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Prediction of mass transfer parameters and thermodynamic properties using the refractance Window™ technique for drying of Yam (*Dioscorea Trifida*) paste

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Abstract

Dioscorea trifida tuber contains starch, vitamins, minerals and bioactive compounds. It is perishable, requiring dehydration treatment to increase shelf life. This study aimed to investigate the mass transfer parameters and thermodynamic properties of *Dioscorea trifida* using Refractance Window (RW) drying (70, 80, and 90 °C). It was observed that the dehydration process occurred in a short time (40 min). The moisture diffusivity and the mass transfer coefficient were determined using the Dincer and Dost model. The diffusivity coefficients ranged from 2.62×10^{-6} at 6.13×10^{-6} $m^2 s^{-1}$, the mass transfer coefficient ranged from 3.46×10^{-4} at 4.04×10^{-4} $m s^{-1}$ and the estimated of activation energy was 44.091 kJ mol^{-1}. In the *Dioscorea trifida* Refractance Window drying, the enthalpy and entropy are positive and negative, respectively, decreasing with increasing temperature and thus indicating that the process is endothermic. Gibbs free energy increases with increasing temperature, indicating that the process does not occur spontaneously.

Keywords: food matrices; mathematical modeling; yam; thermodynamic properties; dincer and dost model.

Practical Application: Use of the Refractance Window™ technique for the production of dehydrated foods with maintenance of functional properties, with low production cost and application on a small scale.

1 Introduction

Dioscorea trifida tuber originates from South America and occurs very frequently in the northern region of Brazil, where it is commonly known as purple yam (Castro et al., 2012; Andres et al., 2017), and is abundantly available for a short period during the summer. *Dioscorea trifida* tubers contain a substantial amount of starch, vitamins, minerals, and important bioactive compounds (Oliveira et al., 2007; Ramos-Escudero et al., 2010; Pérez et al., 2011). Therefore, it has attracted the attention of researchers and the food industry as a potential source of ingredients for various foods such as bread, cookies, creamy soups, cake fillings, among others (Teixeira et al., 2013; Techeira et al., 2014). However, *Dioscorea trifida* is perishable and has a limited shelf life after harvesting. Although the dehydration process can be used to produce value-added products and to extend the shelf life and increase year-round marketing of the products, the conventional drying methods often use high temperatures that can degrade heat-sensitive bioactive compounds. Several studies have reported the Refractance Window (RW) technique for juice concentration, fruit and vegetable dehydration, and even yogurt powder production (Raghavi et al., 2018; Tontul et al., 2018). In the RW drying system, the material to be dried is placed on polyester film which is partially transparent to infrared radiation and this film will be in contact with the surface of hot water, circulating through the reservoir. The thermal energy of hot water is efficiently transferred through the polyester film to wet food material by means of conduction and radiation (infrared), which, in turn, results in higher rate of mass transfer. This process facilitates drying of food material in shorter time with minimal changes in product quality, such as nutrient content and color, as compared to conventional drying methods (Abonyi et al., 2002; Raghavi et al., 2018; Durigon et al., 2018).

Optimal control of the drying process is fundamental and requires complete information on the drying behavior of the materials, requiring an accurate model capable of predicting water removal rates and describing the drying performance of each product under certain conditions (Khatchatourian et al., 2013). Mathematical models are an effective tool in the development, design, and improvement of drying systems and analysis of mass transfer phenomena during the drying process (Silva et al., 2014; Morais & Gut, 2015; Zarein et al., 2015; Qiu et al., 2018). Dincer & Dost (1995) developed analytical models to characterize the mass transfer during drying of objects presenting regular geometry (plate, cylinder, and sphere) and based on the assumption that the effective moisture diffusivity during drying process remains constant. This model is more simplified when compared to the diffusion model based on Fick's second law, and allows the determination of important parameters for the design, simulation, and optimization of the drying process.

However, to the best of our knowledge, there are no studies on mass transfer parameters and thermodynamic properties of the dehydrated tuber by Refractance Window (RW) technique in the literature. Therefore, this study aimed to evaluate the applicability of the analytical model developed by Dincer &

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Dost (1995) using the experimental data of *Dioscorea trifida* paste subjected to RW drying, and to determine the mass transfer parameters and thermodynamic properties involved in the drying process.

