Vol. 18, No. 3 (2019) 851-864
Revista Mexicana de Ingeniería Química
Ingeniería de alimentos

WATER AND PHYTOCHEMICALS DYNAMIC DURING DRYING OF RED HABANERO CHILI PEPPER (*Capsicum chinense*) SLICES

DINÁMICA DEL AGUA Y FITOQUÍMICOS DURANTE EL SECADO DE RODAJAS DE CHILE HABANERO ROJO (*Capsicum chinense*)

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Received: August 20, 2018; Accepted: October 15, 2018

Abstract
Habanero pepper (*Capsicum chinense*) is a source of phytochemicals or bioactive compounds that have shown benefits in human health. For phytochemicals extraction process, a prior drying is required. Therefore, the aim of this study was to evaluate the effect of drying temperature (30, 50 and 70 °C) on total capsaicinoids and total carotenoids, and the coefficients of water diffusivity during drying of sliced red habanero pepper. The results show that the bioactive compounds, present a major stability at high temperature (70 °C), the effective diffusivity increased with drying temperature and the activation energy of the process was calculated to 39.575 kJ mol^{-1} K^{-1}. Several theoretical aspects of average water diffusivity are discussed.
Keywords: capsaicin, *Capsicum chinense*, carotenoids, convective drying, effective diffusivity, phytochemicals.

Resumen
El chile habanero (*Capsicum chinense*) es una fuente de fitoquímicos o compuestos bioactivos que han demostrado beneficios para la salud humana. El secado del fruto es una etapa importante previa a su extracción. Por lo tanto, el objetivo del presente estudio fue evaluar el efecto de la temperatura de secado (30, 50 y 70 °C) sobre la evolución de los capsaicinoides y carotenoides totales además de evaluar la difusividad promedio del agua durante el secado de rodajas de chile habanero rojo. Los resultados muestran que los compuestos bioactivos presentan buena estabilidad a 70 °C, que fue la temperatura mas alta probada. La difusividad efectiva aumenta con la temperatura de secado y la energía de activación del proceso es de 39.575 kJ mol^{-1} K^{-1}. Se discuten algunos aspectos teóricos de la difusividad promedio del agua.
Palabras clave: capsaicina, *Capsicum chinense*, carotenoides, secado convectivo, difusividad efectiva, fitoquímicos.

1 Introduction

Peppers (*Capsicum ssp.*) are important fruits that are widely used in food as a spice or as an ingredient. Capsicum fruits are a significant source of phytochemical compounds such as carotenoids and capsaicinoids. Different authors have shown that these compounds have antioxidant, antifungal and anti-carcinogenic properties and, therefore, it is possible to use peppers in a wide variety of applications in the pharmaceutical, chemical and food industries (Chinn *et al*., 2011; Giuffrida *et al*., 2013; Reyes-Escogido *et al*., 2011; Segura Campos *et al*., 2013; Wahyuni *et al*., 2011). The hotness of capsicum fruits is due to the presence of capsaicinoids within the vegetable material. Capsaicin is the most abundant capsaicinoid and it usually represents 50-85% of this group of compounds (Giuffrida *et al*., 2013). The *Capsicum chinense* (red habanero chili pepper) is a very important crop in Mexico, and demand for it is increasing in both national and international markets (Pino *et al*., 2007; Ruiz-Lau *et al*., 2011). *Capsicum oleoresin* is an important extract due to the content of bioactive compounds (capsaicinoids and carotenoids). Oleoresin must be extracted with solvent in solid-liquid extraction processes and these

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https://doi.org/10.24275/uam/izt/dcbi/revmexingquim/2019v18n3/Olguin
issn-e: 2395-8472

require that water is first removed from the chili fruit (Fernández, Gracia *et al*., 2012). Drying is one of the most traditional processes for food preservation and it is also used as a pretreatment of vegetable materials prior to extraction processes. However, it is well known that during hot-air drying, vegetables undergo physical, structural, chemical and nutritional changes that can affect quality attributes like texture, color, flavor, and nutritional value (Castañeda-Pérez *et al*., 2018; Montoya-Ballesteros *et al*., 2014). Drying involves simultaneous heat and mass transfer (Herman-Lara *et al*., 2005; Hernández-Díaz *et al*., 2013; García-Alvarado *et al*., 2014). Heat transfer properties in food are well understood (Herman-Lara *et al*., 2005; Hernández-Díaz *et al*., 2013) and therefore the design of a drying process requires the estimation of mass transfer properties. In this respect it is important to carry out a correct analysis of the diffusivity of water in order to optimize the process, to reduce operating costs and to maximize the quality of the pepper fruit. The most important property is the average water diffusivity, which can be evaluated from drying dynamics in the linear zone of moisture vs semi-log time representation (Páramo *et al*., 2010) and with the appropriate theoretical considerations with respect to product geometry. In addition to water diffusivity, it is important to evaluate the changes in the principal phytochemical compounds during the drying process to identify the appropriate conditions that result in the retention of phytochemicals. The drying dynamic both for mass transfer and phytochemical evolution is important in the development of an integrated approach for the drying process vs compounds that have human health benefits, i.e., to optimize nutrient retention. In the work reported here, the evolution of water and principal phytochemicals (capsaicin, dihydrocapsaicin and total carotenoids) of red habanero chili pepper (*Capsicum chinense*) fruits during a drying process at different temperatures (30, 50 and 70 ºC) and a constant air velocity (1.5 ms^{-1}) were evaluated. The water dynamic was assessed in order to estimate the average water diffusivity. The lack of published work on the air-drying of red habanero chili pepper and the evolution of water and phytochemicals during drying justifies the interest in the present work.

2 Materials and methods

2.1 Chemicals

Hexane and acetone (analytical grade) were acquired from JT Baker (Ecatepec, Morelos, Mexico). Ethanol, NaCl and magnesium carbonate (analytical grade) were obtained from Golden Bell Reagents (Guadalajara, Jalisco, Mexico). The water was obtained from a Milli-Q water deionization system (Millipore, Bedford, MA, USA). The methanol used for the chromatographic separation was HPLC grade and was purchased from Merck (Darmstadt, Germany). The reference standards of capsaicinoids, i.e., capsaicin (97%) and dihydrocapsaicin (90%), were purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO, USA).

