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Moisture sorption isotherms and hysteresis of soybean grains

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ABSTRACT. Knowledge of the relationship between the equilibrium moisture content of the product and the air that involves it is essential to guarantee product quality and preserve its characteristics during storage. This trend can be studied by sorption isotherms. Thus, this study aimed to obtain desorption and adsorption isotherms of soybean grains to determine the mathematical model that best fits the experimental data and analyze the hysteresis phenomenon. Soybean grains with a moisture content of 21.95% (db) were used to verify the desorption process. The grains were dried until 3.50% (db) for the adsorption process. The static-gravimetric method was employed to determine the equilibrium moisture content of the grain at different temperatures (10, 20, 30, 40, and 50°C) and relative humidity levels (0.10 to 0.92%). Eight mathematical models were fitted to the experimental data. The modified Halsey model satisfactorily represented the desorption and adsorption phenomena of soybean grains. The equilibrium moisture content of soybean grains increased along with an increment in water activity. The increase in temperature led to a reduction in the equilibrium moisture content of soybean grains at a constant water activity. The equilibrium moisture content values obtained by desorption are higher than those obtained by adsorption, indicating the hysteresis phenomenon at the studied temperature range. The isotherms obtained for the desorption and adsorption process were classified as type III because of the high oil content in soybean grains.

Keywords: adsorption; desorption; equilibrium moisture content; *Glycine max* (L.) Merr.; mathematical modeling.

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Introduction

A country must be able to store its agricultural production for several years to ensure the food security of the population, protecting itself from the impacts of adverse weather conditions or armed conflicts, factors that may difficult or become inviable the agricultural production for food supply (Oliveira, Corrêa, Oliveira, Baptestini, & Vargas-Elías, 2017). Furthermore, the storage of agricultural products should contribute to quality maintenance of the products retrieved from production fields, ensuring its commercialization with lower depreciation of market value (Ziegler et al., 2016).

The relationship between the equilibrium moisture content of the product and the relative air humidity at a given temperature should be evaluated to preserve the soybean (*Glycine max* (L.) Merr.) quality characteristics during storage (Raji & Ojediran, 2011; Mahanti & Das, 2015; Arslan-Tontul, 2020). Most agricultural products, including soybean, can lose or gain moisture from the environment, converging, constantly, to keep a relationship of equilibrium between its moisture content and the air conditions. Equilibrium moisture content is reached when the partial pressure of water vapor in the product is equal to the partial pressure of water vapor in the surrounding air (Ashour, Korjenic, & Korjenic, 2015).

The equilibrium moisture content of a hygroscopic material is dependent on the path used to achieve this equilibrium (Corrêa, Botelho, Botelho, & Goneli, 2014). Thus, two equilibrium moisture content (adsorption or desorption) can be found for a value of relative air humidity. This trend occurs because the biological material may have a lower or higher value of moisture content than the equilibrium for the environmental conditions. The sorption process is not entirely reversible, causing a difference between equilibrium moisture content values obtained by desorption and adsorption, which is known as hysteresis (Peleg, 2020).

The desorption isotherm in hysteresis has higher values of equilibrium moisture content than the adsorption isotherm at a given water activity (Zeymer, Corrêa, Oliveira, Baptestini, & Campos, 2019). According to Brooker, Bakker-Arkema, and Hall (1992), this phenomenon occurs because grains are porous

materials formed by narrow capillaries of small diameters and tubes of higher diameter. Pores of small diameters control the draining out of capillaries during the desorption process, resulting in a reduction of the relative humidity of the porous space within the grain. Capillary is not filled up when the grain gains moisture during adsorption, not reaching the previous moisture content.

The relationship between the equilibrium moisture content of grains and the relative air humidity at a constant temperature may be expressed using mathematical models. Studies on the use of non-linear models to estimate equilibrium moisture content of different foodstuff have been extensively reported. The parameters of fitted models are used to predict answers through the experimental data. Thus, mathematical models may be useful tools to describe, estimate, and simulate equilibrium moisture content of stored products under different conditions of temperature and relative air humidity, assisting farmers and industry in decision-making and strategies of post-harvest management.

