

Thermodynamic properties and moisture sorption isotherms of *Matricaria pubescens* leaves

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Abstract: Experimental adsorption and desorption behavior of *Matricaria pubescens* was determined using the gravimetric static method at 30 °C, 40 °C and 50 °C, for water activity ranging from 0.057 to 0.898. Experimental data were compared with eight widely recommended sorption models (Caurie, BET, Henderson-Thomson, Smith, Halsey, Oswin, Peleg, and Adam and Shove) in literature. Peleg and Halsey models were found to be the most suitable for describing sorption isotherm curves. The net isosteric heats of sorption were also calculated by using the Clausius-Clapeyron equation.

Keywords: *Matricaria. p.*, sorption isotherm, modeling, isosteric heat of sorption.

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1 Introduction

Matricaria pubescens, or Saharan chamomile is an endemic plant from Northern Africa, belonging to the family *Asteraceae*, it has a very pleasant smell. Flowering takes place in the spring in the Northern Algerian Sahara. Several researches have highlighted the presence of essential oils, alkaloids, flavonoids, phenols, tannins, steroids and glycosides in the aerial part of this plant (Gherboudj et al., 2012; Metrouh-Amir and Amir, 2018; Cherif et al., 2017; Kherraz et al., 2019). The plant has antibacterial and antiseptic properties (Makhloufi et al., 2012; Metrouh-Amir et al., 2015). The plant is locally called 'ouazouza', it is a much appreciated medicinal plant and it is used by the local Saharan population for the treatment of coughs, allergies and eye problems, gastrointestinal troubles and calculus and to

perfume and preserve the melted butter (Ma ĩza et al., 1996; Ozenda, 2004; IUCN, 2005).

In Algeria, collection of medicinal and aromatic plants for the manufacture of cosmetics, pharmaceuticals as well as flavors for food products, is a virgin field, despite the important plant resources distributed on the coasts, plains, mountains, steppes and the Sahara. The vast majority of species are harvested and dried in the open air, then delivered and commercialized in bulk (Reguieg, 2011).

Fresh plants have a high water content of 80% and above, and are exposed to various microbial attacks. Water in a plant has the same properties as the pure water, so it serve as a medium for various chemical, enzymatic and microbial reactions, which lead to the ultimate loss of quality and nutritional value. At lower humidity values, water is strongly bound and cannot intervene in deterioration reactions. Therefore, it is not the water content that plays a role in the deterioration process but rather its condition, its nature, and availability in the plant. Changing water availability by

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reducing water content can prevent plant species from deteriorating (Joachim and Heindl, 2007).

According to the literature, no work on sorption isotherms and thermodynamics properties has been reported for *Matricaria pubescens*, for that, this study is an attempt to establish moisture adsorption and desorption isotherms of *M. pubescens* leaves, at water activities and temperatures from 0.057 to 0.898 and from 30 °C to 50 °C, respectively.

2 Material and methods.

2.1 Sorption isotherm determination

Matricaria pubescens leaves used for the sorption experiments were harvested a little before their flowering period, in the Bechar region, located in the South West of Algeria. The harvest was carried out in the period from March to May 2018.

The leaves used in the experiments were chosen randomly to complete the selected sample size. A precision analytical balance with a resolution of 0.0001 was used, to measure the mass of the samples. A forced ventilation oven was used to obtain the required temperature conditions for each test. The required humidity conditions have been obtained using saline solutions which generate different humidity conditions depending on the temperature.

The equilibrium water content of the leaves of *M. pubescens* at temperatures 30 °C, 40 °C and 50 °C, were determined by the gravimetric method, which is based on the use of saturated saline solutions to obtain a constant relative humidity of the ambient air (according to the methodology recommended by the European project Cost90) (Spiess and Wolf, 1983).