2.2 Plant material

Good-quality fresh red habanero chili pepper fruits were acquired from the local market of Veracruz, Ver., Mexico. The fruits were then washed with water to remove dust and stored at 4 °C. The fruits were sliced to 5 ± 1 mm with a mandolin vegetable slicer (METALTEX model 194875). The final sliced average dimensions were 26.66 mm of diameter and 1.92 mm of wall thickness.

2.3 Drying kinetics experiments

A tray dryer (Apex Water Systems & Lab Equipment, Model A39854-14, Chennai, Tamil Nadu, India) was used to perform the drying kinetic experiments. The air velocity was set at 1.5 ms^{-1} and measured with a digital anemometer with turbine (PROVA, model AVM-01). The temperatures evaluated were 30, 50 and 70 °C. Pepper samples were dried in a thin layer. Representative samples were taken at different times in each drying process. The samples were immediately vacuum packed in polyethylene bag, using a vacuum food packer (FoodSaver 3800 series), and stored at -20 °C prior to analyses. Drying curves were obtained by periodic determination of the weight and moisture content of the red pepper samples. Each drying kinetic was replicated three times.

2.3.1 Water determination

1 g of red habanero chili pepper slices was placed in a vacuum oven (Lab-Line Instruments model 3618-1)

at 60 °C, 60 kPa until constant weight was achieved. The water content (dry or wet basis) was calculated by weight difference. The analysis was performed in triplicate.

2.3.2 Total capsaicinoids determination

Capsaicin and dihydrocapsaicin contents were determined according to the method reported by Barbero *et al*. (2008) with some modifications. 0.5 g dry matter (d.m.) of fruit was placed in 50 mL of absolute ethanol at 50 ºC and an ultrasound-assisted extraction was carried out in an ultrasonic bath (Westprime Systems Inc.; mod. B90-055H, Chino, California, USA) with a volume of 3.6 L at 45 kHz and 100 W. The separation and quantification of capsaicinoids were carried out on a UHPLC (ACQUITY UPLC H-Class, Waters, Milford, MA, USA) system equipped with an ACQUITY UPLC Quaternary Pump System, an ACQUITY UPLC Auto Sampler with temperature control adjusted at 15 °C, a column oven set at 50 ºC for the chromatographic separation and an ACQUITY UPLC® Fluorescence (FLR) Detector. Empower 3 software (Waters) was used to control the equipment and for data acquisition. Capsaicin and dihydrocapsaicin were analyzed on a Waters ACQUITY UPLC BEH C18 column (50 × 2.1 mm I.D., particle size 1.7 μm). The chromatographic method involved a gradient with two solvents: acidified water (0.1% acetic acid, solvent A) and acidified methanol (0.1% acetic acid, solvent B), using a solvent flow of 0.8 mL min^{-1}. The gradient used for the chromatographic separation of the capsaicinoids (time, solvent B) was: 0 min, 10%; 2 min, 50%, 4 min, 50%; 4.5 min, 55%; 5.5 min, 55%; 6 min, 60%; 7 min, 60%; 9 min, 70%; 10 min, 100%; 15 min, 100%. The temperature of the column was kept constant at 50 °C. The wavelengths employed for fluorescence detection were 278 nm (excitation) and 310 nm (emission). The injection volume was 3 μL. The total capsaicinoids content is expressed as the sum of capsaicin and dihydrocapsaicin. Each determination was performed in duplicate.

2.3.3 UHPLC calibration

The UHPLC method was used to obtain calibration curves for C and DHC (y = 2046823.16x + 61597.37 for C and y = 2218821.73x + 45669.04 for DHC), which are the two capsaicinoid standards that are commercially available. Regression equations and correlation coefficients (R^2) were calculated using Microsoft Office Excel software (0.9998 for C and 0.9999 for DHC). The limits of detection (0.054 mg L^{-1} for C and 0.046 mg L^{-1} for DHC) and quantification (0.181 mg L^{-1} for C and 0.153 mg L^{-1} for DHC) were determined as the analyte concentration corresponding to the standard deviation of the signal of the blank values (n = 10) plus 3 or 10 times, respectively, divided by the slope of the linear regression.

2.3.4 Total carotenoids determination

The total carotenoids (TC) determination was performed according to AOAC method 941.15 (AOAC, 2006) for dried plants, with slight modifications based on Rodriguez-Amaya and Kimura (2004) and Vazquez *et al*. (2017). The dried samples were extracted with 25 mL of a mixture of acetone/hexane (30:70 v/v) for 30 min. Fresh samples were ground and macerated (with a mortar and pestle) in 25 mL acetone/hexane (4:6) and 0.1 g of magnesium carbonate for an analogous extraction to the dry sample. Each extract was then partitioned with water and a solution of 5% NaCl to remove the acetone. The upper phase (hexane + carotenoids) was recovered and reconstituted with hexane to a volume of 25 mL. The sample absorbance at 450 nm was determined with a UV-visible spectrophotometer (Thermo Fisher Scientific, Mod. Genesys 10S, Waltham, Massachusetts, USA). The total carotenoids were calculated using the equation:

$$C = \frac{\left[A_{450}/\left(A_{1cm}^{1\%}100\right)\right]V}{w_s}10^3 \tag{1}$$

Where C is the total carotenoids content in μg/g of sample; A_{450} is the absorbance at 450 nm; $A_{1cm}^{1\%}$ is the absorbance in a 1 cm cell of a carotenoid solution at 1 mg/100 mL (0.25 at 450 nm) reported in Rodriguez-Amaya and Kimura (2004); V is the reconstituted solution volume (25 mL); and w_s is the sample dry matter (g). The determinations were carried out in duplicate.

2.4 Estimation of average water diffusivity

Páramo *et al*. (2010) demonstrated that the average diffusivity of water during drying must be evaluated within the linear zone of the semilog representation of dimensionless moisture (Ψ) vs time, fitted to the appropriate analytical solution of Fick's second law. Chili pieces are hollow cylinders (Fig. 1) of 1 mm or less initial thickness and 5 mm or less initial height.

