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Comparison between desorption isotherm curves of ryegrass (*Lolium multiflorum* L.) and flax (*Linum usitatissimum* L.) seeds

Comparação entre curvas isotérmicas de dessorção de sementes de azevém (*Lolium multiflorum* L.) e linhaça (*Linum usitatissimum* L.)

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ABSTRACT

It is necessary to determine the sorption isotherms of seeds to develop adequate systems of storage and drying. The chemical composition of a product affects the sorption process; products with a high oil content adsorb a lower amount of moisture from the environment than products with a high carbohydrate content. Given the importance of the hygroscopicity of different agricultural products, this work aimed to determine, model and evaluate the difference between desorption isotherms of ryegrass and flax seeds grown at different temperature and relative humidity conditions. Ryegrass and flax seeds, which contained initial moisture contents of 10.4 and 8.7% (db), respectively, were used. The equilibrium moisture content of the seeds was determined using a static-gravimetric method at different temperatures (10, 20, 30, 40, and 50 ± 1 °C) and relative humidity values (between 11 and 96 ± 2%), in three replicates. Seven mathematical models were adjusted to the equilibrium moisture content experimental data of the seeds. The Chung Pfost model best fit the experimental data of ryegrass seeds, whereas the Smith model was determined to be the best fit for flax seeds. The equilibrium moisture content of the seeds was found to decrease as the temperature increased when the value of water activity was constant. The desorption isotherms of ryegrass seeds (Type II) and flax seeds (Type III) are different, according to Brunauer's classification, which is caused by the composition (starch and oil content) of each product.

Index terms: Equilibrium moisture content; chemical composition; mathematical modeling; Chung Pfost; Smith.

RESUMO

A fim de desenvolver sistemas adequados de armazenamento e secagem, é necessário determinar as isotermas de sorção das sementes. A composição química do produto influencia o processo de sorção; produtos com alto teor de óleo absorvem menor quantidade de umidade do ambiente, quando comparados a produtos com alto teor de carboidratos. Devido à importância de compreender a higroscopicidade de diferentes produtos agrícolas, este trabalho teve como objetivo determinar, modelar e avaliar a diferença entre as isotermas de dessorção de sementes de azevém e linhaça, em diferentes condições de temperatura e umidade relativa. Utilizaram-se sementes de azevém e linhaça com teor de água inicial de 10,4 e 8,7% (bs), respectivamente. O teor de água de equilíbrio das sementes foi determinado pelo método estático-gravimétrico em diferentes valores de temperatura (10, 20, 30, 40, e 50 ± 1 °C) e umidade relativa (entre 11 a 96 ± 2%), em três repetições. Sete modelos matemáticos foram ajustados aos dados experimentais do teor de água de equilíbrio das sementes. O modelo Chung Pfost foi o que melhor se ajustou aos dados experimentais das sementes de azevém, enquanto o modelo Smith foi escolhido para as sementes de linhaça. O teor de água de equilíbrio das sementes diminuiu à medida que a temperatura aumentou, para um valor constante de atividade de água. As isotermas de dessorção de sementes de azevém (Tipo II) e de linhaça (Tipo III) são diferentes, de acordo com a classificação de Brunauer, devido à composição dos produtos (teor de amido e óleo).

Termos para indexação: Teor de água de equilíbrio; composição química; modelagem matemática; Chung Pfost; Smith.

INTRODUCTION

Ryegrass (*Lolium multiflorum* L.) is a forage widely cultivated in southern Brazil. Its high dry matter yield and high plant and seed nutrition value contribute to ryegrass being one of the primary winter forages cultivated (Skonieski et al., 2011). Culture production success requires seeds of good physical and physiological quality (Finch-Savage; Bassel, 2016; Araujo et al., 2019). Thus, determining how to improve postharvest procedures, such as drying and storage, is of extreme importance in acquiring high quality seeds.

Flax (*Linum usitatissimum* L.) is an oleaginous product extensively cultivated worldwide, characterized by higher productivity in cold and humid climate conditions. It is considered to be a double use crop, due to its primary products: fiber retrieved from the plant and the extracted oil from the seeds (Anastasiu et al., 2016). Demand for these products has been increasing considerably over the most recent decade, due to the human health benefits offered. However, production of this culture has decreased over time. One possible contributor to this trend is the seed quality offered to producers retrieves a lower net income in comparison to other products (Zuk et al., 2015).