2 Materials and methods

2.1 Plant material and sample preparation

Dioscorea trifida (DT) tubers were purchased from the local market in Belém do Pará (Brazil) and transported to the laboratory. DT tubers were sanitized with a chlorinated solution at 200 mgL^{-1}. The bark (pericarp) was separated manually from the mesocarp using a knife. The mesocarp was washed, cut into approximately 1x1 cm pieces, and homogenized in a food processor (WALITA, RI 3148 SP, Brazil) until a homogeneous paste was obtained. The resulting paste was stored at -5 °C until used for further analysis.

2.2 Refractance Window drying

A batch scale laboratory-operated dryer was constructed using the same principle described by Costa et al. (2019), with some modifications (Figure 1). The drier consisted of a metal container (0.9 m x 0.15 m x 0.10 m) with circulation of hot water from a thermostatic bath (Quimis, Q214M2, Brazil), a digital PID temperature controller (Minipa, MT 1044, Brazil) and a 0.20 mm thick mylar film (type D, DuPont, USA). The mylar film was attached to the top of the metallic container. The drying temperatures were 70, 80, and 90 °C. The paste of DT was warmed at room temperature for 2 hours to reach the same equilibrium temperature before drying experiments. For drying the DT sample, 200 g of purple yam paste was spread over the surface of the mylar film forming a flat plate with a 50 mm side and 3.0 mm thickness. Both the initial moisture and the equilibrium moisture were determined according to the AOAC methodology No. 934.06 (Association of Official Analytical Chemists, 1990) using a vacuum oven (Marconi, MA030, Brazil) and an analytical balance (Shimadzu AY220, Japan) with an accuracy of ± 0.0001 g. The equilibrium moisture reached, as well as the time required to reach it, were specific to each drying regime studied (70, 80 and 90 °C). Each treatment was realized in triplicate.

2.3 Data analysis

In this study to explain the moisture transfer mechanism into the sample and at its surface during drying, the following hypothesis was considered: that the moisture transfer during drying is controlled by the diffusion mechanism. In this mechanism water moves from a region with high concentration toward a region at low concentration assuming that the moisture gradient inside biomaterial is the only driving force of the motion. Under this approach the following conditions have been assumed: (i) constant thermophysical properties of the solid and the drying medium; (ii) the effect of heat transfer on the moisture loss is negligible; (iii) The moisture diffusion in one-direction (perpendicular to the slab surface); (iv) and finite internal and external resistances to the moisture transfer within the solids (referring to $0.1<Bi_m<100$). Hence, the transient moisture diffusivity equation in Cartesian coordinates and dimensionless can be written in the following form (Akpinar & Dincer, 2005) (Equations 1 and 2):

$$\frac{\partial \varphi}{\partial t} = D\frac{\partial}{\partial y}\left(\frac{\partial \varphi}{\partial y}\right) \tag{1}$$

$$\varphi = W - W_e \tag{2}$$

where ϕ is moisture content difference (kg kg^{-1} d.b), D_m is moisture diffusivity ($m^2\ s^{-1}$), t is time (s) and y is space coordinate.

Equation 1 is subject to the following initial and boundary conditions (Equations 3, 4 and 5):

