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MOISTURE SORPTION ISOTHERMS AND HEAT OF SORPTION OF ALGERIAN BAY LEAVES (*Laurus nobilis*)

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ABSTRACT

The moisture sorption isotherms of Algerian bay leaves (*Laurus nobilis*) were determined experimentally in this work. The equilibrium moisture contents of the leaves were measured at 40, 50, and 60 °C using static gravimetric method. Six mathematical models were tested to fit the experimental data of sorption isotherms and predict the hygroscopic behavior during storage or drying. Peleg model was found to be the best fitting model for describing the sorption curves. The net isosteric heat of sorption was computed from the equilibrium data at different temperatures by applying the integrated form of the Clausius-Clapeyron equation.

The net isosteric heat of sorption is inversely proportional to the equilibrium moisture content and is found to be an exponential function of moisture content.

Keywords: Adsorption, desorption, equilibrium moisture content, *Laurus nobilis* leaves, net isosteric heat, sorption isotherms, statistical analysis.

INTRODUCTION

The vegetal world is full of resources and virtues, from which man draws not only food but also its active substances, which often provide a benefit to the organism sometimes affected with insidious disorder. Algeria has splendid gardens and evergreen lands with virtuous plants; they are frequently used for several applications; pharmacies, medicines and food among them the bay leaves. This tree belongs to the Lauraceae family and is native from the southern Mediterranean region (Baba-Aissa 1999). Currently, the plant is cultivated in many countries such as Turkey, Algeria, France, Greece, Morocco, Portugal, Spain, Belgium, Canary Islands, Mexico, Central America and the southern United States. Bay leaves are green smooth, leathery glossy top and paler underneath; they are also widely used in traditional medicine thanks to its medicinal properties: anti parasitic, stimulant, anticonvulsant, stomachic, pedicle (Beloued 1998, Ben-Jemaa *et al.* 2012), antidiabetics (Afifi *et al.* 1997)and can act against the gastro-intestinal problems, rheumatism, diuretic, urinary problems and stones (Sellami *et al.* 2011).

The plants are used fresh, nevertheless they undergo physical, chemical and microbiological changes influenced by the moisture content of the product, water activity and storage temperature (Lahsasni *et al.* 2002).This will generate degradation quality of product in a few days thus drying of the plant prior to their storage is therefore needed for maintaining them stable.

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The sorption isotherms are extremely important quantitative measures in the conservation, the storage and the packaging of the finished products (Rajia and Ojediran 2011, Sargent *et al.* 2010). They represent the relationship between the equilibrium moisture content and the relative humidity and allow to draw insights about the distribution and intensity of connections of water in the existing products and the hygroscopic equilibrium.

Several methods are given in the literature to determine experimentally the sorption isotherms. Mathematical models have been highlighted to better describe the sorption isotherm; over 77 equations with two or more parameters have been proposed in the literature (Boudhrioua *et al.* 2008). Among these models: Model of GAB (Van der Berg and Bruin 1981), Peleg (Peleg 1993), Smith (Smith 1947), white and Eiring (Castillo *et al.* 2003), Henderson Thompson (Thompson *et al.* 1968) and Caurie (Castillo *et al.* 2003).

On the other hand, one of the essential parameters that must be known for the storing and drying processes is the isosteric heat of sorption. It represents the energy released during the adsorption; during desorption it symbolizes the energy needed to break the intermolecular forces between the molecules of the water vapor and the surface of the adsorbent (Rizvi 1995).

It is established using the sorption isotherms data and serves for the design and optimization of the dryers.

Over the past two decades, a large number of studies have focused on the sorption isotherms of aromatic and medicinal plants (Bahloul *et al.* 2008, He *et al.* 2013, Boudhrioua *et al.* 2008, Bejar *et al.* 2012, Martinez *et al.* 2014).

So this study was undertaken to determine the sorption isotherms for bay leaves at three temperatures (40, 50, 60 °C) using the static gravimetric method. In addition, analytical models were used to adjust the experimental data and determine the net isosteric heat of sorption of the plant at different moisture contents.

MATERIALS AND METHODS

Experimental procedure

Bay leaves used in this study were grown in the Bouzaréah region located at 4 km from the Algiers city centre, Algeria.