To develop and obtain adequate storage and drying systems it is necessary to determine the equilibrium relationship between the moisture content of seeds and the relative humidity of the surrounding environment at several temperatures (Corrêa et al., 2015). Knowledge of desorption isotherms is fundamental to improving the drying process, because it provides information to determinate the final point of this process. In addition, most drying models use the difference between the real moisture content and the equilibrium moisture content as a measure of the mass transfer driving force (Temple; Van Boxtel, 1999). Sorption isotherms are used to characterize the moisture state within solids; furthermore, they are useful for predicting the domain of the physical, chemical and microbiological stability of biosystems after drying. Finally, sorption isotherms may be used to determine more adequate storage conditions for products (Monte et al., 2018).

The chemical composition of a product directly affects the sorption process. According to Brooker, Bakker-Arkema and Hall (1992), products with a high oil content adsorb a lower amount of moisture from the environment when compared to products with a high carbohydrate content. Moreover, the cultivar, maturation stage, physical aspects and shape of a product are key factors to acquire the equilibrium moisture content of hygroscopic materials (Goneli et al., 2018). Oh, Lee and Hong (2018) report that the modeling precision of desorption isotherms is linked to the product type and composition. Materials with different amounts of oil and carbohydrates may present different types of desorption isotherms and require different models to describe them.

Ryegrass seeds are rich in starch and sugar, which causes the seeds to essentially be hydrophilic, requiring more energy and time in the desorption process (Marchesan et al., 2015). However, the high content of oil in flax seed can represent approximately 50% of the seed mass. Thus, flax seeds are hydrophobic, resulting in a faster desorption process, with lower energetic costs (Cloutier et al., 2011). Due to the differences in the chemical composition of biological materials, the determination of sorption isotherms for each product is indispensable (Sormoli; Langrish, 2015).

In an effort to better understand how the hygroscopicity of different agricultural products assures drying and storage success, this work aimed to determine, model and evaluate the difference between the desorption isotherms of ryegrass and flax seeds in different temperature and relative humidity conditions.

MATERIAL AND METHODS

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

Ryegrass seeds (*Lolium multiflorum* L.) and flax seeds (*Linum usitatissimum* L.), purchased at a local market in the city of Viçosa-MG, Brazil, had initial moisture contents of 10.4 and 8.7% (dry basis, db), respectively. The static-gravimetric method was used to determine the equilibrium moisture content of the seeds obtained by desorption.

A saturated salt solution was prepared and placed within desiccators to control the relative humidity of the air (11 to 96 ± 2%) (Table 1).

Table 1: Relative humidity of the air related to saturated saline solutions at temperatures of 10, 20, 30, 40, and 50 °C.

Saline Solutions	Temperature (°C)				
	10	20	30	40	50
LiCl	0.13	0.11	0.11	0.12	0.11
$CaCl_2$	0.40	0.35	-	-	-
$Ca(NO_3)_2$	0.59	0.55			
NaCl	0.76	0.76	0.76	0.75	0.75
KBr	-	0.84	-	-	-
K_2SO4	-	-	-	0.96	-
$MgCl_2$	-	-	0.32	-	-
KNO_2	-	-	0.48	-	-
KNO_3	0.96	0.93	0.91	-	-
$MgCl_2$ x $6H_2O$	-	-	-	0.32	0.31
$Na_2Cr_2O_7$	-	-	-	0.50	0.46

Source: Dhingra and Sinclair (1995).

Containers holding 30 g of seeds were placed in triplicate above the salt solutions. Desiccators were stored in Bio-Oxygen Demand (B.O.D.) incubator (model 347 CD/brand Fanem/Carandiru, SP, Brazil) at different temperatures (10, 20, 30, 40 and 50 ± 1 °C).

During the desorption process, samples were weighed daily with the aid of a digital analytical scale (model AY220/brand Marte/São Paulo, SP, Brazil), and hygroscopic equilibrium was reached when the mass variation remained invariable or lower than 0.01 g for three consecutive measurements of weight. After hygroscopic equilibrium was reached, the moisture content of each sample was determined using the gravimetric method, using an oven with forced air circulation at 105 ± 1 °C for 24 h in three repetitions, according to Brasil (2009).

