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Pequi pulp (*Caryocar brasiliense* Cambess): Drying kinetics and thermodynamic properties

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The objective of this study was to evaluate the drying kinetics of Pequi pulp (*Caryocar brasiliense* Cambess) at temperatures of 40, 50 and 60°C, and the thermodynamic properties for this process. Eleven mathematical models commonly used to represent the drying process of agricultural products were fitted to experimental data. The Fick's second law was used to determine the diffusion coefficients of Pequi fruits through the drying kinetics. The model of Midilli best represented the drying process of Pequi pulp. The calculated effective diffusivity was 4.69988 × 10^{-14}, 5.277436 × 10^{-14} and 5.609491 × 10^{-14} (m^2s^{-1}) for temperatures of 40, 50 and 60°C, respectively, and the energy activation for the process was 7694.94 J mol^{-1}. The enthalpy decreased with increasing temperature, with values of 5091.41, 5008.27 and 4925.13 ($Jmol^{-1}$) for temperatures of 40, 50 and 60°C, respectively. The entropy values found were -251.01, -250.38 and -250.05 ($J.Mol^{-1}K^{-1}$) for the same temperatures. The values obtained from the Gibbs free energy for the drying of Pequi pulp increased with increasing temperature. The obtained data were consistent to the drying process, and the mathematical equations were effective to explain the migration of water within the product.

Key words: Effective diffusivity, drying models, enthalpy, entropy, Gibbs free energy.

INTRODUCTION

Pequi (*Caryocar brasiliense* Cambess., Caryocaraceae) is a typical fruit from the savannah ecosystem ("Cerrado") with high nutritional value, being economically exploited by the regional population in the fresh form or in the preparation of juices, ice creams, liqueurs, jams and traditional dishes; however, the fruit is not widespread throughout Brazil due to its high perishability (Machado et al., 2013).

Pequi fruits give a sharp and distinctive flavor of regional cuisine, leading to customer acceptance and satisfaction (Geöcze et al., 2013). Pequi pulp is rich in lipids (33.4 g.100 g^{-1}) and is an important source of dietary fiber (10.02 g.100 g^{-1}), has protein content of 3 g.100 g^{-1} and provides about 358 Kcal.100 g^{-1} of material, which correspond to 18 g.100 g^{-1} the caloric needs of an adult on a diet of 2,000 kcal. In addition, there is a predominance of unsaturated fatty acids in the pulp lipids, with oleic acid showing the highest occurrence.

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Table 1. Mathematical models applied to drying curves.

Model	Model description
Approximation	$RX = a \cdot \exp(-k \cdot t) + (1 - a) \cdot \exp(-k \cdot b \cdot t)$
Two-term	$RX = a \cdot \exp(-k_0 \cdot t) + b \cdot \exp(-k_1 \cdot t)$
Two- term exponential	$RX = a \cdot \exp(-k \cdot t) + (1 - a) \cdot \exp(-k \cdot a \cdot t)$
Handerson & Pabis	$RX = a \cdot \exp(-k \cdot t)$
Logarítmo	$RX = a \cdot \exp(-k \cdot t) + c$
Midilli	$RX = a \cdot \exp(-k \cdot t^n) + b \cdot t$
Newton	$RX = \exp(-k \cdot t)$
Page	$RX = \exp(-k \cdot t^n)$
Thompson	$RX = \exp((-a - (a^2 + 4 \cdot b \cdot t)^{0,5})/2 \cdot b)$
Verma	$RX = a \cdot \exp(-k \cdot t) + (1 - a) \cdot \exp(-k_1 \cdot t)$
Wang & Singh	$RX = 1 + a \cdot t + b \cdot t^2$

Where, t: drying time, h; k, k_o, k_1: drying constants h^{-1}, and a, b, c, n: model coefficients.

To meet Pequi market during the offseason, the conservation of Pequi pulp is basically by freezing and in the form of acidified canned products. The use of other conservation techniques such as dehydration/drying can provide other ways of use and application, preserving the pulp and increasing the life of the product, in addition to promoting the development of differentiated products (Lewicki, 2006).

Dried fruits and vegetables have gained commercial importance and its growth on a commercial scale has become an important sector of the agricultural industry. The lack of adequate treatment causes considerable damage and waste of seasonal fruits in many countries, which is estimated at 30-40% in developing countries. It is necessary to remove the moisture content of the fruit to a certain level after harvest to prevent the growth of mold and bacterial action (Azharul Karim and Hawlader, 2005).