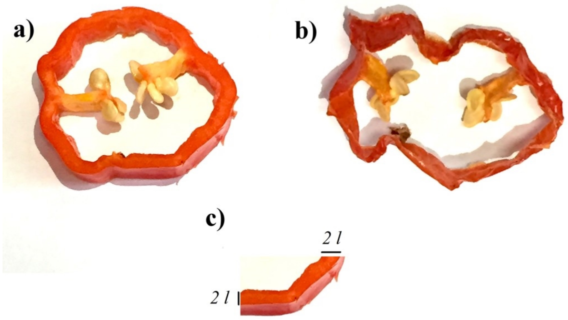

Fig. 1. Red habanero chili pepper slices (a) dried slices (b) and the half thickness of chili slices l (c).

Given this geometry and size, the samples may be approximated to a 2D flat slab drying on both sides. The analytical solution of Fick's second law for a 2D flat slab in contact with an infinite volume solution and interfacial resistance negligible (Vargas-González *et al*., 2017) is,

$$\Psi = \frac{X - X_e}{X_0 - X_e} = \frac{8^2}{\pi^4} \sum_{n=1}^{\infty} \sum_{k=1}^{\infty} \frac{1}{(2n-1)^2(2k-1)^2} \exp\left\{ -\left[\frac{(2k-1)^2\pi^2}{4} \left(\frac{l}{l_y} \right)^2 + \frac{(2n-1)^2\pi^2}{4} \right] \frac{Dt}{l^2} \right\} \quad (2)$$

Where X, X_0 and X_e are instant, initial and equilibrium moisture contents on a dry basis, l is the half thickness of a chili slice wall, l_y is the half height of a chili slice, D is the average water diffusivity and t is the time.

Therefore the asymptotic behavior is,

$$\Psi = \frac{8^2}{\pi^4} \exp\left\{ -\left[\frac{\pi^2}{4} \left(\frac{l}{l_y} \right)^2 + \frac{\pi^2}{4} \right] \frac{Dt}{l^2} \right\} \quad (3)$$

In semilog (Ψ vs t) representation,

$$\ln \Psi = \ln\left(\frac{8^2}{\pi^4} \right) - \left[\frac{\pi^2}{4} \left(\frac{l}{l_y} \right)^2 + \frac{\pi^2}{4} \right] \frac{Dt}{l^2} \quad (4)$$

Or

$$\ln \Psi = \delta + mt \quad (4a)$$

With,

$$m = -\left[\frac{\pi^2}{4} \left(\frac{l}{l_y} \right)^2 + \frac{\pi^2}{4} \right] \frac{Dt}{l^2} \quad (5)$$

Where m is the slope of semilog representation of Ψ vs t in the linear zone and δ is the intercept of Eq. (4a) but it is not necessarily the expression of Eq. (4) because at the beginning of drying the sample temperature is not equal to drying medium. However, Páramo *et al*., (2010) demonstrated that the slope of Eq. (4) and (4a) is defined by Eq. (5) independently of initial drying stages. The average water diffusivities were estimated from the slope obtained from linear regression of experimental $\ln \Psi$ vs t in the linear zone at different temperatures. The 95% confidence interval for the slopes (and therefore for diffusivities) were calculated with,

$$m_0 = m \pm t_{0.975(v)} \sqrt{s_m^2} \quad (6)$$

Where m_0 represents the 95% confidence interval for the linear regression calculated slope (m), s_m^2 is the estimated variance of the linear parameter (m), $t_{0.975(v)}$ is the abscissa of the t-student cumulative probability function for 0.975 probability, v represents the degrees of freedom ($v = n - 2$) and n is the number of drying dynamics points. If the half thickness of the chili slice wall is smaller than the half height of the chili slice, such that $\left(l/l_y\right)^2 \ll 1$, then Eqs. (2) to (5) would be reduced to Fick's second law analytical solution for a 1D flat slab.

The effect of absolute temperature (T) on water average diffusivities was assumed to follow an Arrhenius dependency,

$$\ln D = \ln D_0 - \frac{E_a}{RT} \quad (7)$$

Where E_a is the activation energy for diffusion ($J \cdot moL^{-1}$) and R is the gas constant (8.3143 $J \cdot moL^{-1} \cdot K^{-1}$).

3 Results and discussion

3.1 *Drying kinetics of red habanero chili pepper*

According to expectations, treatment with the lowest temperatures led to longer drying times, whereas the use of higher temperatures required less time for drying due to increase in the temperature difference between the drying air and the product and to the resultant water migration. The loss of moisture with respect to the time at different temperatures for red habanero chili pepper slices is represented in Fig. 2. Other authors reported different drying times depending on the geometries of samples employed. For example, Kaleemullah and Kailappan (2006) reported drying times of 26.0-13.0 h at 50-65 °C for whole fresh samples.

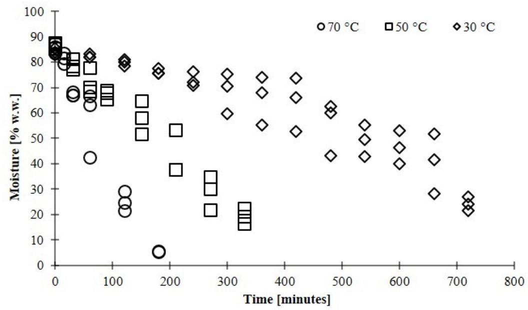

Fig. 2. Batch drying curves for red habanero chili pepper slices (n = 3) at 30 °C, 50 °C, and 70 °C.

When the fruits are sectioned, the contact surface increases and the drying time is reduced. For halved fruits, Pontes *et al*. (2009) reached similar moisture contents at 10.0-2.3 h at 50-70 °C in their study of smell-pepper drying. In the work reported here, low moisture levels were reached in less time because the product was cut into 5 mm slices before drying. The same final moisture contents at different temperatures (50, 60 and 70 °C) were reached after drying times of 13.3, 5.0 and 1.5 h, respectively (Fig. 2). On using similar temperatures and geometries, the drying process is approximately three times faster in comparison with the results of Faustino *et al*. (2007), who reported drying times for slices of 3-4 mm thickness of green bell pepper of 36.5 h at 30 °C and 5 h at 70 °C. However, the required time to reach constant weight for these samples differs from results reported by other authors under similar conditions. As example Arslan and Özcan (2011) reported 25 h and 9 h at 50 °C and 70 °C respectively during red bell-pepper slices (1 cm thickness) drying. Vega *et al*.(2007) required 9.3 h at 50 °C and 4.7 h at 70 °C for red bell pepper (*Capsicum annuum* L.) cubes (1 cm^3) drying. These differences could be explained due to the physico-chemical properties of different capsicums (composition, cultivar and ripening stage). Drying can be modeled in order to understanding the process phenomenon also to optimize the process. However, it is essential to define the geometry and the dimension of the system, which implies the direction of heat and mass transfer (diffusivity of water) in the defined geometry (Castro, Mayorga, & Moreno, 2018). In section 3.3 some modeling concepts of the drying process will be discussed.