Seven mathematical models (Equation 1 to 7) were fitted to the experimental data of the equilibrium moisture content of ryegrass and flax seeds (Table 2).

Table 2: Mathematical models used to predict the hygroscopicity of ryegrass and flax seeds.

Model name	Model	Equation number
Modified Henderson (Thompson; Peart; Foster, 1968)	$X_e = \left[\frac{In(1-a_w)}{-a(T+b)}\right]^{\frac{1}{c}}$	(1)
Modified Halsey (Iglesias; Chirife, 1976)	$X_e = \left[\frac{\exp(a-bT)}{-In(a_w)}\right]^{\frac{1}{c}}$	(2)
Modified Oswin (Chen; Morey, 1989)	$X_e = (a+bT)\left[\frac{a_w}{1-a_w}\right]$	(3)
Harkins-Jura (Harkins, 1945)	$X_e = \frac{\exp(a-bT)}{c-In(a_w)}$	(4)
Copace (Corrêa; Martins; Melo, 1995)	$X_e = \exp\left[a-(bT)+(ca_w)\right]$	(5)
Chung Pfost (Chung; Pfost, 1967)	$X_e = a-bln\left[-(T+c)ln(a_w)\right]$	(6)
Smith (Smith, 1947)	$X_e = a-(bT)-cln(1-a_w)$	(7)

X_e – equilibrium moisture content, (% db); a_w – water activity (decimal); T – temperature (°C); a, b and c – coefficients dependent upon the product.

Models were adjusted to experimental data using nonlinear regression analysis for the Gauss-Newton method. To select the model that best predicted the equilibrium moisture content, the determination coefficient (R^2), mean relative error (MRE), estimated standard error (SEE) and randomness of residual values were considered. The MRE and SEE values of each model were calculated using Equations 8 and 9, respectively:

$$MRE = \frac{100}{n}\sum_{i=1}^{n}\frac{\left|Y_i - \hat{Y}_i\right|}{Y_i} \quad (8)$$

$$SEE = \sqrt{\frac{\sum_{i=1}^{n}\left(Y_i - \hat{Y}_i\right)^2}{GLR}} \quad (9)$$

where MRE is the mean relative error, %; SEE is the estimated standard error, % db; Y_i is the observed value, % db; Ŷ is the estimated value by the model, % db; n is the number of observed data; and DF is the residual degrees of freedom (number of observed data minus 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 the ryegrass and flax seeds. AIC (Equation 10) is an evaluator that uses more complex selection, as verisimilitude is a qualitative character. BIC (Equation 11) is similar to AIC in terms of verisimilitude, but it presents different penalties regarding the number of estimated parameters (Burnham; Anderson, 2004). Lower values of both parameters indicate a better fit of the model (Akaike, 1974; Schwarz, 1978).

$$AIC = -2logL + 2p \quad (10)$$

$$BIC = -2logL + pln\left(N - r\right) \quad (11)$$

where p is the number of parameters in 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.

RESULTS AND DISCUSSION

Tables 3 and 4 present the parameters of the mathematical models fitted to the hygroscopic equilibrium experimental data of ryegrass and flax seeds, respectively, obtained by desorption. Furthermore, the coefficient of determination (R^2), mean relative error (MRE), estimated standard error (SEE), AIC, BIC and residual plot values are also presented.

Analysis of Tables 3 and 4 reveals that all mathematical models presented coefficient of determination (R^2) values that were higher than 96% for both products. According to Sheskin (2004), R^2 defines the model success, as it evaluates the experimental data variation. However, this statistical parameter is not a reliable decision-making tool for nonlinear models, being an indicative index of the best model (Botelho et al., 2019). A model adequately represents experimental data when the MRE is less than 10% (Costa et al., 2015) and when SEE values are lower (Draper; Smith, 1998).

MRE indicates the percentage of the average error present in the model, corresponding to the model's estimated curve. Concerning the desorption isotherms of ryegrass seeds (Table 3), the Modified Halsey, Copace and Smith models are inadequate to represent the experimental data (MRE > 10%), whereas the Smith model is the only model that fulfills this criterion for the desorption of flax seeds (Table 4).

