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Adsorption isotherms and vaporization latent heat of malagueta pepper seeds

Isotermas de adsorção e calor latente de vaporização das sementes de pimenta malagueta

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Abstract

The aim of this study was to determine and model malagueta pepper seeds adsorption isotherms, as well as to estimate the vaporization latent heat released during adsorption process. Hygroscopic balance moisture contents were determined by static method, using six saline solutions and three temperatures. Clausius-Clapeyron equation used to calculate malagueta pepper seeds vaporization latent heat. From obtained results, it was concluded that temperature increase promotes malagueta pepper seeds moisture content reduction, in constant water activity. The Modified Oswin model properly adjusted to the experimental data, satisfactorily representing pepper seeds adsorption isotherms. The energy released during the adsorption process, represented by vaporization latent heat, increases with moisture content reduction, ranging from 2762.92 to 2495.56 kJ $kg^{-1}$for moisture contents from 0.056 to 0.134 (db).

Additional keywords: *Capsicum frutescens* L.; energy; hygroscopicity; seeds.

Resumo

Objetivou-se neste trabalho determinar e modelar as isotermas de adsorção das sementes de pimenta-malagueta, assim como estimar o calor latente de vaporização liberado durante o processo de adsorção. Os teores de água de equilíbrio higroscópico foram determinados pelo método estático, utilizando seis soluções salinas e três temperaturas. A equação de Clausius-Clapeyron foi utilizada para calcular o calor latente de vaporização das sementes de pimenta-malagueta. A partir dos resultados obtidos, conclui-se que, em atividade de água constante, o aumento da temperatura promove a redução do teor de água das sementes de pimenta. O modelo de Oswin Modificado ajustou-se adequadamente aos dados experimentais, representando satisfatoriamente as isotermas de adsorção das sementes de pimenta. A energia liberada durante o processo de adsorção, representada pelo calor latente de vaporização, aumenta com a redução do teor de água, variando de 2.762,92 até 2.495,56 kJ kg^{-1} para os teores de água de 0,056 até 0,134 (b.s.).

Palavras-chave adicionais: *Capsicum frutescens* L.; energia; higroscopicidade; sementes.

Introduction

Capsicum genus belongs to the Solanaceae family, and has over 30 cultivated species in tropical and subtropical regions of the world (Yaldiz et al., 2010). Pepper berries vary in size, shape, flavor, color, pungency (Li et al., 2011a), and chemical composition (Jarret et al., 2013). Such characteristics justify peppers and their seeds high consumption and use in food and nutrition, medicine, and cosmetics (Dagnoko et al., 2013).

Pepper seeds are considered by-products by food processing industries, since problems related to industrial waste are becoming increasingly common (Silva et al., 2013). Although being classified as industrial by-products (Li et al., 2011a; Silva et al., 2013), studies have shown that *Capsicum* genus species seeds have promising chemical composition and biological activities for food and medicine use. Oils that are rich inhighly nutritious unsaturated fatty acids and linoleic acids (Embaby & Mokhtar, 2011; Li et al., 2011a), crude fat sources, carbohydrates and some minerals (Embaby & Mokhtar, 2011), antioxidant properties (Sim& Sil, 2008), and tumor cells antiproliferative effects (Jeon et al., 2012) are associated with *Capsicum* genus species seeds.

As with many other agricultural products, in order to correctly dry and store pepper seeds, it is necessary to know the relation between the product and the surrounding air, temperature and relative humidity desirable for product quality conservation (Goneli et al., 2010; Corrêa et al., 2014).

Pepper seeds, as well as other agricultural products, are hygroscopic, i.e., they have the capacity to give or absorb water from the environment, in order to reach their moisture content balance (Resende et al., 2006). Such phenomena are known as desorption and adsorption (Corrêa et al., 2014).

Agricultural products hygroscopic behavior may be analyzed through isothermal curves, or hygroscopic balance curves, which is the relation between a given product moisture content and water activity balance to a specific temperature (Resende et al., 2006; Clabera-Olivera et al., 2011; Al-Mahasneh et al., 2014).

