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Sorption isotherms and drying kinetics of grapefruit seeds

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ABSTRACT. The drying of grapefruit seeds, by-products from grapefruit processing, was studied at 40, 50, 60 and 70°C and at three air velocities, 0.6, 1.0 and 1.4 m s^{-1}. Sorption isotherms of grapefruit seeds were obtained at each temperature by the static method using saturated salt solutions. The Henderson model adequately described the sorption isotherms, over the entire temperature range. Drying rates indicated that the drying of grapefruit seeds took place under the falling rate period. Effective moisture diffusivity in grapefruit seeds ranged from 4.36×10^{-10} to 6.82×10^{-10} m^2 s^{-1}. The temperature dependence of the effective diffusivity followed an Arrhenius relationship, and the activation energies were 12.23, 11.29 and 11.79 kJ mol^{-1} for dried grapefruit seeds under air velocities of 0.6, 1.0 and 1.4 m s^{-1}, respectively. Three thin-layer models were used to predict the drying curves, Page, Lewis, and Henderson-Pabis model. The Page model presented the best fit for all drying air temperatures and velocities studied.

Keywords: grapefruit seeds, drying, moisture content, sorption isotherm.

Isotermas de sorção e cinética de secagem de sementes de pomelo

RESUMO. A secagem das sementes de pomelo, subproduto do processamento da fruta, foi estudada a 40, 50, 60 e 70°C e a três velocidades de ar, 0,6; 1,0 e 1,4 m s^{-1}. As isotermas de sorção das sementes foram obtidas, a cada temperatura, pelo método estático usando soluções salinas saturadas. O modelo de Henderson descreveu adequadamente as isotermas de sorção no intervalo de temperatura estudado. As taxas de secagem indicaram que a secagem das sementes de pomelo ocorre no período de taxa decrescente. A difusividade efetiva da umidade variou entre $4{,}36 \times 10^{-10}$ e $6{,}82 \times 10^{-10}$ m^2 s^{-1}. A influência da temperatura na difusividade efetiva seguiu uma relação de Arrhenius e a energia de ativação foi de 12,23; 11,29 e 11,79 kJ mol^{-1} para as velocidades de 0,6; 1,0 e 1,4 m s^{-1}, respectivamente. Três modelos empíricos foram usados para predizer as curvas de secagem – Page, Lewis e Henderson-Pabis. O modelo de Page apresentou melhor ajuste para todas as condições estudadas.

Palavas-chave: sementes de pomelo, secagem, umidade, isotermas de sorção.

Introduction

The grapefruit (*Citrus paradisi* Macf.) is one of the most popular citrus fruits in the United States and Mexico. In particular, Mexico has increased its world share of grapefruit production from 2.7% in 1990 to 8.0% in 2008, becoming the second largest grapefruit producer following the U.S. (FAO, 2010). Grapefruit is primarily eaten fresh, and it is commonly used in fruit cups or fruit salads. It is also easily processed into juice, syrup, jelly, and vinegar. The juice is marketed as a beverage in a variety of ways, including fresh, canned, dehydrated or concentrated, and frozen (SALUNKHE; KADAM, 1995).

The industrial processing of grapefruit generates a large amount of wastes that can range between 49 and 69% of the initial weight. These wastes include peel, segment membranes, seeds, and other by-products. Although a portion of citrus waste can be used for pectin extraction or pelletized for animal feed (MAMMA et al., 2008), a large fraction is disposed in landfills every year.

This landfilling is not favored due to both economic concerns such as high transportation costs and lack of disposal sites and the environmental concern that the wastes have a high organic content (TRIPODO et al., 2004).

Some studies have reported the use of citrus waste for production of biofuels, limonene and pectin (POURBAFRANI et al., 2010), extraction of functional fibers (MARÍN et al., 2007), seed oil (ANWAR et al., 2008; WAHEED et al., 2009) and energy production by thermal degradation of seeds (HERNÁNDEZ-MONTOYA et al., 2009).