The selected model and its parameters obtained to represent the sorption isotherms of a product are specific to this product. It cannot be used to represent another species, which may result in significant differences in the equilibrium moisture content. Brooker et al. (1992) stated that the chemical composition of a product directly affects the sorption process: grains with higher oil content adsorb a lower amount of moisture from the environment than grains with higher starch content. Furthermore, variety, maturity stage, and physical and sanitary conditions, along with the path used to reach equilibrium (desorption or adsorption), are criteria that must be considered for the establishment of the equilibrium moisture content of a determined product.

Thus, considering the importance of the hygroscopicity of products, this study aimed to obtain sorption isotherms of soybean grains under different psychrometric conditions of the air, determine the mathematical model that best fits the experimental data, and analyze the hysteresis phenomenon.

Material and methods

The study was conducted at the Laboratory of Physical Properties and Quality of Agricultural Products belonging to the National Training Center in Storage (CENTREINAR), located at the Federal University of Viçosa, Viçosa, Minas Gerais State, Brazil.

Soybean grains of the variety DM 68I69 IPRO from Campo Verde, Mato Grosso State, Brazil, were used. This variety stood out in the 2019/2020 growing season in Brazil due to its high production potential, precocity, resistance to diseases, and high adaptability to different Brazilian regions. The grains were manually harvested with an initial moisture content of 21.95% (dry base, db), homogenized, placed into low-density polypropylene bags, and transported to Viçosa. Previously to the analyses, the samples were stored in BOD chambers (Fanem 347 CD) at a temperature of $4 \pm 1^\circ\text{C}$ for 5 days to maintain and standardize the moisture content of soybean grains.

The desorption process was carried out with soybean grains with an initial moisture content of 21.95% (db). The grains were dehydrated in a forced-air circulation oven (Gehaka 400-3ND) at $60 \pm 2^\circ\text{C}$ until reaching a final moisture content of 3.50% (db).

The static-gravimetric method was employed to reach the equilibrium moisture content of the soybean grains for both desorption and adsorption processes (Brasil, 2009) under different conditions of temperature (10, 20, 30, 40, and 50°C) and relative humidity (11 to $92 \pm 2\%$). All combinations were performed using three replicates.

Saturated salt solutions were used to maintain the relative air humidity stable at different temperatures (Table 1). The salt solutions, diluted in distilled water, were placed inside hermetic desiccators, occupying approximately 3 cm at the bottom of the desiccators. Aluminum recipients containing approximately 30 g of soybean grains were placed in triplicates above and without contact with the salt solutions (Figure 1). The desiccators with the samples were placed in BOD chambers (Fanem 347 CD) to control the internal temperature required for the experiment. A data logger was placed inside the desiccators to record the relative humidity and temperature of storage.

The samples were daily weighted during desorption and adsorption procedures using an analytical scale (Marte AY220). Hygroscopic equilibrium was reached when the mass variation of soybean grains remained invariable or lower than 0.01 g for three consecutive weighings. The moisture content of each sample was determined by the gravimetric method, using a forced-air circulation oven at $105 \pm 1^\circ\text{C}$ for 24h in three replicates, according to Brasil (2009).

Eight mathematical models were used to determine the desorption and adsorption isotherms of soybean grains (Table 2). These models, often used to represent the hygroscopicity of agricultural products, were fitted to the experimental data of equilibrium moisture content of soybean grains, obtained at each combination of temperature and relative air humidity, for the desorption and adsorption processes.

Table 1. Values of relative air humidity (decimal) relative to saturated salt solutions at temperatures of 10, 20, 30, 40, and 50°C.

Salt solution	Temperature (°C)				
	10	20	30	40	50
LiCl	0.13	0.11	0.11	0.12	0.11
CaCl ₂	0.40	0.35	0.32	0.32	0.31
Ca(NO ₃) ₂	0.59	0.55	0.49	–	0.46
NaCl	–	–	0.76	0.75	0.75
KBr	0.81	0.84	–	–	–
K ₂ SO ₄	–	–	–	0.92	–