Remaining statistical parameters must be considered to determine the proper model. SEE indicates the average error of the model and lower values indicate a greater ability by the model to describe a physical process (Draper; Smith, 1998), in the present study, the desorption process. Table 3 shows that the Chung Pfost model presented the lowest SEE values, whereas in Table 4, the Smith model had the lowest magnitudes of this parameter.

AIC and BIC were determined using other statistical parameters that selected the best model for predicting the equilibrium moisture content of the seeds. According to Ferreira Junior et al. (2018), the best model may be more precise as the criteria consider other factors such as the parametrization degree of the models compared. Considering the lowest values of AIC and BIC, the Chung Pfost model exhibited the best fit to the experimental data for the hygroscopic equilibrium of ryegrass seeds. However, for the equilibrium moisture content of flax seeds, the Smith model best fit the data.

Table 3: Model parameters fitted to the equilibrium moisture content of ryegrass seeds, obtained by desorption, with analyzed statistical parameters.

Model	Parameters*	R^2 (%)	SEE (decimal)	MRE (%)	AIC	BIC	Residual plot
Modified Henderson	a = 0.00016 b = 27.30824 c = 1.83349	99.16	0.75	6.80	52.00	56.18	Random
Modified Halsey	a = 4.843509 b = 0.020076 c = 2.007359	98.09	1.13	13.69	69.32	73.50	Biased
Modified Oswin	a = 13.77001 b = -0.10935 c = 2.71318	99.17	0.74	8.18	51.77	55.95	Biased
Harkins-Jura	a = 2.8752 b = 0.0108 c = 0.5232	99.15	0.75	7.57	52.28	56.46	Biased
Copace	a = 1.771080 b = 0.010648 c = 1.753426	98.68	0.94	10.67	61.54	65.72	Biased
Chung Pfost	a = 31.98376 b = 5.90103 c = 22.81437	99.34	0.66	5.67	46.97	51.15	Random
Smith	a = 7.666143 b = 0.116596 c = 8.671001	98.34	0.99	11.45	59.78	63.96	Biased

* Significant at 1% probability according to a t-test.

Table 4: Model parameters fitted to the equilibrium moisture content of flax seeds, obtained by desorption, with analyzed statistical parameters.

Model	Parameters*	R^2 (%)	SEE (decimal)	MRE (%)	AIC	BIC	Residual plot
Modified Henderson	a = 0.00080 b = 21.95261 c = 1.64004	98.34	0.64	13.28	45.77	49.95	Biased
Modified Halsey	a = 3.4734 b = 0.0244 c = 1.8821	96.74	0.90	21.60	59.81	63.98	Biased
Modified Oswin	a = 7.774562 b = -0.074514 c = 2.510142	98.01	0.70	15.30	49.54	53.72	Biased
Harkins-Jura	a = 2.238718 b = 0.013650 c = 0.435856	98.49	0.61	14.25	43.75	47.93	Biased
Copace	a = 1.121089 b = 0.013445 c = 1.925965	98.31	0.65	15.60	46.16	50.34	Biased
Chung Pfost	a = 17.64801 b = 3.49790 c = 14.48074	99.05	0.49	12.53	43.97	48.15	Biased
Smith	a = 4.353000 b = 0.084726 c = 5.149479	98.96	0.51	9.41	35.92	40.10	Random

* Significant at 1% probability according to a t-test.

Furthermore, analysis of the residual plot is required to verify if the model is capable of describing the phenomenon under study. A random residual plot must have residual values near the horizontal zone and should not form geometric figures (Zeymer et al., 2019). Examples of the analysis of the residual plots are presented in Figures 1 and 2.

The Modified Halsey (Figure 1A) and Modified Oswin (Figure 2A) models present a biased distribution of the residues, exhibiting defined geometric figures. In contrast, the Chung Pfost (Figure 1B) and Smith (Figure 2B) models obtained randomness residues, with positive and negative values distributed near the horizontal line, which makes them suitable for predicting the equilibrium moisture content of ryegrass and flax seeds.

Finally, Figure 3 presents an adequate correlation between the estimated and observed values of the equilibrium moisture content of ryegrass and flax seeds, respectively. Thus, the Chung Pfost and Smith models were selected to represent the desorption isotherms of ryegrass and flax seeds, respectively.