Six saturated saline solutions (KOH; MgCl₂; K₂CO₃; NaNO₃; KCl and BaCl₂) have been prepared by dissolving an appropriate amount of salt in distilled

water at a temperature above that of equilibrium to ensure that they remain saturated when cooled. The experimental device used consists of six glass jars of 1 L each, with hermetic closure. Each jar is filled to a quarter of its volume with a saturated solution of prepared salt. A solid layer of salt was maintained throughout the equilibration period to ensure that the solutions always remained saturated. A glass tripod was also placed in each jar to deposit the sample. The jars are placed beforehand in the temperature-controlled oven for 24 hours to stabilize at the temperature of the experiment. As shown in Table 1, saturated saline solutions provide water activity ranging from 0.07 to 0.89 (Greenspan, 1977). The duplicate samples, each 0.02g (± 0.0001 g) for desorption and 0.01g (± 0.0001 g) for adsorption, were weighed and placed in the jars at the top. The jars containing the saline solutions and the samples were then hermetically closed and then placed in an oven at fixed temperature (30 °C, 40 °C and 50 °C) for equilibration. The samples used in the adsorption were dried at 50 °C for 24 hours. The samples were weighed every two days until there was no change in mass. At this stage, the equilibrium between the product and its environment is reached, then the equilibrium water content of the sample was determined using the oven method, in which the temperature was maintained at 105 °C for 24 hours (AOAC, 1990).

The hygroscopic equilibrium of *Matricaria. p* leaves was reached in eight days for desorption and six days for adsorption. The difference in mass before (m_h) and after (m_s) drying in the oven gives the equilibrium moisture content in dry base, as follows:

$$X_e = \frac{m_h - m_s}{m_s} \quad (1)$$

X_e equilibrium moisture content in dry base

m_h mass before drying

m_s mass after drying

Table 1 Selected salts used for preparing saturated salt solutions and their corresponding water activities

Salt	Water activity		
	30 °C	40 °C	50 °C
KOH	0.0738	0.0626	0.0572
MgCl ₂	0.3238	0.3159	0.3054
K ₂ CO ₃	0.4317	0.423	0.4091
NaNO ₃	0.7275	0.71	0.6904
KCl	0.8362	0.8232	0.812

2.2 Modeling of sorption isotherms

Eight mathematical equations (Caurie, BET, Henderson Thomson, Smith, Halsey, Oswin, Peleg, and Adam and Shove) were used to describe the adsorption and desorption isotherms of *M. pubescens* leaves, in the range of temperature varying from 30 °C to 50 °C. The expressions and parameters of the eight models used for data fitting are shown in Table 2. Nonlinear regression analysis was used to estimate the constants of the models from the experimental results of the sorption isotherms.

The efficiency of a model is determined from statistical parameters such as the correlation coefficient (*r*), the standard error (*S*) and the mean relative deviation (*P*), defined as follows:

$$S = \sqrt{\frac{\sum_{i=1}^N (X_{e_i} - X_{e_{pre_i}})^2}{N - n_{param}}} \tag{2}$$

$$r = \sqrt{1 - \frac{\sum_{i=1}^N (X_{e_i} - X_{e_{pre_i}})^2}{\sum_{i=1}^N (\bar{X}_e - X_{e_i})^2}} \tag{3}$$

$$P(\%) = \frac{100}{N} \sum_{i=1}^N \left(\frac{X_{e_{cal_i}} - X_{e_i}}{X_{e_i}} \right) \tag{4}$$

$$\bar{X}_e = \frac{1}{N} \sum_{i=1}^N X_{e_i} \tag{5}$$

where

X_{e_i} : is the experimental value of equilibrium moisture content

X_{e_{pre_i}} : is the calculated value of equilibrium moisture content by using the tested model

N: number of experimental points.

n: is the number of parameters of the particular model

\bar{X}_e is the arithmetic average value of the experimental equilibrium moisture content.

Table 2 Mathematical models used to describe desorption and adsorption isotherms of *M. pubescens* leaves

Models names	Models equations	References
Caurie	$X_e = e^{(A+B a_w)}$	Caurie (1970)
BET	$X_e = \frac{A B a_w}{(1 - a_w)(1 + (A - 1)a_w)}$	Brunauer et al. (1938)
Henderson-Thompson	$X_e = \left(\frac{\ln(1 - a_w)}{-A(T + B)} \right)^{\frac{1}{C}}$	Henderson (1952)
Smith	$X_e = A - B \ln(1 - a_w)$	Smith (1947)
Halsey	$X_e = A \left(-\frac{B}{\ln(a_w)} \right)^{1/C}$	Halsey (1948)
Oswin	$X_e = A(a_w / (1 - a_w))^B$	Chia-Chung Chen (1990)
Peleg	$X_e = A a_w^B + C a_w^D$	Peleg (1993)
Adam and Shove	$X_e = A + B a_w + C a_w^2 + D a_w^3$	Chirife and Iglesias (1978)

Where *A*, *B*, *C* and *D* are parameters of the equations, *T* is temperature (°C), *X_e* is equilibrium moisture content (kg/kg d.b) and *a_w* is the water activity.

