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Sorption Isotherm Characteristics of Distillers Dried Grains with Solubles (DDGS)

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Abstract. Distillers Dried Grains with Solubles (DDGS) is widely recognized as a highly nutritious animal feed ingredient. With the exponential growth of the fuel ethanol industry in the past several years, significant quantities of distillers grains are now being produced. To effectively utilize these feeds in the domestic market, however, these coproduct streams are increasingly being transported greater distances, and must be stored in various structures until final use. Unfortunately, DDGS flow is often problematic, as it frequently becomes restricted by caking and bridging during storage and transport. This issue may arise from a number of factors, including storage moisture, temperature, relative humidity, particle size, time, or temperature variations. The objective of this study was to develop sorption isotherms for DDGS with varying soluble levels, in order to provide facility designers and operators with appropriate storage and transport information. Equilibrium moisture contents (EMC) of DDGS with four different soluble levels (10, 15, 20 and 25% db) were determined using the static gravimetric method at 10, 20, 30 and 40°C over four equilibrium relative humidity (ERH) conditions, ranging from 0.560 to 0.910. The sorption capacity of DDGS increased with increasing temperature and soluble level, and followed a type III isotherm, which is commonly observed in high sugar foods. The observed EMC values for 10, 15, 20 and 25% (db) solubles ranged from 8.61% to 47.07% (db), 11.58% to 83.49% (db), 13.72% to 90.70% (db), and 15.03% to 132.01% (db), respectively. Nine models were applied to fit the isotherm data. The Modified Henderson and modified Chung-Pfost models did not fit the data, however, as the regression coefficients did not converge. The Modified Halsey and modified Exponential models, on the other hand, were found to perform well for the isotherm data. As there was no common model to predict the sorption isotherms of DDGS with various soluble levels, a new EMC model was developed. This new model, termed the GMR (Ganesan-Muthu-Rosentrater) model, incorporated soluble level as one of the effects, along with temperature and moisture content. The GMR model ($R^2=0.9395$ $F=977.55$), followed by a new modified Exponential 2 (NME2) model ($R^2=0.9368$ $F=934.43$), produced the best fits for DDGS with varying soluble levels.

Keywords. DDGS, Sorption isotherms, EMC, ERH, flowability, Solubles, GMR model, type III isotherm.

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Introduction

Distillers Dried Grains with Solubles (DDGS) is one of the coproducts resulting from dry grind ethanol processing. DDGS is as a valuable source of protein, ruminally undegradable protein (RUP), energy, and water soluble vitamins and minerals for animal feeds. Distillers grains are energy dense as they often contain relatively high fat (10-12%) content, and are nearly devoid of starch. DDGS typically contains 86 to 93% (d.b.) dry matter, 26 to 34% (d.b) crude protein, and 3 to 13% (d.b) fat (Rosentrater and Muthukumarappan, 2006). With the tremendous growth of the U.S. ethanol industry in the past decade, significant quantities of distillers grains are now being produced. In 2006-2007, DDGS production is forecasted to be over 10 million metric tons, due to increasing demand for fuel grade ethanol, and concurrently the growth of the industry to meet this demand. Moreover, exports of DDGS to Europe, a major outlet for these feed materials, might decrease in coming years. As a consequence, it is important to increase the use of distillers grains in domestic market. To achieve this, however, they will have to be economically hauled to greater distances. DDGS is usually transported through the rail system. During transportation, DDGS can be subjected to varying temperature and humidity levels, and are stored in bins and silos until use; sometimes this may be a relatively long time. The storability and resulting flowability of DDGS is currently becoming very problematic. DDGS storage and flow behavior depends on the physical and chemical properties of the material itself, as well as external conditions such as air temperature, relative humidity, and compression (i.e., storage bin size, as well as time in storage). At times, even small differences in these factors can cause caking and bridging (i.e., poor flowability) in DDGS. The transportation and storage of DDGS, and the resulting flow behavior under various storage conditions are important considerations, especially in light of the growth of the industry, because these materials must be handled at the plant, during transportation, and upon arrival at the final destination.