Different models often represent the sorption process of different products, such as ryegrass and flax seeds. This trend is explained by a product's physical characteristics and chemical composition. Regarding the products in the present study, ryegrass seeds are rich in starch and sugar (hydrophilic) and flax seed is rich in oil (hydrophobic).

In accordance with the present research, the Chung Pfost model has been used to represent the sorption phenomena of starchy products, such as okra seeds (Goneli et al., 2010), sweet sorghum seeds (Ullmann et al., 2016), sugar beet seeds (Corrêa et al., 2016), and rice grains (Zeymer et al., 2019). In contrast, the Smith model has

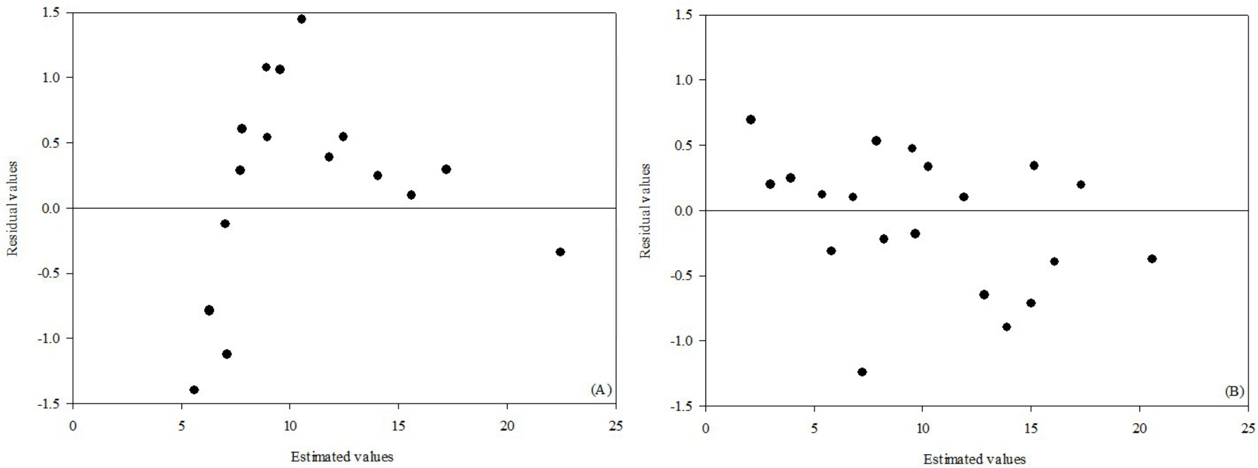

Figure 1: Residual plots of Modified Halsey (A) and Chung Pfost (B) models fit to the desorption data of ryegrass seeds.

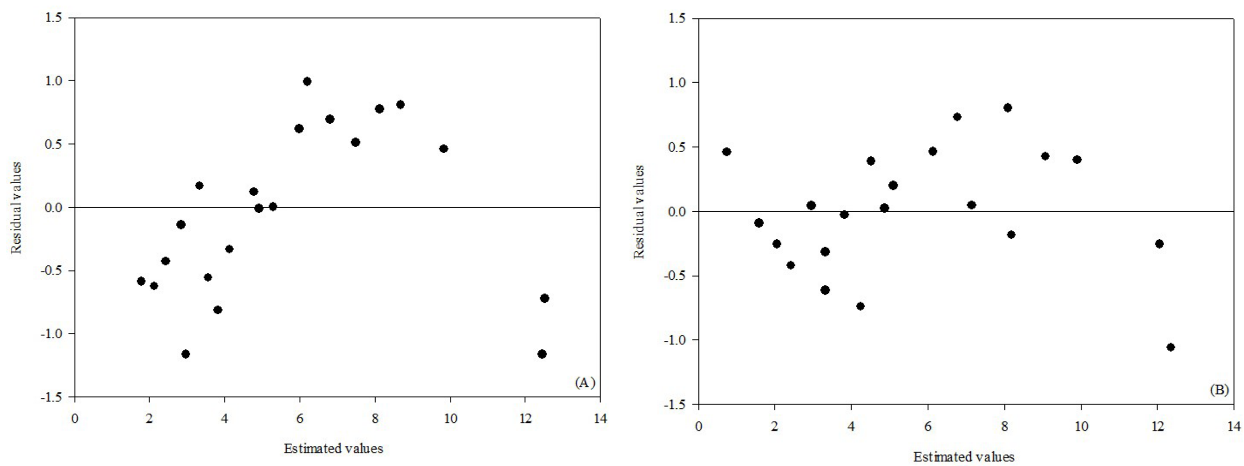

Figure 2: Residual plots of Modified Oswin (A) and Smith (B) models fit to the desorption data of flax seeds.

been used to satisfactorily represent the hygroscopicity of oilseeds, such as jatropha seeds (Ramos; Mancini; Mendes, 2014) and sunflower seeds (Campos et al., 2019).