Oven drying is an inexpensive process, but often leads to degradation of labile compounds and/or oxidizable substrates such as carotenoids and lipids. To overcome these limitations, drying is generally carried out at moderate temperature (40-60°C) (Durante et al., 2014).

The drying process consists of the removal of most of the moisture content of a product, causing unfavorable conditions for the continuity of metabolic activity and growth of microorganisms (Martinazzo et al., 2007). The study on the required parameters of drying kinetics is important in order to improve the drying process and obtain a quality product that meets consumer demands (Cano-Chauca et al., 2004).

The use of mathematical models for the representation of the drying process is crucial, considering that the information generated is of great value for designing, development and improvement of processes and equipment, as well as for the prediction of drying times.

MATERIALS AND METHODS

Pequi fruits were purchased from local market of Rio Verde, Goiás State, Brazil, and transported to the Laboratory of Fruits and Vegetables - Federal Institute Goiás - Rio Verde Campus, Goiás, Brazil. In the laboratory, they were received and sanitized in 150 ppm chlorine solution for 15 min and subsequently dried. Then, Pequi fruits were sliced with an average thickness of 2.33 mm, vacuum packaged, and stored in low-density polyethylene bags until time of drying in oven.

Drying the Pequi pulp

Pequi samples were dried in a Marconi oven model MA 035 - Piracicaba - Brazil, with forced air ventilation and air flow rate of 7.728 kg.$(m^2 s)^{-1}$ at three temperature conditions: 40, 50 and 60°C. During drying in perforated trays, samples were weighed from 20 to 20 min up to obtaining water content of 0.111 (decimal, db), determined at 105 ± 1°C for 24 h (AOAC, 2000). The entire drying process was carried out in three replicates.

Temperature and relative humidity of the environment external to the drying chamber were monitored using a thermohygrometer, and the internal temperature was monitored by a thermometer placed inside the drying chamber. The relative humidity inside the drying chamber was obtained by means of the basic psychrometric principles, using the GRAPSI software.

Drying kinetics

The following expression was used to determine the moisture content in the Pequi pulp during drying:

$$RX = \frac{X - X_e}{X_i - X_e}$$

Where, RX is the humidity ratio, dimensionless; X is the moisture content at time t, decimal dry basis (kg water, kg^{-1} dry matter); X_e is the equilibrium water content of the product, decimal dry basis (kg water, kg^{-1} dry matter); and X_i is the initial moisture content, decimal dry basis (kg water, kg^{-1} dry matter).

The modeling is intended to adjust one or more models throughout the studied range of this variable (Corrêa et al., 2010). The experimental drying data of Pequi pulp were fitted to mathematical models often used to represent the drying of agricultural products, as presented in Table 1.

The liquid diffusion model for flat plate geometry with known thickness (Fick's law and eight-term approximation equation) was fitted to experimental Pequi pulp drying data in accordance with the following expression:

$$RX = \frac{X - X_e}{X_i - X_e} = \frac{8}{\pi^2} \sum_{N_t=0}^{\infty} \frac{1}{(2N_t + 1)} \exp\left[-(2N_t + 1)^2 \pi^2 D \frac{t}{4L^2} \right]$$

Where, N is the number of terms; D_{eff} is the liquid diffusion coefficient, m^2s^{-1}, and L is the half the sample thickness, m.

The relationship between the effective diffusion coefficient and the increase in the drying air temperature was described by the Arrhenius equation.

$$D_{eff} = D_0 \exp\left(-\frac{E_a}{RT}\right)$$

Where, Do is the pre-exponential factor; Ea is the activation energy, $kJ.mol^{-1}$; R is the universal gas constant, 8.134 $kJ.kmol^{-1}.K^{-1}$, and T is the absolute temperature, K.

Thermodynamic properties

The thermodynamic properties of Pequi pulp drying process were obtained by the method described by Jideani and Mpotokwana (2009).

$$\Delta H = E_a - RT$$

$$\Delta S = R\left(\ln A_0 - \ln\left(\frac{k_B}{h_P}\right) - \ln T\right)$$

$$\Delta G = \Delta H - T\Delta S$$

Where, ΔH = enthalpy, J mol^{-1}; ΔS = entropy, J mol^{-1}; ΔG = Gibbs free energy, J mol^{-1}; kB = Boltzmann constant, 1.38 x 10-23 J K^{-1}, and hp = Planck's constant, 6.626 x 10-34 J s^{-1}.

