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Bulk Density and Angle of Repose of Distiller's Spent Grain Under Different Drying Methods and Soluble Concentrations

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ABSTRACT The bulk density and angle of repose (AOR) of distiller's spent grain (DSG) fractions dried using oven-drying, air-drying and superheated steam (SHS) drying techniques were investigated. The analysis was done for particle sizes 300 μm , 425 μm , 600 μm and 850 μm . For DSG fractions dried using three different drying methods, the bulk density values varied from 0.379- 0.435 g/cm^3 and AOR values ranged from 46.0 - 50.4 degrees. When DSG samples were incorporated with condensed distiller's solubles (10-30%) and dried under SHS drying conditions, the bulk density values varied from 0.391- 0.444 g/cm^3 and AOR values ranged from 46.5- 49.8 degrees. SHS drying method produced samples with higher porosity compared with air-drying and oven-drying methods.

Keywords: distiller's spent grain, bulk density, angle of repose, drying, superheated steam, condensed distillers solubles.

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INTRODUCTION Distiller's spent grain (DSG) is the major by-product of distilleries. As the production of biofuels is increasing globally, the availability of DSG is also increasing at an exponential rate. According to Renewable Fuel Association (2010), the DSG production has increased significantly over the last decade to reach 23 million metric tons in the 2008 marketing year (Clementson and Ileleji, 2010). After drying, DSG contains about 86–93% dry matter, 26–34% db (dry basis) crude protein, and 3–13% (db) fat (Tjardes and Wright, 2002; Rosentrater and Muthukumarappan, 2006). Currently, DSG is mainly used as a protein source for cattle and poultry feeds. The marketability of DSG to domestic and international markets is a key factor in determining the profitability of a distillery. DSG is increasingly being transported in big containers by rails or trucks to greater distances before entering into the market, hence handling and logistics are essential for determining the marketability of DSG (Clementson and Ileleji, 2010; Ganesan et al., 2008a). During shipping and storage, caking and bridging of DSG are the common problems that hinder its flowability (Ganesan et al., 2008a). The flowability of DSG is influenced by a number of factors including storage moisture, temperature, relative humidity, particle size, storage time, ageing and pressure (Ganesan et al., 2008b). Small variability in the aforementioned parameters can lead to caking/bridging of the granular materials, which in turn can induce severe damages to shipping and storage containers (Ganesan et al., 2008a). Such damages could result in unnecessary expenses related to renting extra machinery, labour, unloading charges, and railcar downtime (Schlicher, 2005; Rosentrater, 2006a; Tumuluru et al., 2010). Thus, knowledge of flow characteristics of DSG is essential for the ease of conveying, blending, packaging and for optimizing the designs of storage containers (Kamath et al., 1994).

Drying is commonly used as a means of increasing the shelf life of DSG. The type of drying technique employed has an impact on the nutritive value and physical characteristics of DSG (Ezhil, 2010). Superheated steam (SHS) drying is a promising technology that has the potential to remove and/or prevent the off-flavors and aroma in food products (Speckhahn et al., 2010). It utilizes steam under superheated conditions to remove moisture out of the materials. No oxidative reactions are possible due to the absence of oxygen during the process (Mujumdar, 1991). SHS drying technique has significant benefits over hot air drying process including higher drying rates, better quality of the dried products, and energy savings (Shibata and Mujumdar, 1994; Taechapairoj et al., 2003; Tang and Cenkowski, 2000).

Particle size is a key factor for determining the flow characteristics of bulk solids. Properties such as bulk density, angle of repose (AOR), and compressibility of bulk solids are dependent on the particle size of a material (Ganesan et al., 2008a). A small change in particle size can cause significant variations in flowability. Farelly and Valentin (1967/68) found that particle size is the most important factor governing the 'structure' of the powder compact, and the interparticulate force which governs the strength of the structure. DSG is a heterogeneous material having particles of different size fractions (Ileleji et al., 2007). Particle segregation occurs when different sized particles are lodged, causing the particle size distribution of a heterogeneous bulk to change with time during discharge (Shinohara et al., 1968). Therefore, it becomes essential to separate DSG into various particle sizes before studying its physical characteristics.

Bulk density is defined as the mass of particles that occupies a unit volume of the container. It decreases with an increase in particle size, as well as with an increase in equilibrium relative humidity (Yan and Barbosa-Canovas, 1997). Bulk density is an important property for determining the size of transport vehicles and storage vessels for granular solids and powders (Ganesan et al., 2008a). Maintaining a consistent bulk density can minimize handling and transportation costs of bulk solids (Ileleji and Rosentrater, 2008). It was reported by Ganesan et al. (2008a) that, there exists a gap in scientific data on the influence of the bulk density of DSG on its flow characteristics.

