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Thermodynamic properties and moisture sorption isotherms of *Artemisia herba-alba*

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Abstract - *Sorption isotherms of Artemisia herba-alba were determined at three temperatures (30, 40 and 50 °C) and in the range of water activity varying from 0.0572 to 0.898. The hysteresis effect was distinctly observed in the range of temperature tested. Five sorption models were used to fit the experimental data. The GAB equation was the best model describing the equilibrium moisture data for desorption. The modified Halsey equation was the most suitable model for describing adsorption isotherms. Thermodynamic properties such as differential enthalpy and entropy were determined from moisture sorption data, using the Clausius-Clapeyron equation. The experimental data showed that enthalpy-entropy compensation theory was applicable for the moisture sorption behaviour of Artemisia herba-alba.*

Résumé - *Les isothermes de sorption des feuilles d'absinthe sont déterminées pour 30, 40 et 50 °C pour des activités de l'eau allant de 0.0572 à 0.898. Le phénomène d'hystérésis a été observé. Le modèle de GAB est le meilleur pour la description des courbes de sorption. Les résultats obtenus affichent une bonne concordance entre les données expérimentales et les prédictions théoriques. Le calcul des propriétés thermodynamiques fournit une bonne compréhension des propriétés de l'eau et de l'énergie requises dans les phénomènes de sorption. Les chaleurs isostériques de sorption des deux menthes sont calculées à l'aide de l'équation de Clausius-Clapeyron. Les résultats expérimentaux montrent que la théorie de compensation de l'enthalpie et de l'entropie a été bien vérifiée.*

Keywords: *Artemisia herba-alba* - Sorption isotherms - Thermodynamic properties.

1. INTRODUCTION

Artemisia herba-alba was known for its therapeutic and medicinal properties. It was used in both traditional and modern medicine. *Artemisia herba-alba* is a characteristic plant of the steppes of the Middle East, North Africa and Spain. In Morocco it is widespread in the South Desert and the Atlas Mountains. *Artemisia herba-alba* has a wide use in traditional medicine, for treatment of gastric disturbances, such as diarrhoea, abdominal cramps and for healing external wounds [1]. In Morocco, this plant is used, in diabetes treatment although the ant hyperglycaemic effect of this plant has been reported before [2]. From the analysis of the oil samples, it could be deduced that a noticeable chemical polymorphism typified this taxon. Four groups of essential oils exhibited a single compound with percentages near 30 % or higher: davanone, 1,8-cineole, chrysanthenone and cis-chrysanthenol. Two further oil types showed p-cymene and cis-chrysanthenyl acetate as major components in moderate amounts (ca. 20 %) [3].

An important factor in the quality loss of dried medicinal and aromatic plants during storage is the water activity (a_w) witch influences the biochemical reactions and stability of dried products. Some of these reactions are lipid oxidation, caking, agglomeration and degradation of vitamins and lycopene [3-5]. Knowledge of sorption isotherms of agricultural products is necessary to control dehydration and storage processes. A large number of models have been proposed in the literature for the sorption isotherms [6]. It is usual for each investigator to report

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which model best fits the experimental data. It is therefore necessary to understand the moisture sorption characteristics of dried *Artemisia herba-alba*.

Thermodynamic functions calculated from sorption isotherms allow the interpretation of experimental results in accordance with the statement of the theory. The functions include heat of sorption, differential enthalpy and differential entropy. Enthalpy-entropy compensation theory is used to evaluate physical and chemical phenomena such as sorption reactions. This theory states that in order to minimize free energy changes due to these phenomena, compensation arises from the nature of the interaction between the solute and solvent causing the reaction and that the relationship between the enthalpy and entropy for a specific reaction is linear [8, 9]. Enthalpy change provides a measure of the energy variations occurring on mixing water molecules with sorbent during sorption process. Entropy defines the degree of order or randomness existing in the water-sorbent system and helps interpretation of process such as dissolution, crystallization and swelling [7]. The enthalpy-entropy compensation theory has been widely investigated for many different physical and chemical processes [8, 9].

The aims of this work are to determine the sorption isotherms for *Artemisia herba-alba* at 30, 40 and 50 °C, to fit a suitable model for describing the sorption characteristics, to calculate the net isosteric heat of water sorption from experimental data, and to determine the enthalpy-entropy compensation for water adsorption and desorption characteristics of *Artemisia herba-alba*.