Crude oil makes up more than 30% of grapefruit seeds. The oil has a reddish-brown color, a pleasant nutlike aroma and an intensely bitter taste (NOULTE; VON LOESECKE, 1940). According to Anwar et al. (2008), the oil from the grapefruit seeds has a good potential for use in edible products and in various industrial applications. The oil consists mainly of linoleic and palmitic acids which makes up approximately 36 and 32% of the oil, respectively. The total unsaturated fatty acid content is approximately 64% and the percentage of essential fatty acids (linoleic and linolenic), which have potential health benefits, is about 40.46%. The oil is also an important source of tocopherol, especially α-tocopherol, which exists in the oil at a concentration of about 380 mg kg^{-1}.

It is possible that grapefruit seeds, which are generally discarded as an agro-industrial waste, could emerge as a valuable commodity for the production of useful oil and other value-added products. However, for that to happen, it is necessary to reduce the moisture content of the seeds in order to increase the storage life of these seeds. Thus, drying the seeds is essential for both stabilizing the seeds to prevent microbial and chemical degradation, and making the seeds in a form suitable for oil extraction (ROBERTS et al., 2008).

High temperatures and long drying periods of seeds may have deleterious effects on the quality of the oil obtained from the seeds. Careful control of moisture is critical for quality assurance of dry foods during storage. Mathematical modeling and simulation of drying curves under different conditions are important to obtain a better control of this unit operation. Models are often used to study the variables involved in the process, by predicting the drying kinetics of the product, and optimizing the operating parameters and conditions (KARATHANOS; BELESSIOTIS, 1999).

The main objectives of this research were to identify the best model that describes the water sorption isotherms and to determine the effect of drying air temperature on the drying kinetics of grapefruit seeds, aiming to facilitate the production of oil from by-product of grapefruit seeds.

Mathematical development

Six sorption isotherm models (Table 1) were tested to determine how well they fit dried grapefruit seeds sorption isotherm data.

The goodness of fit for each model can be evaluated based on the relative percent error (PE) (Equation 7), which compares the absolute difference between the predicted moisture contents with the experimental moisture contents. Relative percent errors values lower than 10 % indicate a good fit (McLAUGHLIN; MAGEE, 1998).

Most existing models for the study of drying kinetics are based on the Fick's second law. Crank (1975) represented several analytical solutions, based on the assumptions of uniform initial moisture distribution, negligible external resistance, temperature gradients and shrinkage during drying, besides constant diffusion coefficient.

The analytical solution of the diffusion equations for a sphere was chosen to calculate the effective moisture diffusivity of grapefruit seeds, according to the Equation (8). This analytical solution was selected based on a preliminary analysis of the comparison between the geometries of infinite cylinder, sphere and infinite plate. The sphere geometry resulted in the best fit for the experimental data.

Table 1. Sorption isotherm models.

Model	Equation
GAB (LABUZA et al., 1985b)	$M_{eq}=X_m\left[\frac{(C-1)Ka_w}{(1+(C-1)Ka_w)}+\frac{Ka_w}{(1-Ka_w)}\right]$ (1)
Hailwood-Horrobin (HAILWOOD; HORROBIN, 1946)	$M_{eq}=a_w/\left(A+Ba_w+Ca_w^2\right)$ (2)
Henderson (HENDERSON, 1952)	$M_{eq}=a_w/\left(A+Ba_w+Ca_w^2\right)$ (3)
Peleg (PELEG, 1993)	$M_{eq}=k_1a_w^{n_1}+k_2a_w^{n_2}$ (4)
Oswin (OSWIN, 1946)	$M_{eq}=A\left[a_w/\left(1-a_w\right)\right]^B$ (5)
Chung-Pfost (CHUNG; PFOST, 1967)	$M_{eq}=A+B\left(ln\,a_w\right)$ (6)

where:

