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Drying kinetics and thermodynamic properties of bitter melon (*Momordica charantia* L.) leaves

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ABSTRACT: Bitter melon (*Momordica charantia* L.) is a versatile plant that can be consumed as a food and has therapeutic applications. Studying its drying process is important to maintain their leaf quality during storage. The objective of this study was to evaluate the drying kinetics of bitter melon leaves and determine their thermodynamic properties. The leaves were placed in polyethylene trays and subjected to drying in an oven at temperatures of 20, 30, 40, and 50 °C until reaching hygroscopic equilibrium. The experimental data were fitted to several non-linear regression models to characterize the drying process. The Arrhenius model was used to obtain the coefficients of diffusion and the activation energy, which were used to calculate the enthalpy, entropy, and the Gibbs free energy. Midilli and Page were the best models to represent the drying kinetics of bitter melon leaves at temperatures of 20, 30, 40, and 50 °C. Increases in the drying air temperature increased the Gibbs free energy and water diffusivity in the interior of the leaves. Enthalpy and entropy decreased as the temperature was increased.

Key words: diffusivity, Midilli, Page, moisture, medicinal plant

Cinética de secagem e propriedades termodinâmicas das folhas do melão-de-são-caetano (*Momordica charantia* L.)

RESUMO: O melão-de-são-caetano (*Momordica charantia* L.) é uma planta versátil que pode ser consumida como alimento e possui aplicações terapêuticas. Desta forma, o estudo sobre o processo de secagem é de suma importância para manter a qualidade de suas folhas durante o armazenamento. Objetivou-se, com esta pesquisa, avaliar a cinética de secagem das folhas de melão-de-são-caetano, bem como determinar suas propriedades termodinâmicas. As folhas foram dispostas em bandejas de polietileno e submetidas a secagem em estufa nas temperaturas de 20, 30, 40 e 50 °C até atingir o equilíbrio higroscópico. Diversos modelos de regressão não linear foram ajustados aos dados experimentais para caracterizar o processo de secagem. A partir do modelo de Arrhenius foram obtidos os coeficientes da difusão e a energia de ativação para o cálculo da entalpia, entropia e energia livre de Gibbs. Os modelos de Midilli e Page foram os melhores para representação da cinética de secagem das folhas de melão-de-são-caetano nas temperaturas de 20, 30, 40 e 50 °C. O aumento da temperatura do ar de secagem aumenta a energia livre de Gibbs e a difusividade de água no interior das folhas. A entalpia e a entropia diminuem com o aumento da temperatura.

Palavras-chave: difusividade, Midilli, Page, teor de água, planta medicinal

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Introduction

Bitter melon (*Momordica charantia* L.) is a common vine found in the coastal region and interior of Brazil that is known by its use in cooking and medicine (Joly, 1998). It is a medicinal plant that can be easily obtained; studies report its use in several countries, such as Brazil, China, Colombia, Cuba, Ghana, Haiti, India, Mexico, Malaysia, Nova Zealandia, Nicaragua, Panama, and Peru (Assis et al., 2015).

Drying is one of the main processes that assist in the maintenance of agricultural products, which is needed for a better use and maintenance of the active principle of bitter melon leaves (Rodovalho et al., 2015).

The activation energy represents the difficult level of water molecules to overcome the energy barrier in their migration in the interior of a product (Corrêa et al., 2007). It enables the determination of thermodynamics properties that provide information about the mechanism of control of water vapor sorption (Viganó et al., 2012).

Information on thermodynamic properties in the drying process of leaves is important for designing the drying equipment, calculating the energy required for the process, studying properties of the adsorbed water, evaluating food microstructures, and studying the physical phenomena that occur on the surface of agricultural products (Corrêa et al., 2010).

Therefore, the objective of this study was to evaluate the drying kinetics of bitter melon leaves at temperatures of 20, 30, 40, and 50 °C and determine their thermodynamic properties.

Material and Methods

The experiment was conducted at the Laboratory of Plant Physiology of the Federal Institute Goiano (FIG), in Ceres, GO, Brazil (15°18'49"S, 49°36'12"W, and altitude of 630 m), in August 2015. Bitter melon leaves were randomly collected in regions near the FIG campus, at the first hours of the morning without dew on the leaf surfaces.