Sorption isotherms are curves representing moisture content balance, through experimental data or empirical models. However, for a mathematical model to be properly used, certain physical properties knowledge is required (Stefanini & Roa, 1980), such as vaporization latent heat, which is defined as the energy amount required to remove water from the product in desorption (Rodriguez-Arias et al., 1963; Corrêa et al., 1998); or the energy released by the product during adsorption (Wang & Brennan, 1991).

Given agricultural products water sorption hygroscopicity and energy demand importance, the objective of this study was to obtain and model malagueta pepper seeds adsorption isotherms, as well as to determine product vaporization latent heat.

Material and methods

The study was conducted in the Agribusiness Food Laboratory of the Goiano Federal Institute of Education, Science and Technology - *Campus* Ceres, Goiás state. Malagueta pepper seeds (*Capsicum frutescens* L.) were used, which were manually extracted from berries harvested at the institution Experimental Sector.

After extraction, seeds were subjected to pre-drying, under laboratory conditions, for 48 hours. Subsequently, they were subjected to drying in an oven without forced ventilation, adjusted to provide a temperature of 70 °C for 48 hours, until achieving a moisture content of approximately 0.025 (db) (Goneli et al., 2010).

Hygroscopic balance moisture contents were determined using the static-gravimetric method for different controlled temperature conditions (30, 40 and 50 °C), being supplied by a drying oven, and water activity between 0.290 and 0.900 (decimal).

Samples, which contained approximately 1.0 g seeds, were placed in sorption recipients and put inside hermetic recipients containing saturated salt solutions (Ferreira et al., 2011; Silva & Rodovalho, 2012).

Salt solutions were used to promote the different water activities shown in Table 1.

Table 1 -Saturated salt solutions water activity values (A_w, decimal) (Greenspan, 1977).

Solution[1]	CaCl	NaI	$NaNO_3$	NaCl	KCl	$MgSO_4$
A_w	0.290	0.362	0.731	0.751	0.836	0.900

[1]CaCl: Calcium chloride; NaI: Sodium iodide; $NaNO_3$: Sodium nitrate; NaCl: Sodium chloride; KCl: Potassium chloride; $MgSO_4$: Magnesium sulphate.

Periodically, samples were weighed on an analytical balance (0.0001 g) until reaching hygroscopic balance moisture content, i.e., until there was no weight change on three successive weighings (Ferreira et al., 2011; Sousa et al., 2013).

When reaching hygroscopic balance moisture content, it was determined using the oven method, at 105 ± 3 °C for 24 h, in three repetitions (Brasil, 2009).

Mathematical models that are often used to represent seeds and grains hygroscopic behavior were adjusted to the experimental data, and equations are shown in Table 2.

In order to adjust the mathematical models, non-linear regression analysis was carried out through Gauss Newton method. In order to check each model adjustment degree, regression coefficient significance was considered through t test, adopting 1% significance level. Determination coefficient (R^2) magnitude and mean relative error values (P) were determined by Equation 21, and estimated average error (SE) was determined by Equation 22. For a good mathematical model adjustment, P must be lower than 10% (Mohapatra & Rao, 2005), and SE must be the nearest to zero.

$$P = \frac{100}{n}\sum\frac{\left|Y-\hat{Y}\right|}{Y} \quad (21)$$

$$SE = \sqrt{\frac{\left(Y-\hat{Y}^2\right)}{GLR}} \quad (22)$$

Wherein: Y - experimental value; $\hat{Y}$ - Value estimated by the model; n - number of experimental observations; GLR - model degrees of freedom (number of observations minus the number of model parameters).

Othmer et al. (1940), from Clausius-Clapeyron studies, proposed Equation 23 to measure steam partial pressure, which is contained in porous systems:

$$Ln(Pv) = \frac{L}{L'}\cdot Ln(Pvs) + C \quad (23)$$

Wherein: Pvs - free water saturation vapor pressure, for a given balance temperature (T); Pv - free water vapor pressure, in order to determine balance temperature (T); L - product water vaporization latent heat, kJ kg^{-1}; L'- free water vaporization latent heat, to the balance temperature, kJ kg^{-1}; C - integration constant.

