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Moisture sorption isotherms and thermodynamic properties of tow mints: *Mentha pulegium* and *Mentha rotundifolia*

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Abstract - *Moisture sorption isotherms of Mentha pulegium and Mentha rotundifolia were determined at 30, 40 and 50 °C using the static gravimetric method. GAB, modified Halsey and Peleg models were fitted to the experimental data and the agreement between experimental and predicted values was satisfactory. Calculation of the thermodynamic properties was further used to provide an understanding of the properties of water and energy requirements associated with the sorption behaviour. Isosteric heats of sorption were calculated through direct use of moisture isotherms by applying the Clausius-Clapeyron equation. The differential enthalpy and entropy decreased with increasing moisture content and were adequately characterized by a power law equation. A plot of differential heat versus entropy satisfied the enthalpy-entropy compensation theory.*

Résumé - *Les isothermes de sorption de la menthe pouliot et de la menthe ronde sont déterminées pour 30, 40 et 50 °C à l'aide de la méthode gravimétrique statique. Les modèles de GAB, Halsey modifé et Peleg sont utilisés 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. L'enthalpie différentielle et l'entropie diminuent avec l'élévation de la teneur en eau et la théorie de compensation de l'enthalpie et de l'entropie a été bien vérifiée.*

Keywords: Mentha pulegium - Mentha rotundifolia - Sorption isotherms - Thermodynamic properties.

1. INTRODUCTION

Experimental evaluation of sorption characteristics can aid in the improvement of the processing quality of medicinal and aromatic plants. In the literature, numerous equations have been suggested to represent the relationship between equilibrium moisture content and relative humidity [1]. The choice of these plants is justified by their abundance and importance for the agriculturists of the region of Marrakech (Morocco). *M. pulegium* and *M. rotundifolia* are the most popular mints used for medicinal and aromatic purposes in Morocco. *M. pulegium* is beneficial in the following cases: general metabolism, nervous system, skin, respiratory system, liver, digestive system and reproductive system.

The sorption curves express the hygroscopic equilibrium states of a given product. Their determination constitutes an indispensable stage for better understanding the problems of modelling the drying processes [2, 3]. Using an experimental approach, these equilibrium curves are determined by the saturated salt solutions method. The experimental sorption curves are described by (GAB), modified Halsey and Peleg equations. The GAB model is recognised as the most widely utilised and versatile and was recommended by European COST 90 Project [4]. It represents a kinetic model based on multi-layer and condensation, and has been successfully applied to various products [5-10].

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Further analysis of sorption isotherm data by application of thermodynamic principles can provide much important information regarding the dehydration process energy requirements, food microstructure and physical phenomena on the food surfaces, water properties and sorption kinetic parameters [11].

The isosteric heat of sorption (Δh_d) or differential enthalpy of sorption, gives a measure of the water-solid binding strength. A rapid computational procedure, commonly used for its determination, is the application of the Clausius-Clapeyron equation to the sorption isotherms, at constant moisture content [12-16].

The differential entropy (ΔS_d) of a material is proportional to the number of its available sorption sites at a specific energy level [17, 18]. Physical phenomena, such as sorption reactions, are often evaluated on the basis of the enthalpy-entropy compensation theory. This states that compensation arises due to changes in the solvent-solute interaction, and that a linear relation exists between the enthalpy and entropy of the reaction [19, 20].

The objectives of the present study are to:

- experimentally investigate the adsorption and desorption isotherms of two mints in the temperature range from 30 to 50 °C;
- describe the experimental data using selected mathematical models;
- determine the thermodynamic functions (differential enthalpy, differential entropy);
- evaluate the application of the enthalpy-entropy compensation theory.

2. MATERIALS AND METHODS

2.1 Experimental procedure

All materials used in the experiments were produced in Marrakech (Morocco) and obtained from local markets in Marrakech. Fresh samples were used in desorption experiments. Samples used in adsorption experiments were dried in an oven at 105 °C for 24 h.

The adsorption-desorption isotherms were determined by the standard static gravimetric technique. This method is based on the use of saturated salt solutions to maintain a fixed relative humidity. The salts used were KOH, ($MgCl_2$, $6H_2O$), K_2CO_3, $NaNO_3$, KCl and ($BaCl_2$, $2H_2O$). These salts (**Table 1**) have a range of water activity from 0.05 to 0.9 [21].

Table 1: Water activities of the saturated salt solutions at three temperatures used in the sorption experiments [21]

	Water activity		
Salt	**30 °C**	**40 °C**	**50 °C**
KOH	0.0738	0.0626	0.0572
$MgCl_2$, $6H_2O$	0.3238	0.3159	0.3054
K_2CO_3	0.4317	0.4230	0.4091
$NaNO_3$	0.7275	0.7100	0.6904
KCl	0.8362	0.8232	0.8120
$BaCl_2$, $2H_2O$	0.8980	0.8910	0.8823

Figure 1 shows the various fresh and dried products. The experiment apparatus is shown in (Fig. 2). It consists of six glass jars of 1 litter each with an insulated lid. Each glass jar contains a different salt solution so as to have a water activity that varies from 0.05 to 0.9, and they are immersed in a thermostated water bath adjusted to a fixed temperature for 24 h so as to bring the salt solutions to a stationary temperature.