$$\varphi(y,0) = \varphi_i = (W_i - W_e) = \text{Cte.} \tag{3}$$

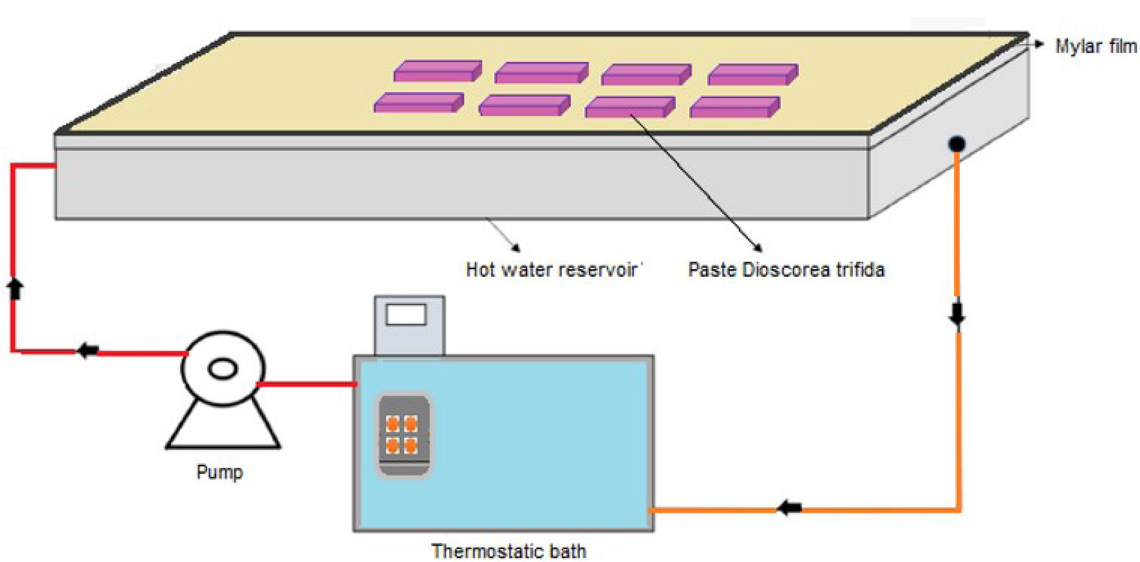

Figure 1. Schematic diagram of a batch-type RW drying system.

$$\left[\frac{\partial \varphi}{\partial y}(0,t)\right]=0 \quad (4)$$

$$-D_m\left(\frac{\partial \varphi}{\partial L}(L,t)\right)=k_m[\varphi(L,t)-\varphi_o] \quad (5)$$

where y = L is thickness (m) and k_m is moisture transfer coefficient, $m.s^{-1}$

The dimensionless moisture (Φ) content can be represented in terms of moisture content at any point of the solid object as (Equation 6):

$$\Phi=\frac{W-W_e}{W_i-W_e} \quad (6)$$

Solution to the governing equation (i.e., Equation 1) under the corresponding boundary conditions gives dimensionless moisture distribution at any point for the slab object is given as following form (Equation 7)(for details see Dincer & Dost, 1995; Dincer & Dost, 1996):

$$\Phi=\sum_{n=1}^{\infty} A_n \cdot B_n \quad (7)$$

The solution can be simplified when F_o> 0.2 values are negligibly small. Thus, the infinite sum in Equation 7 is well approximated by the first term only, i.e (Equations 8, 9 and 10) (Dincer & Dost, 1995):

$$\Phi \cong A_1 B_1 \quad (8)$$

where for slab geometry:

$$A_1=\exp[0.2533Bi/(1.3+Bi)] \quad (9)$$

$$B_1=\exp(-\mu_1^2 F_o) \quad (10)$$

Considering that drying has an exponentially decreasing trend, as proposed by Dincer & Dost (1996), the equation for the objects subject to drying, by introducing lag factor (G, dimensionless) and drying coefficient (S, 1 s^{-1}) is (Equation 11):

$$\Phi=G\exp(-St) \quad (11)$$

The drying coefficient shows the drying capability of an object or product per unit time and the lag factor is an indication of the internal resistance of an object to the heat and/or moisture transfer during drying. These parameters are useful to evaluate and represent a drying process. The value of the dimensionless moisture content can be obtained using the experimental moisture content measurements from Equation 6.

Both Equations 8 and 11 are in the same form and can be equated to each other by having G = A_1. Therefore, the moisture diffusivity for an infinite slab is given by the following Equation 12:

$$D_m=\frac{S L^2}{\mu_1^2} \quad (12)$$

where µ1 is the first root of the transcendental characteristic equation (Equation 7) and can be calculated with respect to Biot number (Bi) for slab geometry by using the following simplified expression (Equation 13) (Dincer & Hussain, 2002):

$$\mu_1=a\tan(0.640443Bi+0.380397) \quad (13)$$

The moisture transfer coefficients (k_m) can be obtained in terms of the lag factor used the Biot number (Bi) which is defined as (Equation 14):

$$B_i=\frac{k_m L}{D_m} \quad (14)$$

To determine the mass transfer parameters based on the Dincer and Dost model, the following procedure was applied:

i) Using the least square curve-fitting method, the dimensionless moisture content values and drying time were regressed in the form of Equation 11 and the lag factor (G) and drying coefficient (S) were determined;

ii) The Biot number was calculated through Equation 9;

iii) The value of μ_1 was determined from Equation 13;

iv) The moisture diffusivity was calculated using Equation 12;

v) The moisture transfer coefficient was obtained from Equation 14.