Relative humidity of the air (interstitial as well as head space) in a storage container, such as a bin silo, or even a rail car, also affects a bulk material's properties. Many bulk materials are hygroscopic, and thus the exposure to humid conditions results in increased moisture content of the material, and can promote the development of particle cohesion, which leads to an increase in bulk strength (i.e., caking, bridging, etc.) (Marinelli and Carson, 1992). The angle of internal friction has been found to decrease with an increase in storage humidity (Peleg and Mannheim, 1973). For example, when prilled urea was tested in a shear cell at 70°F and 50% RH, it showed no arching, whereas prilled urea tested at 90°F and 95% RH showed a central core with a pond of urea-saturated water surrounding it (Johanson, 1978). As another example, the consequences of atmospheric humidity (2 to 100% RH) on the flow characteristics of PS304 plasma spray feedstock powder were studied. The flow was more affected when the RH was above 66%; and, in fact, there was no flow observed at 88% RH (Stanford and Corte, 2002). Fitzpatrick et al (2004) noticed a greater effect on the cohesiveness of skim milk powder when it is exposed to 46% RH and 20°C.

Temperature also has a substantial effect on granular solid and powder flowability. A drastic temperature effect is the freezing of moisture contained within granular materials and on particle surfaces. The resulting ice bonds weaken the flow (Irani et al, 1959; Johanson, 1978; Fitzpatrick et al, 2004). Moreover, severe caking can occur whenever a granular material undergoes a change in crystallinity or other physical properties due to temperature variations (Johanson, 1978). Temperatures of both the wall surface and the bulk material may affect the wall friction angle (Marinelli and Carson, 1992).

The equilibrium moisture content (EMC) is defined as the moisture content at which a hygroscopic material neither gains nor loses moisture at a given environmental (temperature and relative humidity combination) condition. The equilibrium relative humidity (ERH, also known as water activity, a_w) measures the vapor pressure generated by the moisture present in a hygroscopic material in this environmental condition. A moisture isotherm thus describes the relationship between a material's moisture content and the ERH at a given temperature over a range of moisture levels. Moisture isotherms have been published for several grains and food materials. A number of theoretical, semi-theoretical and empirical isotherm equations have been developed to model the observed relationships between EMC,

ERH, and temperature of these materials. For example, Kaleemullah and Kailappan (2004) investigated the EMC of red chilies for various RH and temperatures applicable to specific storage conditions. Thin layer moisture adsorption rates of wheat and barley were determined at grain storage conditions (Casada, 2002). Ertugay and Certel (2000) determined moisture sorption characteristics of wheat, barley, rye, oat, and corn at different temperatures. Isotherms for Australian canola varieties were determined and modeled using the Halsey equation (Cassells et al, 2003). Adsorption isotherms for barley at low and high temperatures were determined using a thin layer technique, and the modified Chung-Pfost equation was found to be most appropriate for modeling the adsorption process for barley (Basunia and Abe, 2005).

EMC isotherms for DDGS at various relative humidity and temperatures are essential to optimizing storage and transport conditions. As of now, however, there has been no study done on the sorption phenomenon of DDGS for range of temperatures and relative humidities, let alone examining the effects of various soluble levels. Thus, the objectives of the study were twofold:

- 1) Develop sorption isotherms for DDGS with varying soluble levels (10, 15, 20 and 25% db) at four constant temperatures (10, 20, 30 and 40°C) and four relative humidities (60, 70, 80 and 90%), using appropriate saturated salt solutions, and to determine the most suitable mathematical model for describing these isotherms.
- 2) Develop a new EMC model incorporating soluble level as one of the effects, in addition to temperature and moisture content.

Materials and Methods

Sample Collection and Preparation

Samples of Condensed Distillers Soubles (CDS) and Distillers Dried Grains (DDG) were obtained from a commercial ethanol plant in South Dakota, and were stored in sealed plastic buckets (the DDG at room temperature, and the CDS at 4°C ± 1°C) until needed. The soluble content of both the DDG and the CDS were determined, and DDGS with 4 different soluble levels (10, 15, 20, and 25% db) were prepared using the methodology developed by Ganesan et al (2005). The moisture content of the DDG and CDS were determined using Method 44-19 (AACC, 1995). Initially the DDG had approximately 10% (db) solubles and 6.1% (db) moisture content. To obtain the EMC data over a wide range of temperatures and relative humidities, it was essential that the experimental samples had a constant initial moisture content. So, prior to experimentation, the DDGS samples that had been prepared with the various soluble contents were dried to a moisture content of 6.1% (db).

