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Hysteresis and thermodynamic properties of water sorption in ‘Malagueta’ pepper seeds

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Key words:

Capsicum frutescens L.
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ABSTRACT

The objective of this study was to determine hysteresis, enthalpy, entropy, enthalpy-entropy compensation theory and Gibbs free energy for water adsorption and desorption in ‘Malagueta’ pepper seeds. Hygroscopic equilibrium moisture contents were determined by the static gravimetric method, with water activity in the range from 0.29 to 0.90 and temperatures of 30, 40 and 50 °C. The hysteresis of the ‘Malagueta’ pepper seeds reduces with the increase of temperature. Enthalpy, entropy and Gibbs free energy of adsorption and desorption increase with the reduction of the moisture content of the seeds. The enthalpy-entropy compensation theory is valid for the sorption processes. The sorption of water between seeds and the surrounding air is a non-spontaneous process.

Palavras-chave:

Capsicum frutescens L.
entalpia
entropia
energia livre de Gibbs

Histerese e propriedades termodinâmicas de sorção de água em sementes de pimenta malagueta

RESUMO

Objetivou-se, neste trabalho, determinar histerese, entalpia, entropia, teoria da compensação entalpia-entropia e a energia livre de Gibbs para os processos de adsorção e dessorção de água em sementes de pimenta malagueta. Os teores de água de equilíbrio higroscópico foram determinados pelo método estático gravimétrico, com atividade de água na faixa de 0,29 a 0,90 e temperaturas de 30, 40 e 50 °C. A histerese das sementes de pimenta malagueta reduz com o aumento da temperatura. A entalpia, entropia e energia livre de Gibbs de adsorção e dessorção aumentam com a redução do teor de água das sementes. A teoria da compensação entalpia-entropia é válida para os processos sortivos. A sorção da água entre as sementes e o ar envolvente é um processo não espontâneo.



INTRODUCTION

Peppers and bell peppers of the genus *Capsicum* are traditionally used in Brazilian cuisine. Red pepper products, usually from the 'Malagueta' variety, pungent or non-pungent, characterize the peppers as one of the world's most important spice commodities (Heinrich et al., 2015). It is estimated that in 2016 the world production of fresh peppers and bell peppers has reached 51.9 million tons (FAOSTAT, 2018), which can be justified by the use of production technologies such as high-quality seeds.

Due to the hygroscopic behavior, the moisture content of seeds is influenced by the temperature and relative humidity of the air (Silva & Rodvalho, 2016). This behavior directly interferes with the biological activity and longevity of these seeds (Silva et al., 2015b).

The hygroscopicity of a product is defined as its ability to release or absorb water from the environment, phenomena known as water desorption and adsorption (Corrêa et al., 2014). The difference between adsorption and desorption isotherms (hysteresis) allows the evaluation of chemical and microbiological deterioration and stability of the product during storage (Goneli et al., 2016b).

From the isotherms, it is possible to determine thermodynamic parameters related to water sorption, such as enthalpy, entropy, enthalpy-entropy compensation theory and Gibbs free energy (Nkolo Meze'e et al., 2008; Oliveira et al., 2013; Silva et al., 2016). These thermodynamic parameters are essential to analyze the binding forces between water molecules and the product during the sorption processes (Costa et al., 2015; Yogendrarajah et al., 2015) and determine the minimum energy required in drying processes (Costa et al., 2013).

This study aimed to determine hysteresis, enthalpy, entropy, enthalpy-entropy compensation theory and Gibbs free energy related to water adsorption and desorption in 'Malagueta' pepper seeds.

MATERIAL AND METHODS

The experiment was conducted at the Food Laboratory of the Federal Institute of Goiás - Campus of Ceres, GO, Brazil (15° 16' 30" S; 49° 35' 54" W). Pepper seeds of the 'Malagueta' variety (*Capsicum frutescens* L.) were manually extracted from ripe fruits (completely red), harvested between January and March 2011 in the experimental area of the same institution.

