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Determination of the hygroscopic equilibrium and isosteric heat of aji chili pepper

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Capsicum baccatum
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thermal property

ABSTRACT

This study focuses on the determination of the hygroscopic equilibrium and isosteric heat of the aji chili pepper (*Capsicum baccatum*) under different controlled temperature and relative air humidity conditions. In addition, the objective was to adjust the model among the existing literature models that best represent the isothermal sorption behavior, as well as propose a new model to represent this phenomenon. Having obtained the mathematical models and experimental data, the best model and parameters that represent the hygroscopicity and the isosteric heat satisfactorily was determined. The temperatures used were 30, 55, and 70 °C, with water activity levels from 0.11 to 0.84. The model that best fit the data had a R² value of 0.97. The integral isosteric heat of sorption for 'Dedo-de-Moça' chili pepper within a moisture content from 0.07 to 0.55 (dry basis) ranged from 3641.66 to 2614.38 kJ kg⁻¹.

Palavras-chave:

Capsicum baccatum
isotermas de sorção
propriedade térmica

Determinação do equilíbrio higroscópico e calor isostérico de pimenta Dedo-de-Moça

RESUMO

Esta pesquisa focou na determinação do equilíbrio higroscópico e do calor isostérico da pimenta Dedo-de-Moça (*Capsicum baccatum*) sob diferentes condições controladas de temperatura e umidade relativa do ar. Foi ajustado o modelo que melhor representa o comportamento isotérmico de sorção entre os modelos descritos na literatura, bem como a proposta de um novo modelo e parâmetros que satisfazem com representatividade o fenômeno de higroscopicidade e calor isostérico. As temperaturas utilizadas foram 30, 50 e 70 °C, com níveis de atividade de água variando de 0.11 a 0.84. O calor isostérico integral de sorção da pimenta Dedo-de-Moça, para o teor de água entre 0.07 a 0.54 (b. s.), variou de 3641.66 a 2614.38 kJ kg⁻¹. O modelo que melhor se representa o comportamento do equilíbrio higroscópico e calor isostérico é o novo modelo proposto no presente estudo, com R² igual a 0.97.



INTRODUCTION

Peppers have been used since the American continent was discovered. The pepper is an important fruit, used in cooking, in allopathic and natural medicine, and even as a means of defense (Reifschneider & Ribeiro, 2008). In addition, peppers contain vitamins, flavonoids, carotenes, and metabolites with antioxidant properties (Veras et al., 2012).

The aji chili pepper, variety Dedo-de-Moça (*Capsicum baccatum*), is highly perishable, and some processes, such as drying, are necessary to increase shelf life and improve handling, transport, and storage (Veras et al., 2012). Thus, drying is a fundamental part in the conservation of the desirable qualities of plant products harvested with high moisture contents (Goneli et al., 2014).

The relationship between the moisture content of a certain product and the relative humidity at equilibrium at a given temperature can be expressed by mathematical equations, so-called sorption isotherms or hygroscopic equilibrium curves (Goneli et al., 2014). The hygroscopic behavior of diverse agricultural products has been studied by various researchers using various mathematical models, such as Chung-Pfost, Copace, modified GAB, modified Halsey, Henderson, modified Henderson, Oswin, Sabbah, and Sigma-Copace (Brooker et al., 1974; Hubinger et al., 2009; Ferreira et al., 2011; Silva & Rodvalho, 2012; Teixeira et al., 2012; Costa et al., 2013; Santos et al., 2015; Goneli et al., 2016). However, to develop isotherms that represent this equilibrium relationship, empirical mathematical models are used.

Studies of the Dedo-de-Moça chili pepper are scarce and mainly concern its hygroscopicity. Thus, this study aimed to determine the sorption isotherms and the isosteric heat of the Dedo-de-Moça pepper and fit different mathematical models to the dataset, finally selecting the model that best represents these phenomena.

