

Cinética da secagem e difusão efetiva das sementes de melancia

Drying kinetics and effective diffusion of watermelon seeds

Cinética de secado y difusión efectiva de semillas de sandía

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Resumo

As sementes de melancia (*Citrullus lanatus*) apresentam elevado teor de água, e necessitam passar pelo processo de secagem para assegurar o armazenamento e manter a viabilidade da

semente. Neste cenário, o estudo da cinética de secagem permite a descrição do processo por meio de modelos matemáticos e obtenção de informações, que auxiliam na compreensão do fenômeno em questão. Assim, objetivou-se ajustar estatisticamente oito modelos matemáticos, a fim de selecionar o que melhor representasse o comportamento da secagem da semente de melancia, determinar o coeficiente de difusão, a energia de ativação e a taxa de secagem das sementes em diferentes temperaturas. As sementes foram secas em um secador experimental nas temperaturas de 40; 45; 50; 55 e 60 °C, com velocidade do ar de 0.8 m s⁻¹, em quatro repetições. Para os ajustes dos modelos fez-se análise de regressão não-linear, pelo método de Gauss-Newton. Conclui-se que os modelos que melhor representam a cinética de secagem de sementes de melancia são Aproximação da difusão para as temperaturas de 40; 50 e 55 °C, e Page para 55 e 60 °C. As maiores taxas de secagem são observadas nas temperaturas mais elevadas. Os coeficientes de difusão apresentaram magnitude de 7.69684x10⁻¹⁰ e 1.27585x10⁻⁹ para o intervalo de 40 a 60 °C. E a energia de ativação foi de 12.2641 kJ mol⁻¹.

Palavras-chave: Coeficiente de difusão; Energia de ativação; Taxa de secagem, Teor de água; Temperatura.

Abstract

Studying the drying kinetics allows describing the process by mathematical models and obtaining information that helps to understand the phenomenon in question. Considering that watermelon (*Citrullus lanatus*) seeds have high moisture content and need to be subjected to drying, the objective was to select the model that best represents the drying behavior of watermelon seeds and determine the diffusion coefficient, activation energy and drying rate of these seeds at different temperatures. The seeds were dried in an experimental dryer at temperatures of 40, 45, 50, 55 and 60 °C, with air velocity of 0.8 m s⁻¹, in four replicates. The models were fitted with non-linear regression analysis using the Gauss-Newton method. It was concluded that the models that best represent the drying kinetics of watermelon seeds are Approximation of Diffusion for temperatures of 40, 50 and 55 °C and Page for 55 and 60 °C. The highest drying rates are observed at the highest temperatures. The diffusion coefficients presented magnitude of 7.69684×10⁻¹⁰ and 1.27585×10⁻⁹ for the range from 40 to 60 °C, and the activation energy was 12.2641 kJ mol⁻¹.

Keywords: Activation energy; Diffusion coefficient; Drying rate; Moisture content; Temperature.

Resumen

Las semillas de sandía (*Citrullus lanatus*) tienen un alto contenido de agua y deben pasar por el proceso de secado para garantizar el almacenamiento y mantener la viabilidad de las semillas. En este escenario, el estudio de la cinética de secado permite la descripción del proceso a través de modelos matemáticos y la obtención de información, que ayudan a comprender el fenómeno en cuestión. Por lo tanto, el objetivo era ajustar estadísticamente ocho modelos matemáticos, para seleccionar el que mejor representara el comportamiento de secado de la semilla de sandía, para determinar el coeficiente de difusión, la energía de activación y la velocidad de secado de las semillas a diferentes temperaturas. Las semillas se secaron en un secador experimental a temperaturas de 40 °C; 45; 50; 55 y 60 °C, con velocidad del aire de 0.8 m s^{-1} , en cuatro repeticiones. Para ajustar los modelos, se realizó un análisis de regresión no lineal utilizando el método de Gauss-Newton. Se concluye que los modelos que mejor representan la cinética de secado de las semillas de sandía son la aproximación de la difusión a temperaturas de 40 °C; 50 y 55 °C, y Page a 55 y 60 °C. Las tasas de secado más altas se observan a las temperaturas más altas. Los coeficientes de difusión tenían una magnitud de 7.69684×10^{-10} y 1.27585×10^{-9} para el rango de 40 a 60 °C. Y la energía de activación fue de $12.2641 \text{ kJ mol}^{-1}$.