$A, B, C, D, K, k_1, k_2, n_1$, and n_2 are constants;

X_m is the moisture content (dry basis) corresponding to a monolayer;

a_w water activity (relative humidity of salt solutions);

M_{eq} equilibrium moisture content (dry basis).

$$PE(\%)=\frac{100}{n}\sum_{i=1}^{n}\frac{\left|M_{exp,i}-M_{predict,i}\right|}{M_{exp,i}} \quad (7)$$

$$MR=\frac{M_t-M_{eq}}{M_0-M_{eq}}=6\sum_{n=1}\frac{1}{n^2\pi^2}exp\left(-n^2\pi^2\frac{D_{eff}}{r^2}t\right) \quad (8)$$

where:

MR represents the unaccomplished moisture content or moisture ratio;

M_t is the moisture at any time *t* during drying;

M_0 the initial moisture content;

M_{eq} the equilibrium moisture content;

r is the sphere radius;

D_{eff} is the effective moisture diffusivity representing the conductive term of all moisture transfer mechanisms.

The temperature dependence of the effective diffusivity has been shown to follow an Arrhenius relationship (RIZVI, 2005):

$$D_{eff} = D_0 \, exp\left(-E_a / RT\right) \quad (9)$$

where:

D_0 is the pre-exponential factor of the Arrhenius equation;

E_a is the activation energy;

R is the universal gas constant;

T is the absolute air temperature.

Simplified drying models have been used to quantify the drying kinetics of various grains, some seeds (CORRÊA et al., 1999; ROBERTS et al., 2008; SACILIK, 2007) and some fruit (KALETA; GÓRNICKI, 2010; SANTOS et al., 2010). Three empirical models were used to quantify the drying kinetics of grapefruit seeds, Page model (Equation 10), Lewis model (Equation 11) and the Henderson-Pabis model (Equation 12).

$$MR = \frac{M_t - M_{eq}}{M_0 - M_{eq}} = \exp\left(-kt^N\right) \quad (10)$$

$$MR = \frac{M_t - M_{eq}}{M_0 - M_{eq}} = \exp\left(-kt\right) \quad (11)$$

$$MR = \frac{M_t - M_{eq}}{M_0 - M_{eq}} = a\exp\left(-kt\right) \quad (12)$$

where:

k is the drying rate constant;

N and *a* are constants.

The goodness of fit for each model can be evaluated based on the root mean square error (RMSE). The predicted moisture ratio was compared with the experimental moisture ratio using the root mean square error as shown in the following equations (McMINN, 2006). As RMSE approaches to zero, the closer the prediction is to the experimental data.

$$RMSE = \left[\frac{1}{n}\sum_{i=1}^{n}\left(MR_{exp,i} - MR_{predict,i}\right)\right]^{\frac{1}{2}} \quad (13)$$

Material and methods

Raw material

Grapefruit seeds were obtained from grapefruits (*Citrus paradisi* Macf.), variety Red. Grapefruit is a hybrid of lemon and orange. The red grapefruit were produced by a mutation of the white variety. The red colour is due to its antioxidant content, which is higher than the white variety. The seeds, representing part of waste material from grapefruit juice processing, were manually separated from the waste using an 8 mm screen. After separation, the seeds were lightly washed with distilled water to remove the remaining pomace.