MATERIAL AND METHODS

Dedo-de-Moça chili peppers from São Paulo state, sold by the Centro de Abastecimento do Estado da Guanabara (CADEG-RJ, Guanabara State Supply Center), were used. The experiments were carried out at the Fluminense Federal University, Niterói, Rio de Janeiro, Brazil (22° 54' 11.6" S 43° 6' 59.5" W). The fruits were selected based on the same visual characteristics of the stage of maturity, color, and shape. In addition, the fruits were obtained at the same point of sale.

The samples were cut in a crosswise manner to obtain a material with a homogeneous geometric shape. Then, they were dried in a laboratory dryer at 45 °C for approximately 12 h, and, in sequence, placed in woven bags made of plastic, properly identified, and weighed. Each test was performed with three replicates.

The moisture content of the pepper samples was determined according to Analytical Standards (IAL, 2005).

The hygroscopicity tests were performed in a biochemical oxygen demand (BOD) incubating chamber with temperature and relative air humidity control. The samples ranged from

approximately 1.029 to 1.289 g of hot pepper in woven nylon plastic bags to ensure the greatest contact surface of the material with the environment.

The temperatures of the experiments were controlled by the BOD chamber and the relative humidity was obtained from the used solutions. The tested temperatures were 30, 55, and 70 °C and the experimental period was 96 h. The solutions used for the experiment and their respective relative humidity at equilibrium as a function of ambient air temperature were performed according to Greenspan (1977).

For the verification and quantification of the hygroscopic equilibrium, the samples were weighed before being placed in the incubating chamber and after being removed from the laboratory oven after entering thermal equilibrium with the surrounding environment under airtight conditions. The different equilibrium moisture contents for the various combinations of temperature and relative air humidity were thus assessed.

In accordance with Teixeira et al. (2012), the mathematical models obtained in the literature for the hygroscopicity of agricultural products used for hot pepper analysis were chosen at random as a function of the temperature and the relative humidity of the air to check which model best represented its isotherm. The models used are shown in Eqs. 1 to 10:

- Chung-Pfost

$$U_e = a - b \cdot \ln[-(T + c) \cdot \ln(RH)] \quad (1)$$

- Copace

$$U_e = \exp[a - (b \cdot T) + c(c \cdot RH)] \quad (2)$$

- Modified GAB

$$U_e = \frac{ab \left(\frac{c}{T}\right) RH}{\left\{ (1 - bRH) \left[1 - bRH + b \left(\frac{c}{T}\right) RH \right] \right\}} \quad (3)$$

- Modified Halsey

$$U_e = \left[\frac{\exp(a - bT)}{-\ln(RH)} \right]^{\frac{1}{c}} \quad (4)$$

- Henderson

$$U_e = \left[\frac{\ln(1 - RH)}{(-a \cdot T_{\text{abs}})} \right]^{\frac{1}{b}} \quad (5)$$

- Modified Henderson

$$U_e = \left\{ \frac{\ln(1 - RH)}{[-a \cdot (T + b)]} \right\}^{\frac{1}{c}} \quad (6)$$

- Oswin

$$U_e = \frac{(a - b * T)}{\left[\frac{(1 - RH)}{RH} \right]^c} \quad (7)$$

- Sabbab

$$U_e = a \left(\frac{RH^b}{T^c} \right) \quad (8)$$

- Sigma-Copace

$$U_e = \exp \left\{ a - (bT) + [c \exp(RH)] \right\} \quad (9)$$

- Proposed model

$$U_e = \exp \left\{ (aa_w) + (T^b) + \left[\left[\frac{(T - a_w)}{a_w} \right]^b \right]^c \right\} \quad (10)$$

where:

- U_e - equilibrium moisture content of the product (dry basis (d.b.));
- RH - relative humidity;
- a_w - water activity (decimal);
- T - ambient air temperature (°C);
- T_{abs} - absolute temperature of the ambient air (K); and,
- a, b, and c - parameters that depend on the nature of the product.