2.3 Determination of the net isosteric heat of sorption

The net isosteric heat of sorption can be determined from the sorption data, using Equation 6, which is derived from the Clausius-Clapeyron equation. The application of the latter to sorption isotherms at different temperatures, is a procedure widely used for the calculation of the isosteric heat of sorption or the differential enthalpy of sorption, which is valid only at constant water contents (Rockland and Beuchat, 1987).

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

Where *q_{st}* is the net isosteric heat of sorption in (KJ mol⁻¹), *a_w* is the water activity, *T* is the temperature in

(K), *R* is the universal gas constant (KJ mol⁻¹ K⁻¹), *Q_{st}* is the isosteric heat of sorption in (KJ mol⁻¹), while *L_V* is the latent heat of vaporization of pure water (43.53 KJ mol⁻¹) at 35 °C. The net isosteric heat represents the energy of fixing of the water to the substrate, ie the additional heat to the heat of vaporization of pure water, which it would be necessary to supply to the product to dehydrate it. The net isosteric heat of sorption is calculated from the slopes of the isosteres, obtained by plotting the curves ln (*a_w*) as a function of (1 / *T*), for a constant water content. We then draw the curve *q_{st}* = *f* (*X_e*). Isosteric heat is calculated by the following

relation:

$$Q_{st} = q_{st} + L_V \quad (7)$$

3 Results and discussion

3.1 Sorption isotherms

The initial water content of the leaves of *Matricaria*. *p* is about 2.33 kg of H₂O per kg dry matter. The adsorption and desorption isotherms obtained for 30 °C, 40 °C and 50 °C, are shown in Figures 1-3.

Sorption isotherms have a type II sigmoidal appearance, according to the BET classification. This type of curve is typical for plant products, as reported by Touati et al. (2008) and Ait-Mohammed et al. (2004). We note that, at constant temperature, the equilibrium water content increases with increasing water activity. Two flex zones are observed, one from 0.1 to 0.3 and the other from 0.6 to 0.7.

The isotherms are therefore divided into three distinct zones. According to Aguilera and Stanley (1999), in zone I (water activity between 0.05 and 0.2), a minimum of water is contained in the product, and the water molecules present are closely linked to the active sites of the substrate (for example: polar groups of molecules), mainly by hydrogen bonds. In zone II (water activity between 0.2 and 0.6), the water is more weakly bound, first as a multilayer above the monolayer, then when the content of water increases, this water successively fills the micro and macropores in the system. In this region, chemical and biochemical reactions requiring water are beginning to take place because of the increased mobility of solutions. In zone III, (water activity between 0.6 and 0.9), excess water is present in the macrocapillaries, presenting almost all of the properties of water. Microbial growth becomes the major reaction of deterioration in this region. The Figures also show the effect of hysteresis between adsorption and desorption on almost all of the water activity range, for the three temperatures, in which the water content in the isotherm of desorption is higher than that of adsorption, to the same water activity. One of the hypotheses used to explain the phenomenon of hysteresis is to consider a pore connected to its environment by a small capillary, during adsorption, as the relative

humidity increases, the capillary begins to fill, when the pore is empty. Only when partial vapor pressure in the air is greater than the vapor pressure in the capillaries, will be the passage of water through the pores. Once saturated, the pore is full of liquid, this liquid can only escape when the partial pressure of the ambient air becomes below the vapor pressure of the liquid in the capillary, and as the pore system generally has a wide range of capillary diameters, as a result, the difference between adsorption and desorption will be observed (Mujumdar, 2014).

Figures show also a decrease in water content at equilibrium with increase in temperature, at constant water activity, this can be explained by the variation of the enthalpy of the connection or dissociation of water, or by the increase in water solubility, as the temperature increases (Wrolstad et al., 2005). Similar results for many species of plants have been reported (Touati et al., 2007; Lamharrar et al., 2007).