Sorption procedure

The grapefruit seeds were dried until constant weight before being submitted to equilibrium moisture content experiments. Sorption isotherms at several water activities were determined by the static gravimetric method (LOPES-FILHO et al., 2002) at 40, 50, 60, and 70ºC. Three samples of grapefruit seeds of 1-2 g were weighed in small receptacles and placed into desiccators with eleven saturated salt solutions (NaOH, LiCl, $KC_2H_3O_2$, $MgCl_2$, K_2CO_3, $Mg(NO_3)_2$, NaNO2, NaCl, KCl, BaCl2, $CuSO_4$) corresponding to a range of water activities from 0.02 to 0.97, which were placed in a temperature-controlled chamber. Water activities at each salt solutions and temperature were obtained from Labuza et al. (1985a) and Young (1967). In order to prevent mould growth, a formaldehyde drop was added to each jar whose water activity was greater than 0.75. The required equilibration time was 4 to 5 weeks, based on the change in weights of samples expressed on a dry basis, which did not exceed 0.1 % (0.001 g g^{-1} d.b). The equilibrium moisture content was determined in a vacuum oven, at 60°C for 48h (AOAC, 1990).

Drying kinetics

The drying equipment (Figure 1) was a pilot scale tray dryer that consisted of three sections i.e., an airflow rate control system, a drying air heating section, and a drying chamber. The drier was equipped with a process control system based on Fieldbus technology, that was supplied by SMAR Industrial Equipment Ltd. (Sertãozinho, São Paulo State, Brazil). The dryer was previously described elsewhere (NICOLETI et al., 2001).

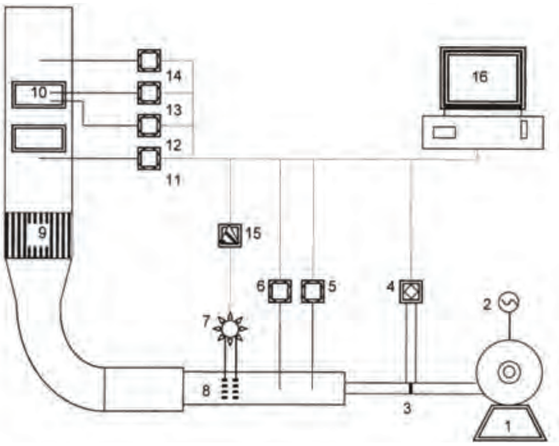

Figure 1. Schematic diagram of the drying equipment. (1) Centrifugal fan. (2) Frequency modulator. (3) Orifice plate. (4) Pressure transmitter. (5,6) Dry and wet bulb temperature transmitters. (7) Power converter. (8) Electric resistances. (9) Honeycomb. (10) Drying compartment. (11,14) Air temperature transmitters. (12) Inner product temperature transmitter. (13) Surface product temperature transmitter. (15) 4 to 20 mA current converter. (16) Computer.

In order to evaluate the effect of air temperature on the drying process, four temperatures (40, 50, 60 and 70°C) were used. These temperatures were chosen carefully to avoid thermal damages to the oil in grapefruit seeds. Drying was stopped when the weight of the test sample reached a constant value. The effect of air velocity on the drying (0.6, 1.0 and 1.4 m s^{-1}) was also tested. Moisture contents at each time interval was calculated from both weight loss data and dry solid weight of the sample, which was determined at the end of drying by the vacuum oven-drying method at 60°C for 48h (AOAC, 1990).

Results and discussion

Equilibrium moisture content

Equilibrium moisture contents versus water activity for grapefruit seeds at different temperatures are shown in the Figure 2. The moisture at each water activity represents the mean value of three replications. It is important to note that the reached equilibrium moisture content of the grapefruit seeds was lower when compared with other products, such as fruit or vegetable.

The six mathematical models presented in the Table 1 were used to fit the experimental data of grapefruit seeds. The statistical results shown in the Table 2 indicated that Henderson model presented the best adjustment under the given conditions. The determination coefficient (R^2) was very close to the unit and the PE was under 5%, indicating that this model is effective to describe the water sorption isotherm for grapefruit seeds under the given conditions. Sogi et al. (2003) analyzed the water sorption isotherm for tomato seeds and also obtained this model to fit their experimental data. It was not found an intersection of the isotherms ('crossing-over') at high water activity, suggesting a low fraction of monosaccharides in this product.

