

Moisture Sorption Isotherms And Thermodynamic Properties Of *Urtica Dioica* Leaves

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Abstract

Urtica dioica is a Moroccan endemic plant of used for its virtues in traditional medicine. Thus, it is necessary to study the effect of preservation processes on the storage conditions of the plant. The static gravimetric method was used to determine sorption isotherms of *Urtica dioica* leaves at three temperatures (40, 50 and 60 °C) and in the range of water activity (a_w) ranging from 0.0572 to 0.898. Six mathematical models were used to fit the experimental data. The Enderby and Peleg models were found to be the most suitable for describing the sorption curves. The optimal water activity for conservation of *Urtica dioica* leaves was determined. Isothermic heats of desorption and adsorption were calculated by applying the Clausius-Clapeyron equation to the sorption isotherms at different temperatures; it decreased with increasing moisture content. A linear relation exists between the enthalpy and entropy of the sorption reaction.

Keywords: Enthalpy, Entropy, Hysteresis, Mathematical models, Sorption isotherms

Introduction

The medicinal and aromatic plants have a great importance for both the pharmaceutical industry and the traditional medicine. The stability of a

dehydrated medicinal plant is influenced by its water activity. *Urtica dioica* is known as nettle in Morocco Its root and leaf, which contains active pharmacological compounds influencing cytological and physiological processes in the body, are used for medical purposes (Krystofova et al., 2010). This plant had been used as a diuretic and as a means of treating Arthritis and rheumatic diseases in the past (Bellakhdar, 1997).

The determination of sorption isotherms is a necessary step in the study of the drying and the preservation action of PAM, because it determining the equilibrium moisture content and offers valuable information on the balance hygroscopic product (Lamharrar et al ., 2007). In this study, temperatures of 30°C, 40°C, and 50°C were chosen, to typify tropical storage conditions. Using an experimental approach, the equilibrium curves are determined by the saturated salt solution method. The experimental sorption curves are described by six different models to identify the most appropriate mathematical model for a better description of the product equilibrium state. Then, the experimental data the water activity optimal for the storage of *Urtica dioica* leaves were investigated.

Further analysis of sorption isotherm data by application of thermodynamic principals can provide information regarding differential enthalpy or the isosteric heat of sorption and differential entropy. Differential enthalpy of sorption gives an indication of the amount of bound water existing in the product. The differential entropy of material is proportional to the number of its available sorption sites at a specific energy level (Arsalan and Togrul, 2006). Subsequently we determine the thermodynamic functions (differential enthalpy, differential entropy). Moreover, we recognized the linear relation exists between the enthalpy and entropy of the sorption reaction.

Materials and methods

Experimental procedure

The plant *Urtica dioica* used in our study was collected at Tahanouat, Marrakesh Morocco. The hygroscopic equilibrium could be achieved by a dynamic or static method. In the present work, a static method is used. The method is based on the use of saturated salt solutions to maintain a fixed relative humidity Rh. The mass transfers between the product and the ambient air are assured by natural diffusion of the water vapor. The atmosphere surrounding the product has a fixed relative humidity for every working temperature imposed on the system.

Six salts were chosen { KOH, (MgCl₂, 6H₂O), K₂CO₃, NaNO₃, KCl and (BaCl₂, 2H₂O)} so as to have a range of water activity (a_w) of 0.0572-0.0898 (Greenspan, 1977). The experimental apparatus consisted of six glass jars of 1 liter each with an insulated lid. Every glass jar was filled to quarter

depth with a saturated salt solution. Duplicated samples each of 0.2 g ($\pm 0.0001\text{g}$) for desorption and 0.1 g ($\pm 0.0001\text{g}$) for adsorption were weighed and placed into the glass jars. The weight recording period was about 3 days. This procedure continued until the weight was constant. The equilibrium moisture content of each sample was determined in a drying oven at $105\text{ }^\circ\text{C}$ for 24 h. The hygroscopic equilibrium *Utica dioica* leaves was reached in then days for desorption and eighth days for adsorption.