In order to quantify Equation 23 LL'^{-1}ratio values, which is the ratio of product water vaporization latent heat (L) and free water latent heat (L') values, in a pepper seeds hygroscopic balance moisture content and temperature range condition, methodology proposed by Stefanini & Roa (1980) was used.

Water vaporization enthalpy equation, presented by Rodrigues-Arias, was adjusted to LL'^{-1} ratio values with the inclusion of a parameter in Equation 24, in order to improve LL'^{-1}estimates (Corrêa et al., 1998):

$$\frac{L}{L'} - 1 = a \cdot \exp\left(-b \cdot Xe^{m}\right) \tag{24}$$

Balance temperature (°C) free water vaporization latent heat (kJ kg^{-1}) was calculated using the studied range mean temperature (T), in °C, using Equation 25:

$$L' = 2502.2 - 2.39 \cdot T \tag{25}$$

Table 2 - Mathematical models used to predict seeds and grains hygroscopic behavior.

Model Designation	Model	
$Xe^* = \frac{(Xm \cdot c \cdot a_w) \cdot (1-(n+1) \cdot a_w{}^n + n \cdot a_w{}^{n+1})}{(1-a_w) \cdot [1+(c-1) \cdot a_w - c \cdot a_w{}^{n+1}]}$	BET	(1)
$Xe^* = (a \cdot b \cdot a_w)/\{[1-(c \cdot a_w)] \cdot [1+(b-c) \cdot a_w]\}$	Modified BET	(2)
$Xe^* = [\ln(1-a_w)/(-a \cdot (T^b))]^{1/c}$	Cavalcante Mata	(3)
$Xe^* = [-1/(c \cdot T^d)] \cdot \ln[\ln(a_w)/(-a \cdot T^b)]$	Chen Clayton	(4)
$Xe^* = a - b \cdot \ln(-(T+c) \cdot \ln(a_w))]$	Chung Pfost	(5)
$Xe^* = (-1/b) \cdot \ln[(T+c) \cdot \ln(a_w)/(-a)]$	Modified Chung Pfost	(6)
$Xe^* = \exp[a - (b \cdot T) + (c \cdot a_w)]$	Copace	(7)
$Xe^* = 1/(a \cdot T^b + a_w{}^c)$	Corrêa	(8)
$Xe^* = \frac{(Xm \cdot c \cdot k \cdot a_w)}{(1-c \cdot a_w) \cdot (1-k \cdot a_w + c \cdot k \cdot a_w)}$	GAB	(9)
$Xe^* = \frac{[Xm \cdot (c/T) \cdot k \cdot a_w]}{(1-c \cdot a_w) \cdot [(1-k \cdot a_w + (c/T) \cdot k \cdot a_w)]}$	Modified GAB	(10)
$Xe^* = Xm \cdot [-\ln(a_w)]^n$	Halsey	(11)
$Xe^* = [\exp(a - b \cdot T)/-\ln(a_w)]^{1/c}$	Modified Halsey	(12)
$Xe^* = [\ln(1-a_w)/(-a \cdot T_{abs})]^{1/c}$	Henderson	(13)
$Xe^* = \{\ln(1-a_w)/[-a \cdot (T+b)]\}^{1/c}$	Modified Henderson	(14)
$Xe^* = a \cdot [a_w/(1-a_w)]^b$	Oswin	(15)
$Xe^* = (a + b \cdot T)/[a_w/(1-a_w)]^{1/c}$	Modified Oswin	(16)
$Xe^* = [(a \cdot a_w{}^b) + (c \cdot a_w{}^d)]$	Peleg	(17)
$Xe^* = a \cdot [a_w{}^b/T^c]$	Sabbah	(18)
$Xe^* = \exp\{a - (b \cdot T) + [c \cdot \exp(a_w)]\}$	Sigma Copace	(19)
$Xe^* = a - (b \cdot T) - c \cdot \ln(1-a_w)$	Smith	(20)

Where: Xe *: balance moisture content, % db; a_w; water activity, decimal; T: temperature, °C; T_{abs}: absolute temperature; Xm: molecular monolayer moisture content, % db; a, b, c, n, k: coefficients depending on the product.