Homogeneous leaves without damages caused by pathogens or insects were selected to avoid their effects on the results.

The initial equilibrium water content of the samples was measured after the selection of the product and at the end of the drying by the gravimetric method in a forced-air circulation oven at 103 ± 1 °C for 24 h, with four replications (ASABE, 2010).

The leaves were dried at temperatures of 20, 30, 40 and 50 °C; the temperatures were controlled by a biochemical oxygen demand (BOD) chamber.

The samples were cut using a square template with an area of 17.2 mm^2, and a thin layer was deposited in polyethylene trays. Approximately 2 g of sample per replication were placed in the trays at the beginning of the drying process, using four replications for each temperature, and were weighed periodically in a digital analytical balance with precision of 0.1 mg until the samples reach equilibrium water content with the drying air.

The interval between weighing started with 20 min and increased up to 10 h to monitor the hygroscopic equilibrium.

The water content ratio was calculated during the drying processes, using Eq. 1.

$$RX = \frac{(X - Xe)}{(X - Xi)} \quad (1)$$

where:

RX - water content ratio, dimensionless;
X - leaf water content, kg of water per kg of leaf dry weight;
Xe - leaf equilibrium water content, kg of water per kg of leaf dry weight; and,
Xi - leaf initial water content, kg of water per kg of leaf dry weight.

The bitter melon leaf water content data were fitted to non-linear regression models (Table 1). Experimental data of drying blackberry (*Morus nigra*) leaves were also fitted to these models by Martins et al. (2018).

Table 1. Non-linear regression models used to estimate the drying of bitter melon leaves

Model	Model equation	
Diffusion approach	$RX = a \exp(-k t) + (1 - a) \exp(-k b t)$	(2)
Two-term	$RX = a \exp(-k t) + b \exp(-c t)$	(3)
Two-term exponential	$RX = a \exp(-k t) + (1-a) \exp(-k a t)$	(4)
Henderson and Pabis	$RX = a \exp(-k t)$	(5)
Logarithm	$RX = a \exp(-k t) + b$	(6)
Midilli	$RX = a \exp(-k t^n) + b t$	(7)
Newton	$RX = \exp(-k t)$	(8)
Page	$RX = \exp(-k t^n)$	(9)

RX - Leaf water content ratio (dimensionless); t - Drying time (h); k - Coefficient of drying; a, b, c, and n - Parameters of the models

The best models to represent the bitter melon leaf drying kinetics were selected, considering the significance of the coefficients of regression by the t test at $p \leq 0.05$, and the magnitude of coefficient of determination (R^2), relative mean error (P), and standard deviation of the estimate (SD).

The mathematical model for drying kinetics was selected considering the highest R^2, p-value below 10%, and the lowest SD.

The standard deviation of the estimate (SD) and relative mean error (P) were calculated using Eqs. 10 and 11.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Y - \hat{Y})^2}{DF}} \quad (10)$$

$$P = \frac{100}{N} \sum_{i=1}^{N} \left(\frac{|Y - \hat{Y}|}{Y} \right) \quad (11)$$

where:

N - number of experimental observations;
Y - experimental water content ratio;
$\hat{Y}$ - predicted moisture ratio; and,
DF - degrees of freedom of the residue.

The dimensions and mean thickness of the sampled leaves were used to obtain the coefficient of effective diffusion by adjusting the mathematical model of the net diffusion to eight terms, as described in Eq. 12, considering the product geometry as a plain body, disregarding leaf volumetric contractions (Brooker et al., 1992).

$$RX = \sum_{n=1}^{\infty} \frac{4}{\lambda n^2} \exp\left(-\frac{\lambda n^2}{L^2} D_{ef} T\right) \quad (12)$$

where:

- RX - water content ratio, dimensionless;
- D_{ef} - coefficient of effective diffusion;
- T - time, s;
- n - number of terms;
- L - product thickness, m; and,
- λn - square roots of the Bessel equation of first type, with order 0.

The correlation between increases in the coefficient of effective diffusion (D_{ef}) and increases in the drying air temperature was evaluated by Eq. 13, which represents the Arrhenius model.

$$D_{ef} = D_0 \exp\left(\frac{E_a}{RT_a}\right) \quad (13)$$

where:

- D_0 - pre-exponential factor;
- T_a - absolute temperature, K;
- R - gas universal constant, 8.314 kJ kmol^{-1} K^{-1}; and,
- E_a - activation energy, kJ mol^{-1}.