Duplicate samples each of 0.3 ± 0.001 g for desorption and 0.05 ± 0.001 g for adsorption were weighed and placed into glass jars. The weight recording period was about 4 days. This procedure was continued until the weight was constant. The equilibrium moisture content of each sample was determined by a drying oven whose the temperature was fixed at 105 °C (± 0.1 °C). The time required for equilibrium was three weeks or more depending on water activity and temperature of the bath.

Fresh *M. pulegium* Fresh *M. rotundifolia*

Dried *M. pulegium* Dried *M. rotundifolia*

Fig. 1: Fresh and dried medicinal plants

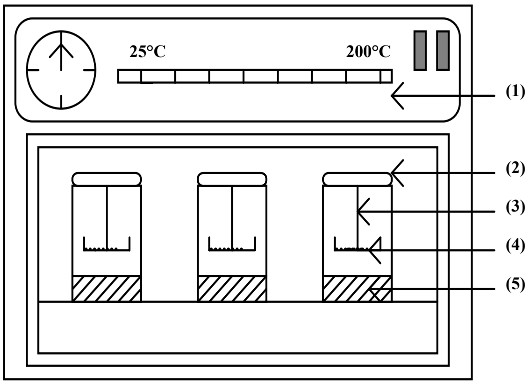

Fig. 2: Experimental apparatus for the sorption isotherms measurement

(1) Thermostated bath ; (2) Glass jar containing salt solution
(3) Sample-holder ; (4) Product ; (5) Saturated salt solution

2.2 Modelling equations

Experimental moisture sorption data can be described by many sorption models. For the purpose of this work, three isotherm equations were chosen to fit the experimental sorption data; the three parameter modified Halsey and GAB models and the four parameter Peleg equation. The selected equations are detailed in **Table 2**.

Nonlinear regression analysis, using the computer programs Curve Expert 3.1, and Origin 6.1, was used to estimate the model coefficients from the experimental sorption data for all samples. The parameters B and C in the GAB equation can be correlated with temperature using the following Arrhenius-type equation [14, 22-27].

Table 2: Equations describing the moisture sorption isotherms

Model	Equation	Ref.
GAB	$M = \frac{A\ B\ C\ a_W}{\left[1 - B\ a_w\right]\left[1 - B\ a_w + B\ C\ a_w\right]}$ $B = B_0 \exp\left(\frac{h_1}{R\ T}\right)$ and $C = C_0 \exp\left(\frac{h_2}{R\ T}\right)$	[10]
Peleg	$M = A\ a_w^c + B\ a_w^D$	[40]
Modified Halsey	$a_w = \exp\left[\frac{-\exp\left(A + B\ \theta\right)}{M^C}\right]$	[41]

$$B = B_0 \exp\left(\frac{h_1}{R\ T}\right) \tag{1}$$

$$C = C_0 \exp\left(\frac{h_2}{R\ T}\right) \tag{2}$$

The suitability of the equations has been evaluated and compared between them using the correlation coefficient (r), mean relative error MRE (%) and the mean square of error MSE [28-31].

$$r = \sqrt{\frac{\sum_{i=1}^{N}\left(M_{pi} - \overline{M}_{ei}\right)^2}{\sum_{i=1}^{N}\left(M_{ei} - \overline{M}_{ei}\right)^2}} \tag{3}$$

$$MRE = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{M_{i,e} - M_{i,p}}{M_{i,e}}\right| \tag{4}$$

$$MSE = \sqrt{\frac{\sum_{i=1}^{N}\left(M_{i,e} - M_{i,p}\right)^2}{d_f}} \tag{5}$$

2.3 Isosteric heat of sorption

The isosteric (differential) 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 (Δh_d) is the amount of energy above the heat of vaporization of water (ΔH_{vap})associated with the sorption process, and was calculated from the experimental data using the Clausius-Clapeyron equation [32].

$$\left[\frac{d\left(\ln a_w\right)}{d\left(1/T\right)}\right]_M = \frac{-\Delta h_d}{R} \tag{6}$$

and

$$\Delta h_d = \Delta H_d - \Delta H_{vap} \tag{7}$$

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

Re-plotting the experimental sorption isotherm in the form $\ln(a_w)$ versus $1/T$, 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 [32, 33].

2.4 Enthalpy-entropy compensation theory

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

$$(-\ln a_w)_M = \frac{\Delta h_d}{R\,T} - \frac{\Delta S_d}{R} \tag{8}$$

By plotting $\ln(a_w)$ versus $1/T$, 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 [34].

The compensation theory proposes a linear relationship between Δh_d and ΔS_d [19] as follows:

$$\Delta h_d = T_\beta \times \Delta S_d + \alpha \tag{9}$$

The isokinetic temperature (T_β) and free energy (α) at T_β were calculated using linear regression.