Free water saturation vapor pressure, Pvs, was calculated through Thétens equation (Equation 26):

$$Pvs = 0.61078 \cdot 10^{((7.5 \cdot T)/(273.3 + T))} \quad (26)$$

Vapor pressure value (Pv) was determined according to Equation 27:

$$Pv = a_w \cdot Pvs \quad (27)$$

In order to quantify pepper seeds water vaporization latent heat for each studied temperature, Equation 24 and Equation 25 were combined, giving Equation 28 (Corrêa et al., 1998):

$$L = (2502.2 - 2.39 \cdot T) \cdot [1 + a \cdot \exp(-b \cdot Xe^{m})] \quad (28)$$

Results and discussions

Table 2 shows correlation coefficients, estimated average errors and average relative errors of mathematical models adjusted to malagueta pepper seeds hygroscopic balance moisture content, obtained by adsorption to different temperature and water activity conditions.

Table 2 -Determination coefficients (R^2), estimated average errors (SE) and average relative errors (P) of the different models adjusted to malagueta pepper seeds hygroscopic balance moisture content, for adsorption process.

Models	R^2	SE	P
	(decimal)		(%)
BET	0.9239	1.8139	12.5828
Modified BET	0.9404	1.6125	11.3995
Cavalcante Mata	0.9594	1.3364	9.7376
Chen Clayton	0.9634	1.2839	9.3940
Chung Pfost	0.9606	1.3170	9.6113
Modified Chung Pfost	0.9606	1.3170	9.6113
Copace	0.9546	1.4115	10.2715
Corrêa	0.9529	1.4378	10.5840
GAB	0.9404	1.6125	11.3992
Modified GAB	0.9359	1.6698	10.9022
Halsey	0.9419	1.5765	12.0335
Modified Halsey	0.9713	1.1269	8.8301
Henderson	0.9430	1.5624	10.6751
Modified Henderson	0.9604	1.3210	9.6292
Oswin	0.9428	1.5654	11.2113
Modified Oswin	0.9730	1.0942	8.5593
Peleg	0.9451	1.5641	11.1069
Sabbah	0.9290	1.7542	11.6208
Sigma Copace	0.9645	1.2526	9.7052
Smith	0.9667	1.2135	9.3004

It was noted that, among adjusted models, the highest values related to determination coefficient were observed for Modified Halsey (R^2 = 0.9713) and Modified Oswin (R^2 = 0.9730) models. Some authors stated that the determination coefficient should not be used alone in non-linear models selection (Goneli et al., 2010; Corrêa et al., 2014). Determination coefficient magnitude is not enough to assess hygroscopic balance models adjustment quality (Rosa et al., 2010).

Thus, in order to better assess models adjustment degree to the experimental data, estimated average error (SE) and average relative error (% P) were determined, which are also shown in Table 2.

Regarding estimated average error (SE), it was observed in Table 2 that Modified Halsey (SE = 1.1269) and Modified Oswin (SE = 1.0942) models had the lowest values between the other studied models. According to Corrêa et al. (2014), the lowest SE value indicates best model adjustment to the experimental data.

For a non-linear model to be used in hygroscopic phenomena representation, the average relative error should be lower than 10% (Rosa et al., 2010). Thus, it is verified in Table 2 that, among adjusted models, Modified Halsey (P = 8.8301%) and Modified Oswin (P = 8.5593%) also had the best statistical adjustments.

Table 3 shows Modified Halsey and Modified Oswin model coefficients that were significant at 1% probability by t test, thus proving these models suitability in the studied phenomenon description.

Table 3 - Modified Halsey and Modified Oswin model coefficients adjusted to malagueta pepper seeds hygroscopic balance moisture contents.

Models	Coefficients		
	a	b	c
Modified Halsey	5.5847**	0.0239**	2.3355**
Modified Oswin	12.2714**	-0.0912**	2.8304**

**Significant at 0.01 by t test.

Based on the evaluated statistical parameters (Table 2 and 3), and due to being a simple equation that adequately describes many biological products sorption isotherms (Chen, 1990), the Modified Oswin Model was selected to estimate hygroscopic balance moisture contents and to describe adsorption isotherms of malagueta pepper seeds (Figure 1).