2. MATERIALS AND METHOD

2.1 Experimental procedure

The sorption method used was the static gravimetric technique, which is based on the use of saturated salt solutions to maintain a fixed relative humidity when the equilibrium is reached. The water activity of the product is identical to the relative humidity of the atmosphere at equilibrium conditions and the mass transfer between the product and the ambient atmosphere is assured by natural diffusion of the water vapour. Six salts were chosen { KOH, ($MgCl_2$, $6H_2O$), K_2CO_3, $NaNO_3$, KCl and ($BaCl_2$, $2H_2O$)} so as to have a range of water activity of 0.0572-0.0898 [5, 6]. The experimental apparatus consisted of six glass jars of 1 litre each with an insulted lid. Every glass jar was filled to quarter depth with a saturated salt solution.

Duplicated samples each of 0.4 g (±0.0001g) for desorption and 0.1 g (±0.0001g) 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 °C for 24 h. The hygroscopic equilibrium *Artemisia herba-alba* was reached in 12 days for desorption and10 days for adsorption.

2.2 Analysis of data

A large number of models have been proposed in the literature for the sorption isotherms [10]. In the present study, the description of the relationship between equilibrium moisture, water activity, and temperature for *Artemisia herba-alba* was verified according to five sorption models (**Table 1**). Nonlinear regression analysis, using appropriate computer programs, was used to estimate the constants of the models from the experimental sorption data.

The correlation coefficient (r) was one of the primary criteria for selecting the best equation to describe the sorption isotherms of *Artemisia herba-alba*. In addition to r, the statistical parameters such as; reduced mean relative error MRE as a %, standard error of estimate of moisture SEE are calculated:

$$\mathrm{MRE} = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{M_{i,\exp} - M_{i,\mathrm{pre}}}{M_{i,\exp}}\right| \tag{1}$$

$$\mathrm{SEE} = \sqrt{\frac{\sum_{i=1}^{N}\left(M_{i,\exp} - M_{i,\mathrm{pre}}\right)^2}{d_f}} \tag{2}$$

where $M_{i,\exp}$ is the i^{th} experimental equilibrium moisture content, $M_{i,\mathrm{pre}}$ is the i^{th} predicted equilibrium moisture content, N is the number of observations, and d_f is the number of degrees of freedom of regression model.

Table 1: Mathematical models applied to the sorption isotherms of *Artemisia herba-alba*

Model name	Model equation	Reference
GAB	$M = \frac{ABCa_w}{\left[1 - Ba_w\right]\left[1 - Ba_w + BCa_w\right]}$	[10]
Modified Henderson	$1 - a_w = \exp\left[-A\left(t + B\right)M^C\right]$	[20]
Modified Chung-Pfost	$a_w = \exp\left[-\frac{A}{t + B}\exp\left(-CM\right)\right]$	[21]
Modified Oswin	$M = \left(A + Bt\right)\left[\frac{a_w}{1 - a_w}\right]^C$	[22]
Modified Halsey	$a_w = \exp\left[\frac{-\exp\left(A + Bt\right)}{M^C}\right]$	[23]

2.3 Isosteric heat of sorption

The isosteric heat of sorption, or differential enthalpy, is an indicator of the state of water absorbed by the solid material. The net isosteric heat of sorption $\left(\Delta h_d\right)$ is the amount of energy above the heat of vaporization of water $\left(\Delta H_{vap}\right)$associated with the sorption process, and was calculated from the experimental data using the Clausius-Clapeyron equation [11, 12].

$$\left[\frac{d\left(\ln a_w\right)}{d\left(1/T\right)}\right] = -\frac{\Delta h_d}{R} \tag{3}$$

and

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

It is a differential quantity, the value of which corresponds to sorbet molecules at a particular equilibrium moisture content (M).

Re-plotting the experimental sorption isotherm in the form $\ln\left(a_w\right)$ versus $\frac{1}{T}$, for specific moisture content, Δh_d was determined from the slop $\left(-\frac{\Delta h_d}{R}\right)$. This procedure is based on the assumption that ΔH_d is invariant with temperature and requires measurement of the sorption isotherms at more than two temperatures [12, 13].