3. RESULTS AND DISCUSSION

3.1 Fitting of sorption models to experimental data

The experimental results for the equilibrium moisture contents of *M. pulegium* and *M. rotundifolia* at each water activity (a_w) for three different temperatures are given in **Table 3** for adsorption and **Table 4** for desorption.

Table 3: Equilibrium moisture contents (% d.b) of two mints: adsorption data

θ (°C)	a_w	*M. pulegium*	*M. rotundifolia*
30	0.0738	8.3333	4.4211
	0.3238	11.8492	6.4854
	0.4317	13.6116	8.9958
	0.7275	20.8333	14.8536
	0.8362	29.1209	24.2739
	0.898	46.4151	36.8209
40	0.0626	7.6225	2.9787
	0.3159	10.2564	5.9623
	0.423	12.5227	6.7623
	0.71	18.8748	12.6789
	0.8232	29.3352	21.2500
	0.891	44.1989	29.2111
50	0.0572	7.0397	2.3404
	0.3054	8.9767	4.6316
	0.4091	11.4130	6.5678
	0.6904	18.0180	12.2622
	0.812	25.9124	20.7113
	0.8823	43.0127	27.0833

Table 4: Equilibrium moisture contents (% d.b) of two mints: desorption data

θ (°C)	a_w	*M. pulegium*	*M. rotundifolia*
30	0.0738	11.3360	8.2949
	0.3238	13.4066	11.0849
	0.4317	16.3636	13.0243
	0.7275	26.4516	21.8341
	0.8362	31.5789	33.6658
	0.898	51.7241	41.1924
40	0.0626	10.5751	7.1298
	0.3159	12.4138	10.0223
	0.423	15.0101	12.5468
	0.71	25.0000	17.2414
	0.8232	30.0866	27.0983
	0.891	45.5399	33.2564
50	0.0572	7.9167	6.3830
	0.3054	9.2369	9.7902
	0.4091	11.6327	11.6071
	0.6904	18.3333	17.0157
	0.812	26.7347	26.8293
	0.8823	44.1860	31.7073

Equilibrium moisture content data obtained for two aromatic plants at different water activities and temperatures are shown in figure 3 and 4. The sorption isotherms have a sigmoid shape (Type II) witch is common for many hygroscopic products.

The results reveal the temperature dependence of the sorptive behaviour, with an increase in temperature decreasing the sorption capacity. Activation of the water molecules due to the increase in temperature causes them to break away from the water binding sites, thus lowering the equilibrium moisture content.

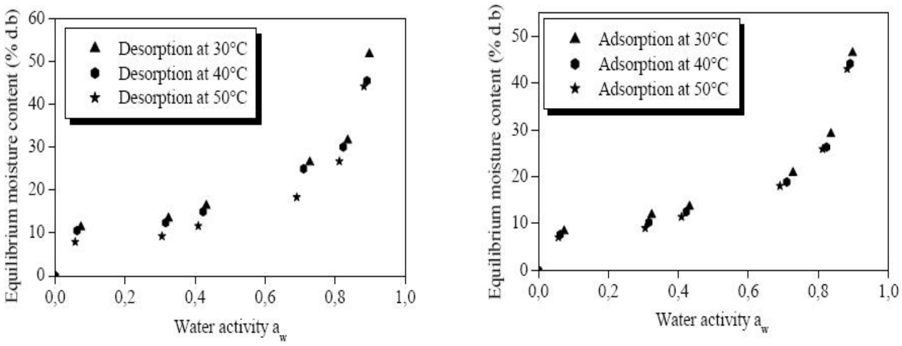

Fig. 3: Influence of temperature on the equilibrium moisture content of *M. pulegium*

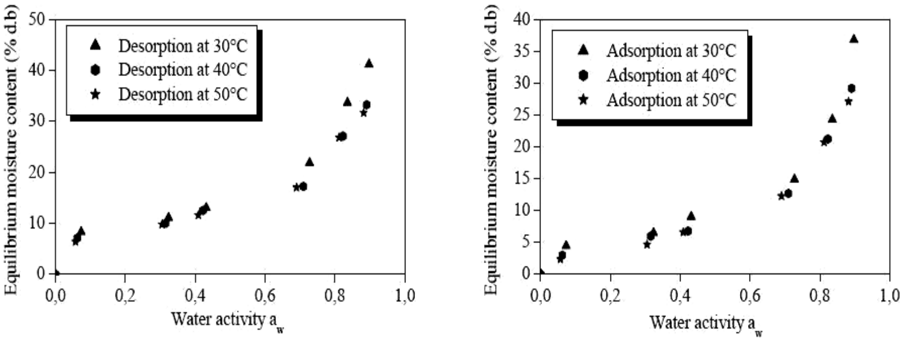

Fig. 4: Influence of temperature on the equilibrium moisture content of *M. rotundifolia*

By comparing the sorption isotherms of the two products at 30, 40 and 50 °C. Figures 5 and 6 shows that the sorption curves of *M. pulegium* and *M. rotundifolia* have similar rates. At constant relative humidity, the equilibrium water content of *M. pulegium* is higher than that of *M. rotundifolia*. This result means that if the storage temperature of a medicinal plant is equal to 40°C, in an ambience whose relative humidity is fixed, *M. rotundifolia* will be dried to a final moisture content that is lower than *M. pulegium's*. The hysteresis phenomenon was observed for the two plants (Fig. 7).