The dependence of D_{eff} on temperature can be determined by a simple Arrhenius expression (Equation 15):

$$D_m=D_o\exp\left(-\frac{E_a}{T_{abs}R}\right) \quad (15)$$

where E_a is the activation energy (kJ mol^{-1}), D_0 is the diffusivity value for infinite moisture content, R represents the universal gas constant, and T_{abs} is the absolute temperature. By plotting ln (D_m) *vs.* $1/T_{abs}$ diagram, E_a and D_0 coefficients can be subsequently related to drying air conditions through non-linear regression analysis.

The thermodynamic properties of the mass transfer process in *Dioscorea trifida* paste subjected to drying by RW was determined according to the method proposed by Jideani & Mpotokwana (2009) (Equations 16, 17 and 18):

$$\Delta H=E_a-RT_{abs} \quad (16)$$

$$\Delta S=R\left(\ln D_o-\ln\frac{k_B}{h_P}-\ln T_{abs}\right) \quad (17)$$

$$\Delta G=\Delta H-\Delta S.T_{abs} \quad (18)$$

where ΔH is the differential enthalpy, kJ mol^{-1}; ΔS is the differential entropy, $kJ.mol^{-1}.K^{-1}$; ΔG is the Gibbs free energy, kJ mol^{-1}; k_B is Boltzmann's constant, 1.38×10^{-23} J K^{-1}; and h_p is Planck's constant, 6.626×10^{-34} J s^{-1}.

Statistical analysis

All analyses were performed in triplicate (n = 3). The significance of the results was analyzed by a one-way analysis of variance (ANOVA). The parameters of the analytical model proposed by Dincer and Dost (Equation 11), Arrhenius equation (Equation 15) were estimated using the software Statistica for Windows 7.0 (StatSoft Inc., Tulsa, OK, USA). The fit quality of the proposed models for the drying kinetics data was estimated using the correlation coefficient (R^2) and the Chi-squared parameter (χ^2).

3 Results and discussion

The moisture content of fresh *Dioscorea trifida* tuber was 77.7 ± 0.1 g 100g^{-1}, and the moisture content of the dried *Dioscorea trifida* subjected to three different temperatures (70, 80, and 90 °C) ranged from 4.75 to 3.72 g 100 g^{-1} (on a wet basis). Figure 2 shows the behavior of the dimensionless moisture content of the sample with the drying time for three different temperatures, as well as the estimated values for the RW drying by the model proposed by Dincer and Dost (Equation 10). The behavior of the replicates for the RW-dried purple face sample showed an average difference between the values of dimensionless moisture, to the majority of the date, lower than the error of the dimensionless moisture measurement, proving the reproducibility of the drying experiments in the prototype assembled for this study, observed for all temperatures studied (Figure 2).

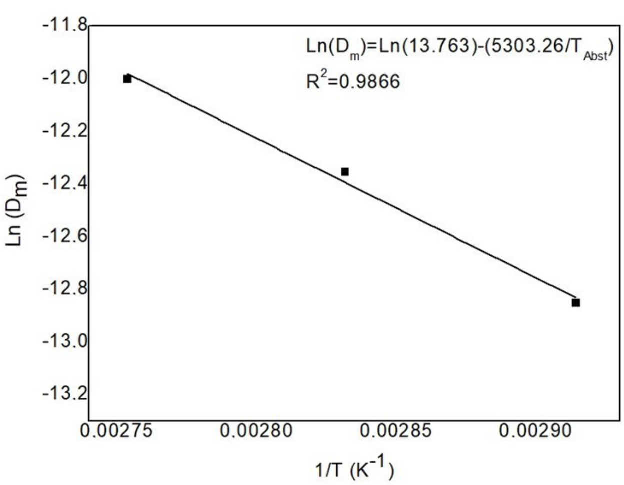

Figure 2. Experimental and predicted average dimensionless moisture content of *Dioscorea trifida*.