Water absorption characteristics and the equilibrium moisture contents of the DDGS were determined at four constant temperatures (10, 20, 30 and 40°C) and four humidity levels (60, 70, 80 and 90%) using a static gravimetric method. Saturated salt solutions were used to maintain a constant relative humidity in each environment. Saturated salt solutions are the most applicable for this type of study, as the three phases (vapor-liquid-solid) systems are independent of changes in their total moisture content: a saturated aqueous solution in contact with an excess of a definite solid phase at a given temperature will maintain a constant humidity within any enclosed space around it. The ERH values of the saturated salt solutions were obtained from the reported data (Spencer, 1926; Hodgman, 1954, 1955; Greenspan, 1977). Nalgene autoclavable plastic desiccators, with a 150 mm inner diameter and a 149 mm height, were used to provide the required hermetic environments. The lower portions of the desiccators, separated from the headspace by 140 mm aluminum plates, contained the specific salt solutions that had been prepared with distilled water. The salts used were of pure laboratory grade (Fisher Scientific Company, Burr Ridge, IL), and the saturated solutions were prepared with excess salt to provide the required humidity environments (Table 1). Approximately 3 g of prepared DDGS (i.e., 10, 15, 20, or 25% db solubles) samples were placed in aluminum dishes and then placed on the aluminum base plate in each desiccator. Each sample was run in triplicate. To prevent microbial growth during the course of the study, crystalline thymol was placed on a petri dish near the samples in each desiccator, following the methods described by Nair et al. (1996), Moreira et al. (2002), and Quirijns et al. (2005). The loaded desiccators were then sealed, and

then placed in four constant temperature conditions (10, 20, 30 and 40°C): a refrigerator provided the 10°C environment, a hot air oven provided the 30°C and 40°C temperatures. For the 20°C environment, the desiccators were placed on the laboratory benchtop, and the room temperature was maintained between 20 and 22°C during experimentation.

The samples were weighed periodically until the difference between successive weighings was less than or equal to 0.001 g. When the samples reached EMC, they were removed from the controlled environments and were tested for moisture content using Method 44-19(AACC, 1995).

Equilibrium Moisture Sorption Isotherm Models

Nine isotherm models that account for temperature effects were selected from the literature and were investigated for applicability to DDGS: the standard Henderson, Chung Pfof, and Sehgal models, and the modified versions of Henderson, Chung Pfof, Smith, Exponential, Oswin, and Halsey models. Four of these models (the modified versions of Henderson, Chung Pfof, Oswin, and Halsey) are, in fact, included in ASABE standards (ASAE, 2005). These nine models (Equations 1 to 9) were evaluated for their ability to fit the EMC-ERH data of DDGS using four different soluble levels. Additionally, these isotherm models have been formatted to express moisture content as a function of relative humidity.

Henderson Model:

$$M = \left[\frac{\ln(1 - H_r)}{-AT} \right]^{\frac{1}{B}} \quad (1)$$

Chung-Pfof Model:

$$M = \left(\frac{-1}{B} \right) \ln \left[\frac{\ln(H_r)T}{-A} \right] \quad (2)$$

Sehgal Model:

$$M = \left(\frac{-1}{B} \right) \ln \left[\frac{\ln(H_r)(\sqrt{T})}{-A} \right] \quad (3)$$

Modified Henderson Model:

$$M = \left[\frac{\ln(1 - H_r)}{-A(T + B)} \right]^{\frac{1}{C}} \quad (4)$$

Modified Chung-Pfof Model:

$$M = \left(\frac{-1}{C} \right) \ln \left[\frac{\ln(H_r)(T + B)}{-A} \right] \quad (5)$$

Modified Halsey Model:

$$M = \left[\frac{-\exp(A + BT)}{\ln(H_r)} \right]^{\frac{1}{C}} \quad (6)$$

Modified Oswin Model:

$$M = (A + BT) \left[\frac{H_r}{1 - H_r} \right]^{\frac{1}{C}} \quad (7)$$

Modified Exponential Model:

$$M = (A - BT)\exp(CH_r) \quad (8)$$

Modified Smith Model:

$$M = (A + BT) - [(C + DT) \ln(1 - H_r)] \quad (9)$$

where M is the equilibrium moisture content (%db), H_r is the equilibrium relative humidity (decimal), T is the temperature ($^{\circ}\text{C}$), and A , B , C , D are empirical coefficients specific to the individual models.