After extraction, the seeds were subjected to pre-drying for 48 h under laboratory conditions to remove excess moisture content and divided into two samples. The first sample, with moisture content of approximately 46.0% (d.b.), was used in the desorption process; and the second one, used for the study on the adsorption process, was subjected to oven drying at temperature of 70 °C for 48 h, until it reached moisture content close to 2.5% (d.b.). The moisture content of the seeds was determined by the gravimetric method in an oven at 105 ± 3 °C for 24 h (Brasil, 2009).

Hysteresis was obtained by the difference between desorption and adsorption equilibrium moisture contents, calculated using the Modified Oswin model (Eqs. 1 and 2,

respectively). These models were recommended to represent the desorption isotherms (Silva & Rodvalho, 2012) and adsorption isotherms (Silva & Rodvalho, 2016) of 'Malagueta' pepper seeds at temperatures of 30, 40 and 50 °C and water activity ranging from 0.29 to 0.90 (dimensionless).

$$X_e = \frac{[17.8500^{**} + (-0.1795^{**})T]}{\left[\frac{a_w}{(1-a_w)}\right]^{\frac{1}{3.2995^{**}}}} \quad (1)$$

$$X_e = \frac{[12.2714^{**} + (-0.0912^{**})T]}{\left[\frac{a_w}{(1-a_w)}\right]^{\frac{1}{2.8304^{**}}}} \quad (2)$$

where:

** - significant at 0.01 by t-test;

X_e - hygroscopic equilibrium moisture content, % d.b.;

T - temperature, °C; and,

a_w - water activity, dimensionless.

The thermodynamic parameters differential enthalpy, differential entropy, enthalpy-entropy compensation theory and Gibbs free energy were calculated using the Eqs. 3-9 (Beristain et al., 1996; Madamba et al., 1996; Nkolo Meze'e et al., 2008).

$$\ln(a_w) = \frac{\Delta h_{st}}{RT} - \frac{\Delta S}{R} \quad (3)$$

$$\Delta S = \frac{\Delta h_{st} - \Delta G}{T_a} \quad (4)$$

$$\Delta h_{st} = T_B (\Delta S) + \Delta G_B \quad (5)$$

$$\Delta G = R T_a \ln(a_w) \quad (6)$$

$$T_{hm} = \frac{n}{\sum\left(\frac{1}{T}\right)} \quad (7)$$

$$T_B = \hat{T}_B \pm t_{m-2, \alpha/2, \sqrt{\text{Var}(T_B)}} \quad (8)$$

$$\text{Var}(T_B) = \frac{\sum\left(\Delta h_{st} - \overline{\Delta G_B} - \hat{T}_B \Delta S\right)^2}{(m-2)\sum\left(\Delta S - \overline{\Delta S}\right)^2} \quad (9)$$

where:

Δh_{st} - differential enthalpy of sorption, kJ kg⁻¹;

ΔS - differential entropy of sorption, kJ kg⁻¹ K⁻¹;

R - universal gas constant (8.314 kJ kmol⁻¹ K⁻¹), equal to 0.4619 kJ kg⁻¹ K⁻¹ for water vapor;

T_a - absolute temperature (K);

- ΔG - Gibbs free energy, kJ kg^{-1} ;
 T_B - isokinetic temperature, K;
 ΔG_B - Gibbs free energy at isokinetic temperature, kJ kg^{-1} ;
 T - temperature ($^{\circ}\text{C}$);
 T_{hm} - harmonic mean temperature, K;
 n - number of temperatures used; and,
 m - number enthalpy-entropy data pairs.

RESULTS AND DISCUSSION

The hygroscopic equilibrium moisture contents of 'Malagueta' pepper seeds obtained in the desorption process are greater than those obtained by adsorption in the entire range of water activity and temperatures studied (Figures 1A, B and C), thus evidencing the phenomenon of hysteresis (Figure 1D).

The hysteresis of the pepper seeds decreased with the increase in temperature (Figure 1D), showing a similar behavior to those observed for 'Bode' pepper (*Capsicum chinense* L.) (Rodvalho et al., 2015), millet (Aviara et al., 2016) and castor beans (Goneli et al., 2016b). For temperatures of 30, 40 and 50 $^{\circ}\text{C}$, the greatest magnitudes of hysteresis (3.54, 2.18 and 1.17% d.b.) were observed for 0.91, 0.77 and 0.44 (dimensionless) of water activity (Figure 1D).