2.4 Enthalpy-entropy compensation theory

The relationship between the net isosteric heat of sorption (Δh_d) and the differential entropy of sorption (ΔS_d) is given by:

$$(-\ln a_w)_M = \frac{\Delta h_d}{RT} - \frac{\Delta S_d}{R} \quad (5)$$

By plotting $\ln(a_w)$ versus $\frac{1}{T}$, for a given moisture content (M), (Δh_d) was determined 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 the dependence of Δh_d and ΔS_d with moisture content to be determined. The compensation theory proposes a linear relationship between Δh_d and ΔS_d [14]:

$$\Delta h_d = T_\beta . \Delta S_d + \alpha \quad (6)$$

The isokinetic temperature (T_β) and constant (α) were calculated using linear regression.

3. RESULTS AND DISCUSSION

3.1 Experimental results

The experimental results of equilibrium moisture contents at 30, 40 and 50 °C at six water activities are given in **Table 2**. As shown in this table, a significant temperature effect on the adsorption and desorption for the full range of water activities was observed for this product. The equilibrium moisture content increases with decreasing temperature at constant water activity (Fig. 1). The sorption isotherms present the characteristic S-shaped curve (Type II), typical of sorption isotherms of many plants and food materials [6, 15, 16].

Table 2: Equilibrium moisture contents of *Artemisia herba-alba* obtained by adsorption and desorption at different water activities and temperatures

30 °C			40°C			50 °C		
a_w	des	ads	a_w	des	ads	a_w	des	ads
0.0738	10.2435	8.9056	0.0626	8.7322	7.9399	0.0572	7.8086	7.0815
0.3238	19.2966	16.8934	0.3159	18.4411	16.2132	0.3054	16.3498	15.6463
0.4317	22.9924	19.8482	0.4230	19.9493	18.4382	0.4091	18.3432	16.3774
0.7275	36.3828	25.7813	0.7100	34.1746	23.1027	0.6904	31.1251	22.2098
0.8362	51.6667	37.9587	0.8232	49.0196	36.3532	0.8120	47.5490	34.5183
0.8980	78.9572	48.4954	0.8910	69.3669	46.4120	0.8823	65.5493	44.0972

The hysteresis effect was observed (Fig. 2). At constant water activity, the equilibrium moisture contents of desorption is higher than the adsorption one. Several hypotheses have been put forward to explain this phenomenon. Al-Hodali [17] explains this hysteresis by considering a rigid structure pore connected to its surrounding by a small capillary. During adsorption, the capillary begins to fill as a result of the rising in water activity, while the pore is still empty. When the partial pressure of the vapour in air becomes greater than the vapour pressure of the liquid in the capillary, the moisture will move into the pore. For desorption, the pore is initially full of liquid at saturation. This liquid can escape only when the pressure of the surrounding air becomes lower than the vapour pressure of liquid inside the capillary. As the system of pores has generally a large range of capillary diameters, it results that differences between adsorption and desorption are observed.

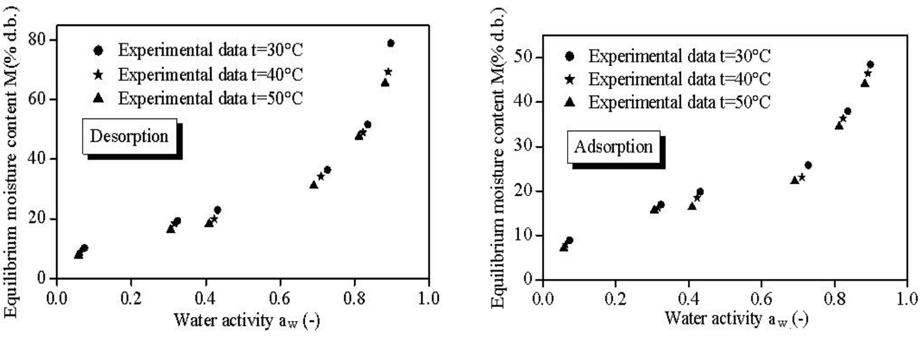

Fig. 1: Influence of temperature on the sorption isotherms of *Artemisia herba-alba*.

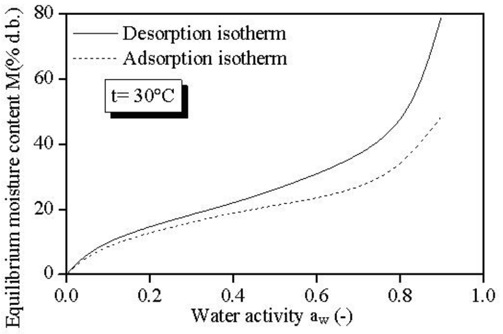

Fig. 2: Sorption hysteresis phenomenon of *Artemisia herba-alba*

3.2 Fitting sorption models to equilibrium moisture data

The results of non linear regression analysis of fitting the sorption equations to experimental data of *Artemisia herba-alba* at three temperatures are presented in **Tables 3** and **4**. For all the models tested, parameters A, B and C are found to be temperature dependent. The observed and predicted sorption isotherms using the different models are shown in Fig. 3.