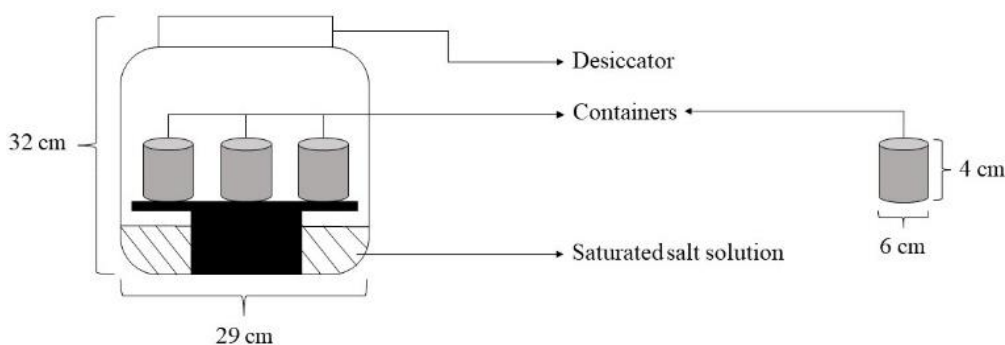


Figure 1. Schematic drawing of the samples and saturated salt solutions inside the hermetic desiccators used in the experiment.

Table 2. Mathematical models used to predict the hygroscopicity of soybean grains through desorption and adsorption processes.

Model	Equation	Number
Chung Pfof	$X_e = a - b \ln[-(T + c) \ln(a_w)]$	(1)
Copace	$X_e = \exp[a - (bT) + (ca_w)]$	(2)
Modified GAB	$X_e = \frac{ab \left(\frac{c}{T}\right) a_w}{\left\{ [1 - ba_w] \left[1 - ba_w + b \left(\frac{c}{T}\right) a_w \right] \right\}}$	(3)
Modified Halsey	$X_e = \left[\frac{\exp(a - bT)}{-\ln(a_w)} \right]^{\frac{1}{c}}$	(4)
Harkins-Jura	$X_e = \frac{\exp(a - bT)}{c - \ln(a_w)}$	(5)
Modified Henderson	$X_e = \left[\frac{\ln(1 - a_w)}{-a(T + b)} \right]^{\frac{1}{c}}$	(6)
Modified Oswin	$X_e = (a + bT) \left[\frac{a_w}{1 - a_w} \right]$	(7)
Smith	$X_e = a - (bT) - c \ln(1 - a_w)$	(8)

In which X_e is the equilibrium moisture content (% db), a_w is the water activity (decimal), T is the temperature (°C), and a , b , and c are coefficients that depend upon the product.

The models were fitted to the experimental data by non-linear regression analysis, using the Gauss-Newton method through the software Statistica 10.0®. The following parameters were considered to select the best model to predict the equilibrium moisture content of soybean grains: coefficient of determination (R^2), the magnitude of the mean relative error (MRE) (Equation 9), standard error of estimate (SEE) (Equation 10), and residual plots. A good mathematical fit is reached when R^2 is close to unity (Kashaninejad, Mortazavi, Safekordi, & Tabil, 2007), MRE is lower than 10% (Gomes da Costa, Silva, Toledo Hijo, Azevedo, & Borges, 2015), SEE is close to zero (Draper & Smith, 1998), and the residuals are randomly distributed, with residual values close to the horizontal range, around zero, without forming defined or geometric figures (Corrêa et al., 2014). According to Kuhn and Johnson (2013), the graphical analysis of predicted dispersion residuals is an important step in the model selection and should not be neglected by the usage of only statistical measures such as RMSE and R^2 .

$$MRE = \frac{100}{n} \sum_{i=1}^n \frac{|Y_i - \hat{Y}_i|}{Y_i} \tag{9}$$

$$SEE = \sqrt{\frac{\sum_{i=1}^n (Y_i - \hat{Y}_i)^2}{DF}} \tag{10}$$

where MRE is the mean relative error (%), SEE is the standard error of estimate (% db), Y_i is the observed value (% db), \hat{Y}_i is the value estimated by the model (% db), n is the number of observed data, and DF is the residual degrees of freedom (number of observed data minus the number of model parameters).

The Akaike information criterion (AIC) and Schwarz's Bayesian information criterion (BIC) were also used as statistical evaluators of the best model to predict the equilibrium moisture content of soybean grains. Lower values of both parameters indicate a better fit of the model (Emiliano, Vivanco, & Menezes, 2014). AIC and BIC values were calculated using the software R version 4.0.2, according to Equations (11) and (12), respectively.

$$\text{AIC} = -2\log L + 2p \quad (11)$$

$$\text{BIC} = -2\log L + p\ln(N - r) \quad (12)$$

in which p is the number of parameters of the model, N is the total number of observations, r is the rank of the matrix X (incidence matrix of fixed effects), and L is the maximum likelihood.