Statistical analysis

Mathematical models were fitted using nonlinear regression by the Gauss-Newton method using a statistical program. Determination of the investigated components was carried out in three replicates. The models were selected considering the magnitude of the determination coefficient (R^2), relative mean error (P) and estimated mean error (SE). Relative mean error values lower than 10% were considered as a criterion for the selection of models, according to Mohapatra and Rao (2005).

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

$$P = \frac{100}{N} \sum \frac{|Y - \hat{Y}|}{Y}$$

Where, Y is the value experimentally observed; Ŷ is the value estimated by the model; N is the number of experimental observations; GLR is the degrees of freedom of the model (number of experimental observations minus the number of coefficients of the model).

RESULTS AND DISCUSSION

Drying kinetics

Pequi pulp showed initial water content of 1.25 dry basis (decimal db) when submitted to the three drying temperatures that promoted relative humidities of 25.96; 15.30 and 9.80%, respectively. Table 2 shows the R², SE and P values for each model considered in this study for different drying temperatures.

The determination coefficient R² is one of the main criteria for choosing the model that best fits the drying process; however, besides R², parameters SE and P are used for determining the adjustment quality (Doymaz, 2012). The choice of the most appropriate model was given by R² > 98 %, SE < 10 % values (decimal) and lower P values.

It was observed that for all temperatures, the models were satisfactory to describe the drying process, except for the model of Wang and Singh. At 60°C, the model of Midilli (RX = a exp (- k t^n) + b t) showed satisfactory R², SE and P values, so, based on the results obtained for other temperatures, this model was chosen to represent the drying process of Pequi pulp.

Radunz et al. (2011), in his work with carqueja, found that the model of Midilli et al. presented adequate fit to the experimental data for the entire temperature range studied (40-90°C). Lima et al. (2007) dried facheiro pulp and concluded that among the models fitted to the drying kinetics data, the equation of Midilli showed the highest determination coefficient and the lowest mean squared deviation values, corroborating this work. Resende et al. (2010) recommends the model of Midilli for drying processes for presenting simple mathematical operations.

Figure 1 shows the moisture content *versus* Pequi pulp drying time curves studied at different temperatures (40, 50 and 60°C) in oven drier with air circulating.

The longest drying time was at temperature of 40°C, about 5.67 h, while for temperature of 50°C, the drying time was around 5.33 h and at 60°C, it was 4.67 h. The drying curves were well-defined, that is, without floating point throughout the process, indicating homogeneity in the dryer.

It was observed that increased temperatures decrease the drying time of Pequi pulp, since it results in rapid evaporation of water present in the solid. Silva et al. (2009) reported that the increase in temperature causes an increase of the drying rate, which suggests the moisture diffusion from within the product to its surface as the physical mechanism predominant throughout the drying process, with no periods of constant drying rate. Silva et al. (2014) concluded that the drying of whole bananas also occurred exclusively during the period of decreasing rate for all temperatures evaluated. Togrul and Pehlivan (2004) found no periods of constant rate throughout the drying process of apricots, grapes, figs,

Table 2. Values of the determination coefficient (R^2), estimated mean error (SE), relative mean error (P) for mathematical models used in the drying of Pequi pulp (*Caryocar brasiliense* Cambess) at 40, 50 and 60°C.

Model description	R^2 (%)	SE (decimal)	P (%)
40°C			
Approximation	99.96	0.0062	2.3054
Two-term	99.96	0.0064	2.3154
Two- term exponential	99.94	0.0070	2.5640
Handerson & Pabis	99.90	0.0092	3.0179
Logarithmic	99.93	0.0076	2.5614
Midilli	99.96	0.0062	2.3031
Newton	99.84	0.0110	3.9719
Page	99.96	0.0059	2.3253
Thompson	99.94	0.0069	2.4894
Verna	99.96	0.0062	2.3054
Wang & Singh	98.31	0.0371	14.23
50°C			
Approximation	99.97	0.005092	1.3392
Two-term	99.98	0.004905	1.2547
Two- term exponential	99.95	0.006570	2.4539
Handerson & Pabis	99.88	0.010360	5.6170
Logarithmic	99.98	0.004828	1.0532
Midilli	99.98	0.004393	1.5906
Newton	99.87	0.010176	5.8201
Page	99.91	0.008733	3.9863
Thompson	99.95	0.006792	2.5855
Verna	99.97	0.005092	1.3393
Wang & Singh	98.54	0.035443	16.267
60°C			
Approximation	98.78	0.0321	7.8128
Two-term	99.91	0.0092	3.3070
Two- term exponential	99.86	0.0103	3.4578
Handerson & Pabis	99.30	0.0234	4.7387
Logarithmic	99.42	0.0221	5.7471
Midilli	99.93	0.0080	2.3108
Newton	98.78	0.0297	7.8126
Page	99.76	0.0138	5.1096
Thompson	99.52	0.0193	6.6302
Verna	99.91	0.0088	3.3069
Wang & Singh	96.31	0.0537	17.911

peaches and plums, confirming the drying curve obtained in this work.