For desorption isotherms, GAB model is found to be the best estimator for predicting the equilibrium moisture of the *Artemisia herba-alba*. This model gives the highest (r) of 0.9994 and the lowest SEE of 0.9582 and the lowest MRE (2.9868 %) at t = 50 °C.

For adsorption isotherms, modified Halsey model is found to be the best estimator for predicting the equilibrium moisture of *Artemisia herba-alba*. This models gives the highest (r) of 0.9926 and the lowest SEE of 2.2902 and the lowest MRE (7.82 %) at t = 30 °C.

3.3 Heat of sorption

The net isosteric heat of sorption (Δh_d) values are calculated from the equilibrium moisture data at different temperatures using Eq. (4) and obtained at different moisture contents [18]. The variation of the heats of sorption of *Artemisia herba-alba* with equilibrium moisture content is shown in Fig. 4.

The net isosteric heat of desorption an adsorption of *Artemisia herba-alba* can be expressed mathematically as a polynomial function of equilibrium moisture content:

Desorption: $$\Delta h_d = -174.6224 + 25.6903\,M - 1.1441\,M^2 + 0.0162\,M^3 \tag{7}$$

Adsorption: $$\Delta h_d = -398.0214 + 56.0717\,M - 2.487\,M^2 + 0.0354\,M^3 \tag{8}$$

The differential entropy is plotted as a function of moisture content in Fig. 5. 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:

Desorption: $$\Delta S_d = -512.2338 + 74.7401\,M + 3.333\,M^2 + 0.0474\,M^3 \tag{9}$$

Adsorption: $$\Delta S_d = -1319.2156 + 182.96417\,M - 8.0501\,M^2 + 0.1139\,M^3 \tag{10}$$

Table 3: Parameters estimation r, SEE and MRE of the six equations fitted to the desorption isotherms of *Artemisia herba-alba*

Equation	t	A	B	C	r	SEE	MRE %
GAB	**30°C**	0.9453	130.134	11.6615	0.9936	3.6779	8.8067
	40°C	0.9249	44.6854	12.1210	0.9982	1.7312	4.9389
	50°C	0.9336	35.8506	11.5966	0.9994	0.9582	2.9868
Modified Henderson	**30°C**	0.0021	-21.4597	1.1322	0.9656	8.4894	23.0528
	40°C	0.0018	-31.2559	1.1832	0.9786	5.9993	18.9418
	50°C	0.0017	-38.3613	1.1287	0.9820	5.3155	17.8739
Modified Chung-Pfost	**30°C**	167.5299	23.7719	0.0502	0.9508	10.1159	22.3777
	40°C	185.2888	17.1569	0.0547	0.9647	7.6840	21.4599
	50°C	188.1861	10.0500	0.0556	0.9636	7.5262	24.4095
Modified Halsey	**30°C**	2.3000	0.0767	1.5750	0.9947	3.3425	4.3386
	40°C	2.3839	0.0571	1.6137	0.9988	1.4258	4.3176
	50°C	2.2657	0.0413	1.5321	0.9992	1.1077	3.7106
Modified Oswin	**30°C**	13.5719	0.3590	1.9407	0.9874	5.1724	12.3653
	40°C	11.1098	0.3127	2.0081	0.9946	3.0273	9.7730
	50°C	6.1868	0.3237	1.9116	0.9956	2.6378	9.6756

Table 4: Parameters estimation r, SEE and MRE of the six equations fitted to the adsorption isotherms of *Artemisia herba-alba*