Thus, the difference of mass before (M_w) and after (M_d) drying determines their equilibrium moisture content ($\text{EMC} = X_{eq}$) of the product at hygroscopic equilibrium:

$$X_{eq} = \frac{M_w - M_d}{M_d} \tag{1}$$

Modelling equations

The relationship between EMC, water activity, and temperature for *Urtica dioica* was predicted in literature by several mathematical models (Al-Muhtaseb et al., 2004). In our study six models have been applied: modified Henderson's model, modified Oswin's model, modified Halsey, model Enderby's model, GAB's model and Peleg's model. The equations expressing those models are given below in Table 1.

X_{eq} represents equilibrium moisture content (%MS); a_w water activity; A, B, C and D coefficients, dependant to the temperature or not, to determine by smoothing the experimental curves of sorption and θ the temperature in $^\circ\text{C}$.

Table 1: Mathematical models used in our study

Model's name	Expression of the model	References
GAB	$X_{eq} = \frac{A B C a_w}{[1 - B a_w][1 - B a_w + B C a_w]}$	(Vanden Berg and Bruin, 1981)
Modified Henderson	$X_{eq} = \left[\frac{-\ln(1 - a_w)}{A(\theta + B)} \right]^{1/C}$	(Thompson et al., 1986)
Peleg	$X_{eq} = A(a_w)^C + B(a_w)^D$	(Peleg, 1993)
Modified Halsey	$X_{eq} = \left[\frac{(-\exp(A + B.\theta))}{\ln(a_w)} \right]^{-1/C}$	(Iglesias and Chirife, 1976)
Modified Oswin	$X_{eq} = (A + B.\theta) \left[\frac{a_w}{1 - a_w} \right]^C$	(Oswin, 1946)
Enderby	$X_{eq} = \left[\frac{A}{1 - B a_w} + \frac{C}{1 - D a_w} \right] a_w$	(Popovski and Mitrevski, 2004)

The correlation coefficient (r) was one of the primary criteria for selecting the best equation to fit the four models to the experimental data. In addition to r , the statistical parameters mean relative error (MRE) as a percentage was used to determine the quality of the fit (Foo and Hameed, 2010).

Levenberg-Marquardt nonlinear optimization method using appropriate software is used for the calculation of model coefficients that describe the equilibrium curves and their statistical parameters: mean relative error (MRE) and the correlation coefficient (r).

Mean Relative Error:

$$\text{MRE} = \frac{100}{N} \sum_{i=1}^N \left| \frac{X_{eq,i,\text{exp}} - X_{eq,i,\text{pred}}}{X_{eq,i,\text{exp}}} \right| \quad (2)$$

Correlation coefficient:

$$r = \frac{\sqrt{\sum_{i=1}^N (X_{eq,i,\text{pred}} - \overline{X_{eq,i,\text{exp}}})^2}}{\sqrt{\sum_{i=1}^N (X_{eq,i,\text{exp}} - \overline{X_{eq,i,\text{exp}}})^2}} \quad (3)$$

Where:

$X_{eq,i,\text{exp}}$ ith Experimental moisture content (%d.b),

$X_{eq,i,\text{pred}}$ ith Predicted moisture content (%d.b).

Determination of the optimal conditions of storage

The study of the sorption isotherms enables us to know the optimal relative equilibrium moisture for the conservation of a product as well as the water content of equilibrium to reach at the end of the drying. Also, it gives users with accurate information to handle a product during storage and conservation (Bolin, 1980).

For this purpose, the optimal water activity of conservation (a_{wop}) was dogged. The whole of the experimental points is gathered on the same graph. Then, we model the isotherm of sorption by a polynomial equation of the third degree. The part closes to horizontality corresponds to the zone of better stability of the product. This process of calculation consists in making a polynomial decomposition of the equilibrium moisture content X_{eq} , for the whole of the experimental results, according to the water activity. This makes it possible to calculate the value for which the derivative second of

X_{eq} is cancelled “inflection point” and consequently optimal relative humidity for conservation.