Angle of repose is an indication of the inter-particulate friction between the particles. AOR can be used to characterize the flow behavior of materials with respect to flowability, avalanching,

stratification and segregation (Ileleji and Zhou, 2008; Frette et al., 1996; Zhou et al., 2002). The AOR values can be used as a base-line data for designing bulk storage structures (Ileleji and Zhou, 2008). Particle size of the material is an important factor affecting the AOR values of the material (Ileleji and Zhou, 2008). Typically, dry materials with a lower AOR value (30–40°) have more flowability, than materials with a higher AOR value (50–60°) (Carr, 1965).

Ganesan et al., (2008b) conducted a study to investigate the effect of addition of solubles to DSG. From the study it was observed that, increased addition of solubles affected the DSG flow negatively (Ganesan et al., 2005). The compressibility of DSG was found to increase with increased level of solubles. Also, DSG with 25% solubles had higher strength, and had the greatest ability to support obstructions to flow when compared to DSG with 10% solubles (Ganesan et al., 2008b). The aim of this study was to investigate the effect of SHS drying, oven-drying and air-drying methods on bulk density and AOR of the DSG material. Also, the study aims to investigate the effect of various percentages of condensed distiller's solubles (CDS) addition on bulk density and angle of repose of the SHS dried DSG material.

MATERIALS AND METHODS

Raw materials Distillers spent grain used for this study was obtained from a local distillery (Mohawk Canada Limited, a division of Husky Oil Limited) in Minnedosa, MB. The raw material was a mixture of corn and wheat in the ratio 9:1.

Initial sample preparation The DSG was centrifuged in a Sorvall General Purpose, RC-3 centrifuge (Thermo Scientific Co., Asheville, NC) to separate the raw material into different fractions. The centrifuge was operated at a relative centrifugal force of $790 \times g$, with a 1000 mL sample container, rotating at a speed of 2200 rpm on a radius of 0.146 m for 10 min. After centrifugation, the supernatant liquid fraction (thin stillage) was discarded; the remaining part contained the semi-solid fractions (condensed distillers soluble-CDS) and the coarse fractions (wet distiller's grain-WDG). The CDS and WDG fractions were bagged in separate airtight containers and kept in the freezer. Before each set of experiments, the required amount of samples were thawed at room temperature for 3 hours. The initial moisture content of the WDG fraction was 74.59 % (wet basis). Moisture content was calculated using the air-oven drying method (AACC standards, 2000).

Drying

Superheated steam drying equipment The SHS drying equipment was designed and fabricated in the Department of Biosystems Engineering, University of Manitoba. The system comprised of a steam generator (boiler), a superheater, conveying pipelines, valves, drying chamber, condensation system, instrumentation, and a control and data acquisition system.

Hot air oven Oven-drying of samples was performed using a laboratory hot air oven (Thermo Electron Corporation, Waltham, MA).

Sieving Sieving was done to separate the raw material into various size fractions. Sieving was performed using the method proposed by American National Standard (ASAE, 2008) for determining and expressing fineness of feed materials. A nest of sieves with US sieve numbers 4, 6, 8, 12, 16, 20, 30, 40, 50, 70, 100 and a bottom pan were used for the analysis.

Bulk density The bulk density values of DSG samples were determined using a standard Carney funnel setup with hopper, funnel, and leveling rod (ASTM, 2006). Bulk density was calculated from the weight and volume of materials filled in a cup of known volume (25 cm³). The mass measurements were done using an electronic balance with an accuracy of 0.001 g (Model Adventurer Pro AV313, Ohaus Corporation, Pine Brook, NJ).

Angle of repose Fixed-base piling angle of repose values were found out experimentally (Ileleji and Zhou, 2008). The apparatus consisted of a metallic funnel (Carney funnel), a stand, and a cylinder of 30 mm diameter. The cylinder was placed on a rotating base so that it could be imaged from different angles. Prior to measuring angle of repose, the material drop height was determined using granular sugar particles and DSG so as to form a conical heap with minimum disturbance. The drop height was found to be 55 mm from the base of the cylinder. The bottom of the funnel was positioned right above the center of the base before each experiment.

