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Research paper

## Modeling of effective moisture diffusivity and activation energy of distillers' spent grain pellets with solubles during superheated steam drying



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#### ARTICLE INFO ABSTRACT Keywords: Drying is an essential unit operation needed for safe storage and handling of the wet Distillers' spent grain (DSG), Distillers' spent grain pellet a major by-product of the ethanol industry. For the simulation and modeling of the drying process, a detailed Distillers' solubles study on different pre-requisite parameters such as the thermo-physical properties and effective moisture dif-Effective moisture diffusivity fusivity of the material to be dried is required. The present study reports the effective moisture diffusivity and Activation energy activation energy of the DSG pellets during superheated steam (SS) drying. Cylindrical DSG pellets at two Superheated steam drying moisture mass fractions (25 and 35%) and three mass fractions of distillers' solubles (0, 10, and 30%) were dried at three SS temperatures (120, 135, 150, 165, and 180 °C) and three SS velocities (0.5, 1.0, and 1.5 m/s), respectively. The experiment-based moisture diffusivity of the DSG pellet with and without solubles was determined by using the drying characteristic and the analytical solution of Fick's law of diffusion. The results showed that the effective diffusivity increased with an increase in SS temperature and velocity and its value was in the range of $2.49 \times 10^{-9}$ to $17.9 \times 10^{-9}$ m<sup>2</sup>/s. The dependency of the moisture diffusivity on temperature and moisture mass fraction was established by using Arrhenius equation and Levenberg-Marquardt optimization algorithm. The model coefficient and constants were compared with the calculated values of instantaneous effective moisture diffusivity with the mean relative percentage deviation $\leq 10\%$ . These findings could serve as a fundamental input for the numerical modeling of SS drying of DSG.

#### 1. Introduction

Distillers' spent grain (DSG), which contains the residue remaining after starch extraction for ethanol production, is the major by-product from the ethanol industry. High levels of protein and digestible fiber combined with low starch content makes this material a suitable raw material for the feed industry [1–3]. When converted to other useful products such as animal feed, dried distillers' grain, with or without solubles, could yield 10–20% additional income to the ethanol industry [4,5]. The increased production of DSG in recent years has resulted in unprecedented interest to identify new value added products from this dietary fiber rich by-product. These products include fish feed [6], cattle feed [7], poultry feed [2], pig feed [8], human food [9,10], and nutritional supplements for simultaneous saccharification and ethanol fermentation [11].

Regardless of the milling method (wet or dry milling), a by-product of the ethanol industry that uses cereal grains as raw materials is the unfermented mashed grain, called wet stillage. This wet stillage is in the form of a slurry with high moisture mass fraction (about 80%) [5]. Approximately, one-third of the grain used for ethanol production ends up as wet stillage [12]. Drying wet stillage mitigates the inconvenience of its storage and transportation. The wet stillage is initially centrifuged to remove excess water and to separate the smaller sized particles (i.e. distillers' solubles) from coarser particles. Depending upon the requirements of the final product, the centrifuged stillage and solubles are then dried either separately or together to yield dried distillers' spent grains (DDGS) or dried distillers' spent grain with solubles (DDGSS), respectively. The quality and market value of the dried product depends on the method and operating conditions of the drying process.

The industrial drying of wet spent grains is done using rotary drum dryers at internal temperatures ranging from 250 to 600°Cs. In order to maintain the throughput of the wet stillage to dried product, the wet stillage is mixed with a batch of previously dried granules of DSG at the entrance of the rotary-drum dryer. The dried granules serve as a core (inert) material to dry the wet DSG coated over it. This approach helps to develop a desired large surface area for heat and mass exchange. But, these multiple passes of the core material through the hot dryer can lead to ignition. This causes either darkening of the DDGS, which affects its nutritional value, or a shutdown of the production line causing monetary losses [13]. The use of superheated steam (SS) for drying such

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wet materials not only eliminates the risk of fire but also improves the colour and quality of the final product [14–16]. The optimization of the drying process using SS is still under research with the aim to improve the quality of dried products; and increase the efficiency and productivity of the drying process.

The SS drying process has different stages of drying such as initial condensation, constant rate drying and falling rate drying. As opposed to air drying where the moisture mass fraction gradually decreases during the warming up period, the moisture mass fraction of the material increases in the initial stage of SS drying due to steam condensation. This phenomenon is referred to as the initial condensation or a reverse drying process [17]. The condensed water either evaporates or is adsorbed by the material depending on the properties of the material causing an increase in the overall moisture mass fraction of the material. This increase in moisture mass fraction affects the drying rate as well as the moisture diffusivity of the material [18]. The duration and quantity of the initial condensation is dependent on the SS temperature and velocity [5]. Therefore, the moisture gain due to the initial condensation is also dependent on the SS condition. Zielinska and Cenkowski [19] reported that different factors such as the initial moisture mass fraction, mass fraction of distillers' solubles in the spent grain, and steam operating conditions affect the moisture diffusivity of DSG. These affect the heat transfer rate and thereby influence the moisture movement inside the material.