To maintain the microbiological safety level of the product, that is, to reduce the risk of contaminants, it is desirable to dry the product to obtain moisture ratio less than 0.15 (decimal db) (Krokida and Philippopoulos, 2005).

Figure 1 shows that constant k of the model of Midilli increases in absolute values with increasing temperature, since higher temperatures lead to higher drying rates; however, the other coefficients of the model of Midilli (*a, n* and *b*) did not show a clear trend as a function of drying temperatures. Reis et al. (2012) also reported increased k constant with increasing temperature when drying basil leaves.

The use of kinetic models is a way to predict the drying process at different temperatures, making mathematical models an interesting tool to be used during the kinetic

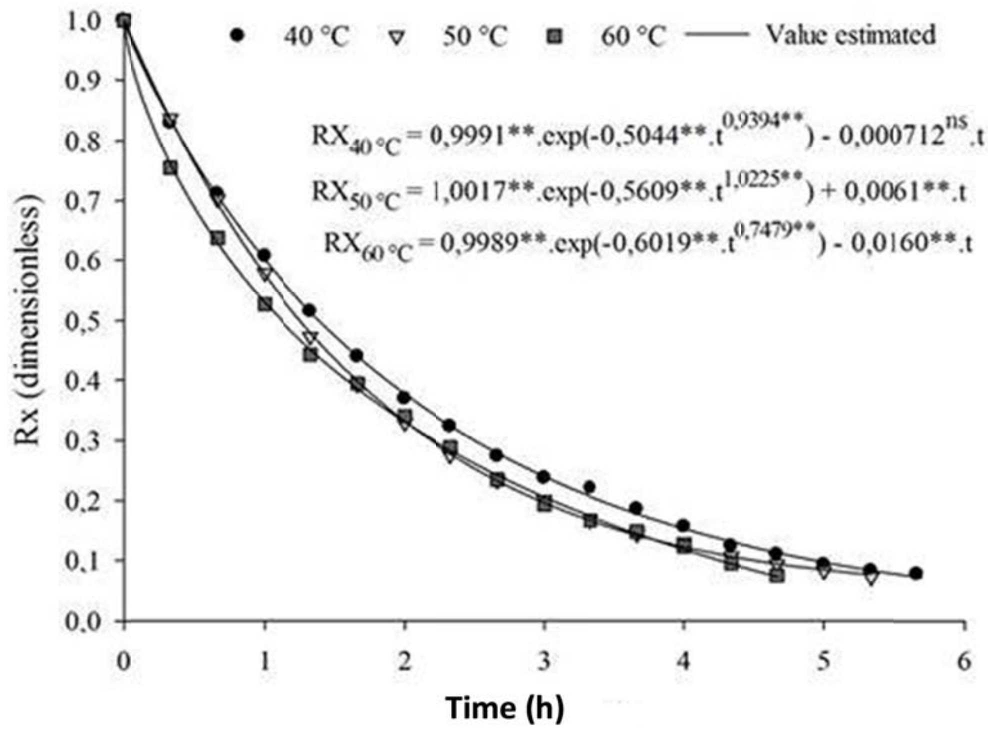

Figure 1. Experimental moisture content values and values estimated by the model of Midilli for drying of Pequi pulp (*Caryocar brasiliense* Cambess) at different temperature conditions.

Table 3. Effective diffusivity and activation energy values for the drying process of Pequi pulp (*Caryocar brasiliense* Cambess) at temperatures of 40, 50 and 60°C.

Property	Temperature		
	40°C	**50°C**	**60°C**
Effective diffusivity ($m^2.s^{-1}$)	$4.6990 x 10^{-14}$	$5.2774 x 10^{-14}$	$5.6095 x 10^{-14}$
Activation energy ($J.mol^{-1}$)		7694.94	

evaluation of a drying process within the temperature range evaluated. Table 3 shows the effective diffusivity results for the different drying temperatures.