3.2 Modeling of sorption isotherms

Tables 3-4 show, respectively, the results of the nonlinear regression analysis of the desorption and adsorption isotherms of the leaves of *Matricaria pubescens*, obtained at 30 °C, 40 °C and 50 °C, the values of the constants of the eight models, as well as their values of (*r*), (*S*) and (*P*) are also given. Models presenting a high value of (*r*) or which tends towards 1, and a small value of (*S*) or tends towards 0, are chosen for fitting the data.

The results indicate that all models are acceptable for predicting equilibrium water content. However, the Peleg and Halsey models gave the best fitting of the adsorption and desorption isotherms of *Matricaria. p* for the three chosen temperatures, with the lowest values of (*S*) and (*P*) and the higher values of (*r*). The equation of Peleg has been found satisfactory for many other plant species (Moreira et al., 2005; Bahloul et al., 2008; Singh et Kumari, 2014), while the Halsey's equation has been adapted for products with type I, II, and III isotherms, and it has been used to describe 220 sorption isotherms for about 69 different foods (Mujumdar, 2014; Rao et al., 2014).

Table 3 Results of fitting of the desorption isotherms of *Matricaria pubescens*

Model	T (°C)	Paramètres				r	S	P(%)
		A	B	C	D			
BET	30	0.1449	12.825	-	-	0.991	0.038	24.454
	40	0.5212	13.679	-	-	0.992	0.029	21.621
	50	19.336	-26.556	-	-	0.989	0.033	32.020
Adam and Shove	30	0.0603	-0.4078	0.5784	0.5784	0.926	0.144	35.893
	40	0.0351	-0.2395	0.4467	0.4467	0.965	0.0816	25.045
	50	0.0331	-0.4062	0.5128	0.5128	0.922	0.0115	49.055
Caurie	30	-5.207	5.426	-	-	0.959	0.084	42.594
	40	-4.174	4.0559	-	-	0.980	0.047	22.470
	50	-7.469	7.8381	-	-	0.976	0.050	51.773
Halsey	30	0.817	0.0081	1.010	-	0.991	0.0373	17.116
	40	0.901	0.0046	1.189	-	0.992	0.035	16.061
	50	0.982	0.0715	1.171	-	0.991	0.037	16.978
Handerson-thompson	30	5.1715	0.584	-38.07	-	0.968	0.083	40.945
	40	6.6403	0.757	-38.00	-	0.984	0.047	25.178
	50	7.7433	0.423	-47.12	-	0.981	0.049	48.213
Oswin	30	0.1223	0.832	-	-	0.982	0.055	28.391
	40	0.1350	0.710	-	-	0.991	0.030	20.451
	50	0.0618	1.113	-	-	0.990	0.032	34.685
Smith	30	-0.011	0.2885	-	-	0.946	0.095	30.007
	40	-0.008	0.2511	-	-	0.976	0.051	28.343
	50	-0.032	0.2382	-	-	0.933	0.083	23.102
Peleg	30	0.3749	1.0614	5.9810	23.9268	0.995	0.035	17.713
	40	20.882	39.224	0.4740	1.5905	0.996	0.027	18.704
	50	0.2356	1.1849	3.813	18.0685	0.999	0.010	16.050