The coefficients of the Arrhenius model were obtained using the logarithm expressed by Eq. 14.

$$\ln(D_{ef}) = \ln(D_0) - \frac{E_a}{R}\frac{1}{T_a} \quad (14)$$

The thermodynamic properties of the dried bitter melon leaves were determined by the method described by Jideanie & Mpotokwana (2009) and Corrêa et al. (2010). Enthalpy (Δh) was calculated using Eq. 15; entropy (Δs) was calculated using Eq. 16; and the Gibbs free energy (ΔG) was calculated using Eq. 17.

$$\Delta h = E_a - RT_a \quad (15)$$

$$\Delta s = R\left(\ln D_0 - \ln\frac{k_b}{h_p} - \ln T_a\right) \quad (16)$$

$$\Delta G = \Delta h - \Delta T_a \Delta s \quad (17)$$

where:

- Δh - specific enthalpy, J mol^{-1};
- Δs - specific entropy, J mol^{-1} K^{-1};
- ΔG - Gibbs free energy, J mol^{-1};
- kb - Boltzmann's constant, 1.38 × 10^{-23} J K^{-1}; and,
- hp = Planck's constant, 6.626 × 10^{-34} J s^{-1}.

Results and Discussion

The experimental data fitted to the models (Table 2), presenting R^2 above 97%, except the two-term exponential model at temperature of 30 °C (61.71%) and diffusion approach model for temperature of 40 °C (92.97%).

The Midilli model showed the highest R^2; high R^2 indicate a better fit of the experimental data to the model (Karizaki, 2016). However, according to Mohapatra & Rao (2005), the use of R^2 as the only evaluation criterion to select drying models is not adequate, thus requiring a joint analysis of fitting indexes.

The Midilli model presented the lowest relative mean error (P) (Table 2). Mohapatra & Rao (2005) recommend the use of joint analysis of all temperatures involved with the drying process with relative mean error lower than 10%.

The Midilli model presented the lowest standard deviation (SD); according to Draper & Smith (1998), the capacity of a model to describe a physical process with reliability is inverse to the SD, therefore, the lower the SD, the better the fit to the model.

Considering the R^2, P, and SD, the Midilli model was chosen to represent the drying kinetics of bitter melon leaves.

Studies on drying of other medicinal plant species have used the Midilli model due to its better fit of the experimental data. This was recommended by Corrêa Filho et al. (2018) who evaluated the drying of parsley plants at temperatures of 40, 50, and 60 °C, and by Goneli et al. (2014a) who evaluated the drying of *Schinus terebinthifolius* leaves at the temperatures of 40, 50, 60, and 70 °C.

The bitter melon leaf drying period decreases as the drying air temperature was increased (Figure 1); this was because of increases in the pressure gradient between the interior of the leaf and the surrounding air. The water content of bitter melon leaves subjected to air drying at temperatures of 20, 30, 40, and 50 °C was low, approximately 3.20 to 0.35 dry basis.

Table 2. Coefficient of determination (R^2), relative mean error (P), and standard deviation of the estimate (SD) as criteria to fit experimental data of bitter melon leaves to drying models, obtained at temperatures of 20, 30, 40, and 50 °C

Model	R^2	P	SD	R^2	P	SD
	(%)		(decimal)	(%)		(decimal)
	20 °C			30 °C		
Diffusion approach	99.92	2.7137	0.00018	99.87	1.8495	0.0003
Two-term	99.93	2.6968	0.00018	99.87	2.0194	0.0003
Two-term exponential	99.92	2.6791	0.00020	61.71	63.5411	0.1088
Henderson and Pabis	99.87	2.4640	0.00030	99.74	4.6480	0.0007
Logarithm	99.94	2.7633	0.00030	99.87	2.1964	0.0003
Midilli	99.95	1.5565	0.00011	99.18	2.1961	0.0003
Newton	99.82	2.2805	0.00045	99.74	4.8128	0.0007
Page	99.89	3.2520	0.00026	98.85	2.4740	0.0004
	40 °C			50 °C		
Diffusion approach	92.97	49.2894	0.02630	99.54	11.6365	0.0012
Two-term	99.97	3.5560	0.00010	99.87	5.3703	0.0003
Two-term exponential	99.89	6.6119	0.00040	99.83	5.1374	0.0004
Henderson and Pabis	99.91	6.6104	0.00030	99.66	9.5608	0.0009
Logarithm	97.56	1.0141	0.00004	99.82	7.4054	0.0004
Midilli	99.99	1.2094	0.00003	99.70	5.1614	0.0008
Newton	99.87	5.8548	0.0005	99.53	11.6363	0.0012
Page	99.86	6.0689	0.0005	99.79	4.5235	0.0006