The hysteresis phenomenon was analyzed after selecting the model that best fitted the experimental data of equilibrium moisture content of soybean grains. It is characterized by the difference between the desorption and adsorption isotherms, analyzing the influence of temperature in this process.

Results and discussion

The parameters of mathematical models fitted to the equilibrium moisture content data of soybean grains obtained for desorption and adsorption are shown in Tables 3 and 4, respectively. Furthermore, the values of coefficient of determination (R^2), mean relative error (MRE), standard error of estimate (SEE), Akaike information criteria (AIC), Bayesian information criterion (BIC), and the analysis of residual values are also presented.

Tables 3 and 4 show that the models presented values of coefficient of determination higher than 96% for desorption and adsorption, except for the Chung Pfof model. According to Sheskin (2004), the coefficient of determination defines the model success, whilst it evaluates the variation in the experimental data. However, Madamba, Driscoll, and Buckle (1996) stated that this parameter cannot be used in isolation as a criterion to evaluate non-linear models. Therefore, other statistical parameters, such as mean relative error (MRE) and standard error of estimate (SEE), were analyzed to determine the best model.

Table 3. Model parameters fitted to the hygroscopic equilibrium of soybean grains, obtained by desorption.

Model	Parameter ^a	R^2 (%)	MRE (%)	SDE (decimal)	AIC	BIC	Residual plot
Chung Pfof	a = 44.3681	95.84	21.71	2.17	92.53	96.51	Biased
	b = 7.3512						
	c = 101.8813						
Copace	a = 1.1639	98.64	10.05	1.25	70.45	74.44	Biased
	b = 0.0055						
	c = 2.5311						
Modified GAB	a = 5.7397	98.61	9.90	1.26	70.84	74.82	Biased
	b = 0.8999						
	c = 400.5952						
Modified Halsey	a = 3.4084	99.56	7.37	0.71	47.94	51.92	Random
	b = 0.0109						
	c = 1.5445						
Harkins-Jura	a = 2.2824	99.14	9.01	0.99	61.30	65.29	Biased
	b = 0.0059						
	c = 0.1803						
Modified Henderson	a = 0.0003	97.53	17.09	1.68	82.32	86.30	Biased
	b = 120.3904						
	c = 1.2309						
Modified Oswin	a = 11.7057	99.09	10.01	1.02	62.40	66.39	Biased
	b = -0.0629						
	c = 1.9717						
Smith	a = 4.3370	98.55	10.62	1.29	71.67	75.66	Biased
	b = 0.0599						
	c = 10.8805						

^aSignificant at 1% probability by the t-test.

Table 4. Model parameters fitted to the hygroscopic equilibrium of soybean grains, obtained by adsorption.

Model	Parameter [*]	R ² (%)	MRE (%)	SDE (decimal)	AIC	BIC	Residual plot
Chung Pfost	a = 44.7268 b = 7.3148 c = 121.1709	95.26	24.80	2.31	95.04	99.02	Biased
Copace	a = 1.0121 b = 0.0052 c = 1.6820	98.52	11.23	1.30	72.03	76.01	Biased
Modified GAB	a = 5.2969 b = 0.9163 c = 396.6801	98.62	10.52	1.26	70.68	74.67	Biased
Modified Halsey	a = 3.1387 b = 0.0101 c = 1.4746	99.50	8.05	0.76	50.59	54.57	Random
Harkins-Jura	a = 2.1726 b = 0.0058 c = 0.1503	99.16	9.33	0.98	60.73	64.72	Biased
Modified Henderson	a = 0.0003 b = 137.9310 c = 1.1500	97.44	18.36	1.71	82.98	86.96	Biased
Modified Oswin	a = 10.9182 b = -0.0572 c = 1.8687	98.99	11.18	1.08	64.48	68.47	Biased
Smith	a = 3.6431 b = 0.0525 c = 10.8609	98.27	12.32	1.41	75.17	79.15	Biased

*Significant at 1% probability by the t-test.

Several authors have agreed that a model fits the experimental data satisfactorily if the MRE value is lower than 10% (Madamba et al., 1996; Kashaninejad et al., 2007; Rosa, Moraes, & Pinto, 2010; Gomes da Costa et al., 2015). According to Kashaninejad et al. (2007), MRE values indicate a deviation from the observed values relative to the curve estimated by the model. Thus, the models Modified GAB, Modified Halsey, and Harkins-Jura presented satisfactory MRE values for desorption, while the models Modified Halsey and Harkins-Jura exhibited magnitudes lower than 10% for adsorption.