Palabras clave: Coeficiente de difusión; Energía de activación; Velocidad de secado, Contenido de agua; Temperatura.

1. Introduction

Watermelon (*Citrullus lanatus*) is one of the most important vegetables produced and consumed in Brazil, since this species found excellent conditions for its development in the country (Torres, 2007). Although the production of seedless watermelon (*Citrullus lanatus* (Thumb.) Matsum & Nakai) is common in countries like the United States of America, it is still incipient in Brazil and other countries, but some small commercial areas have already been implemented (Granjeiro and Cecílio Filho, 2005).

Thus, scientific studies with watermelon seeds in country like Brazil are justified. The most suitable packages for storing watermelon seeds are those that are airtight, an important characteristic as it prevents the seeds from absorbing water during storage. This is because the moisture contents for storage of these seeds are low. Watermelon seeds should be stored with 6.5% moisture content (Harrington, 1972). Therefore, they must undergo a drying process before storage in order to remove excess water, thus ensuring greater stability and safety for

storage.

Among the various post-harvest steps, drying is the most relevant, from the perspectives of energy consumption and formation of processing costs, as well as preservation of quality (Oliveira et al., 2013).

Changes in drying parameters can promote different moisture reduction rates and, although factors such as air flow and relative humidity are important. The moisture reduction rate is mainly influenced by the drying air temperature (Burmester and Eggers, 2010). High moisture reduction rates can result in physical and structural damage, which compromises not only the chemical composition, but mainly the physiological potential of the seed (Ullmann et al., 2010).

Given the importance of the drying process, this step should be predicted, designed and planned in advance. Thus, the use of some techniques, such as the fitting of mathematical models, may bring some contributions (Isquierdo et al., 2013).

The fitting of mathematical models for the drying of agricultural products provides information of fundamental importance for the development of processes and designing of equipment (Siqueira et al., 2013). With this information, it is possible to estimate the drying time and, consequently, the energy expenditure that will determine the cost of the process.

Thin-layer drying kinetics varies with species, variety, environmental conditions and methods of post-harvest preparation, among other factors (Alexandre et al., 2013). Thus, several studies are carried out in order to obtain useful information for understanding the process, such as the effective diffusion coefficient, activation energy and the description of water loss, through the fitting of mathematical models.

The drying kinetics technique has been used in leaves of medicinal plants, such as *Serjania marginata* (Martins et al., 2015), *Bauhinia forficata* (Silva et al., 2017), grains produced on a large scale, “Carioca” beans (Melo et al., 2016), soybean (Botelho et al., 2018) and grains and seeds with characteristics similar to those of watermelon, such as pumpkin grains (Diógenes et al., 2013) and melon seeds (Silva et al., 2018).

The present study aimed to fit mathematical models, represent the drying behavior of watermelon seeds, and determine the effective diffusion coefficient, activation energy and drying rate of these seeds at different drying air temperatures.

2. Methodology

The watermelons were purchased in the market of the city of Dourados, MS, and

subsequently taken to the Laboratory of Post-Harvest Processes of the Federal University of Grande Dourados (UFGD), where their seeds were manually extracted, washed in running water, selected according to size and color, dried with paper towels, placed in plastic bags and stored for 48 h in cold chamber (5 °C), until the drying process was conducted.

After the homogenization period, the moisture contents of the seeds were determined using the standard oven method (Brasil, 2009). After moisture content determination, the seeds were subjected to drying.

The drying process was conducted in an experimental horizontal fixed-bed dryer. The dryer is equipped with a system that accurately controls the drying air flow and temperature, with a sensor connected to the control panel, to obtain a fine adjustment and monitor the drying air temperature. In addition to the data obtained with the sensor, the temperature was monitored from time to time using a mercury thermometer. Air velocity was controlled and kept constant at 0.8 m s^{-1} , using a digital thermo anemometer (Instrutherm brand, AM-100 model).