The sorption isotherms of bay leaves (*Laurus nobilis L.*) were determined for three temperatures 40, 50, and 60 °C using standard static gravimetric method (Lang *et al.* 1981). This method requires a lot of time for reaching the hygroscopic equilibrium; it has the advantage of presenting a more restricted domain of moisture content variation. This method is based on the use of six saturated salt solutions prepared by solubilizing an appropriate amount of salt in distilled water at each temperature. Saline solutions were used to maintain constant relative humidity (Greenspan 1977).

The experimental setup is shown in Figure 1. It consists of six glass jars of 1 liter each with an insulated protective cover for avoiding rehumidification of the samples. Each glass jar is filled to the quarter with a saturated salt solution. An aluminum sample holder containing (0,2 ± 0,0001g) of bay leaves is deposited on a PVC tube, which is placed in the jar. Each jar contains a different salt solution (KOH, $MgCl_2$, K_2CO_3, $NaNO_3$, KCl and $BaCl_2$) in order to have water activities from 0,05 to 0,90 (Table 1) (Dumoulin *et al.* 2004).

Table 1. The water activities of saturated salt solution at 40, 50, 60 °C.

Salt	Water Activity aw (-)		
	40°C	50 °C	60 °C
KOH	0,0626	0,0572	0,0549
$MgCl_2$, H_2O	0,3159	0,3054	0,2926
K_2CO_3	0,4230	0,4091	0,3921
$NaNO_3$	0,7100	0,6904	0,6735
KCL	0,8232	0,8120	0,8025
$BaCl_2$, H_2O	0,8910	0,8823	0,8728

Figure 1. Experimental apparatus for the sorption isotherms measurement.

The jars are placed in an oven at a fixed temperature for 24 hours. In order to obtain desorption isotherms; samples should be moistened so they lose moisture in the jars with saline solution. For adsorption, the samples must be pre-dried in an oven at 50°C to acquire moisture in the jar (Lahsasni *et al.* 2002, Ait Mohamed *et al.* 2005).

The six samples were weighed each three days with a precision balance (± 0,0001g) until equilibrium was reached.

The equilibrium moisture content of each sample was determined in a drying oven at 105 °C (± 0,1 °C) for 24 hours (AOAC 1990).

The difference of mass before and after drying in the oven was used to determine the moisture content *Xe* of the product at equilibrium

$$Xe = \frac{M_w - M_d}{M_d} \quad (1)$$

M_w: the mass of the sample before drying;
M_d: the mass of the sample after drying;
Xe: equilibrium moisture content.

DATA ANALYSIS

Modeling of sorption isotherms

Various models have been proposed and tested in the literature for correlating the equilibrium moisture content with relative humidity. Six models were selected for fitting the experimental data of sorption isotherms of bay leaves for 40, 50 and 60 °C and are reported in Table 2.

For the analysis of nonlinear regression, computer programs are used to estimate the constant models from experimental results. The goodness of fit was determined using, the mean relative percentage deviation (*P %*), standard deviation (*S*) and the correlation coefficient (*r*).

Table 2. The models applied to the experimental sorption data of *Laurus nobilis* leaves.

Name of model	Model equation	Reference
GAB	$X_e = \frac{a\,b\,c\,a_w}{(1-ca_w)(1-ca_w+cb\,a_w)}$	Van der Berg and Bruin 1981
Henderson-Thompson	$X_e = \left[\frac{\ln((1-a_w)}{-a(T+c)}\right]^{1/b}$	Thompson *et al.* 1968
White and Eiring	$X_e = \frac{1}{a+ba_w}$	Castillo *et al.* 2003
Peleg	$X_e = a(a_w)^b + c(a_w)^d$	Peleg 1993
Smith	$X_e = a - b\,(\ln(1-a_w))$	Smith 1947
Caurie	$X_e = exp(\mathrm{a} + \mathrm{ba_w})$	Castillo *et al.* 2003

Where *a, b, c, d* are parameters of the equations, *T* is temperature (°C), *Xe* is equilibrium moisture content (kg/kg d.b.) and a_w is the water activity.