The difference between the hygroscopic equilibrium moisture contents depends on the physical mechanism governing the movement of water from inside the product to its surface (Corrêa et al., 2014). Under high moisture content conditions, when almost all sorption sites are filled, desorption causes the shrinkage of the product and

consequently reduces the available sorption sites (Mohsenin, 1986). Shrinkage reduces the diameter of the pores, leading to less water diffusion (Goneli et al., 2016b), and reduces the binding capacity of the water during a subsequent adsorption (Mohsenin, 1986).

The reduction in the moisture content of pepper seeds caused an exponential increase in the differential enthalpy of desorption and adsorption, with lower values for the adsorption process (Figure 2A). This trend is indicative of the hysteresis phenomenon, as observed for millet (Aviara et al., 2016) and castor bean (Goneli et al., 2016a).

The difference between differential enthalpies of desorption and adsorption decreased with the increase in moisture content, tending to remain constant. According to Madamba et al. (1996), when the moisture content is high, the water comes out more easily from the seeds, thus less enthalpy is used. On the other hand, when the moisture content is low, it is more difficult to remove water and, therefore, there is higher demand of enthalpy.

The values of the differential enthalpy of adsorption, for pepper seeds with moisture contents from 4.6 to 21.3% (d.b.), ranged from 1,153.029 to 97.207 kJ kg^{-1} , while the differential enthalpy of desorption within the moisture content range from 7.0 to 24.7% (d.b.) varied from 1,998.435 to 149.079 kJ kg^{-1} (Figure 2A). In 'Cabacinha' pepper seeds with moisture content from 3.3 to 23.9% (d.b.), the differential enthalpy of desorption varied from 7.279 to 442.933 kJ kg^{-1} (Silva et al., 2016).

The differences between the differential enthalpy values observed for seeds of 'Malagueta' pepper (*C. frutescens* L.) and