The mass of seeds (52 g) was distributed in a single layer on four perforated-bottom trays and subjected to drying at the temperatures of: 40, 45, 50, 55 and 60 °C. The drying process was interrupted when the seeds came into equilibrium. Equilibrium was considered as the lack of mass variation in three consecutive weighing procedures. A moisture content of $6.5 \pm 0.1\%$ (wet basis) was considered in the mathematical modelling of the drying of watermelon seeds.

The mass of seeds along the drying process was measured on semi-analytical scales, with resolution of 0.01 g. After reaching constant mass, the equilibrium moisture content was determined by following the recommendations of the Rules for Seed Analysis (Brasil, 2009).

To evaluate the drying of watermelon seeds, the drying rate of the product was determined according to the following expression (Equation 1).

$$DR = \frac{Mw_0 - Mw_i}{DM (t_i - t_0)} \quad (1)$$

Where DR is the drying rate ($\text{kg kg}^{-1} \text{ h}^{-1}$), Ma_0 is the previous total water mass (kg), Ma_i is the current total water mass (kg), DM is dry matter (kg), t_0 is the total drying time (h) and t_i is the current total drying time (h).

The initial and final moisture contents and those obtained during the drying process

(calculated) were used to determine the moisture ratio (RX) of watermelon seeds along the drying by the following expression (Equation 2).

$$RX = \frac{X - X_e}{X_i - X_e} \quad (2)$$

Where RX is the moisture ratio of the product (dimensionless), X is the moisture content of the product (decimal, d.b.), X_e is the equilibrium moisture content of the product (decimal, d.b.) and X_i is the initial moisture content of the product (decimal, d.b.).

Eight mathematical models used to represent the drying of agricultural products were fitted to the experimental data of watermelon seed drying (Table 1).

Table 1 - Mathematical models used to predict the drying of agricultural products

Model	Model Designation
Approximation of Diffusion	$RX = a \exp(-k t) + (1-a)\exp(-k b t)$ (3)
Henderson and Pabis	$RX = a \exp(-k t)$ (4)
Midilli	$RX = a \exp(-k t^n) + b t$ (5)
Logarithmic	$RX = a \exp(-k t) + c$ (6)
Newton	$RX = \exp(-k t)$ (7)
Page	$RX = \exp(-k t^n)$ (8)
Thompson	$RX = \exp\left(\frac{-a - (a^2 + 4 b t)^{0.5}}{2 b}\right)$ (9)
Valcam	$RX = a + b t + c t^{1.5} + d t^2$ (10)

Where t is drying time (h), k, k_0 , k_1 are the drying parameters (h^{-1}) and a, b, c, d and n are the coefficients of the models.

The mathematical models were fitted by nonlinear regression analysis using the Gauss-Newton method. The magnitudes of the standard deviation of the estimate (SE), mean relative error (P) and coefficient of determination (R^2) were considered. The P and SE values for each model were calculated by Equations 11 and 12.

$$P = \frac{100}{n} \sum \left| \frac{Y - \hat{Y}}{Y} \right| \quad (11)$$

$$SE = \sqrt{\frac{\sum (Y - \hat{Y})^2}{DF}} \quad (12)$$

Where Y is the value observed experimentally, \hat{Y} is the value calculated by the model, n is the number of experimental observations and DF is the degrees of freedom of the model (difference between the number of observations and number of model parameters).