A similar trend was observed for the moisture content of the samples at different drying temperatures, with an exponential decrease with drying time, which was accentuated in the initial 10 minutes of drying and decreased slowly during the process. Therefore, at the end of drying, a lower effect of temperature on drying kinetics was observed, when compared to the beginning of the process, and the moisture transport was controlled by internal factors, i.e. the nature of the material.

Table 1 shows the parameters G and S, estimated by the nonlinear adjustment of the Dincer and Dost model (Equation 11) to the experimental data of the drying kinetics of *Dioscorea trifida* yam subjected to RW drying, the coefficients of determination (R^2) and the chi-square values (χ^2). It can be noted that S value increased and G value decreased with increasing temperature. The one-way ANOVA showed a significant and positive effect ($p < 0.05$) of temperature on G and S parameters. Similar behavior was also observed by Mrkic et al. (2007) in the convective drying of broccoli using the same geometry, and by Corzo et al. (2008) in the convective drying of mango slices at different ripening stages.

Regarding the application of the Dincer and Dost model (Equation 11), the high coefficients of determination ($R^2 > 0.96$) and the low chi-square values ($\chi^2 < 4.06 \times 10^{-3}$) indicate the adequacy of the model to the experimental data (Table 1).

Concerning the B_{iot} number (B_{im}), which represents the relationship between internal resistance and external resistance to mass transfer (when $0.1 < B_{im} < 100$) (Bezerra et al., 2015), as shown in Table 1, the B_{im} values ranged from 0.197 to 0.397 for the RW drying, which indicates the presence of internal and external resistance to moisture transfer Similar behavior was also observed by Rajoriya et al. (2019) in the convective drying of apple slices, with B_{im} values ranging from 0.128 to 0.594. The one-way ANOVA also showed a significant and positive effect ($p < 0.05$) of the temperature on B_{im} values, as expected, thus the temperature increase favored the decrease of the internal resistance, leading to a faster drying process. From a technological point of view, this behavior is an advantage of the RW drying method for the final product quality as it allows good retention of thermo sensitive bioactive compounds.

Using the values of Y, S, and μ_1, the moisture diffusivity (D_m) was then computed from Equation 12. Subsequently, the moisture transfer coefficient (k_m) values were computed by Equation 14. The calculated diffusivity (D_m) and moisture transfer coefficient (k_m) are presented in Table 1. According to the data, the D_m value increased with the increase in temperature from 70 to 90 °C for *Dioscorea trifida* yam subjected to RW drying. This phenomenon is associated with the lower viscosity of water with an increase in temperature. Whereas viscosity is a measure of fluid resistance to flow, the drying conditions have caused

Table 1. Drying coefficient and lag factor values obtained for RW drying of *Dioscorea trifida* paste.

T (°C)	G	S (s^{-1})	R^2	χ^2	B_{iot}	μ_1	$D_m \times 10^6$ (m^2 s^{-1})	$k_m \times 10^4$ (m s^{-1})	ΔH (kJ mol^{-1})	ΔS x10^2 (kJ mol^{-1} K^{-1})	ΔG (kJ mol^{-1})
70	1.060 ± 0.009	0.093 ± 0.002	0.9695	4.06×10^{-3}	0.397 ± 0.0074	0.565 ± 0.034	2.62 ± 0.021	3.46 ± 0.05	41.238	-22.43	118.207
80	1.042 ± 0.004	0.118 ± 0.001	0.9995	6.06×10^{-5}	0.252 ± 0.029	0.496 ± 0.014	4.31 ± 0.022	3.62 ± 0.06	41.155	-22.45	120.437
90	1.034 ± 0.001	0.150 ± 0.003	0.9998	1.60×10^{-5}	0.197 ± 0.007	0.469 ± 0.004	6.13 ± 0.090	4.04 ± 0.05	40.071	-22.47	122.671

an increase in water diffusion in the yam pulp, thus favoring drying (Wang et al., 2014). In addition, the higher effective diffusion coefficient may be due to rising temperatures can lead to an increase in the molecular vibration of water, which also contributed to a faster diffusion.