For data analysis, the water activity (a_w) was considered to be equal to the relative air humidity in decimal format (Teixeira et al., 2012). The parameters for the mathematical models fitted to the experimental data were estimated by nonlinear modeling using the Statistica® 5.0 program.

In the analysis of the representativeness of the model hygroscopicity, the experimental data were compared to the values estimated for each model, checking the percentage of mean relative error (P), mean estimated error (SE), and the chi-square test (χ^2) according to Eqs. 11 to 13, respectively (Ryan, 2009). The capacity of the model to describe this physical process is inversely proportional to the standard deviation of the estimate (Goneli et al., 2014).

$$P = \frac{100}{n} \sum \frac{|Y - Y_0|}{Y} \quad (11)$$

$$SE = \sqrt{\sum \frac{(Y - Y_0)^2}{DF}} \quad (12)$$

$$\chi^2 = \sum \frac{(Y - Y_0)^2}{DF} \quad (13)$$

where:

- Y - value observed experimentally;
- Y_0 - value calculated by the model;
- n - number of experimental observations; and,
- DF - degrees of freedom of the model.

The net isosteric heat of sorption (Q_{st}) is the additional heat necessary to remove the water associated with the product (Teixeira et al., 2012). To calculate Q_{st} , the exponential model of Sopade and Ajisegiri (Eq. 14), which represents the behavior of the isosteric heat of sorption only as a function of equilibrium moisture content (Oliveira et al., 2014), was applied, as well as the Clausius-Clapeyron equation, (Eq. 15), as modified by Wang & Brennan (1991), which considers not only the equilibrium moisture content but also the temperature.

$$q_{st} = A \exp(B.U_e) \quad (14)$$

$$\ln(a_w) = - \left(\frac{q_{st}}{R} \right) \frac{1}{T_{abs}} + C \quad (15)$$

where:

- q_{st} - net isosteric heat of sorption, in Kj kg⁻¹;
- U_e - equilibrium moisture content or water activity (a_w), in decimal format;
- a_w - water activity in decimal format;
- T_{abs} - absolute temperature, in °K;
- R - universal gas constant, 8.314 kJ kmol⁻¹ K⁻¹ (for water vapor = 0.4619 kJ kg⁻¹ K⁻¹); and,
- A, B, and C - adjustment coefficients.

In the Clausius-Clapeyron equation, q_{st} was determined from the slopes of the curves of the chart $\ln(a_w) \times (1/T_{abs})$ for the diverse equilibrium moisture contents on a dry basis from the mathematical model that best fit the experimental data, as described in Eqs. 16 and 17.

$$\ln(a_w) = -(\text{slope of the straight line}) \frac{1}{T_{abs}} + C \quad (16)$$

$$q_{st} = (\text{slope of the straight line}) \times R \quad (17)$$

For practical reasons, in this study, not only the isosteric heat, named the net isosteric heat of sorption, was calculated but also calculated the integral isosteric heat of sorption, which includes the net isosteric heat of sorption and the latent heat of vaporization of free water. The latent heat of vaporization of free water may be represented by Eq. 18 (Brooker et al., 1974).

$$L = 2502.2 - 2.39T_m \quad (18)$$

in which:

- L - latent heat of vaporization of free water, in kJ kg⁻¹;
- and,
- T_m - mean temperature in the range of study, in °C.

From the previously shown data, it is possible to determine the integral isosteric heat of sorption (Q_{st}).

$$Q_{st} = q_{st} + L \quad (19)$$

$$Q_{st} = A \exp(B \times U_e) + L \quad (20)$$

where:

- Q_{st} - integral isosteric heat of sorption, in kJ kg^{-1} ;
- L - latent heat of vaporization of free water, in kJ kg^{-1} ;
- U_e - equilibrium moisture content or water activity (a_w), in decimal; and,
- A and B - coefficients of adjustment.