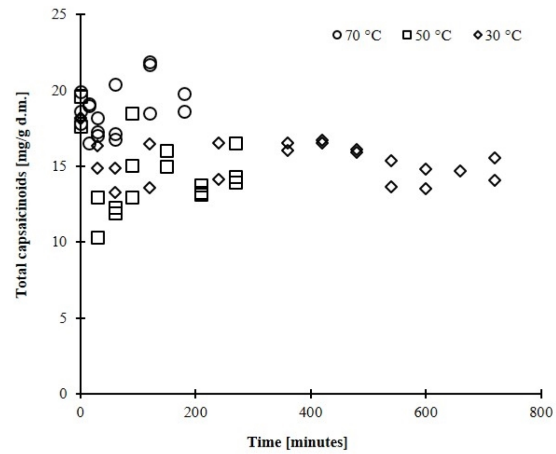

Fig. 3. Total capsaicinoids evolution during drying of red habanero chili pepper slices (n = 2) at 30 °C, 50 °C, and 70 °C.

3.2 Bioactive compounds during drying kinetics

3.2.1 Total capsaicinoids

The red habanero chili pepper is widely known in México as the hottest pepper due to its high concentration of capsaicinoids (Cichewicz & Thorpe, 1996; Pino *et al*., 2007). In this work the initial concentration of total capsaicinoids were 18.06 ± 0.83 mg/g dry matter, lower than the concentration reported by Cisneros-Pineda *et al*., (2007) for orange habanero pepper (62.89 mg/g). The capsaicinoids variation among chili pepper could be depends on the maturity, harvest time, variety, agricultural practices, and environmental factors (Barbero *et al*., 2014; Howard *et al*., 2000).

The evolution of total capsaicinoids during convective drying is represented in Fig. 3. A 19.04 ± 0.93 % decrease of capsaicinoids at the beginning (t <100 min) of drying at 30 and 50 ºC was observed, but capsaicinoids remains constant the rest of process. This behavior had been observed by Montoya-Ballesteros *et al*. (2017) during drying of *Capsicum annuum* L. var. *glabriusculum*, and they concluded that the effect may be due to peroxidase (POD) activity. At 30 and 50 ºC the POD activity explains the initial decrease of capsaicinoids but water lost during drying produces POD inactivation (Bernal *et al*., 1993).

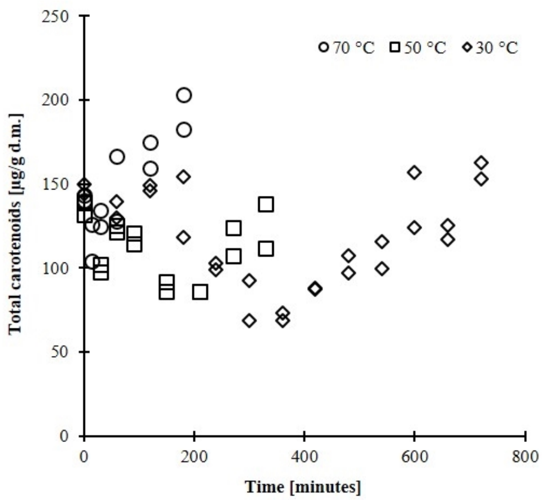

Fig. 4. Total carotenoids evolution during drying of red habanero chili pepper slices (n = 2) at 30 °C, 50 °C, and 70 °C.

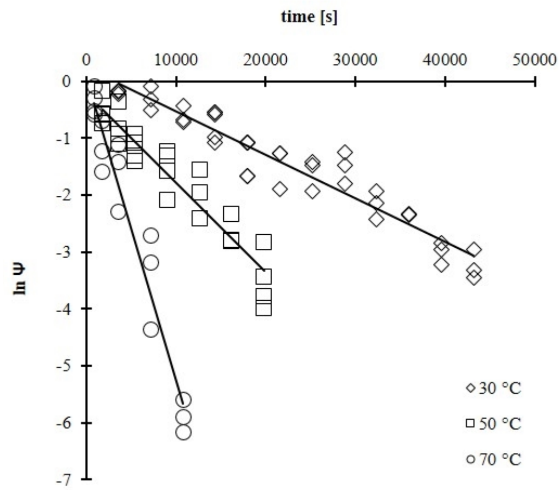

Fig. 5. Natural logarithm of moisture and time relation for red habanero chili pepper slices dried at 30 °C, 50 °C, and 70 °C.

3.2.2 Total carotenoids

The characteristic intense red color of this fruit is due to carotenoid pigments that are mainly synthesized during fruit ripening (Menichini *et al*., 2009). The initial concentrations of total carotenoids were 141.2 ± 6.1 μg/ g dry matter. This concentration is higher in comparison with the values reported by Segura-Campos (2013) for different habanero chili pepper genotypes, which ranged from 10.0 to 12.6 μg/ g sample or by Howard (2000) of 86.6 μg/g dry matter. The evolution of total carotenoids during the drying process at different temperatures is shown in Fig. 4.