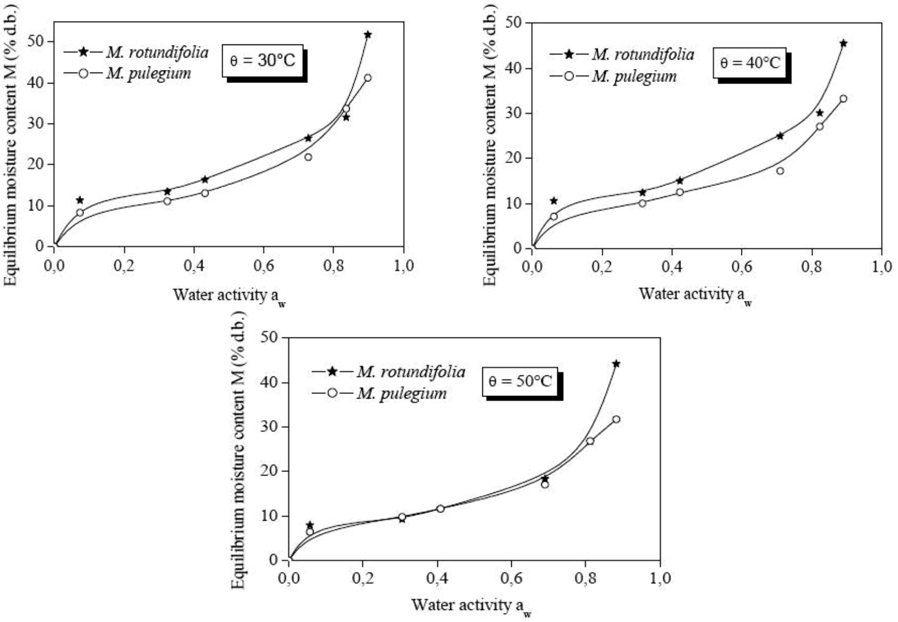

Fig. 5: Comparison of the desorption equilibrium moisture contents of tow mints

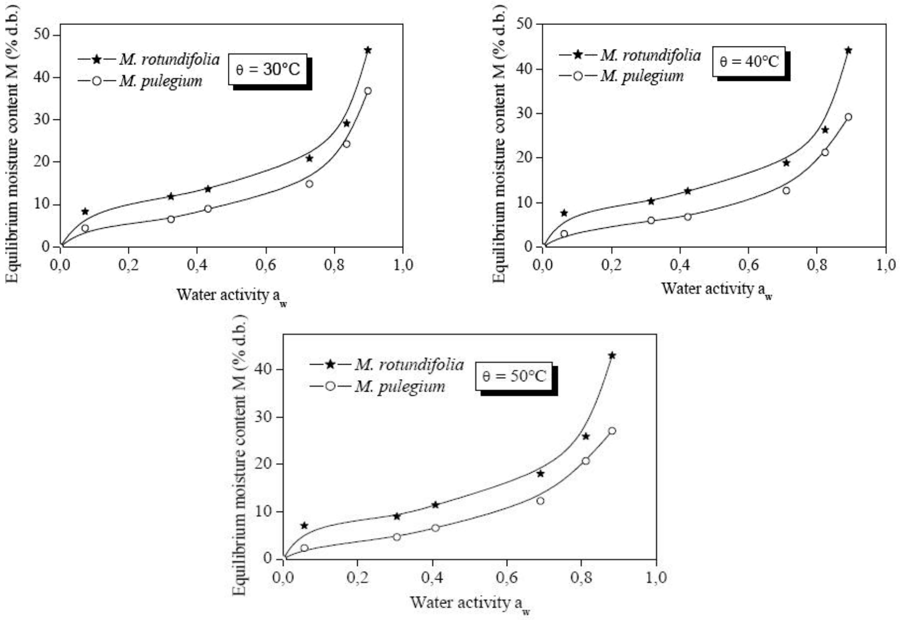

Fig. 6: Comparison of the adsorption equilibrium moisture contents of two mints

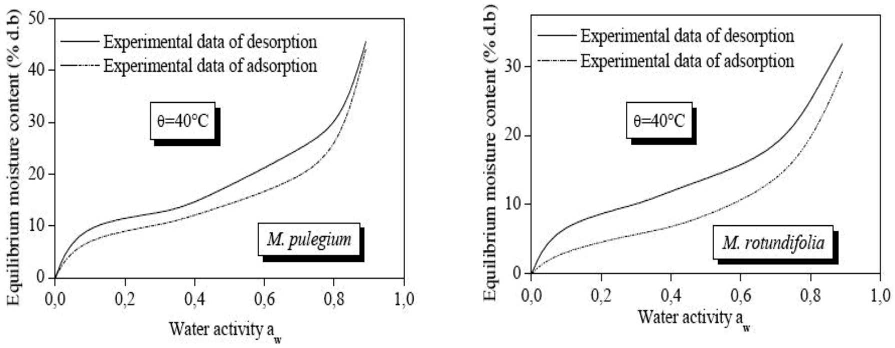

Fig. 7: Hysteresis between adsorption and desorption isotherms for two mints

The sorption relationships detailed in **Table 2** were fitted to the experimental data for all samples. The results of nonlinear regression analysis of fitting the sorption equations to the experimental data are shown in **Tables 5-7**. The moisture content models were compared according to their correlation coefficient (r), mean relative error (MRE) and mean square of error (MSE). A model is considered suitable if the MRE value is less than 10 % [35]. GAB equation gave a satisfactory prediction of the adsorption and desorption equilibrium moisture content of all samples. It should be noted that, the goodness of fit of any sorption model to the experimental data shows only a mathematical quality and not the nature of the sorption process [11].

Table 5: Estimated GAB model coefficients, r, MRE and MSE fitted to the sorption isotherms of two mints

Product	A	B	C	B_0	C_0	r	MRE(%)	MSE
Adsorption								
M. pulegium	0.974	$2.21\ 10^{11}$	5.895	1.582	$2.46\ 10^{15}$	0.995	7.193	1.820
M. rotundifolia	0.957	$2.73\ 10^{1}$	4.370	0.504	$1.82\ 10^{-22}$	0.999	2.954	0.685