A complete understanding of the drying characteristics of the material and the dynamics of drying process are key to the design and modification of such dryers. A development of a numerical model capable of describing the drying process with a good spatial and temporal resolution is an essential step in design. To this, some pre-requisite parameters such as thermo-physical properties of material [20-22] and the moisture diffusivity [23,24] are required. Accurate modeling of the pre-requisite parameters at variable operating conditions of drvers will result in better predictability of results by such models. A detailed research on the effective moisture diffusivity of DSG during drying either by hot air or SS has not been done yet. Moisture diffusivity during drying depends on many events that occur in the material such as liquid diffusion, vapour diffusion, molecular diffusion, hydrodynamic flow, capillary flow, surface diffusion, etc. [25] Hence, a generic term such as 'effective moisture diffusivity' could be used to describe the mechanism of moisture movement in a biological material. This movement of moisture is usually determined by incorporating Fick's law of diffusion into drying experiments [26]. In most drying problems, one-dimensional flow approach is used [27]. The dependency of effective moisture diffusivity on moisture mass fraction [28,29] and temperature [30,31] is a contentious issue that has been studied by many researchers [32-35]. Some researchers believe that the diffusion coefficient is independent of moisture during the first falling drying rate period [36]. This theory is based on Arrhenius type relationship of diffusivity where the temperature during drying describes the effective moisture diffusivity. The assumption of the average temperature of drying media equal to that of the material in the Arrhenius equation [37,38] has been questioned by its critics, especially in the case where a temperature gradient is present in a sample [33,39]. In practice, it is difficult to measure the local temperature of the sample as drying progresses. Hence, it is generally assumed that the average sample temperature is equal to that of the temperature of the drying medium. Therefore, modeling the effective diffusivity as a function of temperature of the drying medium instead of the sample is apparent [33].

The present paper is a continuation to a previous study on determining the pre-requisite parameters for numerical modeling of SS drying of DSG pellets where a section was devoted to the thermophysical properties of DSG pellets at variable moisture and temperature [22]. The present study summarizes the results of the research on the effective moisture diffusivity and activation energy of DSG pellets at different operating conditions of SS drying. The main objective of this paper is to determine the effect of SS temperature and velocity, and mass fractions of distillers' soluble (CDS) in the DSG on the effective moisture diffusivity and activation energy of DSG pellets. Additionally, a model is developed to predict the effective moisture diffusivity of DSG pellets during SS drying.

#### 2. Materials and method

#### 2.1. Sample preparation

The raw materials used for the present study was a by-product mixture of corn and wheat (9:1) stillage. The stillage was obtained from a local ethanol plant in Manitoba (Mohawk Canada Limited, a division of Husky Oil Limited, Minnedosa, MB). It was then stored in a chest freezer at -15 °C in sealed plastic pails. The stillage was thawed overnight at room temperature and thereafter centrifuged at a relative centrifugal force of 790 g using a Sorvall General Purpose, RC-3 centrifuge (Thermo Scientific Co., Asheville, NC). The centrifuge was operated, with four sample bowls of 1 L capacity (filled approximately 75% of volume) rotating at a speed of 36.7 Hz for 10 min.

After centrifugation, the supernatant liquid (excess water with a very small amount of mashed grain soluble) was poured out of the bowl. The semi-solid soluble (distillers' solubles) fraction and the coarse grain fraction were separated manually with a spoon. The centrifugation trials were repeated 10 times to determine the composition of the raw sample in terms of coarse grain fraction, distillers' solubles, and supernatant liquid [13]. The mass of the separated coarse grain fraction, distillers' solubles, and liquid was recorded for each trial. The average mass fraction of the coarse grain fraction, distillers' solubles, and supernatant liquid were  $49.5 \pm 0.5$ ,  $9.5 \pm 0.5$ , and  $40.5 \pm 0.5\%$ , respectively. The separated fractions were placed in airtight polyethylene bags (2 Gauge mils thickness) of 250 g capacity and stored in a freezer at -15 °C. Prior to the experimental trial, a bag was taken out of the freezer and allowed to thaw for 2 h.

#### 2.2. Pre-treatment and mixing

The initial moisture mass fraction of the coarse grain fraction and the solubles were determined using the air-oven drying method [40] using a laboratory oven (Thermo Electron Corporation, Waltham, MA) at 135 °C for 2 h [5,13]. The average moisture mass fraction of whole stillage before centrifugation, coarse grain fraction, and distillers' solubles after centrifugation, were 89.2  $\pm$  0.05, 77.8  $\pm$  0.08 and 82.9  $\pm$  0.1%, respectively.

To prepare the different mixtures of distillers' solubles and coarse grain, calculated amounts of solubles were added to a known mass of coarse grain fraction of DSG. For example, the amount of solubles added to 100 g of coarse grain to obtain a mass fraction of 30% is given by:

$$S = \frac{100 \times 0.3}{(1 - 0.3)} \tag{1}$$

Where, S is the mass of solubles in g.

Three sets of DSG samples were prepared with 0, 10, and 30% of distillers' solubles. The sample with 0% solubles was kept as a control sample. The moisture mass fraction of each mixture was also measured using standard air-oven drying method [40]. Each of these wet mixtures was gently mixed using a spatula for about 2–3 min. The mixed samples were dried in a laboratory oven at a low temperature ( $60 \pm 5$  °C) to ensure that the properties of the samples are not altered [41]. About 20 g of mixture to produce 4–5 pellets was prepared at a time to prevent excessive drying during storage [26]. Each composition of the sample (with 0, 10, 30% solubles) was dried to obtain two different moisture mass fractions (25 and 35%). The selection of moisture mass fraction was done based on the stability of compacted DSG pellets for the selected composition [13,42]. The prepared samples were stored in a

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