The total carotenoids concentration apparently increases during convective drying at high temperatures (70 °C), but at low temperatures the total carotenoids concentration presents variations. These results are consistent with those reported by other authors and this suggests that biosynthesis and continuous degradation of carotenoids occur during drying (Kevrešan *et al*., 2009; Topuz *et al*., 2009). The slow drying allows under-ripening that can modify, by biosynthetic processes, initial concentration of the yellow (zeaxanthin, β-crytptoxanthin and β-carotene) fraction, the red (capsorubin and capsanthin) fraction or both (Mínguez-Mosquera *et al*., 1994). At the same time, the fruit moisture content, together with the variables temperature and drying time, determine the changes (whether positive, negative, or none) in the fruit carotenoid concentration. After the first minutes, the total carotenoids in fruits, metabolically active, maintain complete their enzymatic capacity reached their concentration, not only overcoming the initial loss but reaching concentrations higher than those in the fresh fruit. At minor water content in the sample only slight oscillations around the maximum carotenoid total content was determinate. As a consequence, the total carotenoids apparently remain constant. However, this situation differs from that reported by other authors (Di Scala & Crapiste, 2008; Minguez-Mosquera *et al*., 1994), who found that a higher concentration of carotenoids is observed on increasing the process temperature, which may be due to the shorter drying times. This means that the degradation of the total carotenoids decreases when the residence time is shorter.

These results indicate that to optimize the retention of bioactive compounds it is not necessary to employ a mathematical model for total carotenoid and total capsaicinoids evolution because these compounds remain constant (or increase) upon drying between 40 and 70 ºC. The time of process is then the principal factor for the bioactive compounds preservation, for that the determination of average diffusivity of water vapor allow the knowledge and optimization of process. In the next section the average diffusivity determination is discussed.

3.3 Effective diffusion and activation energy coefficients

Average water diffusivities at the three temperatures were estimated with Eqs. (3) to (5) from the linear zone of the experimental $\ln\Psi$ vs t (Fig. 5) with a 95%

confidence interval. The lower and upper limits are listed in Table 1. The relevance of the fit of Eq. (3) in the linear zone of experimental $\ln \Psi$ vs t is shown in Fig. 5 because, at the beginning of drying dynamic, the behavior is not linear, partially due to the initial effect of Eq. (2) (Páramo *et al*., 2010) and partially due to the initial heat transfer (García-Alvarado *et al*., 2014; Herman-Lara *et al*., 2005). The average of half wall thickness of the slices (Fig. 1) during drying was 0.76 mm and the average slice height was 4.59 mm, therefore $\left(l/l_y\right)^2 \approx 0.03$ and it is not clearly defined whether the 2D solution is required. The average diffusivities were then calculated with Eq. (5) for both the 1D and 2D cases. The results showed a maximum difference of 3% in diffusivity values and therefore the diffusivities were calculated with the 1D assumption. The average diffusivities estimated by this method show that the temperature had a significant effect ($p < 0.05$) because all 95% confidence intervals overlap (Table 1). The average diffusivities obtained differ to those reported by other authors, as can be seen from the data in Table 2. It is possible to speculate that the differences are due to sample shape and size. Some of the values reported in Table 2 are complete outliers. It should be noted that NaCl in water has a diffusivity of around 2×10^{-9} m^{2}s^{-1} (a solute in liquid water). Therefore, the average water diffusivities reported in the order of 10^{-8} m^{2}s^{-1} in Table 2 (Vega *et al*., 2007) are an order of magnitude outside the aforementioned value. In both cases the sample size (characteristic length for diffusion l) may be incorrectly estimated: in one case this value is reported as 1 cm (extremely high) and in another case this distance was not reported. It is important to note that the analytical solution of the mass transfer equation must be selected with the coordinate system that best represents the samples and the appropriate length must be selected. Otherwise, the average diffusivities would be overestimated or under estimated.

As expected, the values obtained for the diffusion coefficient increased with temperature from 1.661 – 2.035×10^{-11} m^{2}s^{-1} at 30 °C to 9.674 – 13.428 × 10^{-11} m^{2}s^{-1} at 70 °C; which demonstrates a decrease in the residence time or in the drying process at higher temperature (Table 1). The Eq. (7) parameters resulted in E_a = 39.575 kJ gmoL^{-1} K^{-1} (Fig. 6) and $D_0 = 1.197 \times 10^{-4}$ m^{2}s^{-1}. Great differences, up to 2 magnitude orders, can be observed with respect to water diffusivities reported by other authors listed in Table 2.

Table 1. Confidence interval (95%) of effective diffusivity at different drying air temperatures.

	Confidence interval of $D_{ef} \times 10^{11}$[m^2/s]		
Temperature	**Lower limit**	**Estimated**	**Upper limit**
30	1.661	1.848	2.035
50	4.098	4.62	5.143
70	9.674	11.551	13.428

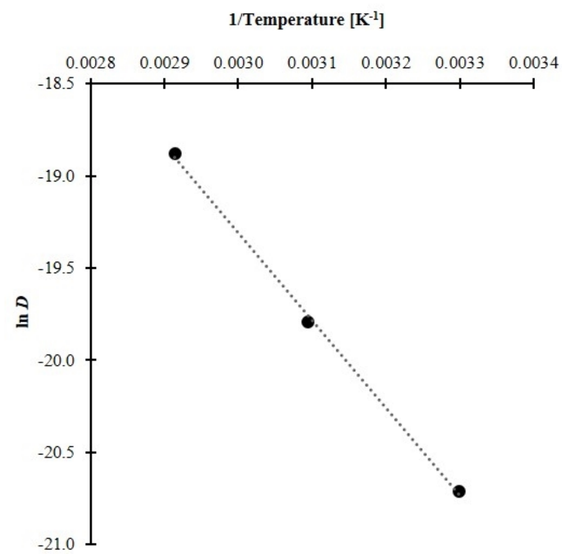

Fig. 6. Representation of Arrhenius equation for the temperature and effective diffusivity relation for red habanero chili pepper slices.

The only explanation for such differences is the characteristic length for diffusion elected. As example, Vega *et al*., (2007) reported water diffusivities of $0.32 - 1.12 \times 10^{-8}$ m^2/s for 50-80 oC. These values are excessive; taking into account that typical diffusivity of small molecules in liquid phase at 50-80 oC is in the order of $1 - 3 \times 10^{-9}$ m^2/s. It is possible that Vega *et al*., (2007) used 0.005 m as characteristic length for diffusion (Table 2), which is the half of cube length but the cube may be hollow like the hollow cylinders of Fig. 1, and therefore this water diffusivity overestimated. The water diffusion in fruits like chili occurs in the fruits wall like can be observed in Fig. 1 and considered in Eq. (4).