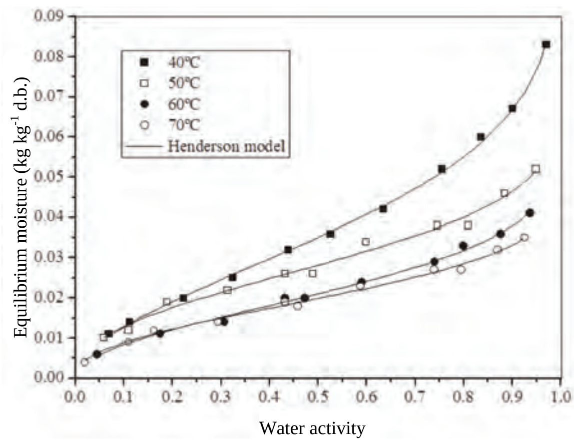

Figure 2. Sorption isotherm of grapefruit seeds at different temperatures.

Table 2. Estimated parameters of Henderson model for grapefruit seeds at different temperatures.

T (°C)	Parameters of Henderson model			
	A	*B*	PE(%)	R^2
40	356.814	1.864	2.049	0.998
50	3620.496	2.399	4.453	0.988
60	1816.316	2.040	3.106	0.996
70	6476.921	2.334	4.280	0.993

Drying kinetics

According to Roberts et al. (2008), dried seeds with moisture content below 0.10 kg kg^{-1} (d.b.) are required for a cold press operation. The time required to dry grapefruit seeds from an initial moisture content of around 1.022 kg kg^{-1} (d.b.) to the final moisture content equal to or below 0.01 kg kg^{-1} (d.b.) was 3.6, 3.2, 2.8 and 2.6 hours at 40, 50, 60 and 70°C of drying air temperature respectively. These times are equal to the three air velocities. Notice in Figure 2 that the 0.01 kg kg^{-1} (d.b.) corresponds to a very low water activity, whose values are below 0.2 to four temperatures. This water activity prevents the microbial action but becomes the seeds susceptible to lipid oxidation. Thus, to prevent lipid oxidation the period that seeds remain in this water activity condition should be observed or the dehydration should be stopped when the water activity reaches values between 0.4 and 0.3, range that ensures the highest lipids stability.

The drying rate decreased continuously throughout the drying period indicating that the drying of grapefruit seeds took place in the falling rate period.

Drying of most food materials occurs in the falling rate period (WANG; BRENNAN, 1992), and moisture transfer during drying is controlled by internal diffusion. The Fick's second law of diffusion has been widely used to describe the drying process during the falling rate period for most biological materials (SARAVACOS; CHARM, 1962). However, thin-layer drying models have been used to describe the drying kinetics of some products, mainly when the geometry of the material is unknown and also due to simplicity of these models. The Table 3 showed the parameters of the Fick's second law and thin-layer drying models. Curves of moisture ratio versus drying time fitted to Fick's second law model are shown in the Figure 3. In the three velocities, the temperature reduced the drying time, higher temperatures reached the same moisture ratio of those under lower temperatures, but in shorter time.

The goodness of fit for each model was evaluated based on the RMSE and R^2 (Table 4). The Fick's second law and Page models proved to better represent the experimental data of grapefruit seeds. Both models presented the highest R^2 and the lowest RMSE values. Lewis and Henderson-Pabis models, although with R^2 greater than 0.9, the RMSE values were higher than the Fick's second law and Page models, i.e., the predicted moisture ratio by these models was very different from the experimental moisture ratio.

The effective moisture diffusivity (D_{eff}) was determined by fitting experimental data to the Equation (8). The Table 3 lists the temperature and velocity dependence of the D_{eff}, increasing with increasing temperature and air velocity. D_{eff} values during drying under the given conditions ranged from 4.36×10^{-10} to 6.82×10^{-10} m² s⁻¹.

Table 3. Parameters of the drying models.