Equation	t	A	B	C	r	SEE	MRE %
GAB	**30°C**	0.8506	64.9499	11.2192	0.9883	2.8674	10.64
	40°C	0.8657	64.8084	10.4798	0.9865	2.9968	9.2868
	50°C	0.8711	51.8324	10.1423	0.9908	2.3814	8.0161
Modified Henderson	**30°C**	0.0032	-28.7183	1.6640	0.9693	4.6224	4.98
	40°C	0.0013	-35.9579	1.6000	0.9634	4.8995	15.5903
	50°C	0.0021	-46.7966	1.5548	0.9688	4.3602	15.6330
Modified Chung-Pfost	**30°C**	194.6346	11.7488	0.0851	0.9735	4.3013	9.74
	40°C	178.8064	-0.5920	0.0871	0.9659	4.7287	12.1432
	50°C	195.3486	-5.3317	0.0885	0.9692	4.3353	13.7481
Modified Halsey	**30°C**	3.4533	0.0784	2.0753	0.9926	2.2902	7.82
	40°C	2.8273	0.0644	1.9744	0.9893	2.6602	9.2286
	50°C	2.4462	0.0529	1.8948	0.9919	2.2333	8.7747

Modified Oswin	**30°C**	12.3176	0.2773	2.6549	0.9888	2.8036	6.17
	40°C	9.8171	0.2454	2.5420	0.9843	3.2276	9.1287
	50°C	8.1775	0.2135	2.4555	0.9875	2.7641	8.5091

3.4 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 Eq. (6). It was assumed that, at specific moisture content, Δh_d and ΔS_d did not vary with temperature [8]. The plot of Δh_d versus ΔS_d, shows a linear relation (Fig. 6), with a coefficient of correlation $r = 1$; this indicates that compensation exists. The parameters T_β and α in (Eq. 6) were calculated from the data by linear regression, and the values are detailed in **Table 5**.

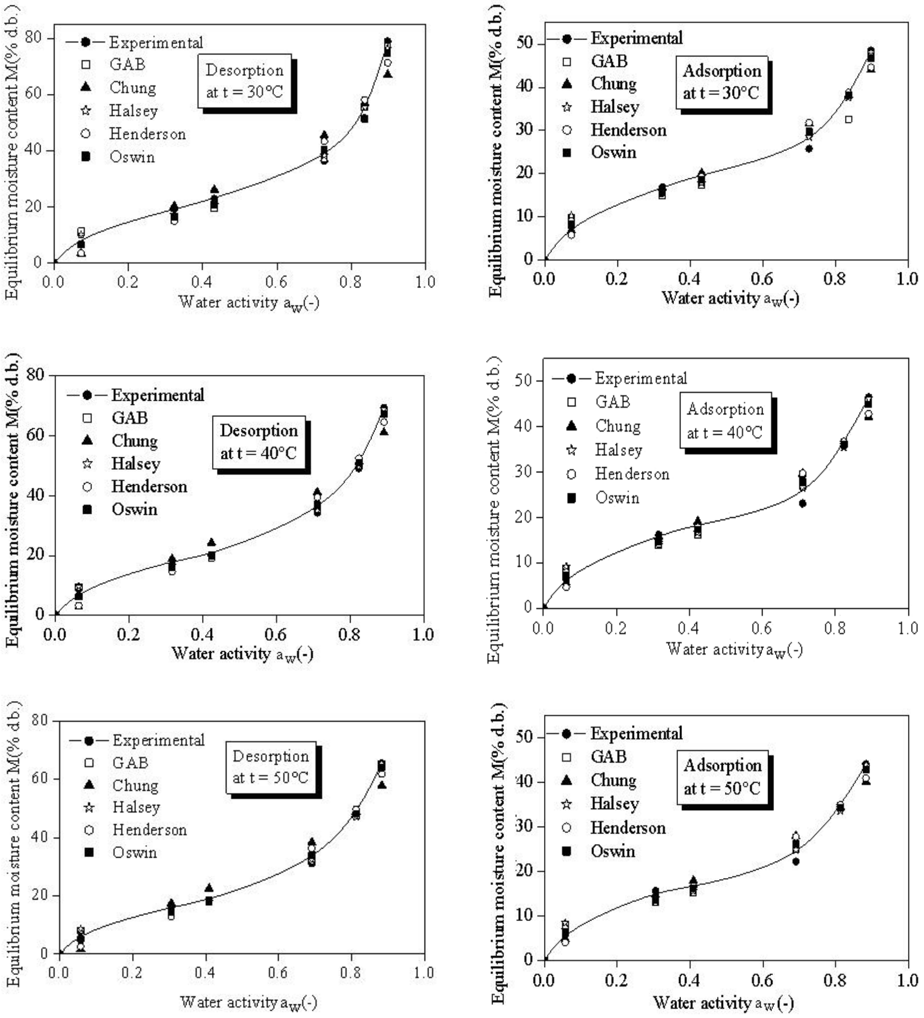

Fig. 3: Predicted and experimental sorption isotherms of *Artemisia herba-alba*.