Desorption								
M. pulegium	0.975	$1.94\ 10^{11}$	6.066	2.311	$1.36\ 10^{4}$	0.994	8.498	1.40
M. rotundifolia	0.903	$6.92\ 10^{2}$	7.885	0.548	$6.74\ 10^{-12}$	0.998	2.037	1.117

Table 6: Estimated Peleg model coefficients, r , MRE and MSE fitted to the sorption isotherms of two mints

Product	A	B	C	D	r	MRE (%)	MSE
Adsorption							
M. pulegium	97.898	18.991	11.673	0.351	0.995	7.193	1.820
M. rotundifolia	42.427	9.253	6.329	0.401	0.999	2.954	0.685
Desorption							
M. pulegium	90.339	14.355	8.841	0.240	0.994	8.498	1.40
M. rotundifolia	48.496	14.176	5.203	0.206	0.998	2.037	1.117

Table 7: Estimated modified Halsey model coefficients, r , MRE and MSE fitted to the sorption isotherms of two mints

Product	A	B	C	r	MRE (%)	MSE
Adsorption						
M. pulegium	1.734	0.030	1.425	0.989	11.963	2.620
M. rotundifolia	1.711	0.034	1.389	0.998	3.499	0.788
Desorption						
M. pulegium	3.098	0.048	1.895	0.989	7.328	2.530
M. rotundifolia	2.878	0.056	1.817	0.996	5.581	1.583

Using these coefficients, the sorption isotherms of *M. pulegium and M. rotundifolia* are predicted by GAB, modified Halsey and Peleg equations. The representation of these results is shown in figures 8 to 10 from which it can be noted that the predicted curve by GAB's model and the experimental data have practically the same rate.

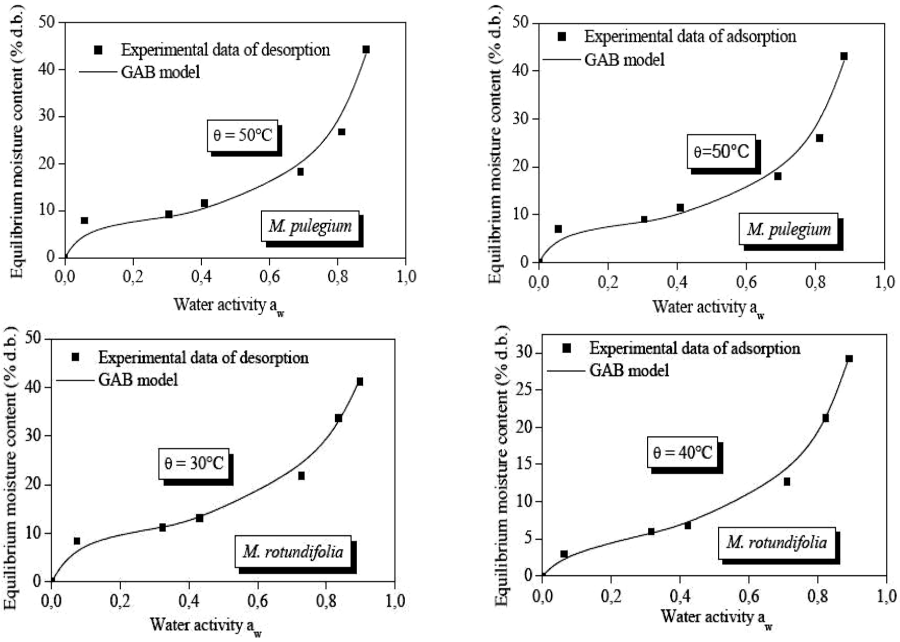

Fig. 8: Comparison between experimental and predicted moisture sorption isotherms of two mints using GAB equation