RESULTS AND DISCUSSION

For temperatures of 30, 50, and 70 °C with a water activity between 0.11 and 0.84, approximately, the mean equilibrium moisture content values obtained were between 0.09 and 0.54 (d.b.) (Table 1).

The experimental points belonging to the sorption curves at different temperatures were obtained from the mean values of the hygroscopic equilibrium moisture content. From those results, for each model, the respective parameters (R^2 , P , and SE) were determined. In Table 2, which follows, the results of the estimates related to the analyses of the hygroscopic equilibrium models observed for the Dedo-de-Moça hot pepper are shown.

Table 1. The mean values of the equilibrium moisture contents (d.b.) for the different combinations of water activity and temperature (°C) after 96 h

Temp. (°C)	Salts	A_w	Equilibrium moisture content (d. b.)
30	Lithium chloride - LiCl	0.11 ± 0.00	0.14
30	Potassium acetate - CH_3COOK	0.22 ± 0.01	0.16
30	Magnesium chloride - MgCl_2	0.32 ± 0.00	0.23
30	Magnesium nitrate - $\text{Mg}(\text{NO}_3)_2$	0.51 ± 0.00	0.36
30	Potassium chloride - KCl	0.84 ± 0.01	0.54
55	Lithium chloride - LiCl	0.11 ± 0.00	0.09
55	Magnesium chloride - MgCl_2	0.30 ± 0.00	0.15
55	Sodium bromide - NaBr	0.50 ± 0.01	0.31
55	Potassium chloride - KCl	0.81 ± 0.00	0.42
70	Lithium chloride - LiCl	0.11 ± 0.00	0.09
70	Potassium chloride - KCl	0.80 ± 0.01	0.31

Table 2. Estimated parameters, coefficients of determination (R^2), mean relative error (P), and mean estimated error (SE) for each model analysed

Model	Parameters			R^2	P (%)	SE (decimal)	χ^2
	a	b	c				
Modified Chung-Pfost	0.7587	0.1433	1.4040	0.92	17.40	0.0881	0.0078
Copace	-1.7352	0.0110	1.7708	0.95	13.00	0.0729	0.0053
Modified GAB	0.6897	0.2785	147.1099	0.94	15.56	0.0719	0.0052
Modified Halsey	-2.0850	0.0232	1.9200	0.92	13.69	0.0954	0.0091
Henderson	0.0199	-	1.6102	0.83	16.25	0.1721	0.0296
Modified Henderson	0.1436	8.6240	1.8554	0.97	8.98	0.0605	0.0037
Oswin	0.3870	-0.0027	2.7164	0.95	11.48	0.0691	0.0048
Sabbah	4.9294	0.8702	0.5992	0.94	12.67	0.0686	0.0047
Sigma-Copace	-2.6122	0.0126	1.0548	0.92	14.66	0.0957	0.0092
GAB	0.1685	10.6947	0.7754	0.78	17.88	0.1416	0.0201
Peleg	-	-	-	0.76	20.86	0.1381	0.0191
Modified BET	156.6900	0.0036	-0.1046	0.76	23.17	0.1423	0.0203
Smith	0.2467	0.0031	0.2071	0.93	16.26	0.0947	0.0090
Andrade	-3.7576	0.2733	-0.2978	0.97	8.69	0.0601	0.0036

The models that did not fit the experimental data satisfactorily had P values greater than 10% and did not adequately represent the phenomenon analyzed. In addition, the R^2 values must be close to one (Teixeira et al., 2012; Mohapatra & Rao, 2005).

Considering the analysis of the results of hygroscopic equilibrium for the Dedo-de-Moça hot pepper, as shown in Table 2, the modified Henderson model showed a satisfactory result; however, the Andrade model fits the phenomenon better. These models may, therefore, be used to represent the phenomenon in other applications because both showed a random distribution of the residues. Thus, the Andrade model was used, which had a coefficient of determination (R^2) of 96.61%, and P , SE , and χ^2 values of 8.69%, 0.0601, and 0.0036, respectively, for the presented results. For a better analysis of the fit of the model to the hygroscopicity, the experimental values of equilibrium moisture content and the values calculated of the sorption isotherms of the model that best fit the experimental data were plotted (Figure 1).