To assist in the decision making and selection of the model to describe the drying of watermelon seeds under each condition, the models that obtained the best fits were subjected to the Akaike Information Criterion (AIC) and Schwarz's Bayesian Information Criterion (BIC). Lower values of AIC and BIC indicate better fit of the model, the latter being the most rigorous criterion (Wolfinger, 1993). According to Gomes et al. (2018), these criteria can also be included for selecting drying models. These information criteria were determined by the Equations 13 and 14.

$$AIC = -2 \log L + 2 p \quad (13)$$

$$BIC = -2 \log L + p \ln (N - r) \quad (14)$$

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

The effective diffusion coefficient of the seeds, for the different drying conditions, was calculated using Equation 15, based on the liquid diffusion theory. This equation is the analytical solution for the Fick's second law, considering the geometric form as a flat slab of the product and eight-term approximation:

$$RX = \frac{X - X_e}{X_i - X_e} = \frac{6}{\pi^2} \sum_{n_i=1}^{\infty} \frac{1}{n_i^2} \exp \left[\frac{n_i^2 \pi^2 D_i t}{9} \left(\frac{3}{L} \right)^2 \right] \quad (15)$$

Where D_i is the effective diffusion coefficient ($m^2 s^{-1}$), L is the thickness of the product (m), t is the drying time (s) and n is the number of terms in the model.

A digital caliper with resolution of 0.01 mm was used to measure the thickness, and the average thickness was obtained using 50 randomly chosen seeds.

The Arrhenius equation was used to evaluate the influence of the drying temperature on the effective diffusion coefficient, according to Equation (16).

$$D_{ef} = D_0 \exp\left(\frac{-Ea}{R T_a}\right) \quad (16)$$

Where D_0 is the pre-exponential factor ($m^2 s^{-1}$), Ea is the activation energy ($kJ mol^{-1}$), R is the universal gas constant, $8.314 kJ mol^{-1} K^{-1}$, and T_a is the absolute temperature (K).

3. Results and discussion

Seeds subjected to drying at higher temperatures had higher average moisture reduction rates (Figure 1), but this behavior can only be observed in the beginning of the process because, after approximately 1 h of drying, the experimental values begin to overlap. This occurs because the difference in the partial water vapor pressure between the grains and the drying air is higher at higher temperatures. In addition, as the drying process progresses, the water becomes more strongly bound and hence more difficult to be removed. Similar results were found by several researchers, working with the drying of different agricultural products (Keneni et al., 2019; Botelho et al., 2015; Rosa et al., 2015; Siqueira et al., 2012).

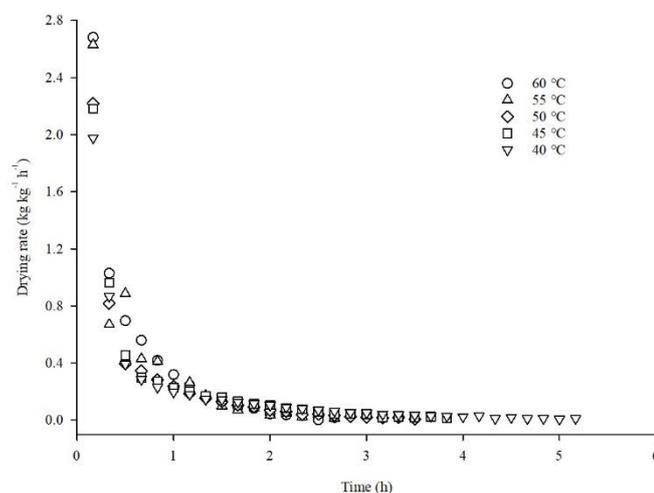


Figure 1 - Drying rate at different drying air temperatures for watermelon (*Citrullus lanatus*) seeds

The drying times for the watermelon seeds to reach the moisture content of $6.5 \pm 0.1\%$ were 5.17, 3.83, 3.50, 3.00 and 2.67 h for the respective temperatures of 40, 45, 50, 55 and 60 °C. Therefore, the increase in drying air temperature reduces the time required for the product to reach the desired moisture content, which is due to the higher rate of water removal, as observed in Figure 1.

Table 2 presents the values of coefficient of determination (R^2), mean relative error (P) and mean estimated error (SE).