Table 2. Effective diffusivity and activation energy of different capsicum fruits at different drying air temperatures.

Author	***Capsicum* fruits**	**Evaluated drying air temperatures (°C)**	**Ea [kJ/(mol K]**	$D \times 10^{11}$ **[m^2/s]**	**Geometry descriptions**	**Applied equation**
Faustino *et al*. (2007)	Green bell pepper	30 °C-70 °C	47.10	90.0-800.0	Slices, diameter: 2.5 cm. Thickness: 3-4 mm	$\Psi = \frac{4}{b_1^2} e^{-Db_1^2 t/r^2}$
Vega *et al*. (2007)	Red bell pepper (Var. *Lamuyo*)	50 °C-80 °C	39.70	320.0-1120.0	Cubes of 1 cm without seeds and placenta	$\Psi = \frac{8}{\pi^2} e^{-D\pi^2 t/4l^2}$
Di Scala *et al*. (2008)	Red pepper	50 °C-90 °C	33.83	50.1-83.2	Slices, 2 x 2 cm Thickness: 5 mm	$\Psi = \frac{8}{\pi^2} e^{-D\pi^2 t/4l^2}$
Arslan and Özcan (2011)	Red bell-pepper	50 °C and 70 °C	NR	40.0 and 131.0	Slices Thickness: 1 cm	$\Psi = \frac{8}{\pi^2} e^{-D\pi^2 t/4l^2}$
Melo, Pereira *et al*. (2015)	Hot pepper (Var. *Cheiro*)	50 °C-70 °C	38.66	89.0-229.3	NR	$\Psi = \frac{8}{\pi^2} e^{-D\pi^2 t/4l^2}$
Deng *et al*. (2017)	Red pepper (*Capsicum annuum* L.)	50 °C-80 °C	48.90	13.8-68.7	Fresh fruit, diameter: 6.3 cm, height: 1.2 cm.	$\Psi = \frac{8}{\pi^2} e^{-D\pi^2 t/4H^2}$
Present work	Red Habanero chili pepper	30 °C-70 °C	39.57	1.7-13.4	Rings, diameter: 2.7 cm, height: 5 mm Fresh wall thickness: 1.92 mm	$\Psi = \frac{8}{\pi^2} e^{-D\pi^2 t/4l^2}$

NR: not reported

As it was stated, food drying involves simultaneous heat and mass transfer in which the heat transfer properties are well understood and therefore the process design requires the estimation of mass transfer properties. (Herman-Lara *et al*., 2005; Hernández-Díaz *et al*., 2013; García-Alvarado *et al*., 2014). These mass transfer properties are the Eq. (7) parameters for habanero pepper which can be directly applied in batch bed drying simulation and its thermal analysis through the Luikov-García Equations (García-Alvarado *et al*., 2014) jointly solved with heat and mass balances in drying air (Heman-Lara *et al*., 2005) and energy equations (Hernández-Díaz *et al*., 2013).

3.4 Simulation of batch bed drying

Some confusion exists over the difference between particle drying and batch bed drying (Vega *et al*., 2007; Castañeda-Pérez *et al*. 2017). Vega *et al*., (2007) described the batch drying of a red bell pepper bed (loading 7 kg/m^2) and used Eq. (4) for diffusivity estimation. Similarly, Castañeda-Pérez *et al*. (2017) describe the batch drying of vibro-fluidized bed for diffusivity estimation. The main difference between particle and batch bed drying is the air variables: in particle drying, air temperature and moisture may be considered constant because the air mass flow is in excess greater than solid particle. In the case of batch bed drying the evaporated water produces an increase in air moisture and the evaporation latent heat produces a decrease in air temperature. Therefore, diffusivities from fixed bed drying have an error in air temperature and in the interface resistance assumptions. In order to demonstrate the described effect a batch bed drying of habanero red pepper was implemented and the air outlet temperature was followed during process. Additionally, the process was simulated by using Luikov- García Equations jointly with heat and mass balances in drying air and the diffusivities estimated in section 3.3.

Luikov-García Equations (García-Alvarado *et al.*, 2014) in 1D rectangular coordinate expressed in dimensionless time (τ) and coordinate axe (ξ) in an ideal mixed stage (j) of a fix bed dryer are,

Heat and mass transfer within red habanero pepper,

$$\frac{\partial T_{\beta j}}{\partial \tau} = \frac{\partial}{\partial \xi}\left(\alpha \frac{\partial T_{\beta j}}{\partial \xi}\right) \quad \text{in} \quad 0 \leq \xi \leq 1 \tag{8}$$

$$\frac{\partial X_{\beta j}}{\partial \tau} = \frac{\partial}{\partial \xi}\left(\phi \frac{\partial X_{\beta j}}{\partial \xi}\right) \quad \text{in} \quad 0 \leq \xi \leq 1 \tag{9}$$

Where, $\tau = D_{ref} t/l^2$, $\xi = z/l$, $\phi = D_0 e^{-E_a/RT}/D_{ref}$, $\alpha = k_\beta / C_p \rho_\beta D_{ref}$.

Heat and mass transfer at interface,

$$-\frac{\partial T_{\beta ji}}{\partial \xi} = \frac{hl(T_{\beta ji} - T_{\gamma j}) + k_c l \rho_\gamma (X_{\gamma ji} - X_{\gamma j})\lambda_{wv}}{k_\beta}, \quad \text{at} \quad \xi = 1 \tag{10}$$

$$-\frac{\partial X_{\beta ji}}{\partial \xi} = \frac{k_c l \rho_j (X_{\gamma ji} - X_{\gamma j})}{D_0 e^{-E_a/RT} \rho_{\beta s}}, \quad \text{at} \quad \xi = 1 \tag{11}$$