Image analysis A Digital camera (Canon Rebel XT, Tokyo, Japan) was used to take the pictures of each heap of the raw material for measuring angle of repose. The images were taken from four different angles at 90° intervals for each conical heap. The images thus obtained were analyzed using Matlab (Version 7, Mathworks Inc., Natick, MA).

Statistical analysis All Statistical analysis was performed using Sigma Stat 3.5 (Systat Software Inc., Chicago, IL).

Experimental procedure Superheated steam drying experiments were carried out in the SHS drying unit. The steam temperature at the inlet of the drying chamber was kept at 150°C and the velocity of steam passing through the drying chamber were set at 1.5 m/s during the SHS drying process. Drying experiments were conducted under or near atmospheric pressure. For the experiments using WDG and CDS, the CDS were added to WDG at 10, 20 and 30% and mixed well before drying in the SHS equipment. Samples of 30 g were placed in thin layers in each run of the experiment in the steam dryer.

Hot air drying of the samples was performed using the laboratory oven at 150°C. The samples were kept in thin layers in aluminum dishes and dried till the desired moisture content was reached. Air-drying of the samples was done at 45°C in the laboratory oven. Hot air oven was preferred in place of open air-drying, so as to prevent the chances of developing moulds on the samples by the prolonged exposure of samples in air. The samples were kept in thin layers in aluminum dishes. All the drying experiments were carried out until the moisture content of the samples reached <10% (db).

After drying, the samples were ground gently using a lab scale mortar and pestle. The ground DSG fractions were sieved using a nest of sieves in a horizontal sieve shaker (Retsch Inc., Haan, Germany) at 300 RPM for 10-15 minutes. The grinding and sieving processes were continued until enough samples were obtained on each sieve for carrying out the experiments. Each size fraction of the sample was separated and kept in airtight container for further experiments. Experiments for bulk density and AOR were repeated 10 times and average values obtained are discussed here.

RESULTS AND DISCUSSION

Influence of drying methods and particle sizes on bulk density of DSG particles Four different particle sizes: 300, 425, 600 and 850 µm were chosen for the analysis. All fractions were maintained at the same moisture content through the entire set of experiments. For both air-dried and oven-dried samples, the bulk density increased significantly ($P \leq 0.001$) with an increase in particle size (Table1). But for SHS dried samples, bulk density has not shown an increase with the increase of particle sizes.

Table 1. Variation of bulk density with particle size for air-dried, oven-dried and SHS dried samples.

| Particle Size (μm) | Bulk Density (g/cm^3) | | |
|---------------------------------|---|---------------|--------------------------|
| | Air-drying | Oven-drying | Superheated Steam-drying |
| 300 | 0.379 (0.003) | 0.392 (0.003) | 0.379 (0.007) |
| 425 | 0.386 (0.004) | 0.417 (0.004) | 0.397 (0.002) |
| 600 | 0.387 (0.004) | 0.433 (0.002) | 0.384 (0.004) |
| 850 | 0.397 (0.002) | 0.435 (0.003) | 0.384 (0.004) |

Values in parenthesis indicate standard deviations ($n= 10$).

Effect of different drying methods on bulk density For the corresponding particle sizes, the bulk densities of air-dried, oven-dried and SHS dried samples were significantly different ($P \leq 0.001$) (Table 1). This gives a clear indication of the dependency of bulk density on the type of drying technique employed.

For SHS dried samples, the bulk density was lower when compared to the other two drying methods, due to an increase in porosity of the material by the influence of steam. Earlier studies indicate that the porosity of granular material increases during steam drying, as higher steam temperature produces samples with a large number of bigger-sized pores (Ezhil, 2010). This will lead to less shrinkage and increased volume of the steam dried material. Also, it was observed that the oven-dried samples have higher bulk density compared to air-dried samples. For a constant particle density, it can be interpreted that air-dried samples have higher porosity than oven-dried samples.

Influence of CDS addition on bulk density of SHS dried DSG material This study was done to analyze the effect of SHS drying on bulk density for different percentages of CDS addition. The study was performed with three different percentages of CDS, i.e., 10, 20, and 30%. The values analyzed for different particle sizes are shown in Table 2. The dried DSG particles became harder with the addition of CDS. Also, for higher concentration of CDS, greater force was needed to grind the particles. The CDS fractions adhered strongly to the DSG particles, which resembles to clay particles sticking to the soil. The CDS acted as a binding agent leading to a reduction in particle volume causing an increase in bulk density. It was observed that the color of DSG became darker with the increase in addition of CDS. A significant difference ($P \leq 0.001$) in the bulk density values was observed among various concentrations of CDS. For most particle sizes, an increase in bulk density was observed with an increase in concentration of CDS from 0 to 30% (Table 2).