Table 2 - Statistical parameters obtained for the eight models used in the thin-layer drying of watermelon (*Citrullus lanatus*) seeds

Models	Temperature														
	60 °C			55 °C			50 °C			45 °C			40 °C		
	R^2	SE	P	R^2	SE	P	R^2	SE	P	R^2	SE	P	R^2	SE	P
3	0.997	0.005	10.2	0.997	0.015	18.4	0.999	0.005	8.9	0.999	0.004	2.9	0.999	0.002	1.5
4	0.986	0.032	30.8	0.977	0.039	37.5	0.960	0.048	38.9	0.966	0.045	30.4	0.944	0.051	37.2
5	0.905	0.090	95.9	0.893	0.090	77.5	0.906	0.078	69.5	0.999	0.008	7.4	0.999	0.007	8.9
6	0.991	0.027	18.2	0.989	0.028	14.7	0.977	0.038	25.9	0.977	0.038	28.0	0.96	0.044	38.9
7	0.981	0.036	36.8	0.97	0.044	44.3	0.943	0.056	49.8	0.947	0.055	43.6	0.904	0.066	56.5
8	0.999	0.008	6.1	0.998	0.012	8.20	0.999	0.008	5.8	0.998	0.011	12.6	0.997	0.013	18.4
9	0.997	0.013	11.7	0.997	0.015	5.90	0.997	0.013	12.9	0.995	0.017	20.9	0.994	0.017	26.7
10	0.992	0.025	19.3	0.99	0.028	16.0	0.979	0.037	23.6	0.982	0.034	24.0	0.964	0.042	32.2

The mean relative error (P) describes the deviation of the observed values in relation to the curve estimated by the model. P values should be lower than 10% (Kashaninejad et al., 2007). Thus, the models that meet this statistical requirement in one or more situations are: Approximation of Diffusion (3), Midilli (5), Page (8) and Thompson (10). Among these, it can be noted that the Approximation of Diffusion model is adequate for the temperatures of 40, 45 and 50 °C, and that the Page model has values lower than 10% for the temperatures of 50, 55 and 60 °C. Midilli and Thompson models are suitable only for one temperature.

The parameters standard deviation of the estimate (SE) and coefficient of determination (R^2) help in selecting the best model to represent the drying kinetics of watermelon seeds. Lower values of SE indicate better fit of the mathematical model (Siqueira

et al., 2012). Thus, in general, the Approximation of Diffusion model has lower values for lower temperatures, and the Page model has lower values for higher temperatures.

Among the eight models analyzed, those with coefficients of determination (R^2) higher than 0.99 were Approximation of Diffusion, Page and Thompson. R^2 values higher than 0.95 indicate good representation of the drying kinetics (Aguerre et al., 1989).

Table 3 shows the values referring to the Akaike Information Criterion (AIC) and the Schwarz's Bayesian Information Criterion (BIC) for selecting the models that fitted best to the drying of watermelon (*Citrullus lanatus*) seeds at the different temperatures. It is observed that the Approximation of Diffusion model fits better to the experimental values of moisture ratio at the temperatures of 40, 45 and 50 °C, while the Page model fits better to those at temperatures of 55 and 60 °C.

The AIC and BIC criteria reinforce the conclusion obtained by the Gauss-Newton method, which indicated that the Page model was adequate for higher temperatures and Approximation of Diffusion was adequate for lower temperatures. AIC and BIC can be used as an additional criterion to select drying kinetics models (Gomes et al., 2018), as they represent a very thorough analysis (Wolfinger, 1993). AIC and BIC was used before to select the best-fitted model for the drying kinetics of sweet potato pulp after pre-selection with the criteria based on the deviation of the estimate, standard mean error and coefficient of determination (Souza et al., 2019).