The magnitude of D_m (Table 1) is similar to those reported by several authors for different biological products using different methods of estimation, such as reported by Falade et al. (2007) found diffusivity values in the range of 0.829×10^{-6}-1.12×10^{-5} m^2 s^{-1} for convective drying of Yam (*Dioscorea alata*) between 50 and 80 °C and at constant air velocity (1.5 m s^{-1}); Mrkic et al. (2007) found diffusivities in the range of 3.58×10^{-6}-1.07×10^{-5} m^2s^{-1} for broccoli slices dried at 60 and 80 °C with a constant air velocity of 2.0 m/s, performed in convective dryer; Furtado et al. (2010) reported D_m values from 1.99×10^{-7} to 4.56×10^{-7} m^2 s^{-1}during drying of *seriguela* pulp using the foam-mat method at 70-80 °C; Guiné et al. (2011) found diffusivity values in the range of 4.08×10^{-8} to 2.35×10^{-7} m^2 s^{-1} for convective drying of pumpkin in the temperature range of 30 °C-70 °C; Aforabi et al. (2014) found diffusivities in the range of 5.27×10^{-8}-2.07×10^{-6} m^2 s^{-1} in drying of cocoyam slices in the temperature range of 50-70 °C for microwave drying; Bezerra et al. (2015) found D_m in the range of 1.05×10^{-8}-6.32×10^{-7} m^2 s^{-1} in the drying of passion fruit peel in the temperature range of 50-70 °C. One-way ANOVA revealed a significant positive effect ($p < 0.05$) of the temperature on moisture diffusivity.

The coefficient k_m calculated by the Equation 14 ranged from 3.46×10^{-4} to 4.04×10^{-4} m s^{-1}. The results of k_m found in this study corroborate the findings in the literature for different foods and drying conditions, such as reported by McMinn et al. (2003) studied potato slabs subjected to convective, microwave, and combined microwave–convective drying in the temperature range of 50-70 °C, found k_m in the range of 0.5×10^{-2} to 0.328×10^{-4} m s^{-1}. Mrkic et al. (2007) found k_m in the range of 1.921×10^{-4}-8.725×10^{-4} m s^{-1} for broccoli slices dried in the temperature range of 60-80 °C. Lemus-Mondaca et al. (2013) found k_m values ranging from 3.10×10^{-7} to 6.05×10^{-6} m s^{-1} in drying of papaya slices at temperatures between 40 and 80 °C. Bezerra et al. (2015) found k_m in the range of 4.53×10^{-7} to 6.062×10^{-7}m s^{-1} for drying of passion fruit peel in the temperature range of 40 at 60 °C. Arranz et al. (2017) during carrot drying at temperatures ranging from 40 to 70 °C, and k_m values in the range of 1.20×10^{-6} to 6.54×10^{-7} m s^{-1}. The influence of temperature on moisture transfer coefficient was positive and significant ($p < 0.05$) for the temperature ranging between 70 and 90 °C. There are few studies on the determination of k_m of yam in the literature, despite their importance for the evaluation of mass transfer or simultaneous heat and mass transfer processes.

Activation energy (E_a) was estimated using Equation 15. The E_a was calculated by plotting ln (D_m) vs the reciprocal of the absolute temperature ($1/T_{abs}$) as presented Figure 3. The results of such fitting gave a regression coefficient of 0.9985 indicating that the quality of such a fitting was good. The value obtained for the E_a in this study was 44.091 kJ/mol and presents reasonable agreement with the data reported by several authors for foods materials. As reported by Xiao et al. (2013), the activation energy for a typical drying operation should range from 12.7 to 110 kJ mol^{-1}. Falade et al. (2007) reported the activation energy ranging from 41.75 to 72.47 kJ mol^{-1} for the convective drying of *Dioscorea alata* and *Dioscorea rotundata* slices. Ju et al. (2016) reported activation energy of 29.53 kJ mol^{-1} for drying of *Dioscorea alata,* while Srikanth et al. (2019) found values from 25.18 to 32.46 kJ mol^{-1} during drying of *Amorphophallus paeonii folius* cubes. Several factors can affect the activation energy, including the variety, ripening stage, sample size, operating conditions, components, and tissue structure of *Dioscorea trifida*.