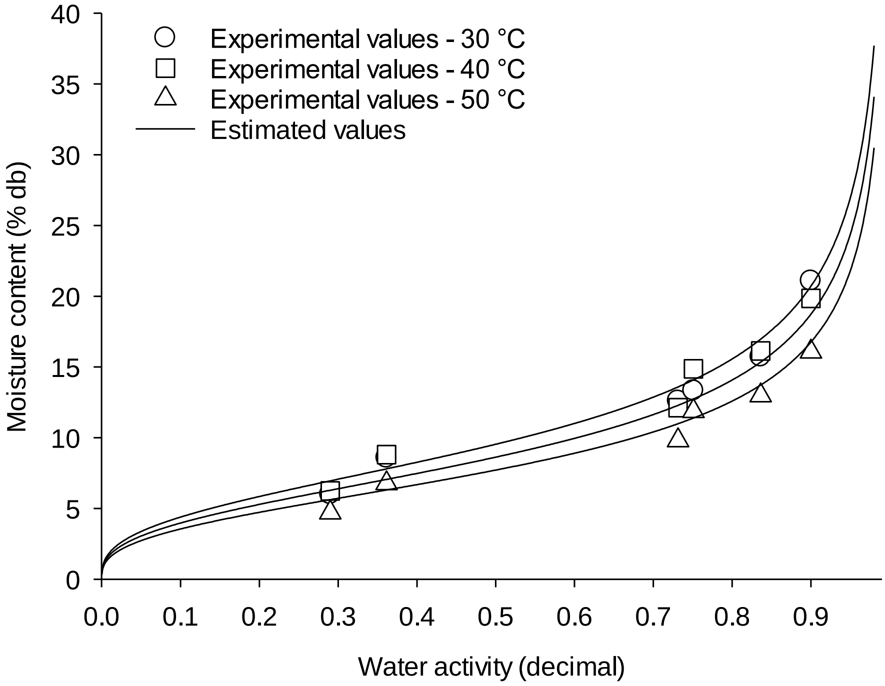

Figure 1 - Malagueta pepper seeds hygroscopic balance moisture content and adsorption isotherms experimental values, estimated by the Modified Oswin model, and obtained for the different temperature and water activity conditions.

In studies with yellow cumarí seeds (*Capsicum chinense*), Ferreira et al. (2011) also recommended the modified Oswin model to estimate hygroscopic balance moisture content for the activity range of 0.29 to 0.9 (decimal), and for temperature between 30 and 40 °C. This same model satisfactorily represented wheat seeds (Li et al., 2011b), corn grains (Smaniotto et al., 2012), crambe fruits (Costa et al., 2013), forage turnip seeds (Sousa et al., 2013), linseeds (Singh & Kumari, 2014), and husked rice seeds (Oliveira et al., 2014a) hygroscopicity.

Looking at Figure 1, it is observed that, for constant water activity, temperature increase caused seeds moisture content reduction. According to Al-Muhtaseb et al. (2004), temperature effect on isothermal absorption is very important, since food are exposed to a range of temperatures during the post-harvest stage.

This behavior was already expected, since the water vapor pressure in the air and on seeds surface is directly proportional to temperature. Furthermore, as seeds have a higher number of water molecules than air, higher product surface water vapor pressure is observed, what means higher water loss for the product to reach hygroscopic balance (Ferreira & Pena, 2003).

It was observed in Figure 1 that malagueta pepper seeds adsorption isotherms estimated by Modified Oswin model have a characteristic sigmoidal shape of type II curves, according to BET classification (Brunauer et al., 1938). This format is considered standard for agricultural products.

Figure 2 shows experimental and estimated values of pepper seeds water vaporization latent heat and free water vaporization latent heat ratio (L L'$^{-1}$), as a function of pepper seeds moisture content.