Differential enthalpy and differential entropy of sorption

The differential enthalpy or isosteric heat of sorption (ΔH_d) indicates the state of absorbed water by the solid material. The net isosteric heat of sorption (Δh_d) represents the quantity of energy exceeding the heat of vaporization of water (ΔH_{vap}) associated with the sorption process.

$$\Delta h_d = \Delta H_d - \Delta H_{vap} \tag{4}$$

The net isosteric heat of sorption can be calculated from the experimental data using the Clausius-Clapeyron equation (Lamharrar et al., 2007).

$$\left[\frac{d(\ln a_w)}{d(1/T)} \right]_{X_{eq}} = \frac{-\Delta h_d}{R} \tag{5}$$

Where:

T Absolute temperature (K)

R Universal gas constant (8.3145 J/mol/K)

This relation allows determining the isotherms at various temperatures in order to calculate the variation logarithmic curve of the water activity according to the reverse of the temperature, for a water content fixed. However, starting from the modified equations, it is possible to obtain an analytical expression of the heat of sorption Δh_d . This approach assumes that isosteric heat ΔH_d does not vary with the temperature (Tsami, 1991). The relationship between the net isosteric heat Δh_d and the differential entropy ΔS_d of sorption is given by:

$$(-\ln a_w)_{X_{eq}} = \frac{\Delta h_d}{R.T} - \frac{\Delta S_d}{R} \tag{6}$$

The experimental sorption isotherm data was plotting in the form of $\ln(a_w)$ versus $\frac{1}{T}$ for specific moisture content (X_{eq}). We determined Δh_d

from the slope $\left(\frac{\Delta h_d}{R} \right)$ and ΔS_d from the intercept $\left(\frac{\Delta S_d}{R} \right)$. Applying this at different moisture contents allowed determining the dependence on Δh_d and ΔS_d .

Enthalpy-Entropy Compensation Theory

According to the theory of compensation, the linear relationship between the enthalpy Δh_d and the entropy ΔS_d for a specific reaction are given by:

$$\Delta h_d = T_\beta \cdot \Delta S_d + \Delta G_\beta \quad (7)$$

The isokinetic temperature (T_β) is a characteristic property of the food surface whose dimension is absolute temperature. It represents the temperature to which all the reactions of the series proceed to the same rate.

The free energy (ΔG_β) provides a criterion to evaluate if the process of the water sorption is spontaneous ($-\Delta G_\beta$) or not ($+\Delta G_\beta$). This process is repeated for several values of equilibrium relative humidity with an aim of underlining the relation between the differential entropy of sorption and the equilibrium moisture content of the studied product. The isokinetic temperature (T_β) and constant (ΔG_β) were calculated using linear regression (Naji et al., 2010).

Results and discussion

Sorption and desorption isotherms

The equilibrium moisture content increases with decreasing temperature at constant water activity (Figure 1). The sorption isotherms present the characteristic S-shaped curve (Type II), typical of sorption isotherms of many plants and food materials (Park et al., 2002; Kouhila et al., 2002; Lahsasni et al., 2004; Ait Mohamed et al., 2005; Jamali et al., 2006; Kouassi Ernest et al 2015).

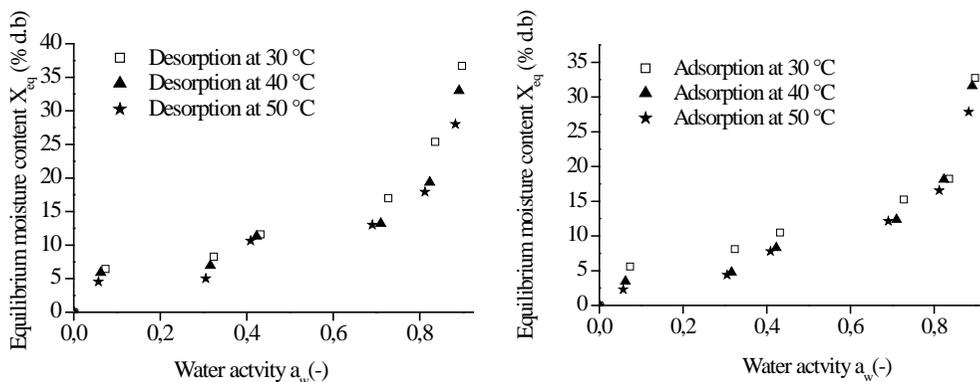


Figure 1: Sorption isotherms of *Urtica dioica* leaves at 30, 40 and 50 °C

The presentation of sorption and desorption experimental data in the same plot show that the plant does not have the same hygroscopic equilibrium behavior (Figure 2). The phenomenon of desorption-sorption is irreversible. For the same constant relative moisture, the water content of desorption is higher than that of adsorption. Indeed, there exists a phenomenon of hysteresis. This characteristic is visible for *Urtica dioica* leaves.