Each parameter was determined using equations (2-5) (Bahloul *et al.* 2008):

$$P = \frac{100}{N}\sum_{j=1}^{N}\frac{Xe_{jcal} - Xe_{j\exp}}{Xe_{j\exp}} \tag{2}$$

$$S = \sqrt{\sum_{j=1}^{N}\left|\frac{\left(Xe_{jcal} - Xe_{j\exp}\right)^2}{N - n_p}\right|} \tag{3}$$

$$r = \sqrt{1 - \frac{\sum_{j=1}^{N}\left(Xe_{jcal} - Xe_{j\exp}\right)^2}{\sum_{j=1}^{N}\left(\overline{Xe} - Xe_{j\exp}\right)^2}} \tag{4}$$

Where:

$$\overline{Xe} = \frac{1}{N}\sum_{j=1}^{N} Xe_j \tag{5}$$

Where: Xe_{jcal} and Xe_{jexp} are predicted and experimental values, respectively. Values of P below 10% are indicative of a good fit (Aguerre *et al.* 1989, Park *et al.* 2002).

Determination of the isosteric heat

The net isosteric heat of sorption can be determined from moisture sorption data using the following equation which is derived from Clausius-Clapeyron relation (equation 6) (Ait Mohamed *et al.* 2005, Janjai *et al.* 2006).

$$Q_{st} = -R\left[\frac{\partial(\ln a_w)}{\partial\left(\frac{1}{T}\right)}\right] \tag{6}$$

Where aw is the water activity, Qst the net isosteric heat of sorption (kJ/mol), R the universal gas constant (kJ/mol K) and T is absolute temperature (K).

The integration of equation (6) leads to equation (7):

$$\ln(a_w) = -\left(\frac{Q_{st}}{R}\right)\frac{1}{T} + K \tag{7}$$

The net isosteric heat of sorption can be calculated from Eq. (7) by plotting the sorption isotherm as $ln\ a_w$ against $1/T$ for a specific moisture content of material and determining the slope which equals Q_{st}/R. This procedure is repeated for all values of the equilibrium water content.

Marquardt–Levenberg (Jamali *et al.* 2006) non-linear optimization method, using the computer programs Curve Expert 1.4, and Origin 8.5 were used to find the best equation for bay leaves sorption isotherms.

RESULTS AND DISCUSSION

Experimental results (desorption and adsorption isotherms)

The hygroscopic equilibrium of bay leaves is achieved respectively after 18 days for desorption and 15 days for adsorption. Figures 2 and 3 show the experimental results of sorption obtained for each working temperature 40, 50 and 60 °C.

The figures of sorption isotherms have a sigmoidal shape and are of type II according to the BET classification, in agreement with the behavior of other medicinal and aromatic plants and food products (Boudhrioua *et al.* 2008, Bejar *et al.* 2012).

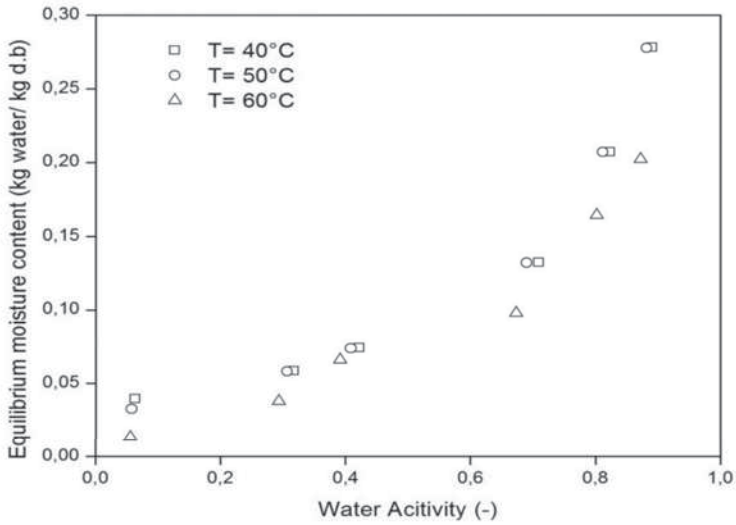

Figure 2. Influence of temperature on the adsorption isotherms of *Laurus nobilis* leaves.