Table 4 Results of fitting of the adsorption isotherms of *Matricaria pubescens*

Model	T (°C)	Paramètres				r	S	P(%)
		A	B	C	D			
BET	30	1.9981	11.7468	-	-	0.993	0.022	21.53
	40	0.2202	20.2012	-	-	0.98	0.031	25.14
	50	1.61	0.0264	-	-	0.98	0.029	42.1
Adam and Shove	30	0.0388	-0.3512	0.4913	0.4913	0.938	0.1106	37.6
	40	0.0296	-0.1126	0.285	0.285	0.97	0.052	23.32
	50	0.0963	-0.3761	-0.735	1.6476	0.921	0.1	52.32
Caurie	30	-5.702	5.7643	-	-	0.97	0.0592	41.88
	40	-4.117	3.597	-	-	0.983	0.03	20.27
	50	8.1779	-7.912	-	-	0.978	0.041	52.99
Halsey	30	2.54	0.0029	1.055	-	0.986	0.057	26.15
	40	1.916	0.0015	1.339	-	0.986	0.031	14.22
	50	0.997	0.0074	0.775	-	0.993	0.027	12.39
Handerson-thompson	30	5.4284	0.5647	-28.21	-	0.978	0.056	38.66
	40	7.645	0.8763	-38.43	-	0.983	0.033	24.92
	50	6.2534	0.4072	-47.79	-	0.983	0.04	48.54
Oswin	30	0.0934	0.8669	-	-	0.989	0.035	23.76
	40	0.1113	0.6229	-	-	0.988	0.025	22.32
	50	0.0493	1.1537	-	-	0.99	0.026	35.89
Smith	30	-0.021	0.2423	-	-	0.954	0.073	34.28
	40	0.0023	0.1762	-	-	0.981	0.032	26.34
	50	-0.03	0.2055	-	-	0.93	0.073	25.18
Peleg	30	0.356	1.4353	5.4538	25.89	0.997	0.022	19.73
	40	0.122	0.4304	0.602	5.948	0.992	0.027	16.5
	50	3.661	18.988	0.2104	1.3	0.999	0.009	17.52