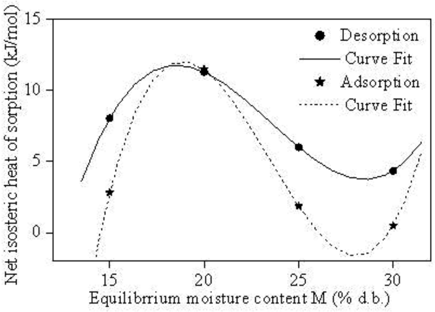

Fig. 4: Net isosteric heat of adsorption and desorption as a function of equilibrium moisture content

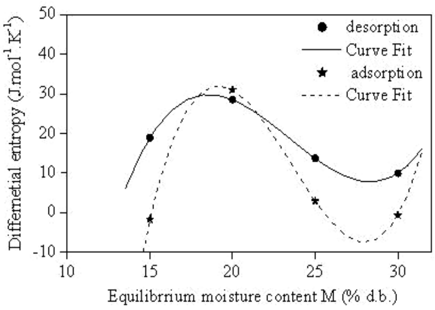

Fig. 5: Differential entropy of adsorption and desorption as a function of equilibrium moisture content

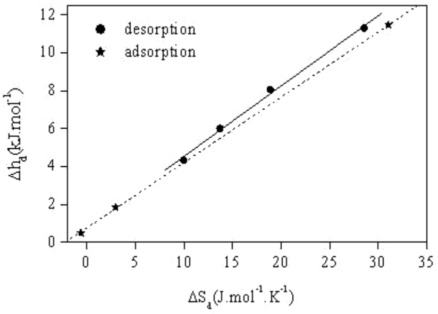

Fig. 6: Δh_d / ΔS_d relationship for adsorption and desorption in *Artemisia herba-alba*

Table 5: Characteristic parameters for Δh_d versus ΔS_d relationship

	T_β (K)	α (J.mol^{-1})	r
Desorption	414.36	381.26	0.9954
Adsorption	352.43	758.01	0.9995

The knowledge of the change of isosteric heat, and thus also of the change of differential entropy, is interesting because it offers an additional level of information to characterize the association of two molecules [19]. Systems can indeed be characterized by constants of the same affinity value, and thus by the same difference in free energy, but to have extremely different contributions enthalpic and entropic, which suggests that different factors are responsible for the stabilization of the product. The entropic term brings information as for the role of the hydrophobic effect on stabilization, this term being generally dominated by the interactions with solvent when this one is water.

The isokinetic temperature T_β represents the temperature to which all the reactions of the series proceed to the same rate and the free energy α at the temperature T_β provides a criterion to evaluate if the process of the water sorption is spontaneous $(-\alpha)$ or not $(+\alpha)$.

4. CONCLUSION

The moisture sorption curves of *Artemisia herba-alba* were experimentally investigated at three temperatures 30, 40 and 50 °C. The isotherms have a sigmoid shape (Type II according to the BET classification) which is common for many hygroscopic products. The hysteresis phenomenon was distinctly observed. Among the sorption models chosen, the GAB model was the best model describing the equilibrium moisture data for desorption, and the modified Halsey model was the most suitable to estimate adsorption isotherms. Net isosteric heat of sorption and differential entropy values of *Artemisia herba-alba* 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 *Artemisia herba-alba*.

NOMENCLATURE

A, B, C	: Model coefficients	t	: Temperature (°C)
ads	: Adsorption	T	: Absolute temperature (K)
des	: Desorption	T_β	: Isokinetic temperature (K)
d.b.	: Dry basis	Δh_d	: Net isosteric heat of sorption (kJ mol^{-1})
M	: Equilibrium moisture content (% d.b.)	ΔH_d	: Isosteric heat of sorption (kJ mol^{-1})
a_w	: Water activity (dimensionless)	ΔH_{vap}	: Heat of vaporization (kJ mol^{-1})
MRE	: Mean relative error (%)	ΔS_d	: Entropy of sorption (J mol^{-1}K^{-1})
SEE	: Standard error of estimate of moisture	α	: Free energy at the temperature T_β (J.mol^{-1})
R	: Universal gas constant (J/mol/K)	r	: Correlation coefficient

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