Parameter estimates for the above models were determined with non-linear regression, using the PROC NLIN procedure of SAS v 9.1 (SAS 2002). This procedure used the Gauss-Newton method to resolve the models, and minimized the mean relative error (MRE) and standard error of the mean (SEM) of the moisture content (% db) data:

$$\text{MRE} = \frac{100}{N} \sum \left| \frac{M_i - \hat{M}_i}{M_i} \right| \quad (10)$$

$$\text{SEM} = \sqrt{\frac{\sum (M_i - \hat{M}_i)^2}{\text{DF}}} \quad (11)$$

where M_i and $\hat{M}_i$ are experimentally observed and predicted equilibrium moisture contents, respectively, N is the number of data points, and DF is the degrees of freedom (number of data points minus the number of coefficients in the model). PROC NLIN directly provides the F -statistic and R^2 values, which were calculated from the output (Model sum of squares / Total sum of squares). Plotting the ERH values (x axis) versus residual EMC (y axis) values gave the residual plots for each fitted model.

The suitability of each of the models were then evaluated and compared using the MRE, SEM, coefficient of determination (R^2), F -statistic, and residual plot patterns as performance measures. An extensive literature survey reveals that these parameters are often used for this type of model performance analysis (Chen and Morey, 1989; Menkov, 2000; Kaleemullah, 2004; Tungsangprateep and Jindal, 2004; Igathinathane, 2005; Basunia, 2005). The data points in the residual plots should be uniformly scattered in a horizontal band centered on the x-axis, and should display no systematic distribution or clear pattern. If the residual plots show a specific pattern, the model should not be accepted (Chen and Morey, 1989; Menkov, 2000). If, however, all the residual plots show a clear pattern, then that parameter should be ignored and the rest of the parameters should be considered for model performance analysis (Kaleemullah, 2004). In this study, a model was considered “good”, when it produced small error terms (i.e., MRE and SEM), high F -statistic and R^2 values, and the residual plots indicated uniformly scattered points (Igathinathane, 2005).

Results and Discussion

ERH and average EMC values obtained for DDGS with 10, 15, 20 and 25% db solubles at various temperatures (10, 20, 30 and 40°C) are shown in Table 2. Generally the EMC values increased with increasing ERH values for all the soluble levels. The minimum and maximum observed EMC values for 10, 15, 20, and 25% db solubles were 8.61% to 47.07% db, 11.58% to 83.49% db, 13.72% to 90.70% db, and 15.03% to 132.01% db, respectively. The highest EMC values were observed for the 0.90 ERH and 40°C temperature combination. In most cases, the EMC values at 25% db soluble content were highest among all the soluble levels. The standard deviation of EMC values varied with ERH values.

Large differences were found at higher ERH values, often because of the inherent difficulty in determining EMC at these elevated humidity levels. Similar issues were reported by other researchers (Maroulis et al., 1988; Yu et al., 1999).

Isotherm Model Fitting and Evaluation

The fitted isotherm coefficients and parameters for the nine models used are shown in Table 3. The subsequent residual plots obtained for each model are shown in Figures 1 through 7. Out of all nine models used, the modified Henderson and modified Chung-Pfost did not fit the data well. In fact, the model coefficients did not converge for these two models. Hence these models were not considered further. The performance parameters (MRE, SEM, F-statistic, R^2 , and residual classification) did not show much similarity among models or soluble levels for the DDGS. But, these comparisons did show that the Sehgal, Chung-Pfost, and Henderson models did not produce acceptable performance.

On the other hand, the modified Halsey, modified Exponential, modified Smith, and modified Oswin models were all determined to have good performance for the DDGS. The residual plots for these models (i.e., modified Halsey, modified Exponential, modified Oswin, and modified Smith) all exhibited a fairly random distribution. Even though modified Smith model produced smaller error terms and a randomized residual pattern, the model was deemed unacceptable due to lower F-statistic and R^2 values compared to these other models. The values of the error terms were not significantly different from the error terms obtained from the other models, but the F-statistic and R^2 values determined to be too low. The Henderson, Chung-Pfost, and Sehgal model all exhibited a systematic distribution in their residual plots, which confirmed that these models were unacceptable for DDGS.