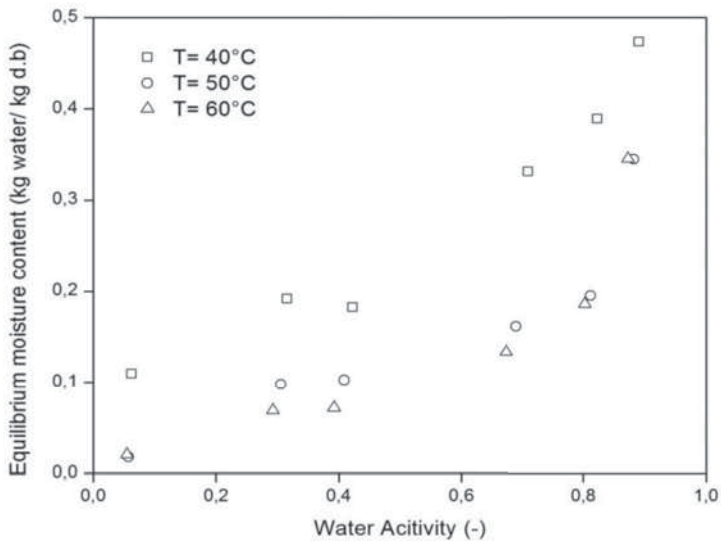

Figure 3. Influence of temperature on the desorption isotherms of *Laurus nobilis* leaves.

These isotherms show that for the same value of water activity, moisture content at equilibrium decreases with increasing temperature. This result can be explained by considering that the temperature increase leads to the decrease of the binding energy between water molecules present in the product, and consequently reduces the attractive forces between the water molecules and sorption sites (Al-Muhtaseb *et al.* 2002, Miranda *et al.* 2012). Similarly, at a constant temperature, the equilibrium moisture content increases with increasing water activity. Similar results were observed in several other studies for aromatic and medicinal plants (Bejar *et al.* 2012, He *et al.* 2013, Martinez *et al.* 2014).

The different equilibrium moisture contents observed at the same water activity, between adsorption and desorption processes constitute the hysteresis cycle (Figure 4) (Salin 2011).It occurs during the adsorption or desorption, due to changes in the structure and porosity.

Moreira *et al.* 2005, can be explained this phenomenon by pre-drying at which the samples were submitted in the case of adsorption. The pre-drying changes and closes the pores and water binding sites in the bay leaves (by shrinkage and/or chemical processes) (Bahloul *et al.* 2008, Bejar *et al.* 2012).

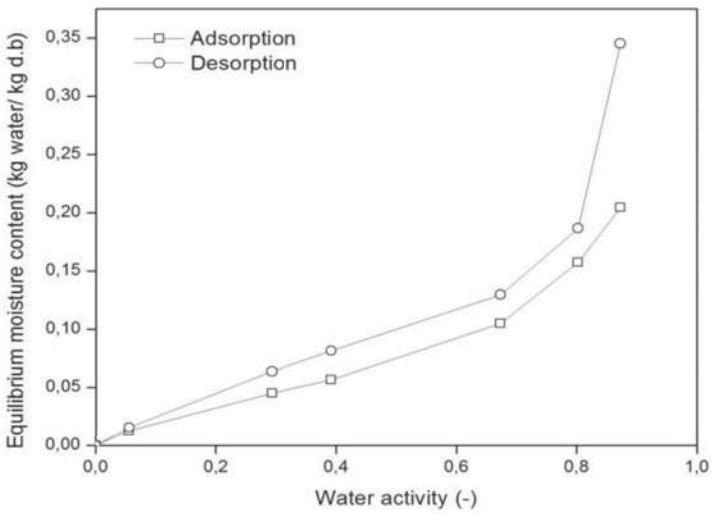

Figure 4. Adsorption and desorption isotherms of *Laurus nobilis* leaves at T=60 °C.

Fitting of sorption models to experimental data

A number of empirical and semi-empirical equations have been proposed to fit the experimental data of the water content at equilibrium. Among these models Henderson Thompson, Curie, Smith, Peleg, GAB, White and Eiring were chosen.

The results of the nonlinear regression analysis of experimental data for the bay leaves and the coefficients of each model, as well as their statistical average ratio (*P*) and the standard error of the estimate (*S*) are presented in Tables 3 and 4.

Table 3. Results of fitting of the desorption isotherms of *Laurus nobilis* leaves.