The ability of a model to describe a physical process is inversely proportional to the SEE value (Draper & Smith, 1998). Therefore, lower SEE values indicate that the model represents the observed data well. Tables 3 and 4 show that the Modified Halsey model presented lower SEE values among the tested models for desorption and adsorption, respectively.

Finally, the Akaike information criterion (AIC) and Schwarz's Bayesian information criterion (BIC) were determined aiming to obtain other statistical parameters to select the best model to predict the equilibrium moisture content of soybean grains (Tables 3 and 4). According to Ferreira Junior, Resende, Oliveira, and Costa (2018), the best model indication can be more precise, as the AIC and BIC criteria consider other factors, such as the analysis of the degree of parameterization of the compared models. Emiliano et al. (2014) state observed that lower AIC and BIC values indicate a better model adjustment compared to the others. Table 4 shows that the Modified Halsey model presented the lowest magnitudes of this parameter for desorption and adsorption. Several authors have used this statistical criterion to select the mathematical model that best describes the sorption curve of agricultural products (Ferreira Junior et al., 2018; Quequeto, Resende, Silva, Silva, & Silva, 2019; Souza, Resende, Moura, Ferreira Junior, & Andrade, 2019; Fonseca et al., 2020; Corrêa, Zeymer, Oliveira, Araujo, & Silva, 2020).

Baptistini et al. (2017a) indicated that an analysis of the residual plot should be performed because the model may exhibit a biased distribution, being unable to represent the experimental data, regardless of the adequate statistical parameters. According to Corrêa et al. (2014), the model must present residual values close to the horizontal zone (around zero) and should not form geometric figures. Figure 2 shows that the trends exhibited by the residuals of the Modified Halsey model for desorption and adsorption are random within the range of the equilibrium moisture content, with positive and negative values.

Thus, according to the statistical results, the Modified Halsey model is recommended to represent the equilibrium moisture content of soybean grains, obtained by desorption and adsorption. Modified Halsey models also represented satisfactorily the hygroscopicity of sunflower seeds (Maciel, De La Torre, Bartosik, Izquierdo, & Cendoya, 2015), green soybean (Yang, Zhu, & Zhu, 2015), rapeseed (Le Duc & Dong, 2016),

castor beans (Goneli, Corrêa, Oliveira, Resende, & Mauad, 2016), pumpkin seeds (Teixeira, Andrade, & Devilla, 2018), chia seeds (Bustos-Vanegas, Corrêa, Zeymer, Baptestini, & Campos, 2018), and soybean expeller (Maciel et al., 2020).

The literature has indicated that Modified Halsey is the best model to predict the equilibrium moisture content of agricultural products with high oil and protein contents, including soybean (Maciel et al., 2015; Maciel et al., 2020). It occurs due to the behavior of the sorption isotherms generated by the model, which shows an abrupt increment with an increase in moisture content under high relative humidity values.

Figure 3 shows the sorption isotherms using the Modified Halsey model and the experimental results of equilibrium moisture content of soybean grains at all evaluated temperatures.