Table 3 - Akaike Information Criterion (AIC) and Schwarz's Bayesian Information Criterion (BIC) for the models that best fit the drying data of watermelon (*Citrullus lanatus*) seeds for different temperatures

Models	40 °C		45 °C		50 °C		55 °C		60 °C	
	BIC	AIC								
3	-317.60	-323.47	-184.65	-189.36	-158.24	-162.60	-96.89	-100.67	-111.21	-113.71
4	-91.17	-95.56	-73.72	-77.26	-63.98	-67.25	-62.37	-65.21	-62.34	-64.84
5	-210.22	-217.55	-150.05	-155.94	-147.48	-152.94	-102.64	-107.36	-107.07	-111.24
6	-98.25	-104.11	-79.96	-84.67	-72.96	-77.33	-73.40	-77.18	-68.01	-71.35
7	-77.38	-80.31	-66.16	-68.52	-59.15	-61.34	-60.15	-62.04	-60.44	-62.11
8	-181.01	-185.17	-139.12	-142.66	-145.30	-148.58	-107.42	-110.26	-121.25	-124.59
9	-112.78	-117.17	-99.48	-103.02	-94.21	-97.49	-96.68	-99.52	-90.05	-92.55
10	-98.60	-105.93	-82.73	-88.62	-71.84	-77.30	-71.75	-76.47	-68.01	-72.18

The Page model fitted best for the temperature range from 60 to 80 °C in the drying of pumpkin (*Cucurbita spp.*) seeds (Jittanit, 2011) and Azuki bean for the temperature range from 40 to 80 °C (Almeida et al., 2019). Better fit of the Approximation of Diffusion model within the temperature range from 40 to 80 °C was verified for pumpkin (*Cucurbita moschata* Duchesne) seeds (Diógenes et al., 2013). Considering that the drying kinetics of watermelon seeds was accurately represented only by the Page model for temperatures of 55 and 60 °C and by the Approximation of Diffusion model for the temperatures of 40, 45 and 50 °C, combining the two criteria, these two models were chosen to represent the drying phenomenon: Page model for high temperatures (55 and 60 °C) and Approximation of Diffusion for low temperatures (40, 45 and 50 °C). The latter has been recommended by other authors who have worked with the drying of seeds with similar characteristics.

Figure 2 presents the values of moisture ratio estimated by the Page and Approximation of Diffusion models, as a function of the drying time for the five temperatures. The models show good fit and accuracy in describing its behavior, demonstrating that temperature and drying time are inversely proportional, i.e., the higher the temperature, the shorter the drying time, a result similar to that was observed before (Siqueira et al., 2012).

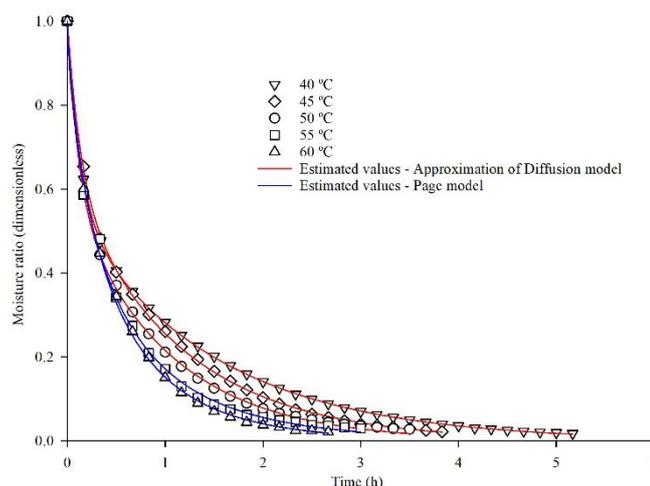


Figure 2 - Moisture ratios estimated by Page and Approximation of Diffusion models for the drying of watermelon (*Citrullus lanatus*) seeds at different temperatures

The result in the Table 4 shows the parameters of the Page model for the temperatures of 55 and 60 °C, and the parameters of the Approximation of Diffusion model for the temperatures of 40, 45 and 50 °C.

Table 4 - Coefficients of Page and Approximation of Diffusion models obtained for the drying of watermelon (*Citrullus lanatus*) seeds at different temperatures

Temperature (°C)	Mathematical models				
	Page		Approximation of Diffusion		
	k	n	a	k	b
60	1.885982	0.763332	-	-	-
55	1.762182	0.712697	-	-	-
50	-	-	0.59314	1.01244	8.77618
45	-	-	0.62005	0.88751	8.279110
40	-	-	0.55921	0.68786	11.23147

It can be observed that the “k” coefficients of the models increase with the elevation of temperature. This parameter is an estimate that represents the effects of temperature and is correlated with the effective diffusivity during the drying process in the falling rate period and with the liquid diffusion controlling the process (Madamba et al., 1996). For the parameter “n”, the same behavior was observed, representing the internal resistance of the product to drying (Corrêa et al., 2007), as the parameters “a” and “b” (Brooker et al., 1992).