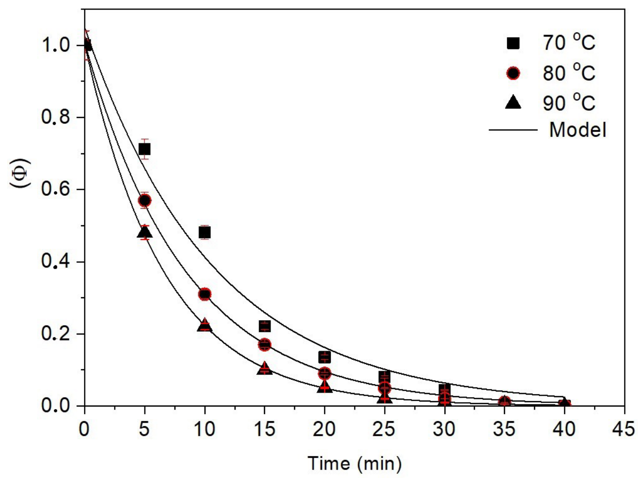

Figure 3. Relationship between moisture diffusivity (D_m) and temperature on purple yam dried by refractance window.

Thus, the effect of temperature on the D_m for RW drying of *Dioscorea trifida* paste can be represented by the following Equation 19:

$$D_m = 13.763 \exp\left(-\frac{5303.26}{T_{abs}}\right) \tag{19}$$

The thermodynamic properties observed for RW drying of *Dioscorea trifida* paste subjected to different temperatures are presented in Table 1. The enthalpy values (ΔH) were positive, pointing to endergonic reactions, that is, heat energy was necessary for the process. However, a reduction in energy demand from 41.238 to 41.071 kJ mol^{-1} was observed with an increase in drying temperature, which corroborates other studies on drying of other agricultural products (Beigi, 2016; Costa et al., 2016; Fayose & Huan, 2016; Chayjan et al., 2011).

All entropy values (ΔS) (Table 1) for RW drying of the *Dioscorea trifida* paste was negative (ΔS <0), indicating no significant increase in the system disorder. The ΔS values decreased with temperature increments, probably due to the decrease in moisture and restricted movement of water molecules, as there are few sites available during the dehydration process. This behavior can also be due to the formation of an activated complex when a substance can present negative entropy when the degree of freedom of translation or rotation is lost during the process.

In contrast to ΔH and ΔS, the Gibbs free energy (ΔG) increased with the increase in temperature, ranging from

118.207 at 122.671 kJ mol-1 for the temperature range studied. Positive ΔG values indicate that RW drying of *Dioscorea trifida* paste is a non-spontaneous process, once it requires additional energy from the environment around the product to reduce the water content. In this case, hot water was a source of external energy. Similar behavior was previously reported by Rajoriya et al. (2019).

4 Conclusion

Refractance windows drying is an effective tool in the drying process of tubers such as *Dioscorea trifida* yam. The drying of *Dioscorea trifida* paste using RW, can be predict by the model developed by Dincer and Dost with good accuracy and reliability to calculate D_m and K_m mass transfer parameters, within the ranges of other agricultural products and with high coefficients of determination and low chi-square values. Unlike other types of drying, it was possible to obtain powdered purple yam, at the three temperatures tested, in times of twenty-five to forty minutes, according to the temperature, in a simple and easy-to-build equipment.

The relationship between D_m and temperature can be described by the Arrhenius equation, which has activation energy of 44.091 kJ mol-1 for the RW drying of *Dioscorea trifida* paste. In addition, the thermodynamic properties pointed to a non-spontaneous process, with positive ΔH and ΔG values, and negative ΔS values. The ΔH and ΔS values decreased with increasing drying temperature, while ΔG values increased within the temperature range evaluated (70 to 90 °C). These findings can be used for the simulation and optimization of the drying process of *Dioscorea trifida* yam paste.

Declaration of competing interest

The authors declare no financial interests or personal relationships to influence the present study.

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