In commercial ethanol plants, the CDS (often referred to as “syrup”) is added back to the wet distillers grains, the combination of which is then dried to produce DDGS. The amount of CDS added will vary for each production cycle, and is often not a systematic process. Considering this, soluble level was incorporated into the study as a key parameter (in addition to temperature and moisture content) in the isotherm model equations. The soluble term was included in multiple modified versions of the Halsey, Exponential and Oswin models, and then evaluated for performance. Only the models which performed reasonably well are discussed subsequently. They include:

New Modified Halsey Model 1 (NMH 1):

$$M = \left[\frac{-\exp(A + BT + S^D)}{\ln(H_r)} \right]^{\frac{1}{C}} \quad (12)$$

New Modified Halsey Model 2 (NMH 2):

$$M = \left[\frac{-\exp(AT + S^B)}{\ln(H_r)} \right]^{\frac{1}{C}} \quad (13)$$

New Modified Exponential Model 1 (NME 1):

$$M = (A - BT + SD)\exp(CH_r) \quad (14)$$

New Modified Exponential Model 2 (NME 2):

$$M = (AT + SB)\exp(CH_r) \quad (15)$$

New Modified Oswin Model 1 (NMO 1):

$$M = (A + BT + S^D) \left[\frac{H_r}{1 - H_r} \right]^{\frac{1}{C}} \quad (16)$$

New Modified Oswin Model 2 (NMO 2):

$$M = (AT + SB) \left[\frac{H_r}{1 - H_r} \right]^{\frac{1}{C}} \quad (17)$$

where M is the equilibrium moisture content (% db), H_r is the equilibrium relative humidity (decimal), T is the temperature ($^{\circ}\text{C}$), S is the soluble level (% db), and A, B, C, D are empirical coefficients specific to the individual models.

The fitted isotherm coefficients and parameters for the new isotherm models are shown in Table 4. All these new isotherm models had higher F-statistic values and error term values compared to the results based on the traditional models. R^2 values decreased comparatively, but not significantly. Models for DDGS ranked in order of decreasing performance were NMH2, NME2, NMO2, NMH1, NME1 and NMO1.

The residual plots (figures 8 to 13) were fairly randomly distributed for all these new isotherm models. Out the six models, NMH2 was found to be the best performing, and has been termed the “GMR” (Ganesan-Muthu-Rosentrater) model. The GMR model ($R^2=0.9395$ $F=977.55$) was considered the best choice to describe DDGS moisture sorption isotherms. The NME2 model ($R^2=0.9368$ $F=934.43$), ranked second. Thus the GMR and NME2 models are recommended for DDGS sorption isotherm analysis and prediction.

Prediction of EMC by GMR Model

The GMR model was subsequently used to predict sorption isotherms for DDGS. The observed and predicted EMC for DDGS with soluble levels of 10, 15, 20 and 25% db, at temperatures from 10 to 40 $^{\circ}\text{C}$, are shown in figures 14 through 17, respectively. The predicted isotherms show the good fit of the GMR model to the observed data. EMC values increased with increasing temperature for all the four soluble levels. This trend thus displays Type III isotherm characteristics, which is common in high sugar foods (Bolin, 1980; Mazza, 1984; Saravacos et al, 1986; Maroulis et al, 1988; Tsami et al, 1990; Ayranci et al, 1990; Lim et al, 1995; Alhamdan and Hassan, 1999). This has been termed cross over behavior for biomaterials. This inversion of the usual dependence of water sorption isotherms on temperature may be attributable to endothermic dissolution of sugars at higher water activities and higher temperatures, resulting in an increased water holding capacity, and thus resulting in high EMC values (Saravacos et al, 1986; Tsami et al, 1990; Ayranci et al, 1990; Falade et al, 2004). Additionally, the DDGS with a soluble level of 25% db exhibited the highest EMC values, and also exhibited the highest variation. This is reflected in the residual plot (figure 9).

Conclusions

The observed EMC values for DDGS with soluble levels of 10, 15, 20 and 25 % db ranged from 8.61% to 47.07% db, 11.58% to 83.49% db, 13.72% to 90.70% db, and 15.03% to 132.01% db, respectively. DDGS at the 25% soluble level produced the greatest EMC values at higher temperatures and humidities. The addition of CDS (soluble materials) affects the composition of DDGS, and the resulting moisture behavior. DDGS showed Type III isotherm characteristics, which is typical for high sugar foods. The GMR model ($R^2=0.9395$ $F=977.55$), incorporated to effect of soluble content in addition to those of temperature and moisture content. It is thus recommended for DDGS moisture sorption

isotherm analysis. The NME2 model ($R^2=0.9368$ $F=934.43$), with fairly random residuals, ranked second in terms of performance. This is an initial step taken towards characterizing the sorption isotherms of DDGS. Future work will investigate on the sorption isotherm characteristics of DDGS on the lower range of water activities ($a_w < 0.6$).