Sorption isotherms are classified according to their form, and there are five different types. The isotherms have a sigmoidal type II pattern (Brunauer et al., 1938). This type of sorption isotherm is widely found in agricultural products (Goneli et al., 2016), for example, castor beans. The type II isotherm is related to synergic effects of Raoult's law, capillary effects, and the interaction of the moisture on the surface of the studied material (Labuza & Altunakar, 2007).

Isosteric heat of sorption is the additional energy necessary to remove the water associated with the hygroscopic material, which is greater than the energy necessary to vaporize the free water contained in the product under the same temperature and pressure conditions (Teixeira et al., 2012). The equilibrium moisture of a hygroscopic material is relevant in a drying study because it determines the minimum moisture content that the product may reach under the determined drying air conditions (Costa et al., 2013).

Based on the previous determination of the best model for the hygroscopicity, the Andrade model was used for the determination of the water activity (a_w) in the characterization of the isosteric heat of sorption of the Dedo-de-Moça hot pepper. The moisture content values were obtained from the model chosen, based on the calculation of $\ln(a_w)$.

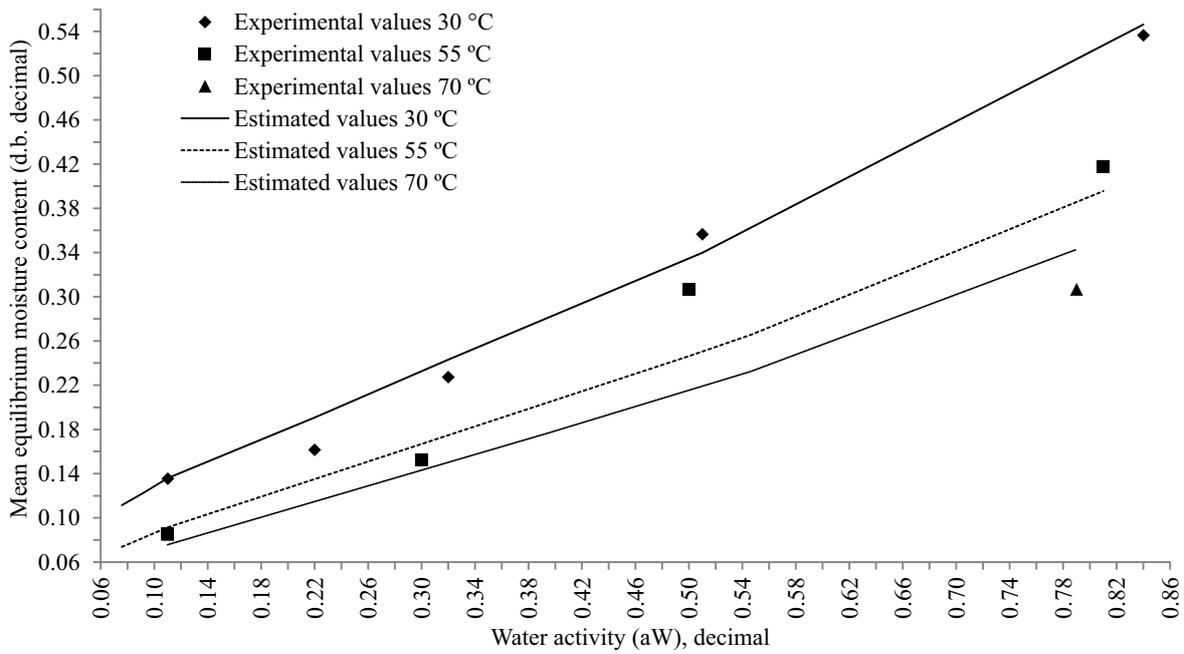


Figure 1. Experimental values and values estimated by the proposed model of the equilibrium moisture content of 'Dedo-de-Moça' chili pepper as a function of water activity (a_w) and temperature