The values of effective diffusion coefficients for the temperature range studied are shown in Figure 3.

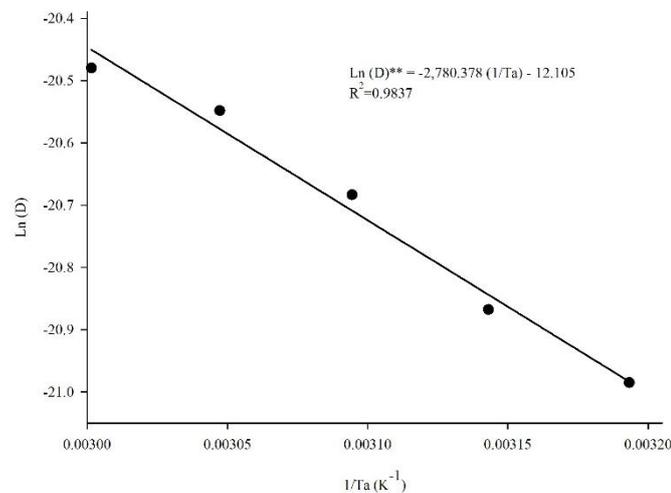


Figure 3 - Effective diffusion coefficient for the temperatures of 40, 45, 50, 55 and 60 °C.

**Significant at 0.01 by t-test

The effective diffusion coefficients ranged between 7.69684×10^{-10} and 1.27585×10^{-09} for the temperature range between 40 and 60 °C. It can be noted that the diffusion coefficient increases with increasing temperature. This occurs due to the reduction in water viscosity when the temperature increases, and variation in this property promotes the diffusion of water through the capillaries of the grain, allowing this fluid to move within the product (Goneli et al., 2009). The effective diffusion coefficients varied between 1.553×10^{-10} and $2.091 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ for the range from 35 to 50 °C in melon seeds (Silva et al., 2018). In pumpkin (*Cucurbita spp.*) seeds, the effective diffusion coefficient varied between 37.62×10^{-11} and $50.96 \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ for the range from 60 to 80 °C (Jittanit, 2011).

The activation energy to trigger the drying process for watermelon seeds was $12.26 \text{ kJ mol}^{-1}$. This value is close to the range of activation energy for agricultural products, varies from 12.7 kJ mol^{-1} to 110 kJ mol^{-1} (Zogzas et al., 1996). This value is very low compared to the one found for melon seeds, which was $55.81 \text{ kJ mol}^{-1}$ (Silva et al., 2018), and to the value of $62.12 \text{ kJ mol}^{-1}$ for pumpkin seeds (*Cucurbita spp.*) (Jittanit, 2011). This is probably due to the form, initial moisture content, chemical composition and structure of the product, besides the imposed drying conditions.

4. Conclusions

The Approximation of Diffusion model is adequate for representing the drying of watermelon (*Citrullus lanatus*) seeds at temperatures of 40, 45 and 50 °C. For higher temperatures (50, 55 and 60 °C), the Page model was the most adequate to represent the phenomenon. The effective diffusion coefficient varied between 7.69684×10^{-10} and 1.27585×10^{-09} for the temperature range between 40 and 60 °C, increasing as the temperature increased. The activation energy to trigger the drying process was $12.26 \text{ kJ mol}^{-1}$.

In future works it is recommended that it be complemented with the evaluation of the physiological quality of the seed, reconciling the mathematical models selected to this parameter. The use of the AIC and BIC criteria in the decision taking of mathematical models of drying kinetics is highly recommended as it reinforces or dispels any doubts that may result from the statistical parameters commonly used in the model selection criteria.

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