V	T	Fick	Page		Lewis	Henderson-Pabis	
(m s⁻¹)	(°C)	D_{eff} (m² s⁻¹)	k (s⁻¹)	N	k (s⁻¹)	k (s⁻¹)	a
0.6	40	4.36×10^{-10}	6.29×10^{-3}	0.670	4.50×10^{-4}	2.00×10^{-4}	0.873
	50	4.98×10^{-10}	7.49×10^{-3}	0.659	5.20×10^{-4}	2.40×10^{-4}	0.874
	60	5.80×10^{-10}	7.58×10^{-3}	0.671	6.10×10^{-4}	3.10×10^{-4}	0.881
	70	6.53×10^{-10}	8.39×10^{-3}	0.668	7.00×10^{-4}	3.20×10^{-4}	0.882
1.0	40	4.64×10^{-10}	9.57×10^{-3}	0.624	4.90×10^{-4}	2.00×10^{-4}	0.849
	50	5.17×10^{-10}	9.00×10^{-3}	0.640	5.50×10^{-4}	2.50×10^{-4}	0.862
	60	5.94×10^{-10}	8.56×10^{-3}	0.658	6.30×10^{-4}	3.10×10^{-4}	0.874
	70	6.73×10^{-10}	9.80×10^{-3}	0.651	7.30×10^{-4}	3.20×10^{-4}	0.874
1.4	40	4.63×10^{-10}	7.85×10^{-3}	0.648	4.90×10^{-4}	2.00×10^{-4}	0.87
	50	5.18×10^{-10}	9.21×10^{-3}	0.637	5.50×10^{-4}	2.50×10^{-4}	0.862
	60	6.01×10^{-10}	9.21×10^{-3}	0.649	6.40×10^{-4}	3.10×10^{-4}	0.869
	70	6.82×10^{-10}	10.4×10^{-3}	0.646	7.40×10^{-4}	3.30×10^{-4}	0.871

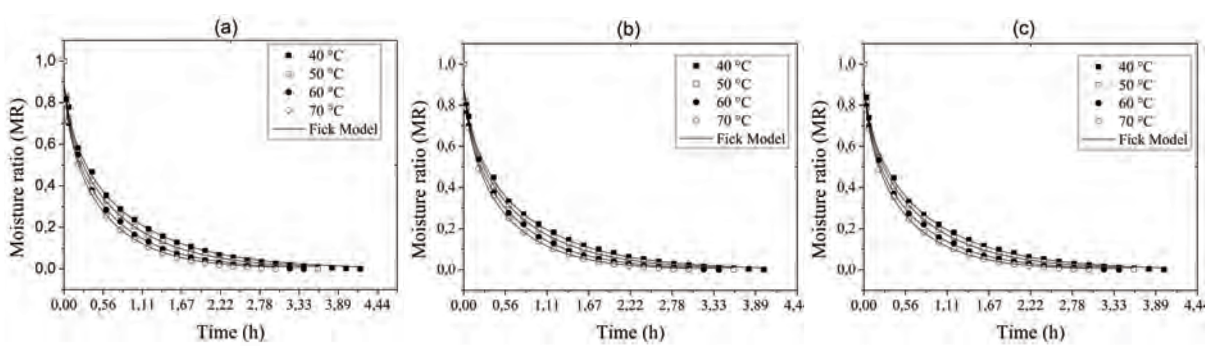

Figure 3. Drying curves: (a) 0.6 m s⁻¹; (b) 1.0 m s⁻¹; (c) 1.4 m s⁻¹.

Table 4. Prediction model Evaluation.