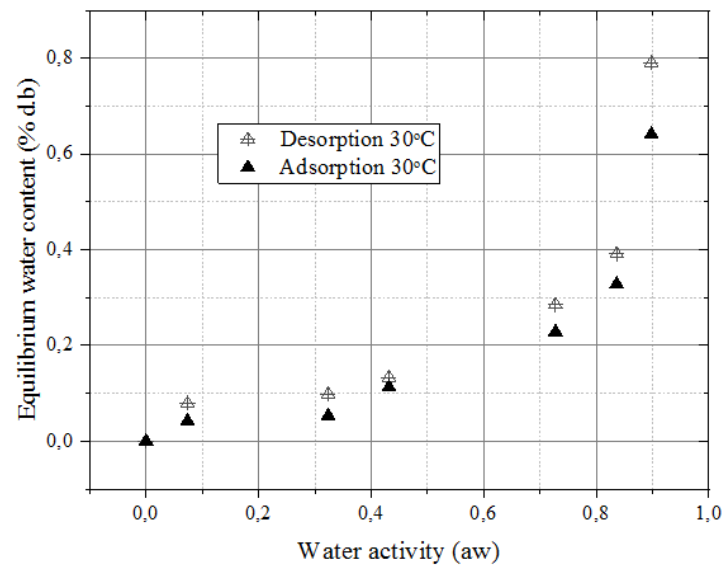


Figure 1 Isotherm of adsorption and desorption of *Matricaria.p* leaves at 30 °C

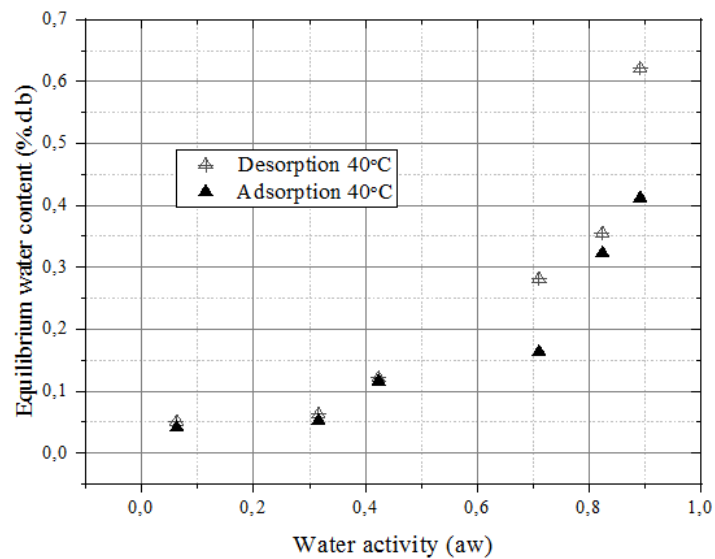


Figure 2 Isotherm of adsorption and desorption of *Matricaria.p* leaves at 40 °C

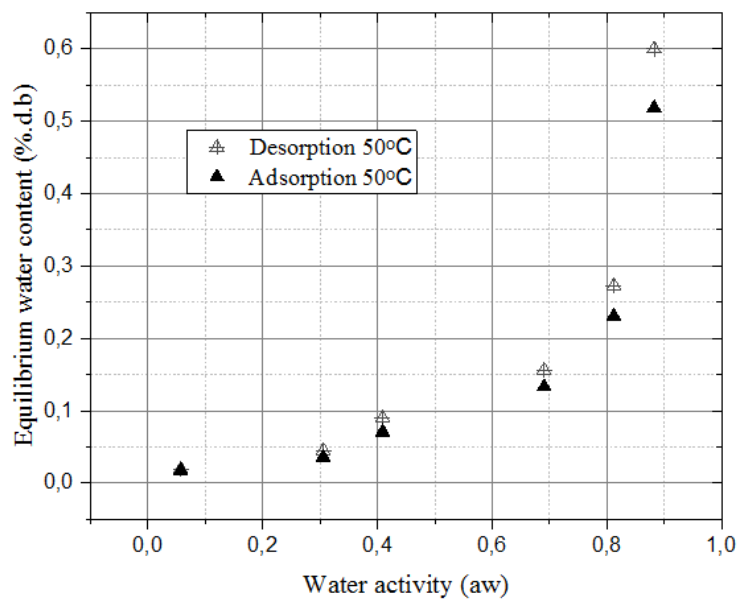


Figure 3 Isotherm of adsorption and desorption of *Matricaria.p* leaves at 50 °C

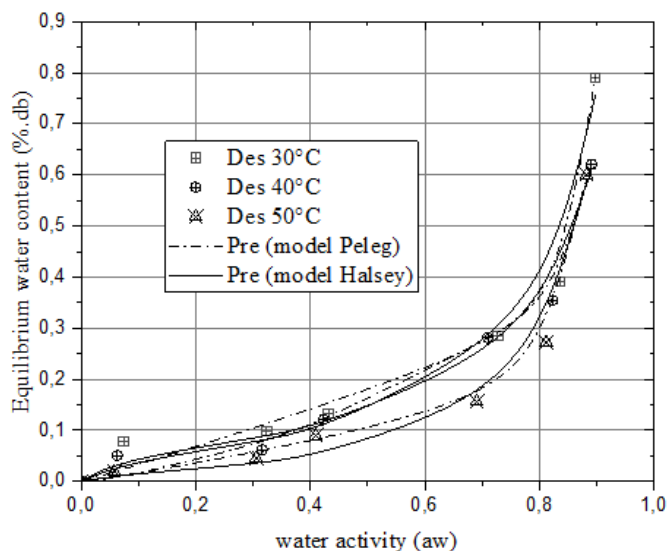


Figure 4 Comparison between experimental and predicted data (Peleg and Halsey model) of desorption of *Matricaria. p* leaves at 30 °C, 40 °C and 50 °C (pre: predicted).

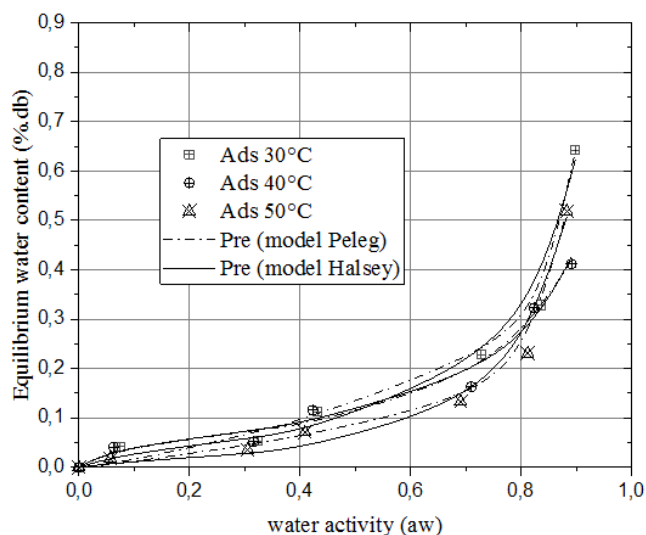


Figure 5 Comparison between experimental and predicted data (Peleg and Halsey model) of adsorption of *Matricaria. p* leaves at 30 °C, 40 °C and 50 °C (pre: predicted).