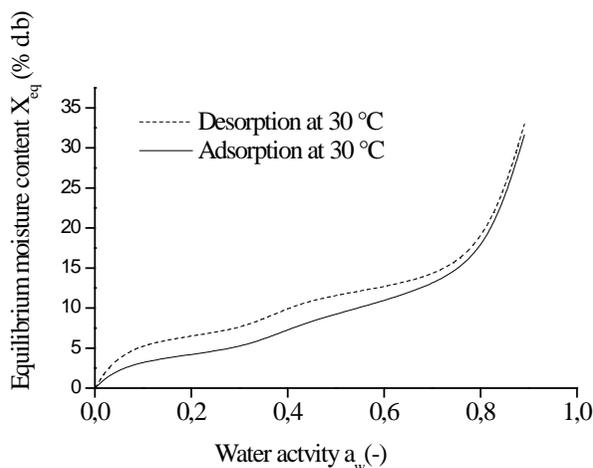


Figure 2: Sorption hysteresis phenomenon of *Urtica dioica* leaves

Several hypotheses have been put to explain hysteresis. One of it is an analogy with a sponge. When the pores do not contain any more water, adsorption does not make it possible to recover the totality of initial water since this one imprisons air in the pores. Another hypothesis states that hysteresis in the sorption isotherm is a consequence of variation in the fraction of bound water present in the adsorption and desorption processes. The bound fraction being always larger on desorption than on adsorption (Sherman, 1947).

Modeling of sorption experimental data

The results of non linear regression analysis of fitting the sorption equations to experimental data of *Urtica dioica* leaves was at three temperatures are presented in Table 2. For all the models tested, parameters A, B and C are found to be temperature dependent. For desorption isotherms, Enderby model is found to be the best estimator for predicting the equilibrium moisture of the *Urtica dioica* leaves. This model gives the highest correlation coefficient (r) of 0.9981 and mean relative error MRE (4.3075 %) at $\theta = 30$ °C. For adsorption isotherms, Peleg model is found to be the best estimator for predicting the equilibrium moisture of *Urtica dioica*

leaves. This models gives the highest (r) of 0.9967 and the MRE (6.6280%) at $\theta = 50\text{ }^{\circ}\text{C}$.

Table 2: Estimated model coefficients, r , MRE of six equation model fitted to sorption isotherm of *Urtica dioica* leaves

Model	Parameters	Desorption			Adsorption		
		θ ($^{\circ}\text{C}$)	30	40	50	30	40
GAB	A	5.5252	4.2602	4.5941	4.4702	3.5516	3.7865
	B	0.9432	0.9706	0.9426	0.9487	0.9944	0.9771
	C	390.1938	110.2862	380.6512	68.6528	412.2234	29.0048
	r	0.9947	0.9721	0.9799	0.9643	0.9934	0.9920
	RME	7.1330	16.7363	12.1912	18.6960	8.6848	11.0096
Henderson modifié	A	0.0028	0.0019	0.0014	0.0023	0.0032	0.0025
	B	-17.4960	-15.8693	-20.5234	-15.5315	1.2706	-7.0323
	C	1.1818	1.1529	1.2235	1.2726	0.8307	0.9293
	r	0.9605	0.9186	0.9489	0.9297	0.9616	0.9725
	RME	21.7143	21.5854	33.4118	25.0296	32.2487	25.6404
Peleg	A	57.0714	95.0904	68.5839	16.7738	94.1110	133.8690
	B	14.0564	13.4275	14.3752	224.0437	13.7622	16.8128
	C	8.4645	13.4848	12.4200	0.5139	13.9519	18.6910
	D	0.3288	0.3403	0.5144	24.0535	0.6584	0.9377
	r	0.9974	0.9933	0.9845	0.9957	0.9955	0.9967
RME	6.1795	9.4255	16.9626	7.9714	13.7854	6.6280	
Halsey Modifié	A	2.7540	2.3549	1.6014	2.4438	1.6943	1.1292
	B	0.0301	0.0214	0.0320	0.3480	0.0123	0.0225
	C	1.6460	1.5653	1.6035	1.6773	1.2729	1.3154
	r	0.9944	0.9718	0.9804	0.9711	0.9900	0.9917
	RME	8.3696	15.8939	15.2886	12.7527	12.7076	10.0679
Oswin Modifié	A	4.6327	0.7189	2.3978	3.1596	0.1649	1.6449
	B	0.2442	0.2371	0.1459	0.2421	0.1835	0.1189
	C	0.4928	0.5147	0.4948	0.4766	0.6580	0.6202
	r	0.9843	0.9539	0.9708	0.9563	0.9817	0.9874
	RME	13.5973	21.5854	21.3011	18.3354	20.5685	14.8797
Enderby	A	316.3100	1.8301	152.5967	121.3757	92.8471	0.7277
	B	-38.7367	1.0478	-20.3168	-10.6550	-14.8069	1.0867
	C	3.6179	250.0000	2.4319	1.1768	2.3258	18.6217
	D	0.9878	-2.8564	1.0164	1.0609	1.0319	-0.3849
	r	0.9981	0.9934	0.9847	0.9954	0.9964	0.9967
RME	4.3075	7.7728	14.1093	6.6280	9.8444	14.1519	