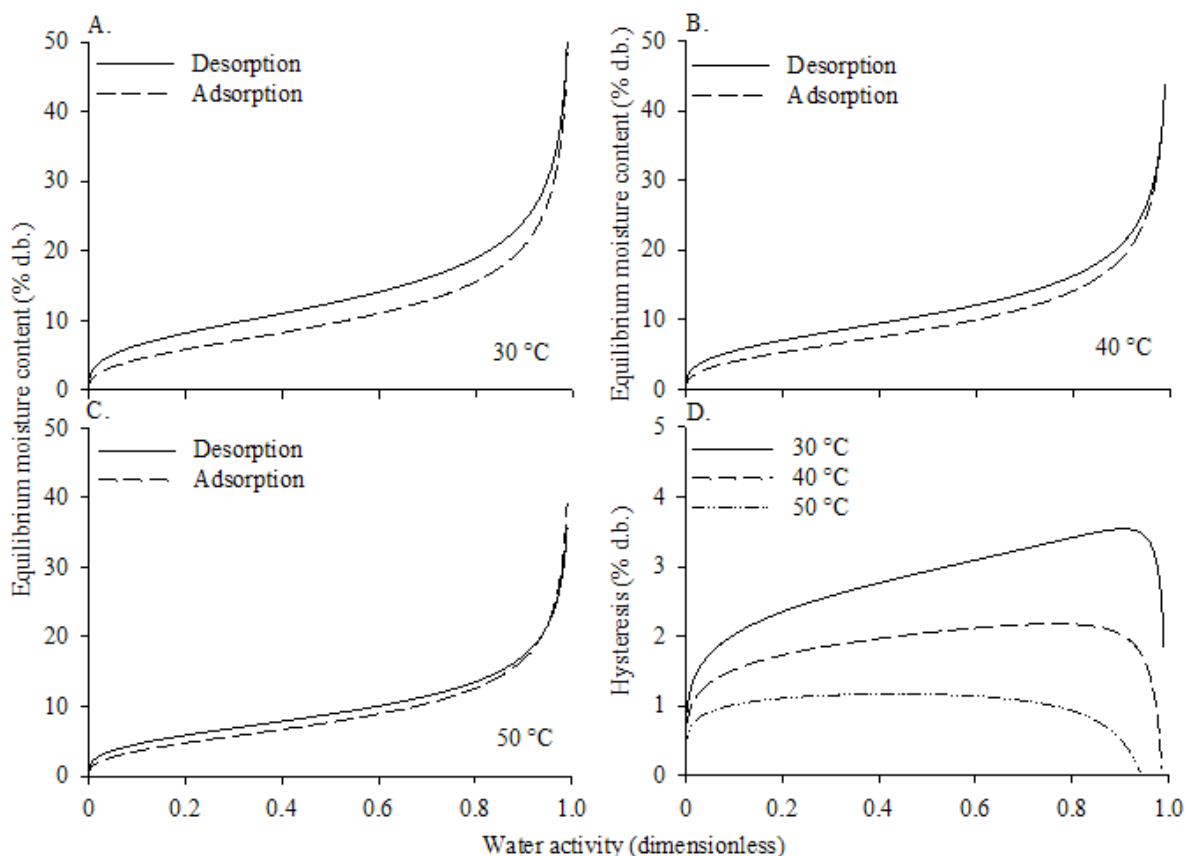


Figure 1. Estimated values of desorption and adsorption isotherms (A, B, C) and hysteresis (D) of 'Malagueta' pepper seeds (*Capsicum frutescens* L.) for different conditions of temperature and water activity

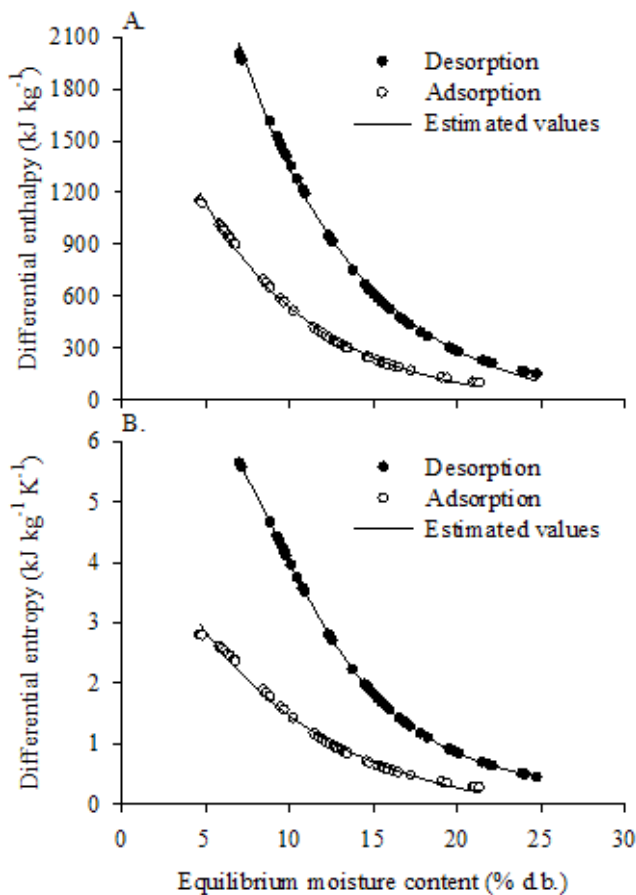


Figure 2. Experimental and estimated values of differential enthalpy (A) and differential entropy (B) of desorption and adsorption as a function of equilibrium moisture contents of 'Malagueta' pepper seeds (*Capsicum frutescens* L.)

'Cabacinha' pepper (*C. chinense* L.) can be attributed to the difference in chemical composition between the two species, as found for two cultivars of corn (Oliveira et al., 2010) and two varieties of millet (Aviara et al., 2016).

For the moisture content range from 4.6 to 21.3% (d.b.) and 7.0 to 24.7% (d.b.), the differential entropy of adsorption and desorption varied from 2.804 to 0.276 kJ kg⁻¹ K⁻¹ and from 5.643 to 0.447 kJ kg⁻¹ K⁻¹, respectively (Figure 2B). It is noted that the reduction in moisture content increases the differential entropy, as well as the difference between adsorption and desorption values.