The temperature ($T_{\beta ji}$) and moistures ($X_{\beta ji}$, $X_{\gamma ji}$) at the interface are related with Raoul's law at interface (García-Alvarado *et al.*, 2014),

$$X_{\gamma ji} = \frac{a_{wj} P_{wj}/P}{1 - a_{wj} P_{wj}/P} \frac{18}{29} \tag{12}$$

with Henderson-García (García-Alvarado *et al.*, 1995) sorption isotherm equation,

$$a_{wj} = 1 - \exp\left(k_1 T_{\beta ji}^{k_2} X_{\beta ji}^{k_3 + k_4 + k_5 T_{\beta j}^2}\right) \tag{13}$$

and extended Antoine Equation (Perry *et al.*, 1997) for water vapor pressure,

$$P_{wj} = \exp\left(73.649 - 7258.2/T_{\beta ji} - 7.3037 \ln T_{\beta ji} + 4.1653 \times 10^{-6} T_{\beta ji}^2\right) \tag{14}$$

Heat and mass balances in drying air describes its temperature and moisture dynamic which are neglected in the studies where diffusivities were estimated from Eq. (4) and fixed bed drying kinetics. Referred balances in drying air (Hernan-Lara *et al.*, 2005) expressed in dimensionless time in an ideal mixed stage of fix bed dryer are,

$$\frac{\partial H_{\gamma j}}{\partial \tau} = \frac{k_c l^2 \rho_\gamma a (X_{\gamma ji} - X_{\gamma j})\lambda}{\varepsilon \rho_j D_{ref}} + \frac{hl^2 a (T_{\beta ji} - T_{\gamma ji})}{\varepsilon \rho_\gamma D_{ref}} + \ldots \tag{15}$$

$$\frac{\partial X_{\gamma j}}{\partial \tau} = \frac{k_x l^2 \rho_\gamma a (X_{\gamma ji} - X_{\gamma j})}{\varepsilon \rho_\gamma D_{ref}} - \frac{G_\gamma l^2 (X_{\gamma j} - X_{\gamma j-1})}{\varepsilon \rho_\gamma D_{ref} V_j} - \frac{h_{out} a_{out} l^2 (T_{\gamma j} - T_{out})}{\varepsilon \rho_\gamma D_{ref}} - \frac{G_\gamma l^2 (H_{\gamma j} - H_{\gamma j-1})}{\varepsilon \rho_\gamma D_{ref} V_j}, \quad \text{in } V_\gamma \tag{16}$$

Where air enthalpy is defined as,

$$H_{\gamma j} = C p_a T_{\gamma j} + (H_{wv}^0 + C p_{wv} T_{\gamma j}) X_{\gamma j} \tag{17}$$

The energy required (Hernández-Díaz *et al.*, 2013) for air drying heating is,

$$E_0 = \int_0^t G_\gamma (H_{\gamma 0} - H_{\gamma out}) dt \tag{18}$$

The energy applied in water evaporation,

$$E_1 = \int_0^t \sum_{j=1}^{n} k_c \rho_\gamma a (X_{\gamma ji} - X_{\gamma j}) \lambda dt \tag{19}$$

Therefore Eq. (20) may be expressed as,

$$\frac{dE_1}{d\tau} = \sum_{j=1}^{n} \frac{k_c l^2 \rho_\gamma a (X_{\gamma ji} - X_{\gamma j})\lambda}{D_{ref}} \tag{20}$$

And the overall thermal efficiency is the ratio between the energy applied in water evaporation over the energy required in air,

$$\eta = \frac{E_1}{E_0} \tag{21}$$

Eqs. (8) to (11) were discretized in the position derivative and solved jointly with (15), (16) and (21) as an ordinary differential equations (ODE) system. Eq. (8) to (22) were solved for every ideal mixed stage (j) assuming that the fix bed is represented by 1 (N = 1), 5 (N = 5) and 10 (N = 10) ideal mixed stages. The total of properties required and the experimental conditions are listed in Table 3.

Table 3. Thermodynamic and transfer properties jointly with process variables used in Eqs. (8)-(21)

Property or Variable	Value or Eq.	Reference
D_{ref}	1.13×10^{-10}m^{2}s^{-1}	Experimental
D_0, E_a	$e^{-9.03}$m^{2}s^{-1}, 3.96×10^4 J gmol^{-1}	Experimental
k_β	0.3 Wm^{-1}K^{-1}	Herman *et al*. (2005)
h, k_c	100 Wm^{-2}K^{-1}, 0.05 ms^{-1}	Herman *et al*. (2005)
k_1, k_3 Eq.(13)	$e^{-0.964}$, 0.723	Fitted to data of
k_2, k_4, k_5 Eq.(13)	0, 0,0	Di Scala & Crapiste (2008)
$\rho_{\beta,s}$	400 kg m^{-3}	Estimated
ρ_γ	Ideal gas equation kg m^{-3}	Calculated
Cp_w, Cp_β	4185, 2000 J kg^{-1}K^{-1}	Herman *et al*. (2005)
Cp_a, Cp_{wv}	1000, 1609 J kg^{-1}K^{-1}	Herman *et al*. (2005)
H^0_{wv}	2.501×10^6 J kg^{-1}	Herman *et al*. (2005)
λ	$H_\gamma - Cp_w T$	Eq. (18)
l, l_y, r	0.0005, 0.0025, 0.0127	Experimental
ε	0.76	Experimental
a	$(1-\varepsilon)A_\beta/V_\beta$ m^{2}m^{-3}	Calculated
A_β	$2\pi(2r+2l)2l_y$ m^2	Hollow cylinder
V_β	$\pi\left[(r+2l)^2 - r^2\right]2l_y$ m^3	Hollow cylinder
V_j	$7.9925\times10^{-4}/N$ m^3	Experimental
G_λ	1.43×10^{-2} kg s^{-1}	Experimental
$T_{\gamma0}, X_{\gamma0}$	70 ºC, 0.017 kg kg dm^{-1}	Experimental
T_{out}	28ºC	Experimental

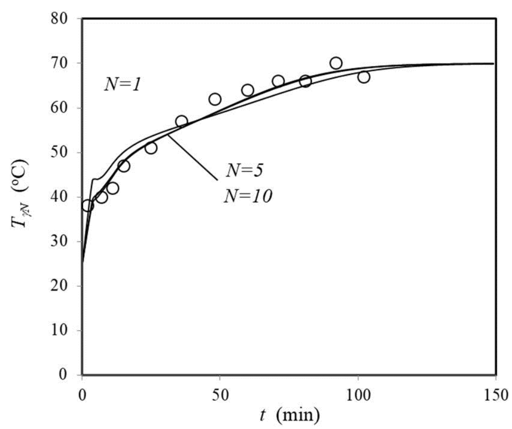

Fig. 7. Experimental (o) and simulated (at N = 1, N = 5 and N = 10) outlet air temperature of a batch fixed bed drying of red habanero chili pepper.