Measurement of optimal water activity of *Urtica dioica* leaves

A good product deserves quality treatment. The heat and process engineering specialists must provide users with accurate information on how

to handle a product during storage and conservation. For this purpose, the optimal water activities of conservation (a_{wop}) were determined. The sorption isotherm curve can be described as a polynomial equation of the third degree, the central part or "stage" is the best area of product stability (Figure 3).

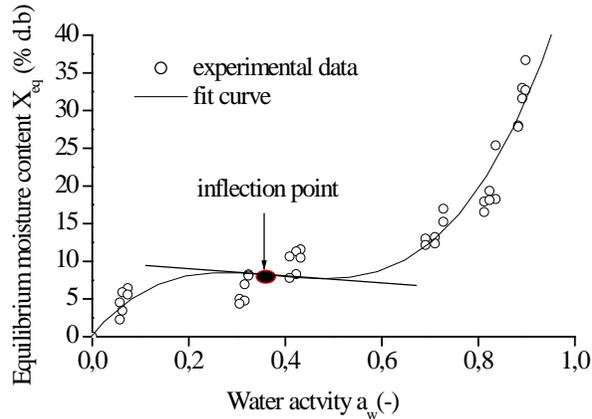


Figure 3: Determination of optimal water activity for conservation of *Urtica dioica* leaves

This calculation method consists of decomposing polynomial of equilibrium moisture content X_{eq} , for all the experimental results for each product based on water activity (equation 7). The value of the optimal water activity for conservation of *Urtica dioica* leaves is $a_{wop} = 0.36$; the optimal water activities are ranged in (0.3-0.4) (Le Meste et al., 2001).

$$X_{eq} = 0.321 + 73.788 a_w + -211.159 a_w^2 + 186.305 a_w^3 \quad r=0.93 \quad (8)$$

Enthalpy and entropy of sorption

The net isosteric heat of sorption (Δh_d) values are calculated from the equilibrium moisture data at different temperatures using Clausius-clapeyron equation (4). The variation of the heat sorption of *Urtica dioica* leaves with equilibrium moisture content is shown in Fig. 4.

The net isosteric heat of desorption and adsorption of *Urtica dioica* leaves can be expressed mathematically as a polynomial function of equilibrium moisture content:

$$\text{Desorption : } \Delta h_d = 58.5349 - 5.0950X_{eq} + 0.1149X_{eq}^2 \quad r=0.9979 \quad (9)$$

$$\text{Adsorption : } \Delta h_d = 55.3856 - 5.9170X_{eq} + 0.1598X_{eq}^2 \quad r=0.9984 \quad (10)$$