		Desorption isotherms						
Models names	T (° C)	Parameters				r	S	P (%)
		a	b	c	d			
GAB	40	0,1408	54,613	0,789	-	0,995	0,018	4,464
	50	0,0556	22,343	0,943	-	0,975	0,0321	27,26
	60	0,0394	35,104	1,013	-	0,992	0,019	16,562
White and Eiring	40	7,788	-6,387	-	-	0,99	0,021	8,467
	50	20,272	-19,577	-	-	0,969	0,0309	46,424
	60	26,792	-27,330	-	-	0,99	0,018	26,01
Peleg	40	0,233	0,268	0,3706	3,69	0,995	0,0218	4,682
	50	5,308	28,564	0,214	0,759	0,998	0,0102	9,786
	60	2,4192	18,862	0,1787	0,838	0,999	0,009	7,7
Smith	40	0,108	0,166	-	-	0,994	0,017	6,128
	50	0,0224	0,132	-	-	0,958	0,0357	26,293
	60	0,00208	0,142	-	-	0,956	0,038	22,93
Caurie	40	-2,338	1,745	-	-	0,992	0,0195	5,513
	50	-3,573	2,686	-	-	0,944	0,0412	32,07524,8
	60	-4,409	3,699	-	-	0,958	0,038	
Henderson Thompson	40	10,002	1,913	-39,004-	-	0,983	0,0335	11,35
	50	10,476	1,173	48,794	-	0,953	0,0436	18,363
	60	8,154	0,807	-58,454	-	0,962	0,041	33,471

P is average relative error; *S* is standard error; *r* is correlation coefficient; a, b, c, d are parameters of the equations; T is temperature (°C)

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Table 4. Results of fitting of the adsorption isotherms of *Laurus nobilis* leaves.

Adsorption isotherms								
Models names	T (°C)	Parameters				r	S	P (%)
		a	b	c	d			
GAB	40	0,045	11,612	0,942	-	0,998	0,0075	6,626
	50	0,044	19,70	0,957	-	0,996	0,0105	9,958
	60	0,0467	6,753	0,894	-	0,995	0,0098	8,784
White and Eiring	40	22,875	-21,687	-	-	0,998	0,006	5,348
	50	22,573	-21,569	-	-	0,996	0,0091	10,019
	60	28,688	-27,390	-	-	0,980	0,0161	38,3
Peleg	40	0,0866	0,2900	0,37	5,577	0,9996	0,004	2,47
	50	0,0966	0,387	0,368	5,457	0,999	0,0032	2,061
	60	0,113	0,747	0,235	6,089	0,996	0,0111	8,3
Smith	40	0,018	0,11	-	-	0,989	0,0155	11,357
	50	0,0149	0,117	-	-	0,992	0,0131	9,781
	60	0,0088	0,0927	-	-	0,993	0,0098	7,99
Caurie	40	-3,911	2,887	-	-	0,986	0,0174	15,034
	50	- 3,905	2,919	-	-	0,991	0,0143	12,745
	60	-4,113	2,861	-	-	0,990	0,0114	14,208
Henderson - Thompson	40	9,772	1,054	-38,906	-	0,982	0,023	19,546
	50	10,443	1,048	-48,776	-	0,987	0,0195	18,956
	60	14,848	1,099	-58,778	-	0,991	0,0125	14,192

P is average relative error; *S* is standard error; *r* is correlation coefficient; a, b, c, d are parameters of the equations; T is temperature (°C).

The results reported in Tables 3 and 4 show that the model of Peleg gives the best fit to the experimental data obtained from the desorption and adsorption isotherms of bay leaves for a range of water activity between 0,05 and 0,9 (Figure 5). Peleg model provides a correlation coefficient from 0,995 to 0,999 and an average relative error (*P %*) is ranging from 4,664 to 9,786% for desorption and 2,47 to 8,3% for the adsorption.

Peleg model was successfully applied to several aromatic and medicinal plants such as olive leaves (Boudhrioua *et al.* 2008, Bahloul *et al.* 2008) and orange leaves (Bejar *et al.* 2012). These leaves have the same texture even though they differ in their geometries. The fitting of the sorption isotherms by Peleg model at different temperatures is displayed in Figure 5.