Figure 4 presents the experimental results of the equilibrium moisture content of ryegrass and flax seeds obtained by desorption, along with the isotherms determined by the selected models.

Figure 4 confirms the temperature influence over the desorption isotherms of ryegrass and flax seeds. At a constant water activity, the equilibrium moisture content diminishes with increasing temperatures, a trend that is reported for most agricultural products (Goneli et al., 2010; Corrêa et al., 2016; Goneli et al., 2018; Botelho et al., 2019; Zeymer et al., 2019). According to McLaughlin and Magee (1998), this trend is explained by the molecules' excitation state. Under high temperatures, the attractive forces between molecules are less due to the increase in the kinetic energy of the water molecules, allowing a break in the connection of water and the sorption sites, which reduces the moisture content of the product.

Five types of isotherms have been described by Brunauer et al. (1940), based on the pores' ability to adsorb gases by mean forces, such as Van der Waals. According to Basu, Shivhare and Mujumdar (2006), the two most commonly found isotherms in food products are type II and III. Figure 5 presents an outline of these isotherm types.

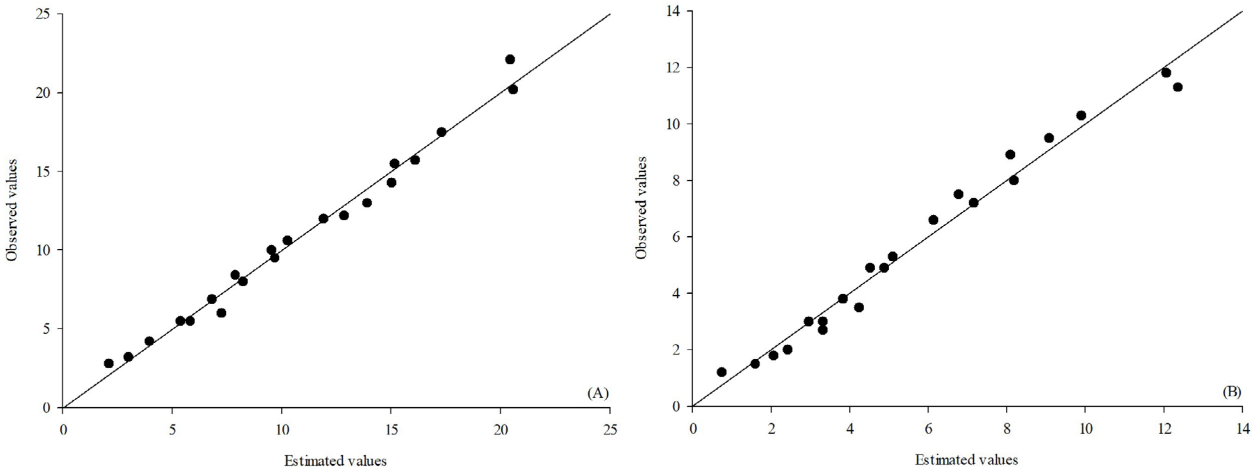

Figure 3: Chung Pfost model (A) and Smith model (B) adjusted to the experimental values for the desorption of ryegrass and flax seeds, respectively.

