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Original Research Paper

The effect of process variables on drum granulation behavior and granules of wet distillers grains with solubles

Kyle V. Probst [a,b], Klein E. Ileleji [b,*]

[a] Department of Grain Science and Industry, Kansas State University, Manhattan, KS, USA
[b] Department of Agricultural and Biological Engineering, Purdue University, West Lafayette, IN, USA

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ABSTRACT

A laboratory scale batch drum granulation process was used to manufacture granules of DDGS by adding condensed distillers solubles (CDS) as a binder to wet distillers grains (WDG), coproducts from corn dry-grind ethanol process, under varying formulation and process conditions. A full factorial experimental design was used to test all combinations of factor levels, which included the amount of CDS binder (30%, 35%, and 40% (w/w)), CDS binder solids content (22% and 38%, wet basis), screen size opening (3.175 and 6.35 mm), and residence time (1, 2.5, 5, and 10 min). Measured response variables included granule size (geometric mean diameter), granule yield (1.68–3.35 mm), bulk density, true density, and porosity. Results show that as the amount of binder added increased from 30% to 40% the granule size significantly increased from 2.08 to 4.45 mm, respectively. Increasing the binder solids content resulted in granules with greater bulk density. Granules produced from the low solids CDS had lower porosity as a result of intraparticle voids in them. Screen sizing influenced nucleation and produced significant differences in the final granule size distribution. The greatest growth was observed for granules prepared with 40% amount of binder and low solids content CDS. Compression testing of wet granule compacts corresponded to observed growth behavior; wet pellets made from higher solids CDS had greater resistance to deformation and enhanced plasticity while the opposite was observed for the low solids CDS. The best yields of granules between 1.68 and 3.35 mm were achieved at ~80% using 35% amount of binder with the high solids content CDS, the small screen size opening and residence times between 2.5 and 10 min.

1. Introduction

Corn ethanol production in the US has increased exponentially in the past decade with 192 plants currently in operation producing over 15 billion gallons per year (RFA 2014). Dried distillers grains with solubles (DDGS) is a major co-product of the dry-grind process with 35.5 million metric tons produced during the 2012–2013 market year [1]. Often priced at $200–300 per ton, DDGS is sold as a feed additive for ruminants, swine, and poultry; it is an important contributor to operation profitability accounting for 27% of the total revenue for dry-grind ethanol production facilities in 2013 [1]. Though DDGS is an integral revenue stream for the dry-grind process, DDGS storage and transportation has been a major hurdle for the ethanol industry mainly due to its inconsistent bulk properties. Development of low-cost technologies capable improving the handling, storage and marketability of DDGS would be highly valuable for the dry-grind ethanol industry.

The dry-grind ethanol process (which accounts for ~80% of the US fuel ethanol industry) produces ethanol, DDGS, and carbon dioxide; 7.7 kg of DDGS is generated per 25.4 kg of corn (one bushel of corn) [2]. As described by Rausch and Belyea [3], the dry-grind process begins when the corn is ground into a coarse, whole flour. Water is added to the flour to form a slurry which is cooked to gelatinize the starch. Alpha-amylase and glucoamylase enzymes are added to break down the starch into usable sugars, which are fermented into ethanol by the yeast *Saccharomyces cerevisiae*. The ethanol is removed via distillation while the remaining whole stillage, the non-fermented slurry portion, is centrifuged. The centrate, known as thin stillage, is condensed by evaporators into a thick syrup known as condensed distillers solubles (CDS) ranging in dry matter content from 23% to 45% [4]. The coarse, solid materials dewatered by the centrifuge are

* Corresponding author at: Department of Agricultural and Biological Engineering, Purdue University, 225 South University Street, West Lafayette, IN 47906, USA. Tel.: +1 (765) 494 1198; fax: +1 (765) 496 1115.
E-mail address: ileleji@purdue.edu (K.E. Ileleji).

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known as wet distillers grains (WDG) or wet cake and have a moisture content ranging from 65% to 75%. The WDG and CDS are combined, known as wet distillers grains with solubles (WDGS), and dried in large gas-fired rotary drum dryers producing DDGS, a bulk granular solid rich in protein, fat, fiber, minerals, and vitamins with a bulk density ranging from 391 to 590 kg/m^3, an average particle size ranging from 790 to 1500 μm, and moisture content from 8% to 13% [5–8].

The handling and transportation of bulk DDGS remains to be a major challenge due to issues with caking, poor flowability, and segregation [5,9]. During railcar transportation, DDGS caking is a real burden resulting in limited flowability and the tendency to avalanche during unloading making DDGS handling not only difficult, but dangerous [5,10]. Additionally, the particle heterogeneity of DDGS can result in segregation, inconsistencies in bulk density and nutrient variability [11,12]. To relieve the issues with DDGS handling, the densification of DDGS using pellet mills was investigated by Rosentrater and Kongar [13], who reported DDGS pelletization would help reduce transportation costs. Unfortunately the high capital investment and operating costs to build and operate a pellet mill has restricted the adoption of this technology by the corn dry-grind ethanol industry.