The differential entropy (ΔS_d) is plotted as a function of moisture content in Figure 4. Once again, the entropy data display a strong dependence on moisture content. The experimental desorption and adsorption data, respectively, conform to polynomial relation as represented by the equations:

$$\text{Desorption : } \Delta S_d = 390.6015 - 65.5339X_{eq} + 3.8877X_{eq}^2 - 0.0784 X_{eq}^3$$

$r=1$ (11)

$$\text{Adsorption : } \Delta S_d = 371.2822 - 65.2823X_{eq} + 3.9958X_{eq}^2 - 0.0817X_{eq}^3$$

$r=1$ (12)

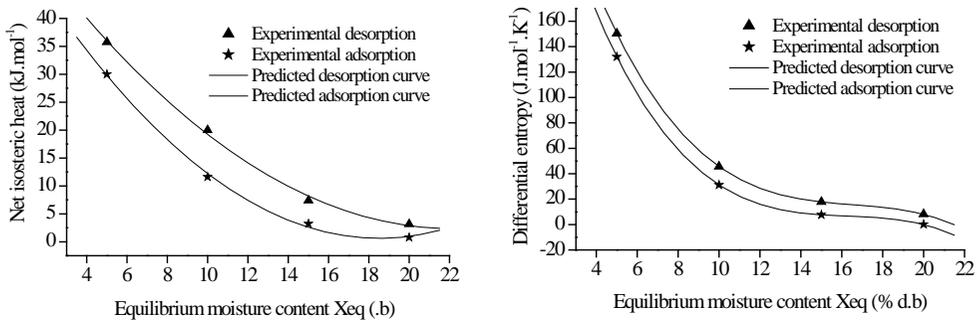


Figure 4: Net isosteric heat and differential entropy of sorption of *Urtica dioica* leaves as a function of equilibrium moisture content

Enthalpy-entropy compensation theory

The Δh_d and ΔS_d values for adsorption and desorption, at given moisture contents, were calculated by linear regression equation using equation 6. It was assumed that, at specific moisture content, Δh_d and ΔS_d did not vary with temperature. Figure 5 shows a linear relation, with a correlation of determination ($r = 1$). This indicates that the compensation exists. The parameters T_β and ΔG_β (Equation 7) were calculated from the data by linear regression, and the values are detailed in Table 3.

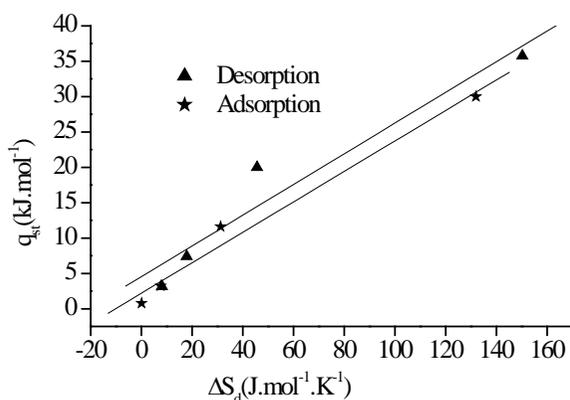


Figure 5: Compensation theory of enthalpy/entropy

Table 3: Characteristic parameters for $\Delta h_d / \Delta S_d$ Relationship

	T_β (K)	ΔG_β (J.mol ⁻¹)
Desorption	216.90	4537.90
Adsorption	215.12	2197.69

Conclusion

It's now widely accepted that sorption data provide valuable information for industrial drying in order to preserve and store the seasonal plants and make them available to consumers all year round. The sorption data may be used not only for the identification of the optimum residual moisture content of the final product, but also for the calculation of the drying time of hygroscopic substances.

The moisture sorption curves of *Urtica dioica* leaves were experimentally investigated at three temperatures 30, 40 and 50 °C. The isotherms have a sigmoid shape (Type II) which is common for many hygroscopic products. The hysteresis phenomenon was distinctly observed. Among the sorption models chosen, the Enderby model was the best model describing the equilibrium moisture data for desorption, and the Peleg model was the most suitable to estimate adsorption isotherms. The optimal water activities of conservation were determined. Net isosteric heat of sorption and differential entropy values of *Urtica dioica* leaves were found to be a polynomial function of moisture content for desorption and adsorption. Enthalpy-entropy compensation theory could be successfully applied to the moisture sorption behaviour of *dioica* leaves.

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