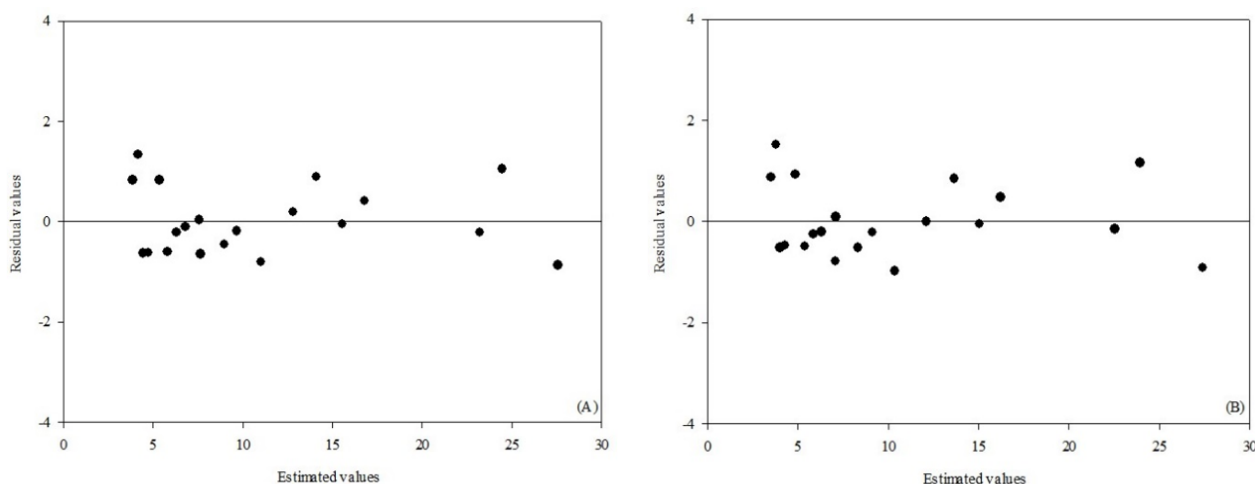


Figure 2. Random residual distribution of the Modified Halsey model for desorption (A) and adsorption (B) of soybean grains.

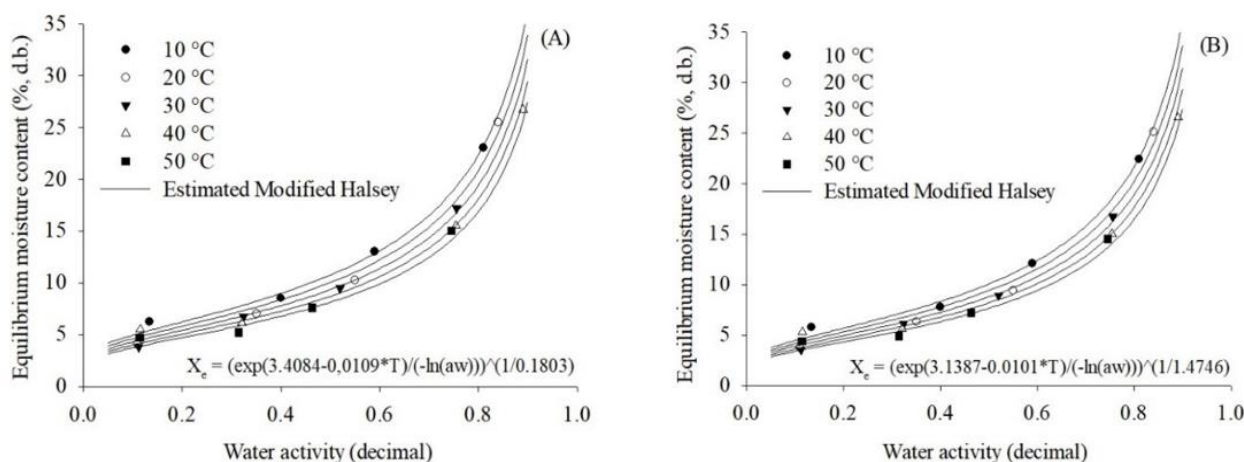


Figure 3. Observed and estimated values using the Modified Halsey model for the equilibrium moisture content obtained through desorption (A) and adsorption (B) of soybean grains at all evaluated temperatures.

Figure 3 shows an adequate correspondence between the values estimated by the model and the experimental data. Furthermore, an increase in the equilibrium moisture content can be observed with an increment in water activity at a constant temperature. According to Alpizar-Reyes et al. (2017), this trend occurs due to a reduction of the water-vapor pressure inside the soybean grain when subjected to lower relative humidity values.

Consequently, the equilibrium moisture content increases almost linearly from low to intermediate values of water activity (0.0 - 0.4). However, the equilibrium moisture content increases rapidly for higher values of water activity since the physical absorption of moisture at strongly active sites occurs at low values of relative humidity, whereas moisture is bound to the material surface under high levels of relative humidity (Vishwakarma, Shivhare, & Nanda, 2011). Analogous behavior was also observed by Baptestini, Corrêa, Oliveira, Cecon, and Soares (2017b), suggesting a similar trend for most products of agricultural origin.

Figure 3 also shows the temperature effect on the sorption isotherms of soybean grains. An increase in temperature at a constant water activity leads to a decrease in the equilibrium moisture content. According to McLaughlin and Magee (1998), this trend is related to the excitation state of molecules. The attractive forces between molecules are lower at high temperatures due to an increase in the kinetic energy of water molecules, allowing the connection between moisture and sorption sites to be broken, which reduces the moisture content of the product.