Table 2. Variation of bulk density with different percentages of CDS addition.

| Particle Size (μm) | Bulk Density (g/cm^3) | | |
|---------------------------------|---|---------------|---------------|
| | 10%CDS | 20%CDS | 30%CDS |
| 300 | 0.391 (0.004) | 0.393 (0.004) | 0.425 (0.002) |
| 425 | 0.395 (0.003) | 0.403 (0.004) | 0.437 (0.005) |
| 600 | 0.410 (0.005) | 0.412 (0.006) | 0.444 (0.007) |
| 850 | 0.413 (0.006) | 0.423 (0.005) | 0.421 (0.004) |

Values in parenthesis indicate standard deviations (n= 10).

The bulk density values obtained from this study concur with the results obtained by other researchers for similar studies involving different treatment methods. Studies done by Rosentrater (2006b) showed that the bulk density of dried distiller's spent grain with solubles produced at six ethanol plants in South Dakota ranged from 391 to 496 kg/m^3 . In another study Bhadra et al. (2009) found ranges of 490–590 kg/m^3 from five plants in South Dakota. Bulk density ranged from 365 to 561 kg/m^3 in a study conducted using DDGS obtained from 69 sources (US Grains Council, 2008). Clementson and Ileleji (2010) conducted a study by varying soluble content and found that the bulk density values varied from 420.47 to 458.05 kg/m^3 .

Angle of repose Angle of repose values were measured for three different particle sizes i.e., 300, 425 and 850 μm , obtained from the three drying techniques (Table 3). The results indicate that the AOR values were significantly different ($P = <0.001$) for different particle sizes. In majority of cases, the AOR values decreased with increase in particle size. This is due to the fact that smaller particles have more cohesive and adhesive forces compared to bigger particles; also, for bigger particles gravity plays a dominant role in controlling their flow. There was no significant difference found among AOR values for different drying methods. This indicates that, the drying method doesn't have a significant influence over AOR.

As per Ileleji and Zhou (2008), AOR values less than 30° is considered as free flowing, between 30° and 35° as good flowing, above 35° as fair flowing, greater than 40° as poor flowing and greater than 50° as very poor flowing. The range of AOR values for spent grain from this study indicate that DSG falls mainly under poor flowing category ($46\text{--}51^\circ$).

Table 3. Variation of AOR with particle size for air-dried, oven-dried and SHS dried samples.

| Particle Size (μm) | Angle of Repose (Degrees) | | |
|---------------------------------|---------------------------|-------------|--------------------------|
| | Air-drying | Oven-drying | Superheated Steam-drying |
| 300 | 50.4 (1.7) | 49.4 (2.0) | 50.0 (2.3) |
| 425 | 47.1 (1.8) | 46.9 (1.9) | 47.2 (2.5) |
| 850 | 46.7 (1.8) | 46.0 (2.3) | 47.2 (2.1) |

Values in parenthesis indicate standard deviations (n= 10).

Among different CDS concentrations from 0 to 30% the AOR values ranged between $46\text{--}51^\circ$, but there was no significant difference observed in AOR values for different percentages of CDS

addition (Table 4). Only for 850 μm , the particles showed a significant difference ($P = 0.032$) of AOR values among different soluble concentrations.

Table 4. Variation of AOR with different concentrations of CDS addition.

| Particle Size (μm) | Angle of Repose (Degrees) | | |
|---------------------------------|---------------------------|------------|------------|
| | 10% CDS | 20% CDS | 30% CDS |
| 300 | 49.5 (2.0) | 49.8 (1.6) | 48.9 (2.4) |
| 425 | 47.9 (1.7) | 47.2 (2.4) | 48.0 (2.4) |
| 850 | 46.5 (2.0) | 47.7 (1.8) | 46.7 (2.2) |

Values in parenthesis indicate standard deviations ($n = 10$).

CONCLUSION The study has shown that both bulk density and AOR values vary with different particle sizes for the same drying technique. The bulk density of DSG material varies significantly with different drying techniques and CDS concentrations. The AOR values indicated that dried DSG material falls under poor flowing category for all drying methods, and the values are independent of the drying technique used. Only for 850 μm the particles showed a significant difference of AOR values among different soluble concentrations. DSG material produced by SHS drying method has higher porosity compared to oven-drying and air-drying methods. The AOR and bulk density values obtained from this study can be used for determining the design specifications of bulk storage structures and transportation vehicles.

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