characteristics. With an increasing demand for fuel ethanol, the DDGS and wet distillers' grains are viewed as a potential bridge feedstock for ethanol production from other cellulosic biomass. The introduction of DDGS or wet distillers' grains as an additional feed to the existing dry grind plants for increased ethanol yield requires a different approach to the compositional analysis of the material. Rather than focusing on its nutritional value, this new approach aims at determining more detailed chemical composition, especially on polymeric sugars such as cellulose, starch and xylan, which release fermentable sugars upon enzymatic hydrolysis. In this paper we present a detailed and complete compositional analysis procedure suggested for DDGS and wet distillers' grains, as well as the resulting compositions completed by three different research groups. Polymeric sugars, crude protein, crude oil and ash contents of DDGS and wet distillers' grains were accurately and reproducibly determined by the compositional analysis procedure described in this paper. © 2007 Elsevier Ltd. All rights reserved.

Keywords: Analysis of DDGS; Compositions; Ethanol; Dry grind process

#### 1. Introduction

Fuel ethanol production from corn grain in the US exceeded 4.9 billion gallons in 2006. Of that production, 82% was from facilities employing some variation of the dry-grind process for ethanol production (Renewable Fuels Association, 2007). Dry-grind processes are characterized

by a lack of a steeping step at the front end of the process, a hallmark of wet milling of corn, and little or no fractionation of the corn kernel components prior to saccharification of the starch and fermentation (Kwiatkowski et al., 2006). In dry-grind processes, the whole grain is ground by hammer mills into a course powder with a mean particle diameter of approximately 1 mm (Rausch et al., 2005). An aqueous slurry of yeast cells and residuals from the ground corn kernels remaining after fermentation pass through a stripper where the ethanol is recovered. The non-volatile components then leave this step as a product called whole stillage (Bothast and Schlicher, 2005). Whole stillage contains the fiber, oil, protein, other unfermented components of the grain, and yeast cells. Whole stillage is usually

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centrifuged to produce a liquid fraction (thin stillage) and a solids fraction (wet distillers' grains). A significant fraction (15% or more) of the thin stillage is recycled as backset to be used as process water at the front end of the process to slurry the ground grain (Kwiatkowski et al., 2006). The remaining thin stillage is concentrated through multiple effect evaporators to produce a syrup called condensed distillers' solubles (CDS) (Ganesan et al., 2006). While wet distillers' grains, syrup, or the combination of both (wet distillers' grains with solubles, WDGS) can be sold as an animal feed, the combination of wet distillers' grains and syrup is often dried to produce dried distillers' grains with solubles (DDGS) in order to greatly lengthen its shelf-life (McAloon et al., 2000).

Composition of DDGS has been of great interest to researchers in the area of animal science, ethanol producers, and especially to people in the feed industry as majority of it has been sold as a feed ingredient for livestock. A major consumer of the DDGS is the animal feed industry. The compositional analysis of DDGS has been focusing on nutritional value of DDGS, such as digestibility, total digestible nutrients, net energy, amino acid and mineral profiles. Compositional analysis of the corn-to-ethanol by-products have utilized established methods for determining the nutritional value of forages and grain animal feeds (Spiehs et al., 2002; Stein et al., 2006). An extensive compositional analysis of DDGS has been completed by several researchers. The averaged composition of 118 samples of DDGS (Spiehs et al., 2002) collected from 10 different dry grind facilities as well as composition of DDGS collected at one plant over a five-year period (Belyea et al., 2004) are summarized in Table 1. Major components of DDGS have been given as crude protein, crude fat and crude fiber. Since DDGS production has tripled in the past decade to an annual production of 12 million metric tons in 2006 (Renewable Fuels Association, 2007), additional efforts are underway to further develop and standardize these methods to insure feed quality as these ethanol

Table 1

Composition of distillers' dried grains with solubles (DDGS), previously reported by Spiehs et al. (2002) and Belyea et al. (2004)

	Spiehs et al., mean value, coefficients of variation	Belyea et al., mean (%)
Moisture content (% total)	11.1	Na
Dry matter content (% total)	88.9 (1.7)	Na
Total mass closure	100.0	
Crude protein	30.2% (6.4)	31.3
Crude fat	10.9% (7.8)	11.9
Crude fiber	8.8% (8.7)	10.2
Starch	Na	5.1
ADF	16.2% (28.4)	17.2
Ash	5.8% (14.7)	4.6
Ash	5.8% (14.7)	4.6

All values are % dry basis except where otherwise noted.

byproducts become a larger share of the animal feed market (American Feed Industry Association, 2007). As a result of a lack of standardization the compositional analysis procedures and resulting composition of the same DDGS were slightly varied depending on the methods applied by each research group.

This paper reports averaged composition of a common lot of DDGS, wet distillers' grains, and thin stillage, measured by three research groups, as well as the detailed analvsis procedures that have been applied by the groups. The composition includes not only the common compositions such as proteins, fat, and ash, but also cellulose, xylan, arabinan, and starch contents that are especially valuable for research on enzymatic hydrolysis and fermentation of DDGS. The carbohydrates present in the fiber component of DDGS (cellulose and hemicellulose) have potential value as a source of fermentable sugars for increased ethanol yield per bushel of corn. Additionally, these polysaccharides are indigestible in monogastric livestock (e.g. swine and poultry) and are of limited value as feed components for cattle. Therefore, the cellulose and hemicellulose in DDGS presents a potential opportunity for implementing cellulose conversion technologies into the current US ethanol industry. Evaluating DDGS as an additional source for fermentable sugars requires a different set of compositional analysis. This includes more detailed chemical analysis, especially on polymeric sugars that can release fermentable sugars upon enzymatic hydrolysis. Application of analytical methods developed for analyzing the composition of cellulosic biomass has been applied to corn-to-ethanol byproducts with varying success in closing the material balance (Mosier et al., 2005; Tucker et al., 2004).

Comparisons between the methods and suggestions for improved analytical techniques are presented. Research results on the DDGS utilization published in this special issue are based on the composition given in this paper. We also include terminology for several terms frequently used throughout this special issue to ease understanding and communication between researchers.

#### 2. Methods

DDGS, wet distillers' grains (wet cake), and thin stillage were obtained from an operating dry-grind ethanol facility, Big River Resources, LLC (West Burlington, IA). Reagents and chemicals, unless otherwise noted, were purchased from Sigma–Aldrich (St. Louis, MO).

### 2.1. HPLC analysis

HPLC analysis of liquid samples was performed on a system consisting of a Varian 9010 Solvent Delivery System, Waters 717plus Autosampler, Aminex HPX-87H column (Biorad, Hercules, CA), Waters 2414 Refractive Index Detector, Waters 2487 Dual  $\lambda$  Absorbance Detector, and a Hewlett Packard HP3396G Integrator. The mobile phase

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