Figure 3 shows that the desorption and adsorption isotherms present a J shape type, known as type III isotherm, according to the classification of Sing (1985). According to Costa, Resende, and Oliveira (2013), type III isotherms indicate that the main constituents of the product (solutes) present low affinity to water molecules, as in the case of soybean grains, which have high oil contents in the composition. Yan et al. (2015) evaluated the equilibrium moisture content of green soybeans and also observed a type III behavior of the isotherms estimated by the Modified Halsey model. The literature shows that most agricultural products, especially foodstuff rich in starch, have sigmoidal-shape isotherms, called type III isotherms (Alpizar-Reyes et al., 2017; Iorfa, Charles, Oneh, & Iorwuese, 2018).

Figure 4 shows the desorption and adsorption isotherms of soybean grains estimated by the Modified Halsey model at each temperature separately.

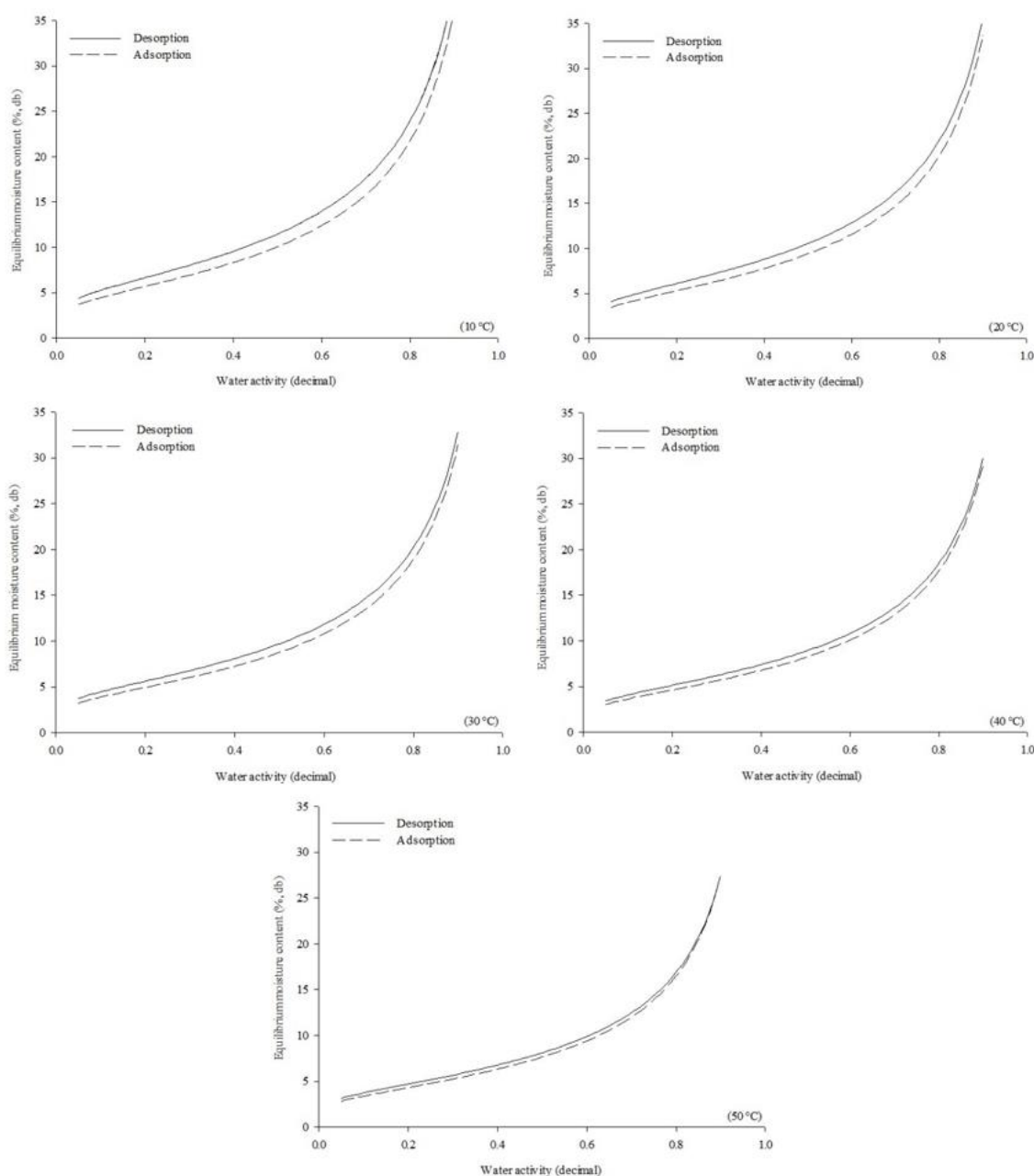


Figure 4. Desorption and adsorption isotherms of soybean grains estimated by the Modified Halsey model for all evaluated temperatures.