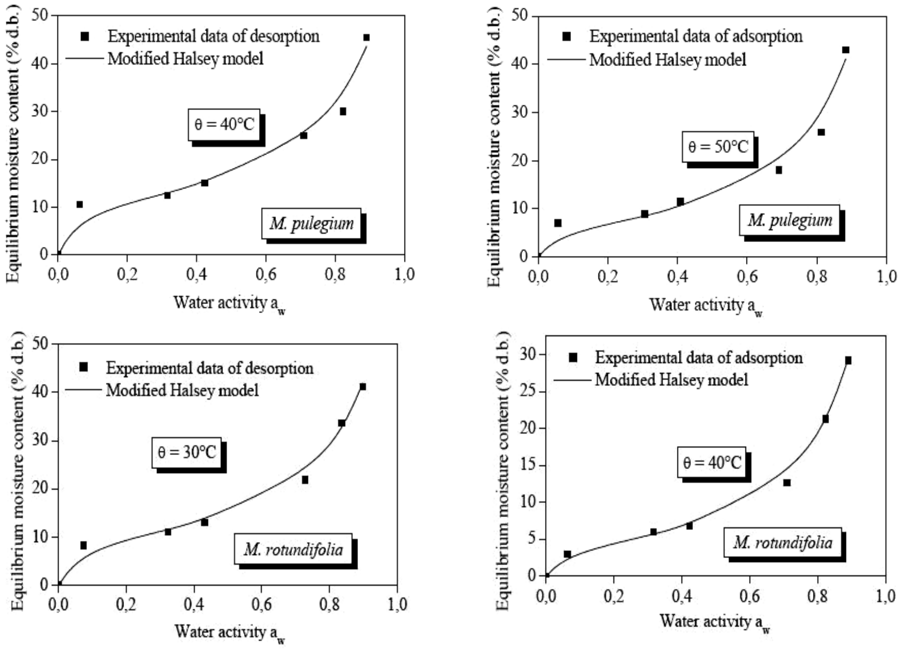

Fig. 9: Comparison between experimental and predicted moisture sorption isotherms of two mints using modified Halsey equation

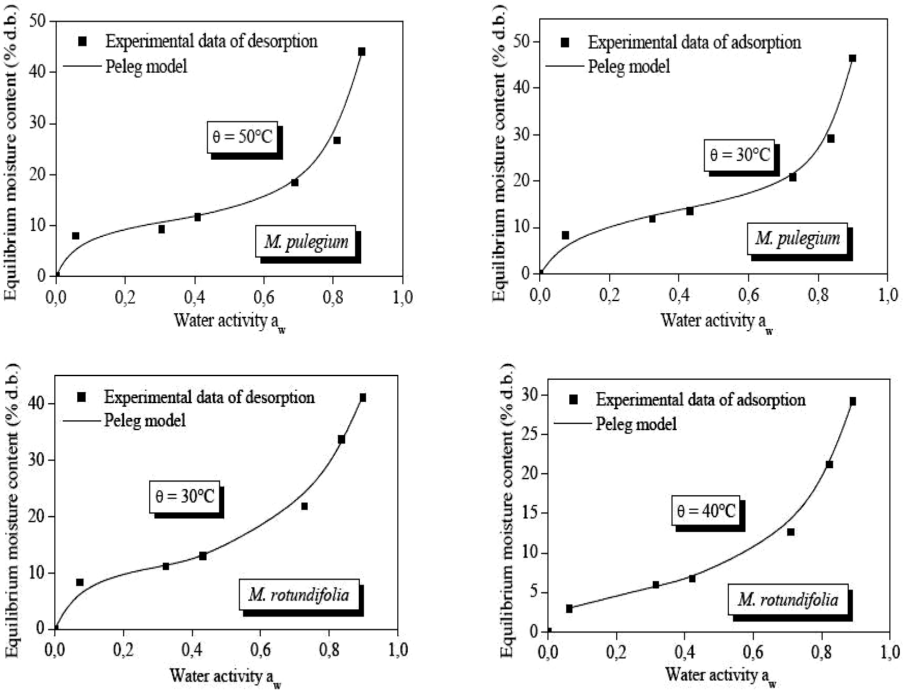

Fig. 10: Comparison between experimental and predicted moisture sorption isotherms of two mints using Peleg equation

3.2 Isosteric heat of sorption

The values of the isosteric heat of sorption, Δh_d, were calculated from the equilibrium data at different temperatures using Eq. (6) and obtained at different moisture contents. The variation of the heats of adsorption and desorption of the two plants with moisture content is shown in figure 11 and 12. Figure 13 shows the variation of isosteric heat with moisture content for each product.