Entropy has been related to the number of sorption sites available at a specific level of energy (Silva et al., 2015a). Therefore, the high values of entropy observed for the lowest moisture contents of pepper seeds demonstrate the reduction of movement and the degree of freedom of water molecules that are strongly bound to the active sites of the seeds. Under this condition, the energy needed to remove water bound to the active sites is greater compared with those that have high moisture content. This is because in the hydrated state, there is greater freedom for water molecules, which are weakly adsorbed to the seeds, to form the monolayer (Silva et al., 2015a; Yogendrarajah et al., 2015).

The high coefficients of determination ($R^2 > 0.99$) obtained by Eq. 5 and presented in Table 1 indicate the high degree of linearity of the enthalpy-entropy ratio for water desorption and

Table 1. Parameters obtained by the enthalpy-entropy ratio for the processes of desorption and adsorption in 'Malagueta' pepper seeds (*Capsicum frutescens* L.)

Process	T_b (K)	ΔG_b (kJ kg ⁻¹)	R^2
Desorption	350.759	-21.110	0.999
Adsorption	402.221	-35.274	0.998

adsorption in pepper seeds. This suggests the existence of the enthalpy-entropy compensation phenomenon for the processes of desorption and adsorption in 'Malagueta' pepper seeds.

Originally applied by Bell (1937), the enthalpy-entropy compensation theory is an important tool in evaluating water sorption mechanisms under different conditions, such as drying processes (Rizvi, 1995). It has been observed that generally the changes in enthalpy and entropy occur simultaneously (Oliveira et al., 2013; Corrêa et al., 2015; Goneli et al., 2016a, b; Silva et al., 2016), which according to Leffler (1995) allows to verify greater molecular interaction or bonds between molecules due to the reduction in the freedom or to the binding of the molecules in the system.

To test the validity of the enthalpy-entropy compensation phenomenon, Krug et al. (1976a,b) suggested that the isokinetic temperature (T_b) should be different from the harmonic mean temperature (T_{hm}). Thus, the magnitude of the isokinetic temperatures (Table 1) for desorption (350.76 ± 3.57 K) and adsorption (402.22 ± 10.04 K) processes compared with the harmonic mean temperature (311.45 K), confirms that the isokinetic theory is valid for sorption processes in 'Malagueta' pepper seeds and corroborate the results obtained for seeds of cucumber (Corrêa et al., 2015), forage turnip (Sousa et al., 2015) and 'Cabacinha' pepper (Silva et al., 2016).

The isokinetic temperature has been used to characterize the temperature at which the product is in equilibrium, i.e., when all reactions occur simultaneously (Goneli et al., 2016a). For Leffler (1955), if $T_b > T_{hm}$, the sorption process is controlled by enthalpy; otherwise ($T_b < T_{hm}$), the process is controlled by entropy. Therefore, the desorption and adsorption processes of 'Malagueta' pepper seeds are controlled by the enthalpy, following the same behavior of various agricultural products (Oliveira et al., 2013; Goneli et al., 2016a; Silva et al., 2016).

The increase in temperature led to reduction in Gibbs free energy of desorption (Figure 3A) and adsorption (Figure 3B), particularly for the desorption process. Gibbs free energy of desorption and adsorption, as well as the effect of temperature on this thermodynamic parameter, decreased at high moisture contents. It is known that Gibbs free energy is related to the work required to make the sorption sites available (Nkolo Mezeè et al., 2008). Therefore, the reduction of this variable with the increment in moisture content demonstrates the increase in the number of sites available for sorption.

The Gibbs free energy of desorption and adsorption showed positive values for the different temperatures and moisture contents analyzed (Figure 3). This trend is characteristic of an endothermic reaction (Goneli et al., 2016a), i.e., a reaction which requires the input of energy in the system for water desorption or adsorption to occur in the seeds (Oliveira et al., 2013; Sousa et al., 2015); in other words, water sorption processes in 'Malagueta' pepper seeds are not spontaneous.