The values of equilibrium moisture content obtained by desorption are higher than those obtained by adsorption (Figure 4), evidencing the hysteresis phenomenon at the studied temperature range. There was also a trend to perform a closed cycle (hysteresis cycle) between the lower and upper limits of water activity as temperature increased. Bingol, Prakash, and Pan (2012) also reported this trend when evaluating the sorption isotherms of corn grains.

Several authors have explained the hysteresis phenomenon. Rizvi (2005) stated that hysteresis is related to the nature and state of foodstuff components, reflecting its potential for structural and conformational rearrangements, also changing the accessibility of energetically favorable polar sites. Vazquea, Chenlo, and Moreira (2003) argued that hysteresis is an irreversible process of physical and chemical alterations within dehydrated foodstuffs, impacting directly its final quality and stability. Iglesias and Chirife (1976) recognized the impossibility of giving a single explanation to hysteresis because foodstuff is a complex combination of several components, which may absorb water independently, but also may interact with each other.

One of the most used theories to explain the hysteresis phenomenon suggests that the sorption sites of the molecular structure of the material are almost filled up with adsorbed water under conditions of a high amount of moisture. The available sorption sites are reduced after drying, along with product shrinkage. It leads to a reduction in the water capacity to bind during future adsorption (Kapsalis, 2017).

Figure 5 shows the effect of temperature and water activity on the variation of hysteresis (equilibrium moisture content of desorption minus equilibrium moisture content of adsorption) of soybean grains, estimated by the Modified Halsey model.

The hysteresis phenomenon is observed in the entire range of water activity (Figure 5) for all tested temperatures, increasing at intermediate values of water activity (0.4 - 0.6). Also, the hysteresis magnitude decreased as temperature increased, as reported by different authors (Corrêa et al., 2014; Aviara, Ojediran, Sa'id, & Raji, 2016; Goneli et al., 2016; Baptestini et al., 2017a; Bustos-Vanegas et al., 2018; Jian et al., 2018; Silva, Rodovalho, & Silva, 2018; Torres, Chenlo, & Moreira, 2018; Zeymer et al., 2019). This trend may be attributed to an increase in the elasticity of the capillary walls and the higher capacity to form hydrogen bonds between dry matter and moisture present in the grain.

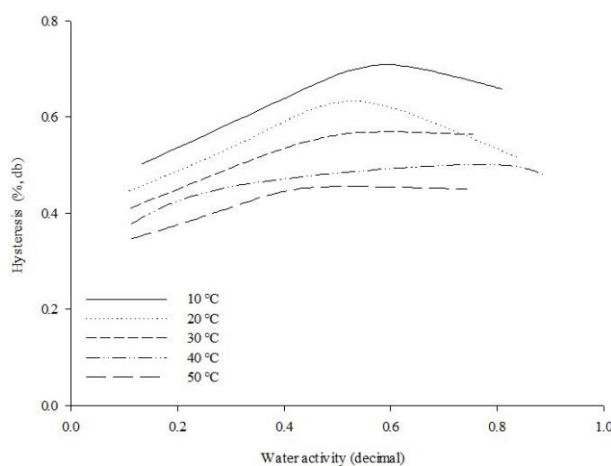


Figure 5. Effect of temperature and relative humidity on the hysteresis phenomenon in soybean grains, estimated by the Modified Halsey model.

Conclusion

The Modified Halsey model represents satisfactorily the desorption and adsorption isotherms of soybean grains. The equilibrium moisture content of soybean grains increased with an increment in water activity. The increase in temperature led to a reduction in the equilibrium moisture content of soybean grains at a constant water activity. The isotherms obtained for desorption and adsorption processes were classified as type III because of the high oil content of soybean grains. The values of equilibrium moisture content obtained through desorption are higher than those obtained by adsorption, evidencing the hysteresis phenomenon. The hysteresis values decrease with an increase in temperature.

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