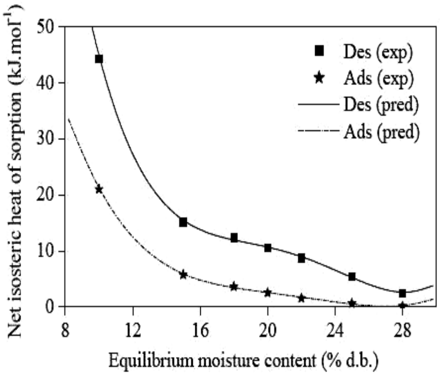

Fig. 11: Isosteric heat of adsorption and desorption of *M. pulegium* as a function of equilibrium moisture content

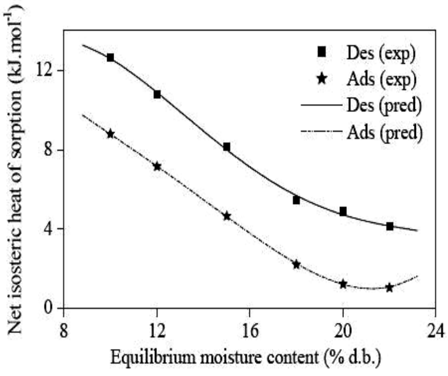

Fig. 12: Isosteric heat of adsorption and desorption of *M. rotundifolia* as a function of equilibrium moisture content

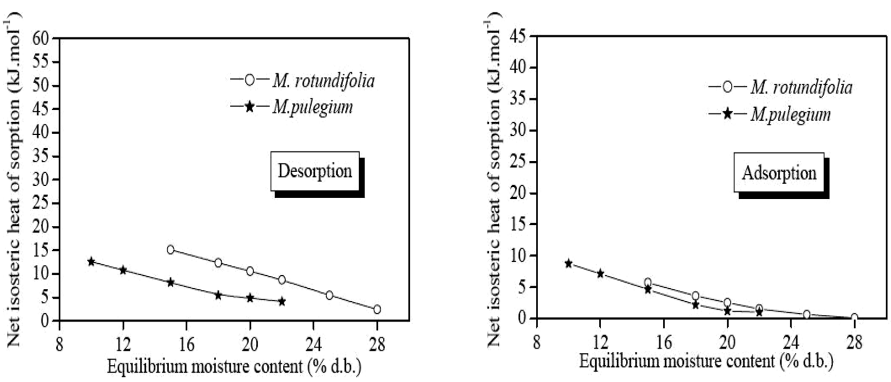

Fig. 13: Variation of net isosteric heat of adsorption and desorption with moisture content for tow mints

As seen in these figures, isosteric heats of sorption are high at low moisture contents (< 8–15%). The heat of desorption is greater than of adsorption at low moisture contents for all samples studied. This indicates that energy required in the desorption process is greater than that in the adsorption process as stated by [35]. The isosteric heats versus moisture content results are adequately represented by polynomial functions.

M. pulegium

Desorption

$$\Delta h_d = 428.981 - 79.035\ M + 5.615\ M^2 - 0.176\ M^3 + 0.002\ M^4 \qquad r = 0.9999$$

Adsorption

$$\Delta h_d = 193.584 - 34.595\ M + 2.385\ M^2 - 0.074\ M^3 + 8.476\ 10^{-4}\ M^4 \qquad r = 0.9996$$

M. rotundifolia

Desorption

$$\Delta h_d = -7.291 + 6.781\ M - 0.729\ M^2 + 0.029\ M^3 - 3.992\ 10^{-4}\ M^4 \qquad r = 0.9978$$

Adsorption

$$\Delta h_d = 22.442 - 2.854\ M + 0.273\ M^2 - 0.016\ M^3 + 3.239\ 10^{-4}\ M^4 \qquad r = 0.9999$$

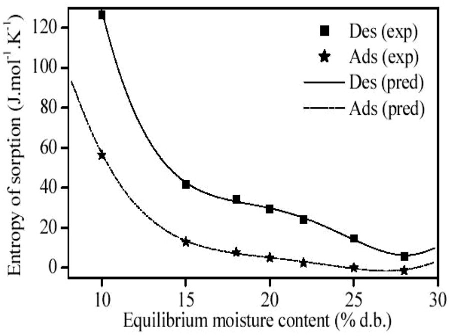

Fig. 14: Differential entropy of adsorption and desorption of *M. pulegium* as a function of equilibrium moisture content

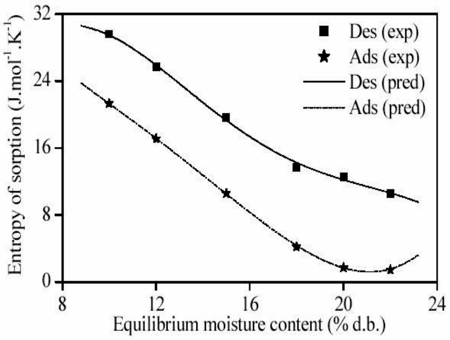

Fig. 15: Differential entropy of adsorption and desorption of *M. rotundifolia* as a function of equilibrium moisture content