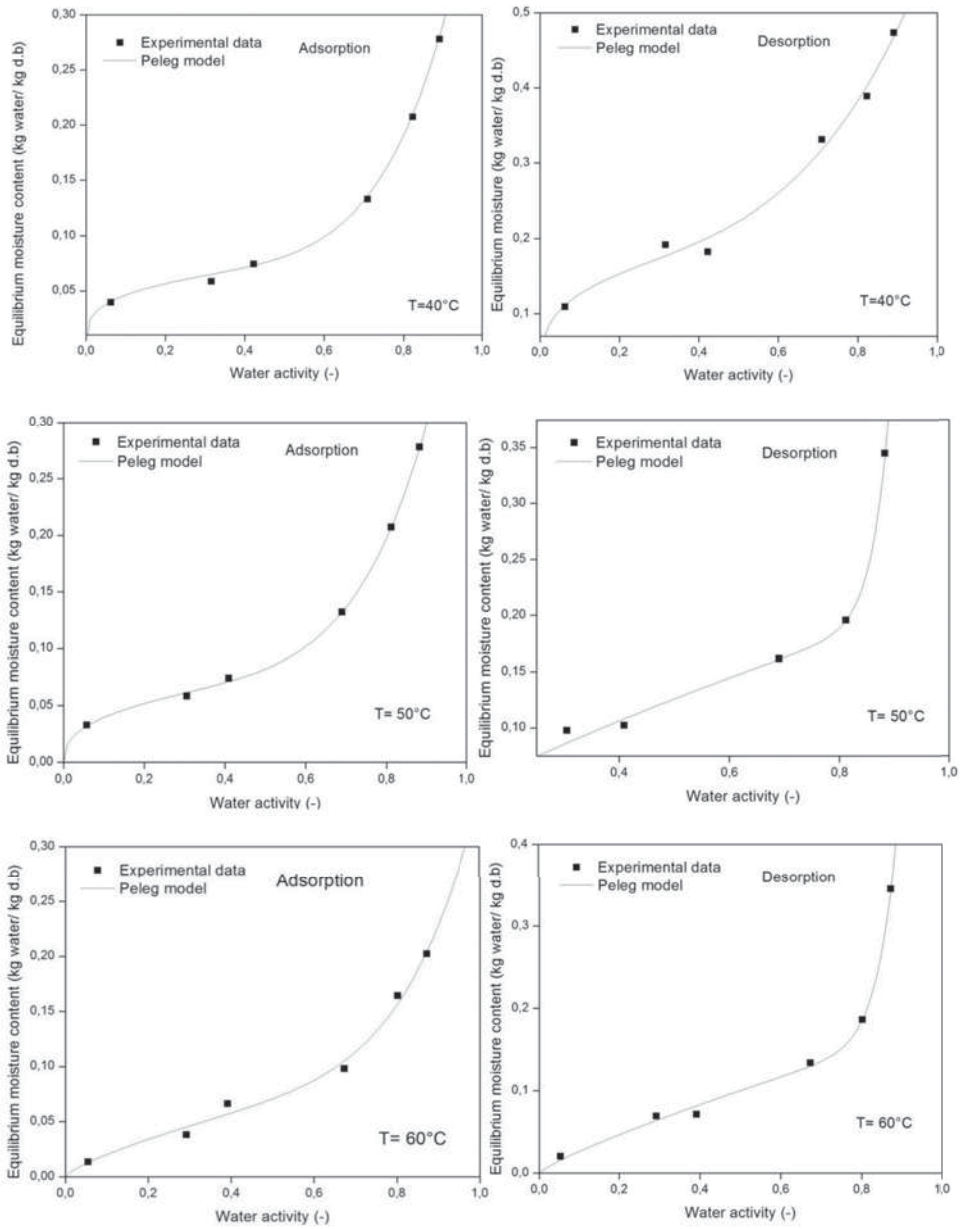

Figure 5. Adsorption and desorption isotherms of *Laurus nobilis* leaves fitted by Peleg model.

Isosteric heat of sorption

Net isosteric heat of sorption of bay leaves was determined from experimental data of sorption isotherms. At each experimental temperature, Peleg model was used to obtain the values of the water activity at constant moisture content.

Net isosteric heat of sorption values were calculated from the slope of the curve (ln (aw) as a function of (1/T)) at constant moisture content as shown in Figure 6.

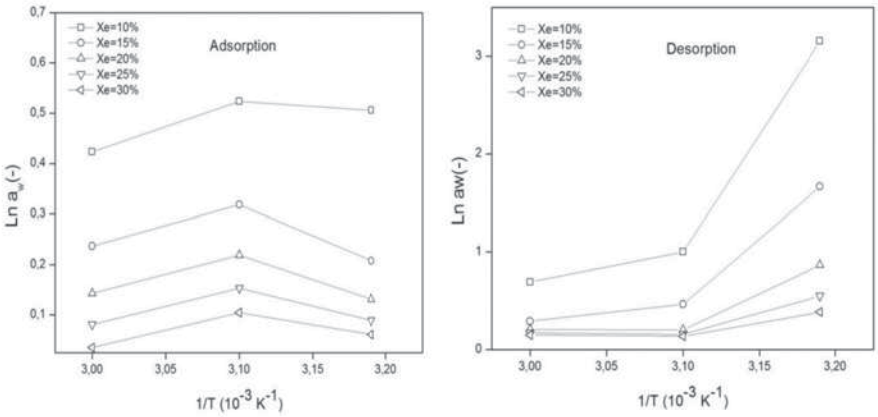

Figure 6. Ln (aw) vs 1/T graphs for calculating the adsorption and desorption heat of *Laurus nobilis* leaves.