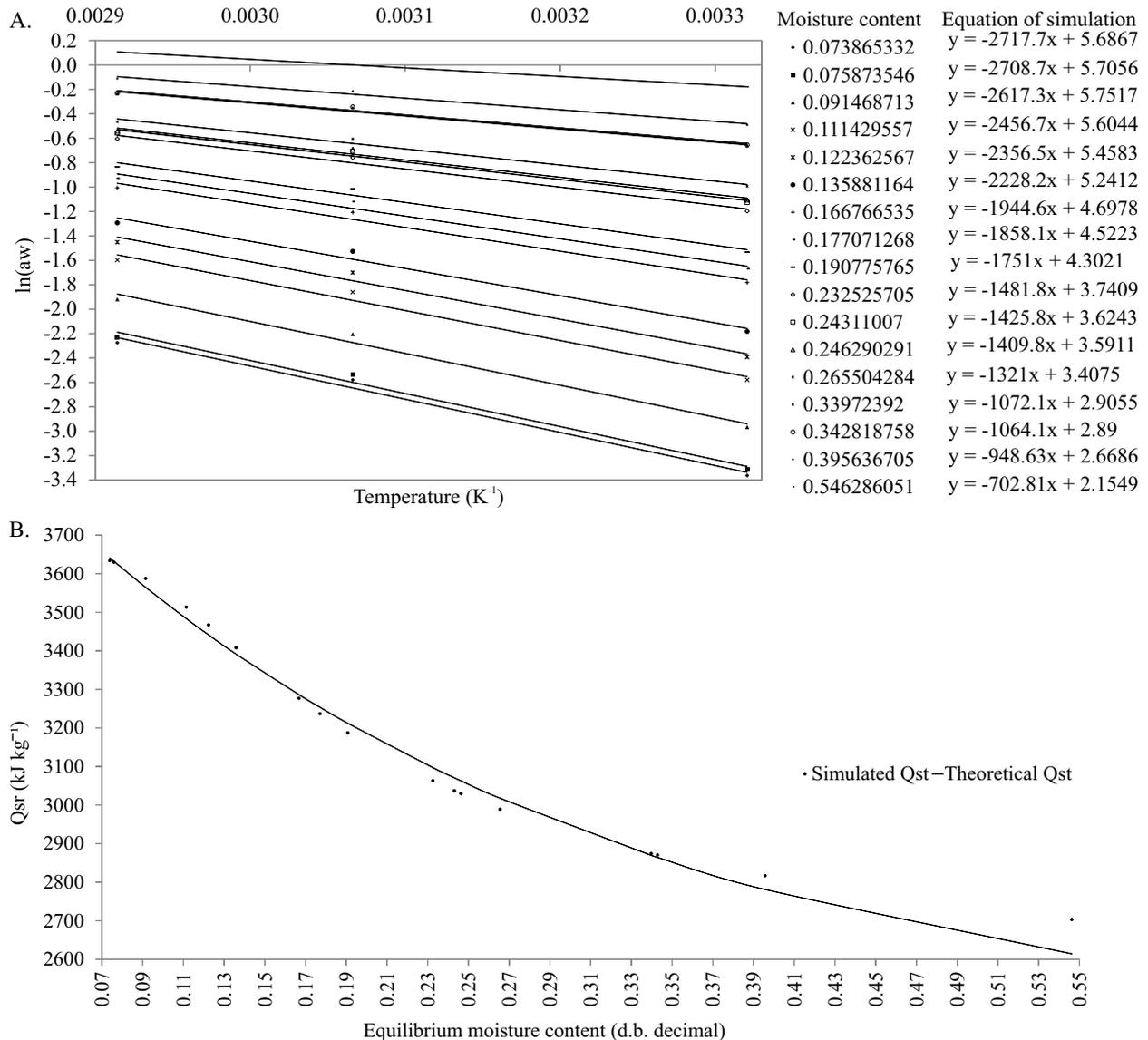


Figure 2. (A) Values of (a_w) for different equilibrium moisture contents (d.b.), $Y = \ln(a_w)$ and $X = K^{-1}$, and (B) theoretical and simulated values of integral isosteric heat of sorption as a function of equilibrium moisture content