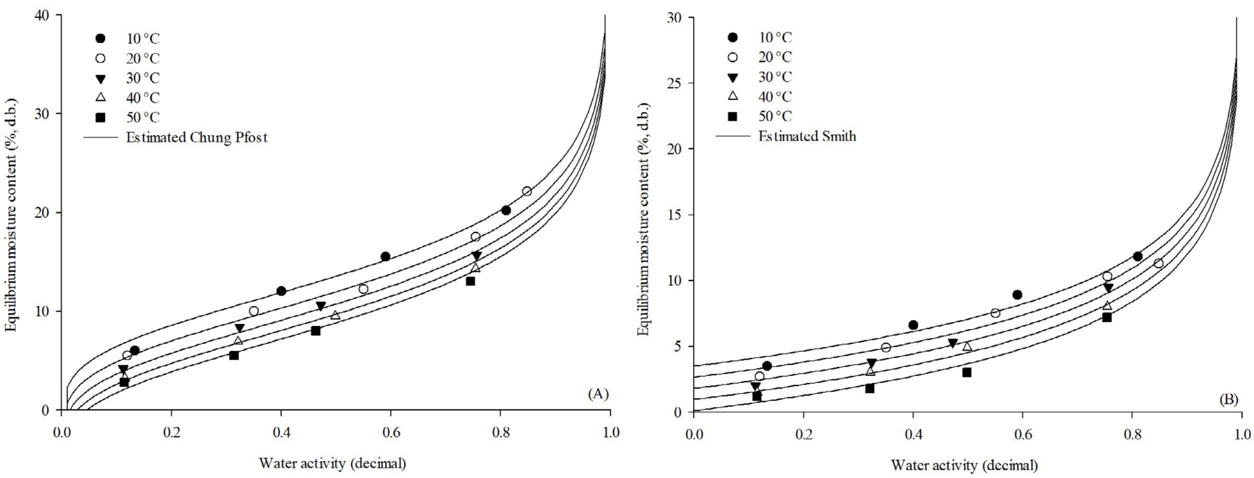

Figure 4: Observed and estimated values, based on the Chung Pfost model (A) and Smith model (B), of the equilibrium moisture content of ryegrass and flax seeds, respectively, obtained by desorption.

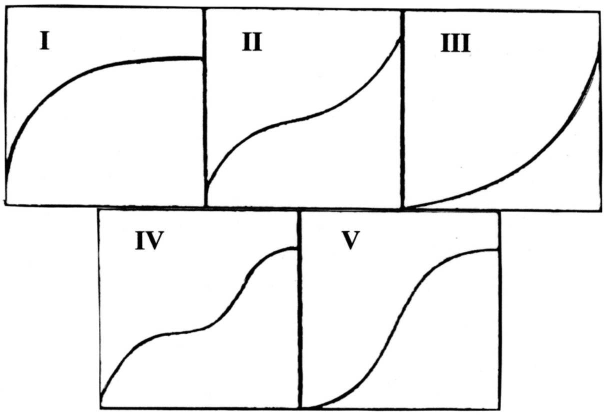

Figure 5: Brunauer classification of isotherm types. Source: Adapted from Brunauer et al. (1940).

Desorption isotherms of ryegrass seeds (Figure 4A) exhibited a sigmoidal shape, characteristic of a type II curve, among Brunauer's five forms of sorption isotherms (Figure 5). Labuza and Altunakar (2007) reported that the shape of type II isotherms is caused by synergistic effects of Raoult's law, in which capillary effects and moisture interactions occur at the material surface. Additionally, Torres and Seijo (2016) indicated that type II isotherms are typically achieved with aggregates of plate-like particles, for example, the material possess nonrigid slit-shaped pores. Several studies verified the same pattern in starchy products (Tran et al., 2015; Velázquez-Gutiérrez et al., 2015; Torres; Seijo, 2016; Gili et al., 2017), such as ryegrass seeds.

However, desorption isotherms of flax seeds (Figure 4B) exhibited characteristics of a type III curve (Figure 5). According to Labuza and Altunakar (2007), the type III isotherms, known as the Flory–Huggins isotherms, are characteristic of food products that are primarily composed of components that permit the passage of light, due to a regular tridimensional arrangement of their molecules. Moisture gain, in these cases, is as low as 0.8 of a_w. According to Costa, Resende and Oliveira (2013), products that present type III isotherms indicate less of an affinity for water molecules, such as flax seeds, which have a high oil content.

CONCLUSIONS

The equilibrium moisture content of flax and ryegrass seeds decreases as temperature increases at a constant value of water activity. Desorption isotherms of ryegrass seeds (Type II) and flax seeds (Type III) are different, according to Brunauer's classification, due to each products' composition (starch and oil content). Based on statistical parameters, the Chung Pfost model best fit the experimental data of ryegrass seeds, whereas the Smith model best fit that of flax seeds. This difference is also due to each products' composition (starch and oil content).

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