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Table 1: Saturated salt solutions used for providing control environments (actual RH provided)

RH (decimal)	Temp (°C)	Saturated Salt Solutions			
		10	20	30	40
0.60		Sodium bromide (0.622)	Sodium bromide (0.591)	Sodium bromide (0.560)	Sodium bromide (0.570)
0.70		Lithium acetate dihydrate (0.720)	Lithium acetate dihydrate (0.700)	Strontium chloride (0.691)	Strontium chloride (0.680)
0.80		Ammonium sulfate (0.821)	Ammonium chloride (0.792)	Potassium bromide (0.803)	Potassium bromide (0.794)
0.90		Strontium nitrate (0.906)	Barium chloride (0.910)	Barium chloride (0.890)	Potassium nitrate (0.890)

Table 2: ERH and mean EMC values for DDGS with 10, 15, 20 and 25% db solubles (S), at different temperatures using the static gravimetric method (n=3 for each treatment combination)

Temperature (°C)	ERH ^[a] (decimal)	10S		15S		20S		25S	
		EMC ^[b] (% db)	S.D. ^[c] (% db)	EMC ^[b] (% db)	S.D. ^[c] (% db)	EMC ^[b] (% db)	S.D. ^[c] (% db)	EMC ^[b] (% db)	S.D. ^[c] (% db)
10	0.622	18.74	0.73	14.15	0.04	17.87	0.46	18.07	0.12
	0.720	21.15	0.29	18.74	0.05	23.41	0.19	24.71	0.34
	0.821	27.85	0.17	25.98	0.16	32.16	0.60	57.66	1.22
	0.906	38.01	0.81	43.41	0.29	56.84	0.57	66.17	1.61
20	0.591	9.56	0.96	21.21	0.07	17.72	0.48	30.15	0.04
	0.700	16.05	0.64	18.11	0.12	32.07	0.40	42.20	0.30
	0.792	21.96	0.00	40.74	0.34	51.22	0.59	55.05	0.21
	0.910	29.96	1.47	42.94	0.41	55.00	2.15	61.54	0.59
30	0.560	8.61	0.01	23.02	1.38	14.42	0.09	15.03	0.08
	0.691	18.42	0.18	28.74	2.30	28.39	0.34	28.39	0.24
	0.803	23.16	1.36	32.20	3.69	32.61	0.56	33.62	0.25
	0.890	35.61	0.46	46.47	2.53	56.73	0.59	58.64	0.68
40	0.570	9.85	0.43	11.58	0.40	13.72	0.12	15.98	0.07
	0.680	23.61	0.23	30.21	0.24	34.08	0.60	35.49	0.19
	0.794	29.34	0.26	32.93	0.70	37.58	0.46	43.08	0.19
	0.890	47.07	1.04	83.49	0.04	90.70	3.22	132.01	10.70

[a] ERH values from standard results (Spencer, 1926; Hodgman, 1954/55; Greenspan, 1977)

[b] EMC = Mean EMC value

[c] S. D. = Standard deviation

Table 3: Fitted isotherm parameters for DDGS with 10, 15, 20 and 25% (db) solubles