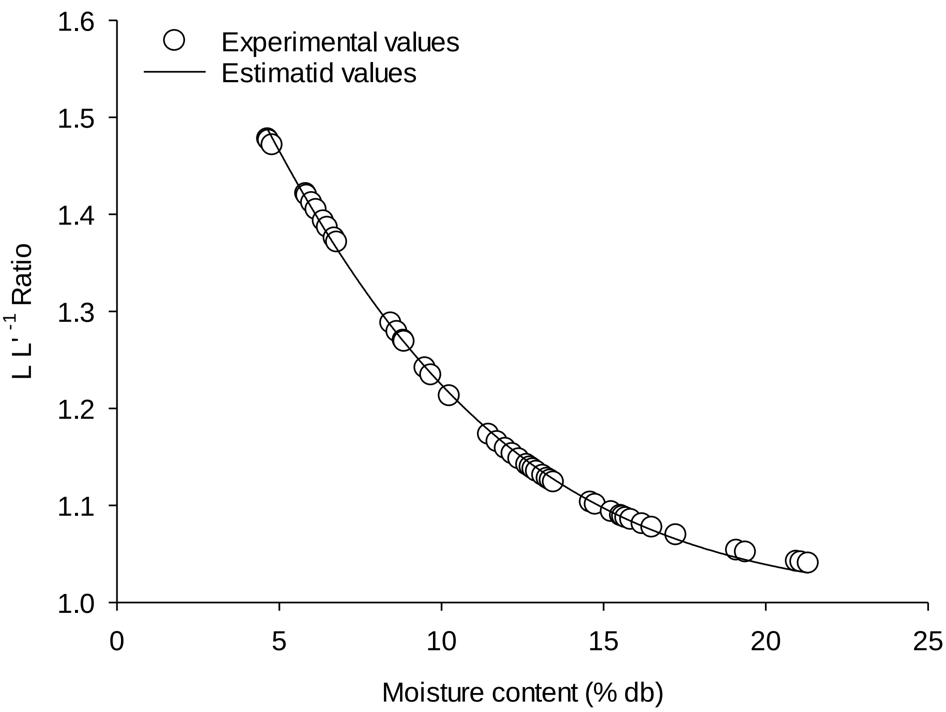

Figure 2 - LL'$^{-1}$ ratio experimental and estimated values, as a function of malagueta pepper seeds water balance contents.

It was noted that the L L'$^{-1}$ ratio is indirectly proportional to seeds moisture content, moisture content reduction promotes L L'$^{-1}$ ratio increase. In addition, it was observed that all L L'$^{-1}$ ratio values were higher than 1, demonstrating that the energy released by seeds to absorb water during the adsorption process is always higher than the energy needed to evaporate pure water. Similar behaviors were observed for coffee beans and soybean (Stefanini & Roa, 1980), popcorn seeds (Corrêa et al., 1998), husked red rice grains (Rodovalho et al., 2009), wheat seeds (LI et al., 2011b), yellow cumarí seeds (Ferreira et al., 2011), castor beans (Ojediran et al., 2013), and tucumã-de-Goiás seeds (Oliveira et al., 2014b).

Table 4 shows Equation 24 "a", "b", and "m" coefficients adjusted to L L'$^{-1}$ ratio experimental data.

Table 4 - Coefficients used to calculate malagueta pepper seeds LL'$^{-1}$ ratio.

a	b	m	R^2
0.7855**	0.0690**	1.2594**	0.9993

** Significant to 0.01 by t test.

It was observed in Table 4 that Equation 24 was properly adjusted to L L'$^{-1}$ ratio experimental data, since it showed high determination coefficient (R^2 = 0.9993) and significant coefficients at 1% probability by t test. Thus, it can be used to determine pepper seeds water vaporization latent heat.

Substituting Table 4 coefficient values in the equation proposed by Corrêa et al. (1998), and adding free water vaporization latent heat, there is Equation 29, which was used to calculate pepper seeds water vaporization latent heat for each temperature.

$$L = (2502.2 - 2.39T)[1 + 0.7855\exp(-0.690Xe^{1.2594})] \quad (29)$$

In Figure 3, water vaporization latent heat curves are shown, as a function of malagueta pepper seeds moisture contents for the three studied temperatures.