It was observed that the effective diffusivity values increase with increasing temperature. This increase is due to increased vapor pressure within the solid, which facilitates the migration of water to the surface of the product. Chen et al. (2012) observed a gradual increase in the effective diffusivity values with increasing temperature during the drying process of biomass.

It is known that the effective diffusivity of different biomaterials varies according to their structure, temperature, and moisture content (Perea-Flores et al., 2012). Madamba et al. (1996) reported that the effective diffusivity coefficients for agricultural products are in the order of 10^{-9} to 10^{-11}, thus differing from the present work in more than 1000 times, which is possibly due to the biological structure of Pequi pulp, since it is rich in lipids and can interfere with the connections of water with the pulp. Oliveira et al. (2012) found low diffusivity coefficients in the drying of corn grains cultivar AG 7088 (from 1.54×10^{13} to 4.85×10^{13}), explaining that these values are due to the chemical composition of the product, and evidencing that to dry an agricultural product, the water must pass through layers of different cell tissues and depending on the chemical composition of these layers, the product presents different characteristics with the environment.

Janjai et al. (2010) obtained similar values for effective diffusivity in drying of lychee, which are on the order of 1.479×10^{-13} and 1.542×10^{-13} at temperatures of 50 and 60°C respectively, explaining that these values vary according to the biological structure of the fruit.

It was observed that the activation energy for the drying of the Pequi pulp was 7.69 kJ mol^{-1} for temperature ranging from 40 to 60°C. For Zogzas et al. (1996), the

Table 4. Values of the enthalpy and entropy variation and the Gibbs free energy for the drying process of Pequi pulp (*Caryocar brasiliense* Cambess) at temperatures of 40, 50 and 60°C.

Thermodynamic properties	Temperature (°C)		
	40	50	60
Enthalpy ($J.mol^{-1}$)	5091.41	5008.27	4925.13
Entropy ($J.mol^{-1}.K^{-1}$)	-251.01	-250.38	-250.05
Gibbs free energy ($J.mol^{-1}$)	83693.70	85919.44	88229.35

activation energy for agricultural products ranged from 12.7 to 110 kJ mol^{-1}. In the present work, the activation energy value found was lower; however, it was higher than that reported by Faria et al. (2012), for the drying of crambe (4.97 kJ mol^{-1}).

Thermodynamically, activation energy is defined as how easy water molecules overcome the energy barrier during migration within the product (Corrêa et al., 2007). It is noteworthy that for drying processes, the lower the activation energy, the higher the water diffusivity within the product (Faria et al., 2012), indicating a facilitated drying process.

Thermodynamic characteristics

Table 4 shows the values of the enthalpy and entropy variation and the Gibbs free energy. The calculation of these energies is relevant, since entropy and enthalpy result in Gibbs free energy, which is a thermodynamic state function representing the maximum amount of energy released in a process occurring at constant temperature and pressure that is free to perform the useful work (Ascheri et al., 2009).

This study shows lower enthalpy values for higher temperatures during the drying process, indicating a smaller amount of energy required for the drying to occur at higher temperatures, that is, enthalpy decreases with increasing temperature. Oliveira et al. (2010) explained that lower enthalpy values indicate lower energy required by the drying process to remove water within the product.

In absolute scale, entropy decreased with increasing temperature. Reduced entropy values can be explained by the fact that when the product is being dehydrated, there is a reduction in the moisture content and the movement of water molecules in the product becomes more difficult (Corrêa et al., 2011). Moreira et al. (2008) explained that negative entropy values are assigned to the existence of chemical adsorption and / or structural modifications of the adsorbent.

Positive values for the Gibbs free energy mean that the drying phenomenon is not a spontaneous process, that is, it requires an energy source for the process to occur. This was expected, since the samples were in an environment with higher humidity (harvest), being subsequently submitted to a process to reduce the humidity values. Corrêa et al. (2011) dried corn cobs and obtained the same trend of increased Gibbs free energy values with increasing process temperature, also observed in this study, which indicates a greater amount of energy with increasing temperature.

Conclusion

Based on experimental data, it was concluded that the removal of water from Pequi pulp occurred during periods of decreasing rate for all temperatures. Among the models investigated in this study, the model of Midilli showed satisfactory data to explain the drying process of Pequi pulp. The constant k of the model of Midilli increases with increasing temperature, as also observed for the diffusion coefficient.

In the drying of Pequi pulp, enthalpy decreases with increasing temperature. The entropy was negative for the entire temperature range studied. The Gibbs free energy was positive for all temperatures, and increased with increasing temperature.

Conflict of Interests

The author(s) have not declared any conflict of interests.

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