Drum granulation is an alternate process that can be used to increase the density, particle size, and uniformity of DDGS. It has been successfully used by others to produce densified granules from other sources of biomass [14,15]. Drum granulation is a wet granulation method that agglomerates smaller particles into larger aggregates by agitating particles using a cylindrical rotating drum in the presence of a liquid binder [16]. The rotating action of the drum provides the mechanical energy needed for particle collision and agglomeration [17]. Three rate processes are used to describe the granulation phenomenon: nucleation, consolidation and growth, and breakage and attrition [18]. Nucleation is the first stage of granulation and is defined as the formation of initial particle assemblies under the strength of a liquid binder. Consolidation and growth occurs when nuclei collide together to form larger sized aggregates. Before growth, nuclei often go through a period of consolidation where particles are squeezed into the core of the nucleate allowing the liquid binder to form a layer around the outside. Variables that affect the degree of consolidation include binder addition, collision velocity (agitation), feed particle size, collision frequency and binder viscosity. Similar to consolidation, growth is affected by particle size, binder viscosity, the amount of binder, and collision velocity. The amount of binder affects the amount of liquid saturation, as more binder is added, the granulation regime moves from nucleation to steady growth to rapid growth and, finally, slurry/over wet-mass [19]. Granule attrition and breakage is affected by the collision forces due to mechanical agitation along with the capillary, viscous, and frictional forces affecting granule deformation and strength [16]. Granule deformation is related to the elastic–plastic behavior of materials, increased granule plasticity often results in less breakage while granules exhibiting semi-brittle behavior often fracture more easily.

Preliminary work by Probst and Ileleji [20] showed that drum granulated DDGS had an increased average particle size, bulk density, and enhanced particle uniformity compared to non-granulated DDGS. Using existing infrastructure such as a rotary drum dryer, granulation has the potential to alleviate the issues surrounding DDGS handling, requiring minimal start-up and operating costs. It should be noted that during the commercial drying of DDGS, some degree of granulation does occur, though unintentional, producing what are known by the industry as 'syrup balls' as a result of poor CDS dispersion throughout the WDG. Controlling the formation of syrup balls using a granulation step has potential to generate more uniform and consistent DDGS with improved handling. Additionally, the distinct spherical nature of granulated DDGS could introduce new, higher value market opportunities outside of animal feed. For example, granulated DDGS could be used as a biobased carrier for fertilizer and other chemicals for lawn, garden and even industrial agriculture applications [21–23]. In order to develop a successful DDGS granulation process to improve DDGS handling and/or provide alternative products, an understanding of the processing parameters on granule characteristics must be known. A laboratory-scale batch drum granulation process was used to produce densified granules of DDGS. This paper attempts to understand the effect of process variables on the drum granulation behavior of WDGS and the bulk properties of DDGS granules. Therefore, the main objective of this study was to determine the effect of granulation time, amount of binder, screen size, and binder solids content on granule deformation, growth and dried granule characteristics. To our knowledge, this is the first study on using drum granulation to produce granules of DDGS.

2. Experimental

2.1. Materials

Wet distillers grains (WDG) and condensed distillers solubles (CDS) were obtained from an ethanol manufacturing operation in the upper northwest part of Indiana (New Energy Corp, South Bend, IN) and stored in a freezer (−20 °C) to prevent spoilage until experimentation. WDG served as the 'feed material' and CDS served as the 'binder' for the granulation process (Table 1). To account for the low end range of dry matter in CDS reported throughout dry-grind processing facilities in the U.S. [4], a portion of the CDS (10 kg) was adjusted to a lower solids content by adding distilled water – approximately 5.79 kg of CDS (38% solids) was added to a 5 gallon bucket and combined with 4.21 kg of warm distilled water (80 °C) and mixed with a wooden paddle until homogenous. The solids content of the CDS was determined gravimetrically by drying a 2 g sample in a convection oven at 80 °C for 12 h.

2.2. Physical properties

A Boerner divider was used to provide representative subsamples prior to analysis. All analyses were performed in triplicate unless otherwise noted.

Moisture content of granules was determined gravimetrically by drying a ground 2 g sample in a convection oven at 105 °C for 3 h (NFTA Method 2.1.4). Prior to drying, granules were coarsely ground in a coffee grinder for 10 s.

Specific gravity was determined according to ASTM D1963-85. CDS was heated on a hot plate with intermittent stirring to 60 ± 5 °C to improve flowability and poured into a pre-weighed Hubbard-type pycnometer (25 mL with 1.6 mm hole in stopper). The pycnometer was filled half-way with CDS, cooled to room temperature and weighed. Distilled water (20 °C) was added to completely fill the pycnometer and placed in a water bath at 25 °C for approximately 1 h. Once constant temperature was reached,

Table 1
Physical properties of WDG and CDS.

Property	WDG[a]	CDS_Low	CDS_High
Geometric mean diameter (mm)	0.93 (0.01)[b]	N/A	N/A
Bulk density (kg/m^3)	312.90 (0.82)	N/A	N/A
True density (kg/m^3)	1689.06 (43.45)	N/A	N/A
Specific gravity	N/A	1.08	1.13

[a] WDG was dried prior to analysis.
[b] Numbers in parenthesis indicate standard deviation.