Comparisons were done between experimental and calculated (Peleg and Halsey models) data of desorption and adsorption isotherms obtained for *Matricaria pubescens* leaves at the three temperatures and are shown, respectively, in Figures 4 and 5.

3.3 Isostatic heat of sorption

The net isosteric heat of sorption of the leaves of *Matricaria. p* obtained for different water contents has been determined, using Equation 6 and the best model used for data fitting. Figure 6 shows, the variation of the isosteric heat of sorption (adsorption and desorption) with the water contents at equilibrium.

As shown in the Figure, the isosteric heat of sorption is higher than the latent heat of vaporization of pure

water. This observation indicates that the binding energy between the water molecules and the substrate is greater than the binding energy between the water molecules in the liquid state. The same conclusions have been reported by Choudhury et al. (2011).

At low water content, the isosteric heat of sorption is high, then it decreases with increasing water content. This increase in isosteric heat of desorption, at low water contents, could be due to the fact that water is tightly bound to the substrate, which corresponds to a high interaction energy. By increasing the water content, the active sites become occupied and sorption takes place on the less active sites, giving rise to a low heat of sorption (Tsami et al., 1990; Iglesias and Chirife, 1976).

We notice that the heat of desorption has a magnitude greater than that of the corresponding heat of adsorption. Iglesias and Cherifi (1976), considered that this, either because of the structural modifications which take place during desorption, which modifies the global energy of the bond to the substrate by cooperative bonds

or by the trapping effect. This phenomenon explains not only the difference between the heat of adsorption and absorption, but it can also explain the difference between the water content of the adsorption and desorption isotherm for a given water activity (Al-Muhtaseb et al., 2002).

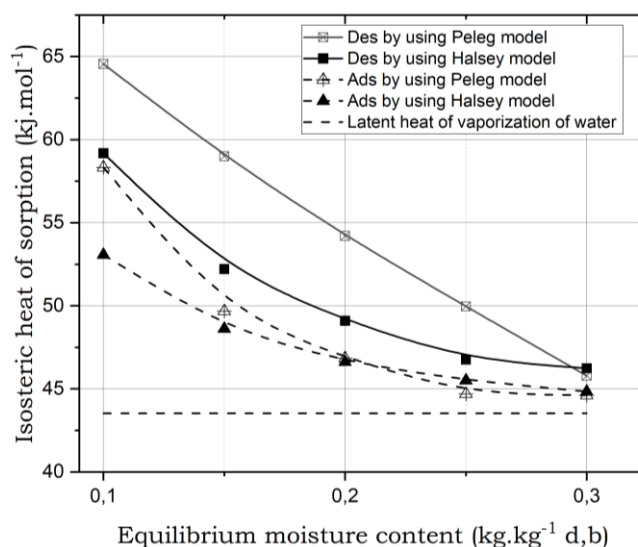


Figure 6 Isosteric heats of desorption and adsorption of *Matricaria. p* as a function of the equilibrium water content

4 Conclusion

An experimental study of the desorption and adsorption isotherms of *Matricaria. p* for three temperatures (30 °C, 40 °C and 50 °C) was carried out. Such study is very important for the design and optimization of drying equipment, and for the prediction of quality, stability, and shelf life of any species. The sorption isotherms obtained have a type II sigmoidal shape. The phenomenon of hysteresis was clearly observed. Eight mathematical equations (Halsey, BET, Henderson-Thomson, Smith, Oswin, Peleg, Caurie and Adam and Shove) were used to describe the adsorption and desorption isotherms of *Matricaria pubescens* leaves, in the temperature range from 30 °C to 50 °C. Sorption isotherm modeling results indicate that all models are acceptable for predicting equilibrium water content. However, Peleg and Halsey's model gave the best fitting of the adsorption and desorption isotherms for the three temperatures chosen, with the lowest values of (S) and (P) and the highest values of (r). The net isosteric heat of sorption of leaves of *Matricaria pubescens*, for different water contents, was also

determined, using the Clausius-Clapeyron equation and the best model used for data fitting.

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