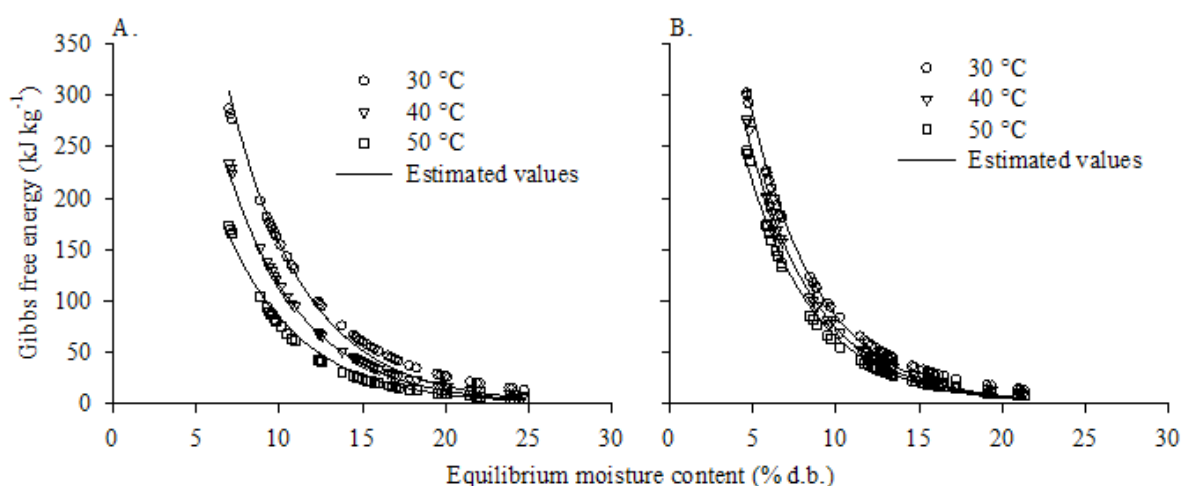


Figure 3. Experimental and estimated values of Gibbs free energy of desorption (A) and adsorption (B) as a function of temperature and equilibrium moisture content of 'Malagueta' pepper seeds (*Capsicum frutescens* L.)

The regression equations fitted to the values of differential enthalpy, differential entropy and Gibbs free energy of desorption and adsorption (Table 2) had a high degree of fit to experimental data ($R^2 > 0.99$) and can be used to estimate these thermodynamic parameters for the desorption and adsorption processes of 'Malagueta' pepper seeds with moisture contents from 7.0 to 24.7% (d.b.) and from 4.6 to 21.3% (d.b.), respectively, at temperatures of 30, 40 and 50 °C.

Table 2. Regression equations and coefficients of determination for differential enthalpy (Δh_{st}), differential entropy (ΔS) and Gibbs free energy (ΔG) of water adsorption and desorption in 'Malagueta' pepper seeds (*Capsicum frutescens* L.)

Regression equations	R ²
Desorption	
$\Delta h_{st} = -77.385^{**} + 5560.307^{**} \exp(-0.137^{**} X_e)$	0.999
$\Delta S = -0.332^{**} + 15.064^{**} \exp(-0.127^{**} X_e)$	0.998
$\Delta G = (-1277.526^{**} \ln(T) + 5757.819^{**}) \exp(-0.220^{**} X_e)$	0.996
Adsorption	
$\Delta h_{st} = -37.972^{**} + 2398.093^{**} \exp(-0.143^{**} X_e)$	0.999
$\Delta S = -0.267^{**} + 5.599^{**} \exp(-0.118^{**} X_e)$	0.997
$\Delta G = (-423.430^{**} \ln(T) + 2361.314^{**}) \exp(-0.237^{**} X_e)$	0.998

**Significant at 0.01 by t-test

CONCLUSIONS

1. The magnitude of hysteresis in 'Malagueta' pepper seeds decreases with the increment in temperature.
2. Differential enthalpy and differential entropy of adsorption and desorption increase with the reduction in the moisture content of pepper seeds, being higher for the desorption process.
3. For the studied ranges of moisture content, the differential enthalpies of adsorption and desorption varied from 1,153.029 to 97.207 kJ kg⁻¹ and from 1,998.435 to 149.079 kJ kg⁻¹, respectively; whereas the differential entropy varied from 2.804 to 0.276 kJ kg⁻¹ K⁻¹ for adsorption and from 5.643 to 0.447 kJ kg⁻¹ K⁻¹ for desorption.
4. The enthalpy-entropy compensation theory is valid for water adsorption and desorption in pepper seeds, and these processes are controlled by enthalpy.
5. The water sorption process in pepper seeds is not spontaneous.

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