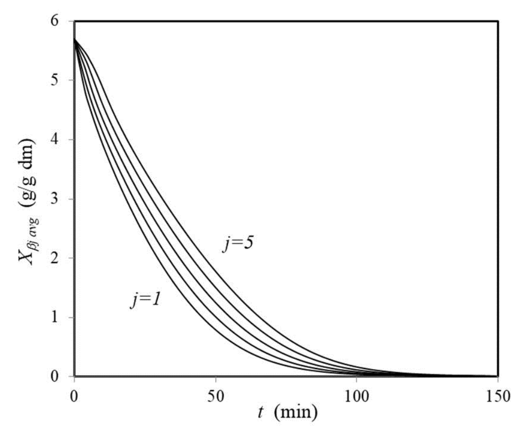

Fig. 8. Simulated average moisture of red habanero chili pepper dried in a batch fixed bed with N = 5 ideal mixed stages.

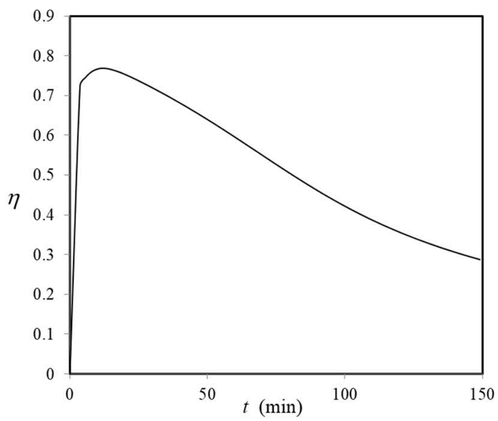

Fig. 9. Thermal efficiency of a batch fixed bed drying of red habanero chili pepper.

The experimental outlet air temperature jointly with simulated at $N = 1$, $N = 5$ and $N = 10$ are plotted in Fig. 7. It is evident that outlet air temperature differs from inlet temperature practically until the end of drying. There is no evidence that $N = 5$ produces different results than $N = 10$, and therefore the rest of the results were obtained at $N = 5$. The simulated habanero red pepper average moistures ($X_{\beta javg} = \int_0^1 X_{\beta j} d\xi$) were plotted in in Fig. 8, in which is evident the different moistures reached at different ideal mixed stages due to the variations in temperature and moisture of drying air. This effect is fundamental for the overall thermal efficiency, plotted in Fig. 9. At the beginning of drying, the thermal efficiency is high (up to 60%) due to the high moisture in product, but the process is incomplete. The overall thermal efficiency decreases with product moisture, and therefore, the drying process must be stopped in an optimal point, in which the product moistures were homogeneous (like can be observed at the end of process in Fig. 8), below to a required value and preserve the maximal thermal efficiency as possible. The estimated diffusivities in this work may be used in order to design an optimal thermally drying process.

Conclusions

The effect of air-drying temperature (30, 50 and 70 °C) on bioactive compounds (capsaicin, dihydrocapsaicin and total carotenoids) for red Habanero chili pepper and the water transport during the drying process were investigated. The results indicate that the greater retention of phytochemicals was achieved with high drying temperatures (70 °C). The capsaicinoids content remained constant during the drying process. The total carotenoids content appeared to increase. These results indicate that optimum drying conditions are directly dependent on energy consumption because the bioactive compounds remain constant between 30 and 70 °C. A simulation model was built and demonstrated that thermal efficiency depends on heat and mass transfer during drying and product final moisture. Therefore, the average water diffusivities estimated with the correct chili slice shape and size are the most relevant properties required to predict chili pepper drying dynamics and thermal efficiency.

Acknowledgements

The authors express their gratitude to Consejo Nacional de Ciencia y Tecnología (CONACyT) for scholarships provided to J.A. Olguín-Rojas and L.A.Vazquez-León. The authors express their gratitude to the Fondo Sectorial de Investigación para la Educación (project no. CB-256084), as well as to Tecnológico Nacional de México (project no. 5922.16-P) and University of Cadiz (OT2017/032) for ?nancial support.

Nomenclature

a	specific area (m^2/m^3)
a_w	water activity
A_{450}	absorbance at 450 nm
$A_{1cm}^{1\%}$	absorbance in a 1 cm cell of a carotenoid solution at 1 mg/100 mL
A	particle transfer surface (m^2)
C	total carotenoids content (μg/g)
D	water average diffusivity (m^2/s)
E	energy consumption (J/kg)
E_a	activation energy for diffusion (J/mol)
G	mass flow (kg/s)
h	heat transfer coefficient (W/m^2K)
k	heat conductivity (W/m/K)
k_c	mass transfer coeficient (m/s)
$k_1 \ldots k_5$	Henderson-Garcia equation parameters (Eq.13)
l_y	half height of chili slices (m)
l	half thickness of chili slices wall (m)
m	slope (1/s)
N	number of ideal mixed stages.
P	pressure (Pa)
r	internal radius of hollow cylinder (Fig. 1) (m)

R	ideal gas constant (8.3143 J/mol/K)
T	temperature (K or °C)
t	time (s)
X	moisture content (kg/kg dm)
z	rectangular spatial coordinate (m)
Greek symbols	
α	thermal diffusivity (m^2/s)
δ	intercept
ε	porosity
η	thermal efficiency
H_{wv}^0	water latent heat at reference value (J/kg)
λ	latent heat of evaporation (J/kg)
ξ	any dimensionless coordinate
ρ	density (kg/m^3)
τ	drying time (s)
ϕ	thermographic coefficient (m^2/s)
Ψ	dimensionless moisture content
Subscripts	
0	at the beginning of the drying process
avg	indicate averaged over space
e	at equilibrium
j	ideal mixed stage
out	indicate ambient condition
v	for vapor
ref	at reference
w	for water
wv	indicate water vapor
β	indicate solid phase
γ	indicate gas phase (air)

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