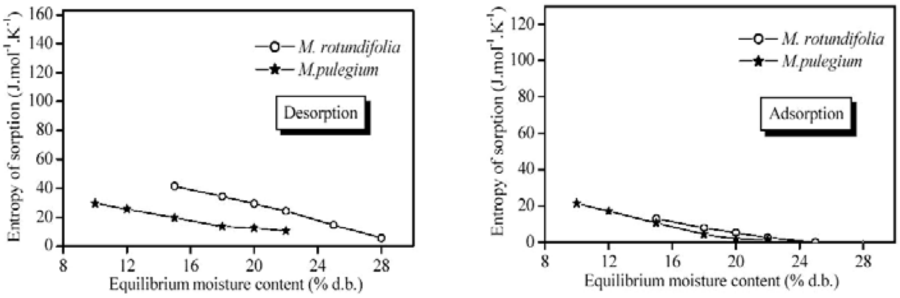

Fig. 16: Variation of differential entropy of adsorption and desorption with moisture content for tow mints

The differential entropy is plotted as a function of moisture content in figure 14 and 15. The variation of differential entropy with equilibrium moisture content for each product is shown in figure 16. Once again the entropy data display a strong dependence on moisture content. The experimental adsorption and desorption data, for each product, conform to a relation as represented by the equations.

M. pulegium

Desorption

$$\Delta S_d = 1280.008 - 237.930\ M + 16.982\ M^2 - 0.534\ M^3 + 0.006\ M^4 \qquad r = 0.9997$$

Adsorption

$$\Delta S_d = 574.611 - 104.909\ M + 7.321\ M^2 - 0.228\ M^3 + 0.003\ M^4 \qquad r = 0.9995$$

M. rotundifolia

Desorption

$$\Delta S_d = -49.756 + 24.702\ M - 2.583\ M^2 + 0.106\ M^3 - 0.002\ M^4 \qquad r = 0.9980$$

Adsorption

$$\Delta S_d = 56.853 - 7.581\ M + 0.743\ M^2 - 0.043\ M^3 + 8.947\ 10^{-4}\ M^4 \qquad r = 0.9999$$

3.3 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. (8). It was assumed that, at specific moisture content, Δh_d and ΔS_d did not vary with temperature ([36]. The plot of Δh_d versus ΔS_d for each product (Fig. 17 and 18) shows a linear relation, with a coefficient of determination $r = 1$; this indicates that compensation exists. The parameters T_β and α (Eq. (9)) were calculated from the data by linear regression, and the values are detailed in Table 8.

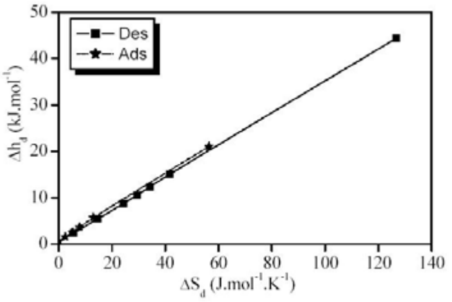

Fig. 17: $\Delta h_d / \Delta S_d$ relationship for adsorption and desorption of *M. pulegium*

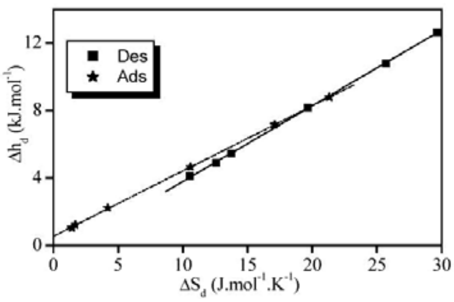

Fig. 18: $\Delta h_d / \Delta S_d$ relationship for adsorption and desorption of *M. rotundifolia*

Table 8: Characteristic parameters for Δh_d versus ΔS_d relationship

	$T_\beta\left(K\right)$	$\alpha\left(Jmol^{-1}\right)$	r
M. pulegium			
Desorption	352.26	315.57	1
Adsorption	391.31	619.75	1
M. rotundifolia			
Desorption	441.49	-520.99	1
Adsorption	386.74	541.8	1

4. CONCLUSION

The sorption curves provide valuable information about the hygroscopic equilibrium of aromatic and medicinal plants. They give a clear idea on the stability domain of the plant after drying. They also yield information on the different kinds of water in the product. So, these curves are indispensable in the pharmaceutical industry, especially in the operation of storage and conservation of aromatic and medicinal herbs [37-39].

The sorption isotherms of two aromatic and medicinal plants have been determined by experiment and then described by GAB, modified Halsey and Peleg models. The saturated salt method can be used successfully for experimental determination of the equilibrium moisture content of aromatic and medicinal plants. The experimental results show that the sorption isotherms of *M. pulegium* and *M. rotundifolia* take a form of the sigmoid type and that GAB, modified Halsey and Peleg models give a better fit for the sorption isotherms of aromatic and medicinal herbs.

The net isosteric heats of sorption can be calculated using the Clausius-Clapeyron equation. The heats of sorption increased with decreasing moisture content. Desorption values are higher than those for adsorption. The differential entropy of sorption can be characterized by a polynomial function. The isosteric heat versus entropy data satisfies the enthalpy-entropy compensation theory.

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