Net isosteric heat of sorption of bay leaves were determined by combining the model Peleg with equation (7) for different moisture content, they are shown in Figure 7 for adsorption and desorption.

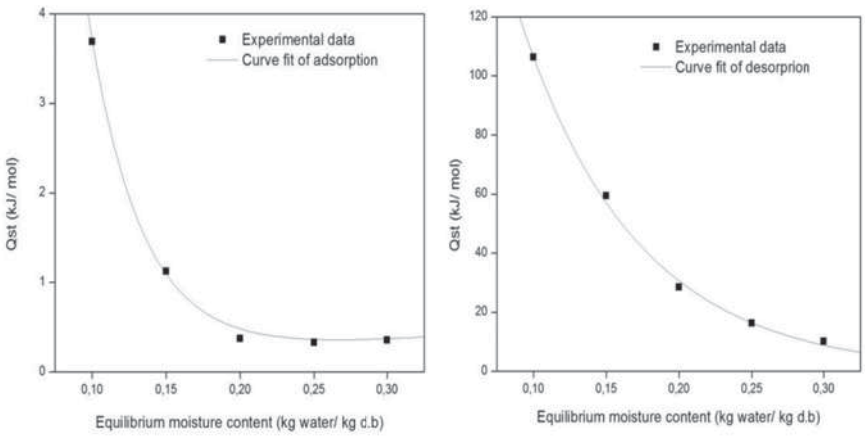

Figure 7. Net isosteric heat for adsorption and desorption for different moisture contents.

These results show that the net isosteric heats decreases with the increase of moisture content, indicating that the maximum energy is being used to remove the water present in the product at low moisture content. This phenomenon could be due to the presence of highly active polar sites on the surface of the product, which are covered with water molecules, forming a monomolecular layer (Tsami 1991). Another explanation to this phenomenon could be the elevation of the isosteric heat of sorption at low humidity due to higher resistance to water movement from the inside to the surface of the samples (Iglesias and Chirife 1976). Similarly, the net isosteric heat of adsorption is lower than that of the desorption.

Similar results for other medicinal plants have been reported in the literature (Boudhrioua *et al.* 2008, Bejar *et al.* 2012, Bahloul *et al.* 2008, Lahsasni *et al.* 2002).

The net isosteric heat of adsorption and desorption of water in bay leaves can be expressed mathematically by an exponential function of the moisture content for the adsorption and desorption:

For adsorption: r = 0,998; S = 0,173

$$Q_{st} = 0{,}157\exp(2{,}76Xe) + 58{,}854\exp(-28{,}21Xe) \quad (8)$$

For desorption: r = 0,999; S = 3,392

$$Q_{st} = 185{,}2\exp(-12{,}5Xe) + 185{,}30\exp(-12{,}44Xe) \quad (9)$$

These mathematical relationships may be used to calculate the heat of sorption of bay leaves for various moisture contents.

CONCLUSIONS

The sorption isotherm curves were determined experimentally for the bay leaves at three temperatures (40, 50, 60 °C) and relative humidity within the range of 5-90% commonly used in the drying and storage of bay leaves. The experimental data of the moisture equilibrium of the plant have been fitted with six mathematical models to determine the best model for predicting desorption and adsorption. Among the selected models, the model of Peleg was the most appropriate for describing the curves of adsorption and desorption. The sorption curves have a sigmoidal shape. The hysteresis phenomenon was observed for the range of temperature studied. The net isosteric heats of sorption were determined for bay leaves by combining the model of Peleg with the Clausius-Clapeyron equation. The heat of sorption was inversely proportional to the amount of moisture and is found to be an exponential function of moisture content. Two equations were developed to describe variations of isosteric sorption heats. These results represent a preliminary phase of the study of the drying kinetics of bay leaves in a convective indirect solar dryer.

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