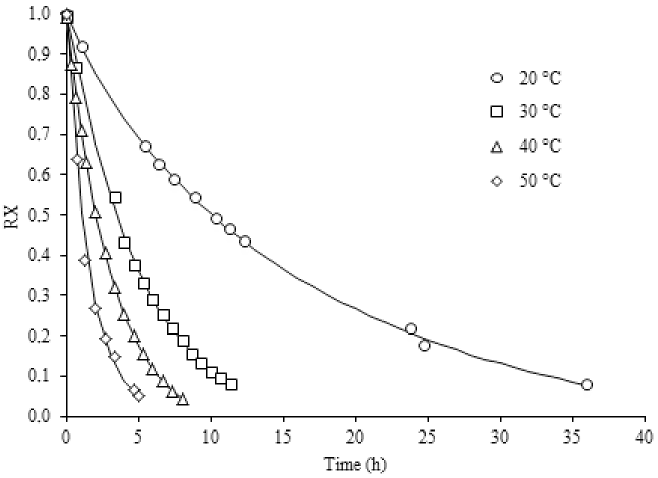

Figure 1. Experimental and estimated values for water content ratio (RX) by estimates of parameters of the Midilli equation as a function of drying time of bitter melon leaves, at the temperatures of 20, 30, 40, and 50 °C

The drying time decreased as the temperatures was increased. This can be explained by increases in the pressure gradient between the drying air and the interior of the leaves, and indicates a greater decrease of water content in less time.

This was also found for the drying of leaves of *Cordia verbenacea*, *Ziziphus joazeiro,* and *Mentha piperita* (Goneli et al., 2014b; Sousa et al., 2015; Gasparin et al., 2017).

The Midilli model is also used for the fit of drying kinetics, as recommended by Martins et al. (2018), who evaluated the drying of blackberry leaves at temperatures of 40, 50, 60, and 70 °C.

The k coefficient of the Midilli model increased from 0.077462 to 0.690506 as the temperature was increased (Table 3). This coefficient can be used to characterize the effect of temperature and is related to the effective diffusivity in the drying process in a decreasing period, because the liquid diffusion controls the drying process (Babalis & Belessiotis, 2004).

The D_{ef} values increased in 4.38, 13.9, 24.1, and 38.1 × 10^{-10} m^2 s^{-1} for the drying air temperatures of 20, 30, 40, and 50 °C, respectively (Table 3). Martins et al. (2015) found similar results for *Serjania marginata* leaves, with a D_{ef} increase from 0.6630 to 12.0712 × 10^{-11} m^2 s^{-1}. According to Goneli et al. (2014a), these increases are dependent on temperature, concentration, vibration of water molecules, and volume of the product. Increase in temperature increases vibration of water molecules in the interior of the product, thus increasing its coefficient of effective diffusion (Goneli et al., 2008).

The D_{ef} of the bitter melon leaf drying was in the range of 10^{-11} to 10^{-9} m^2 s^{-1}, which is consistent with Zogzas et al. (1996), who found D_{ef} within this range for agricultural products.

The slope of the Arrhenius curve (Figure 2), generated from the values of $\ln(D_{ef})$ as a function of the inverse absolute air temperature (1/Ta) during the bitter melon leaf drying, provides the Ea/R ratio, whereas its intersection with the ordinate axis indicates the value of D_0.

The correlation between the coefficient of effective diffusion and drying air temperature has been described satisfactorily by the Arrhenius equation (Martins et al., 2015). According to Gasparin et al. (2017), the linear fit indicates the uniformity of variation of the coefficient of diffusion as a function of temperature.