V	T	Fick		Page		Lewis		Henderson-Pabis	
(m s⁻¹)	(°C)	R^2	RMSE	R^2	RMSE	R^2	RMSE	R^2	RMSE
0.6	40	0.992	0.025	0.996	0.018	0.960	0.058	0.983	0.128
	50	0.992	0.026	0.997	0.016	0.958	0.060	0.981	0.118
	60	0.992	0.027	0.997	0.015	0.962	0.057	0.982	0.096
	70	0.992	0.028	0.998	0.014	0.963	0.060	0.982	0.116
1.0	40	0.990	0.025	0.995	0.021	0.940	0.067	0.975	0.131
	50	0.991	0.026	0.996	0.018	0.949	0.065	0.977	0.112
	60	0.992	0.026	0.997	0.015	0.959	0.060	0.981	0.098
	70	0.992	0.027	0.998	0.015	0.957	0.062	0.978	0.115
1.4	40	0.990	0.025	0.997	0.016	0.952	0.059	0.977	0.133
	50	0.992	0.024	0.996	0.017	0.949	0.062	0.977	0.108
	60	0.992	0.025	0.997	0.016	0.955	0.062	0.979	0.098
	70	0.992	0.027	0.998	0.015	0.955	0.063	0.978	0.111

Similar results for the effective moisture diffusivity under the same conditions were found in the literature, such as 1.75×10^{-10} to 8.03×10^{-10} m² s^{-1} for different varieties of grape seeds (ROBERTS et al., 2008); 5.42×10^{-11} to 9.29×10^{-10} m² s^{-1} for pistachio nuts (KASHANINEJAD et al., 2007); 2.53×10^{-12} to 7.67×10^{-11} m² s^{-1} for quinoa seeds (GELY; SANTALLA, 2007); 8.53×10^{-11} to 1.52×10^{-10} m² s^{-1} for hull-less pumpkin seeds (SACILIK, 2007) and 3.78×10^{-9} to 7.10×10^{-9} m² s^{-1} for red chillies (KALEEMULLAH; KAILAPPAN, 2006).

Temperature dependence of the three air velocities, 0.6, 1.0 and 1.4 m s^{-1}, can be represented by the following equations, respectively.

$$D_{eff} = 4.77\times10^{-08}\, exp\left(-12228/8.31T\right) \quad (14)$$

$$D_{eff} = 3.52\times10^{-08}\, exp\left(-11294/8.31T\right) \quad (15)$$

$$D_{eff} = 4.25\times10^{-08}\, exp\left(-11791/8.31T\right) \quad (16)$$

The activation energy (E_a) was determined from the slope of the Arrhenius plot, ln(D_{eff}) versus $1/T$. It can be seen from Equations 14, 15, and 16 that the E_a varied between 11.29 and 12.83 kJ mol^{-1}, clearly showing the poor influence of air velocity on the drying process. Arrhenius relationship fitted appropriately to the effective diffusivity with determination coefficients above 0.99. The estimated values of activation energy of grapefruit seeds are lower than reported (33.15 kJ mol^{-1}) by Sacilik (2007) for hull-less pumpkin seeds and (30-40 kJ mol^{-1}) by Roberts et al. (2008) for different varieties of grape seeds.

The empirical constants for thin-layer models (Equations 10 to 12) are given in the Table 3. The determination coefficient for the drying rate constant varied from 0.975 to 0.998. According to Roberts et al. (2008), high values of determination coefficients are due to the highly linear plots of the unaccomplished moisture content, probably due to accurate equilibrium moisture contents. However, Lewis and Henderson-Pabis presented the worst RMSE values, indicating that the Page model is the best among the thing-layer tested models to describe the experimental data of grapefruit seeds. Drying rate constants increased with increasing temperature, but the air velocity had a low influence.

Conclusion

According to the results obtained, the sorption isotherm of grapefruit seeds was best described by the Henderson model. The effective diffusivity varied from 4.36×10^{-10} and 6.82×10^{-10} m² s^{-1}. The temperature dependence of the effective diffusivity followed an Arrhenius relationship, and the activation energy was found to range between 11.29 and 12.83 kJ mol^{-1} and was not influenced by the air velocity. The Page model proved to be the best thin-layer model for predicting the drying of grapefruit seeds at each air velocity.

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