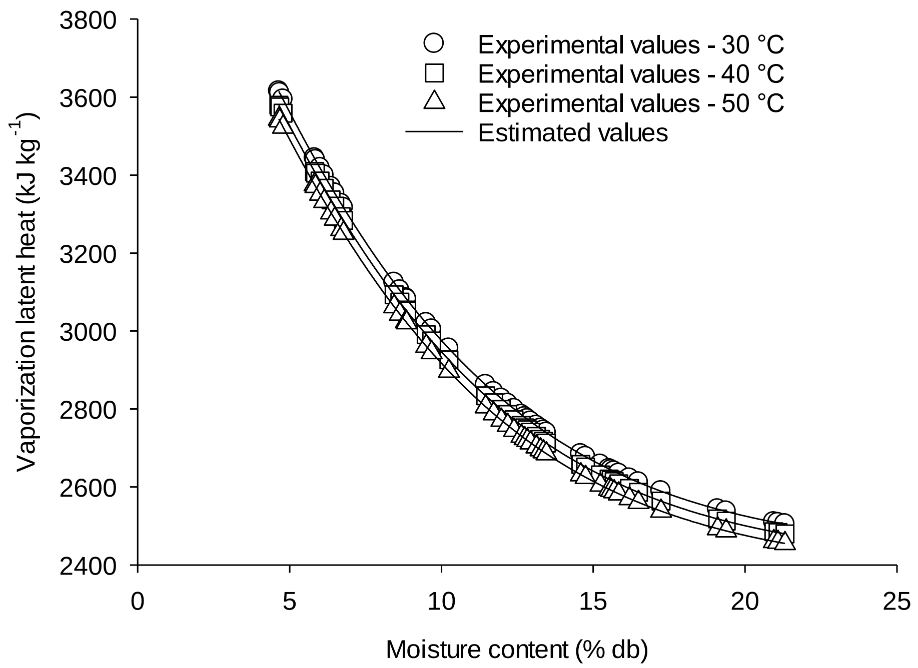

Figure 3 – Malagueta pepper seeds water vaporization latent heat curvesfor different temperatures.

It was noted that seeds water vaporization latent heat values increase with moisture content decrease. Moreover, it was verified that for the same moisture content, temperature increase caused vaporization latent heat reduction. Ferreira et al. (2011), Smaniotto et al. (2012), and Oliveira et al. (2014b), in studies with yellow cumarí seeds, corn grains, and tucumã-de-Goiás seeds, respectively, found similar behavior to that observed in this study.

According to Wang & Brennan (1991), water vaporization latent heat released during adsorption is an indicator of intermolecular attraction forces between the product water vapor adsorption sites. Thus, it can be seen in Figure 2 that the moisture content close to 0.21 L value for malagueta pepper seeds are of 2504.4 kJ kg^{-1} at 30 °C, 2479.8 kJ kg^{-1} at 40 °C, and 2455.1 kJ kg^{-1} at 50 °C. These L values are close to the L' value (2406.6 kJ kg^{-1}), indicating water molecules existence in the free form, with reduced attraction forces.

Pepper seeds water vaporization latent heat values, with moisture content in the range of 0.046 to 0.213 (db), varied from 3615.01 to 2455.14 kJ kg^{-1}. In studies with corn grains in the moisture content range from 0.127 to 0.233 (db), Smaniotto et al. (2012) found that vaporization latent heat varied from 2775.87 to 2468.14 kJ kg^{-1}. Oliveira et al. (2014c) found that, for purging nut seeds with moisture content in the range from 0.056 to 0.134 (db), vaporization latent heat ranged from 2762.92 to 2495.56 kJ kg^{-1}.

Differences in each product vaporization latent heat can be attributed to characteristics and factors related to them (Resende et al., 2006), such as moisture content, temperature (Brooker et al., 1992), and chemical composition.

Conclusions

In constant water activity, malagueta pepper seeds hygroscopic balance moisture content decreases with increasing temperature.

Modified Halsey and Modified Oswin models are the best to represent pepper seeds adsorption isotherms in the studied conditions.

Isotherm curves have a characteristic type II sigmoidal shape, which is common for most agricultural products.

Through moisture content reduction, there was increased energy released by seeds during water adsorption, and pepper seeds water vaporization latent heat values, in the moisture content range from 0.046 to 0.213 (db), varied from 3615.01 to 2455.14 kJ kg^{-1}.

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