Soluble (% db)	Equation	A	B	C	D	F- statistic	MRE (%)	SEM (%)	R ²	Residual classification
10	Modified Exponential	1.270	-0.011	3.544	-	597.91	17.22	4.18	0.976	Random
	Modified Smith	7.166	-0.400	8.920	0.356	90.21	15.95	4.06	0.860	Random
	Modified Halsey	3.351	0.015	1.637	-	481.90	21.18	4.64	0.970	Random
	Modified Oswin	9.299	0.091	1.835	-	493.11	20.68	4.59	0.971	Random
	Sehgal	8.630	0.083	-	-	80.95	22.19	6.39	0.638	Systematic
	Chung Pfof	107.400	0.124	-	-	34.41	30.15	8.03	0.428	Systematic
	Henderson	1.85E-06	3.321	-	-	162.47	38.13	9.30	0.876	Systematic
15	Modified Exponential	0.756	-0.028	3.996	-	308.89	21.09	8.06	0.954	Random
	Modified Smith	12.042	-0.717	4.501	0.847	60.76	21.02	7.77	0.806	Random
	Modified Halsey	2.591	0.031	1.410	-	338.80	20.85	7.72	0.958	Random
	Modified Oswin	6.832	0.284	1.603	-	320.78	20.73	7.92	0.955	Random
	Sehgal	12.093	0.072	-	-	21.98	34.05	14.18	0.323	Systematic
	Chung Pfof	400.600	0.132	-	-	7.61	39.40	15.97	0.142	Systematic
	Henderson	1.05E-13	7.826	-	-	86.59	45.15	16.98	0.790	Systematic
20	Modified Exponential	0.837	-0.017	4.358	-	397.53	16.88	8.30	0.964	Random
	Modified Smith	6.901	-0.763	13.221	0.799	84.04	14.34	8.01	0.850	Random
	Modified Halsey	2.760	0.021	1.327	-	386.75	18.62	8.41	0.963	Random
	Modified Oswin	9.504	0.223	1.492	-	382.81	18.02	8.45	0.962	Random
	Sehgal	7.540	0.049	-	-	44.90	28.05	14.45	0.494	Systematic
	Chung Pfof	113.200	0.080	-	-	17.64	38.56	17.27	0.277	Systematic
	Henderson	1.73E-08	4.195	-	-	92.15	48.47	19.24	0.800	Systematic
25	Modified Exponential	0.690	-0.018	4.736	-	161.04	24.68	15.92	0.915	Random
	Modified Smith	23.057	-1.605	6.692	1.406	38.13	26.96	15.34	0.722	Random
	Modified Halsey	2.467	0.025	1.216	-	164.04	29.01	15.79	0.916	Random
	Modified Oswin	9.669	0.287	1.393	-	156.15	27.24	16.15	0.912	Random
	Sehgal	6.771	0.038	-	-	32.52	29.32	21.79	0.414	Systematic
	Chung Pfof	89.120	0.061	-	-	14.79	37.93	24.76	0.243	Systematic
	Henderson	4.91E-08	3.714	-	-	68.55	47.82	27.04	0.749	Systematic

Table 4: Fitted isotherm parameters for DDGS with 10, 15, 20 and 25% db solubles using new models

Models	A	B	C	D	F- statistic	MRE (%)	SEM (%)	R ²	Residual classification
NMH 1	-0.196	0.024	1.326	0.356	732.350	22.760	10.043	0.9397	random
NMH 2 (GMR)	0.0235	0.353	1.368	-	977.550	22.707	10.036	0.9395	random
NME 1	-0.1127	-0.018	4.305	0.051	702.850	20.680	10.239	0.9373	random
NME 2	0.0167	0.048	4.278	-	934.430	20.561	10.251	0.9368	random
NMO 1	-3.1431	0.222	1.518	0.864	680.220	22.540	10.397	0.9354	random
NMO 2	0.2026	0.534	1.533	-	900.110	22.235	10.432	0.9346	random

Figure 1: Residual plot of Modified Exponential model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 2: Residual plot of Modified Smith Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 3: Residual plot of Modified Halsey Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 4: Residual plot of Modified Oswin Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 5: Residual plot of Sehgal Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 6: Residual plot of Chung-Pfost Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 7: Residual plot of Henderson Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 8: Residual plot of New Modified Halsey 1 (NMH 1) Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 9: Residual plot of New Modified Halsey 2 (NMH 2) / GMR Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 10: Residual plot of New Modified Exponential 1 (NME 1) Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 11: Residual plot of New Modified Exponential 2 (NME 2) Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 12: Residual plot of New Modified Oswin 1 (NMO 1) Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 13: Residual plot of New Modified Oswin 2 (NMO 2) Model for the moisture isotherm data of DDGS at temperatures 10°C to 40°C

Figure 14: Comparison between observed and GMR model predicted sorption isotherms of DDGS with 10% db solubles at temperatures from 10°C to 40°C

Figure 15: Comparison between observed and GMR model predicted sorption isotherms of DDGS with 15% db solubles at temperatures from 10°C to 40°C

Figure 16: Comparison between observed and GMR model predicted sorption isotherms of DDGS with 20% db solubles at temperatures from 10°C to 40°C

Figure 17: Comparison between observed and GMR model predicted sorption isotherms of DDGS with 25% (db) solubles at temperatures from 10°C to 40°C