The activation energy (Ea) obtained by the Arrhenius model was 33.163 kJ mol^{-1}. Silva et al. (2015) found similar result for *Genipa americana* leaves, with Ea of 33.87 kJ mol^{-1}, and Martins et al. (2018) found lower results for blackberry leaves, with Ea of 66.08 kJ mol^{-1}. The lower Ea found for the bitter melon leaves denotes the need for less energy to trigger the liquid diffusion process than that for blackberry leaves. Different activation energy values found for different agricultural products can be attributed to the physical and biological characteristics of the products (Martins et al., 2015).

According to Corrêa et al. (2007), Ea can be explained as the water molecule difficulty to overcome the energy barrier during their migration in the interior of leaves, since the lower the activation energy the higher the water diffusivity in the interior of the leaf during the drying process. The Ea found for the bitter melon leaf drying was within the range (12.7 to 110 kJ mol^{-1}) for agricultural products proposed by Zogzas et al. (1996).

Regarding the thermodynamic properties, the enthalpy (Δh) decreased from 30.726 to 30.477 kJ mol^{-1} as the temperature was increased (Table 4). This indicates the need for a lower quantity of energy for drying at high temperatures (Martins et al., 2015).

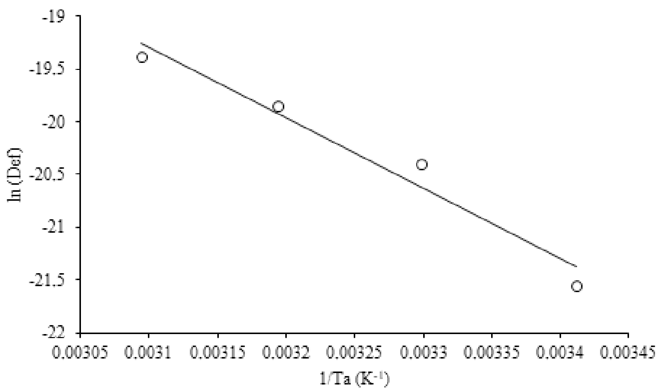

Figure 2. Arrhenius expression for the coefficient of effective diffusion (D_{ef}) as a function of the inverse absolute air temperature during the bitter melon leaf drying

Table 3. Parameters of the Midilli model (a, b, k, n) and coefficient of effective diffusion (D_{ef}) for different bitter melon leaf drying conditions

Temperature (°C)	a	k	n	b	D_{ef} (m^2 s^{-1})	R^2 (%)	P
20	0.997883	0.084531	0.893524	-0.00130	4.38 10^{-10}	98.92	8.91
30	0.991493	0.18	1.06	-0.00134	13.9 10^{-10}	97.54	9.04
40	0.977481	0.33088	0.983439	-0.00419	24.1 10^{-10}	99.02	8.55
50	1.002541	0.686053	0.908001	-1.0 10^{-12}	38.1 10^{-10}	98.33	7.62

R^2 - Coefficient of determination; P - Relative mean error

Table 4. Enthalpy (Δh), entropy (Δs), and Gibbs free energy (ΔG) of the bitter melon leaf drying process

Temperature (°C)	Δh (kJ mol^{-1})	Δs (kJ mol^{-1} K^{-1})	ΔG (kJ mol^{-1})
20	30.726	-0.2276	97.3172
30	30.643	-0.2274	99.5901
40	30.559	-0.2277	101.8658
50	30.477	-0.2279	104.1442

The entropy (Δs) decreased from -0.2276 to -0.2279 kJ mol^{-1} K^{-1}, similarly to the enthalpy (Table 4). According to Corrêa et al. (2010), this result is expected, because decreases in temperature cause lower excitation of water molecules, increasing the order of the water-leaf system. The negative entropy is attributed to chemical adsorption or structural changes in the adsorbent (Moreira et al., 2008).

The Gibbs free energy (ΔG) increased from 97.3172 to 104.1442 kJ mol^{-1} as the temperature was increased. This characterizes an endothermic reaction, i.e., an external energy source is needed to increase the energy level and transform the reagents from the liquid to the vapor state (Ong et al., 2013).

Conclusions

1. Midilli and Page are the models that better represent the bitter melon (*Momordica charantia* L.) leaf drying.

2. Increases in temperature increase removal of water from bitter melon leaves during their drying.

3. The coefficient of effective diffusion and the Gibbs free energy increased as the drying temperature was increased, decreasing the enthalpy and entropy.

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