After determining the values of $\ln(a_w)$, it was possible to represent the curves of the Napierian logarithm of the water activity of the Dedo-de-Moça hot pepper as a function of the inverse of absolute temperature ($1/T_{\text{abs}}$) for different equilibrium moisture contents (d.b.) and their respective linear equations (Figure 2A).

From the slopes of the straight lines, the values of q_{st} can be calculated from Eq. 14. For the determination of Q_{st} in kJ kg^{-1} , as represented by Eq. 17, the value of L was also included, which represents the minimum amount of energy necessary to evaporate the water. For this calculation, the mean temperature used in the study was considered, which was $51.67\text{ }^\circ\text{C}$, resulting in a value for latent heat of vaporization of $2378.7167\text{ kJ kg}^{-1}$. The values of net isosteric heat and integral isosteric heat of sorption obtained from the slope of the straight line and from the latent heat of vaporization, respectively, are shown in Table 3.

After obtaining the above data using the Statistica[®] 5.0 program, the parameters of the equation of integral isosteric heat of sorption for the 'Dedo-de-Moça' hot pepper were obtained (Eq. 21) as a function of the equilibrium moisture content (U_e) (d.b.) and the mean temperature of $51.67\text{ }^\circ\text{C}$.

$$Q_{\text{st}} = 1641.44 \exp(-3.55.U_e) + 2378.7167 \quad (21)$$

$$R^2 = 0.9986$$

As shown in Figure 2B, as the moisture content of the product decreases, there is a notable increase in the amount of energy required to remove water. Therefore, the values of integral isosteric heat of sorption for Dedo-de-Moça hot pepper for moisture contents from 0.07 to 0.5463 (d.b.) ranged from 3641.66 to $2614.38\text{ kJ kg}^{-1}$, which agrees with the results obtained in analyses of the red bell pepper (Vega-Gálvez et al., 2007) and pepper variety bico (Santos et al., 2015).

Table 3. Values of net isosteric heat and integral isosteric heat of sorption for different equilibrium moisture contents (d.b.)

Slope of the straight line	Net Isosteric heat (kJ kg^{-1})	Theoretical integral	Simulated integral
-2717.70	1255.31	3634.02	3641.27
-2708.70	1251.15	3629.87	3632.29
-2617.30	1208.93	3587.65	3564.72
-2456.70	1134.75	3513.47	3483.52
-2356.50	1088.47	3467.18	3441.43
-2228.20	1029.21	3407.92	3391.59
-1944.60	898.21	3276.93	3286.30
-1858.10	858.26	3236.98	3253.70
-1751.00	808.79	3187.50	3212.11
-1481.80	684.44	3063.16	3097.22
-1425.80	658.58	3037.29	3070.70
-1409.80	651.19	3029.90	3062.93
-1321.00	610.17	2988.89	3017.78
-1072.10	495.21	2873.92	2869.65
-1064.10	491.51	2870.22	2864.28
-948.63	438.17	2816.89	2781.20
-702.81	324.63	2703.34	2614.38

CONCLUSIONS

1. The proposed model (Eq. 10) best describes the drying phenomena in the Dedo-de-Moça pepper and may be used in other applications.

2. The equilibrium moisture content of Dedo-de-Moça hot chili pepper varies as a function of relative air humidity for a given temperature.

3. For the integral isosteric heat, there is an increase in energy demand such that, at lower equilibrium moisture contents, the energy needed to remove water from the product is greater.

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