

Moisture sorption isotherms and thermodynamic properties of *Asteriscus graveolens* leaves

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Abstract: *Asteriscus graveolens* is a medicinal and aromatic plant which has been used to treat several diseases. Moisture equilibrium data of the plant leaves were determined by using the gravimetric static method at 30 °C, 40 °C, and 50 °C for water activity (*a_w*) ranging from 0.057 to 0.898. A hysteresis effect was observed. The experimental data of sorption were fitted by eight models (GAB, BET, Henderson-Thompson, modified-Chung Pfoest, Halsey, Oswin, Peleg, and Adam and Shove). The Halsey and Peleg models were the most suitable models for estimating sorption isotherms curves. The net isosteric heats of desorption and adsorption of *A. graveolens* leaves were calculated by applying the Clausius-Clapeyron equation to the sorption isotherms.

Keywords: moisture sorption, isotherm, *Asteriscus graveolens*, Isosteric heat, modelling

Citation: Saad, A., B. Touati, B. Draoui, B. Tabti, A. Abdenebi, and K. Mir. 2016. Moisture sorption isotherms and thermodynamic properties of *Asteriscus graveolens* leaves. *Agricultural Engineering International: CIGR Journal*, 18 (2):276-285.

1 Introduction

Asteriscus graveolens belongs to the family of *Asteraceae*; it is an endemic herbaceous medicinal aromatic plant extending from North Africa to the desert regions of Asia, it is mainly distributed in South-Western Algeria and South-Eastern Morocco (IUCN, 2005). Over the past twenty years, considerable attention has been given to *Asteriscus* genus (Akssira et al., 2006). The phytochemical screening of the leaves and flowers of *A. graveolens* revealed the presence of alkaloids, catechintanins, terpenes, coumarins and cyagenetic compounds (Alilou et al., 2014). Many studies have been focused on *A. graveolens*, describing its flavonoids, sesquiterpenes lactones and humulenes derivatives

content (Cristofari et al., 2012; Ahmed et al., 1991; El Dahmy et al., 1985). The essential oil of this species has been found to display a variety of biological activities such as antioxydant and antifungal activities that might be a natural potential source of preservative and antifungal compound used in food, cosmetics and in pharmaceutical products (Alilou et al., 2014). The essential oil of the plant has also been used in a green approach as corrosion inhibitor of mild steel (Znini et al., 2012).

Extracts from the plant have been used for controlling bayoud disease of date palm caused by *Fusarium oxysporum* sp. *albidenis*ⁱ (Mebarki et al., 2015). This Saharan species is reported to possess antimicrobial and hypoglycemic activities whereas its Asterunculides have shown phytotoxic and cytotoxic activities (Rauter et al., 2015). *A. graveolens* is also used as a natural active ingredient in some cosmetic products such as IBR-Pristinizer which is commercialized and used to protect cells from air pollution (IBR).

Received date: 2015-11-21 Accepted date: 2016-03-14

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The plant has a significant history of medicinal use by Algerian Saharan people for the treatment of disease, such as female infertility, rheumatism, muscle contraction, fatigue, diabetes, headache and infected wounds (Hammiche and Maiza, 2006).

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 (Reguig, 2011).

It is well known that many factors, such as a high drying temperatures or prolonged storage under air and/or at a relatively high temperature and humidity have negative effects on various product quality attributes.

In order to correctly conduct drying and storage operations, it is necessary to know the relationship between air temperature (T) and the relative humidity (RH), and desirable conditions for preserving the product, to obtain this information, sorption isotherms are

Table1 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
BaCl ₂	0.898	0.891	0.8823

indispensable(Arana, 2012). See Table 1 please.

From the literature, no information on sorption isotherms has been reported for *A. graveolens*. Thus, this study is an attempt to establish the moisture adsorption and desorption isotherms of *A. graveolens* leaves, at water activities and temperatures from 0.057 to 0.898 and from 30 °C to 50 °C, respectively. Eight models presented in the literature (GAB, BET, Henderson-Thompson, modified-Chung Pfof, Halsey, Oswin, Peleg, and Adam and Shove) were fitted to the experimental data in order

to verify their adequacy to describe the equilibrium moisture content of *A. graveolens* leaves.

2 Materials and methods

2.1 Description of experimental procedure

Asteriscus graveolens leaves for experiments were obtained from Bechar region of South-Western Algeria. Harvest was done between March and May 2011. Discolored and dried out samples were discarded

2.2 Determination of sorption isotherms

The equilibrium moisture content of *Asteriscus graveolens* leaves at 30 °C, 40 °C and 50 °C were determined by a gravimetric technique, which is based on the use of saturated salt solutions to obtain constant relative humidity of surrounding air.

Six saturated salt solutions (KOH, MgCl₂, K₂CO₃, NaNO₃, KCl and BaCl₂) were prepared by dissolving an appropriate quantity of salt in distilled water at a higher temperature than equilibration to ensure that they remain saturated when cooled.

The experimental apparatus utilized consists of six glass jars of 1L each with insulated lid.

Every glass jar is filled to one quarter depth with a prepared saturated salt solution. A layer of solid salts was maintained during the whole period of equilibration to confirm that the solutions always remain saturated. A tripod was also put in each jar to place *Asteriscus graveolens* leave sample. The jars are then put in a controlled temperature oven for 24 h to be stabilized at experiment temperature. As indicated in Table 1, the saturated salt solutions allow obtaining a water activity ranging from 0.07 to 0.89 (Greenspan, 1977).

Duplicated samples, each of 0.02g ($\pm 0,0001$ g) for desorption and 0.01g ($\pm 0,0001$ g) for adsorption, were weighed using an analytical balance ($\pm 0,0001$ g) and placed into the glass jars. The glass jars, containing saturated salt solutions and *Asteriscus graveolens* leaves samples were then tightly closed and then put in an oven at a fixed temperature (30, 40 or 50 ± 1 °C) for equilibration.

The *Asteriscus graveolens* leaves samples were weighed every two days, until there is no change in mass. Fresh *Asteriscus graveolens* leaves were used for desorption experiments. Samples used for adsorption isotherms were dried 24h in an oven at 105°C until reaching maximum dehydration. The moisture content of each sample was determined in a drying oven at 105°C ($\pm 0,1$ °C) for 24 h (AOAC, 1990). The hygroscopic equilibrium of *Asteriscus graveolens* leaves was reached in eight days for desorption and six days for adsorption.

The difference in mass before (m_w) and after (m_d) drying in the oven gives the moisture content X_e of the product at hygroscopic equilibrium (Equation 1).

$$X_e = \frac{mw - md}{md} \quad (1)$$

2.3 Data analysis

2.3.1 Modeling of sorption isotherms

Eight mathematical equations were used for describing desorption and adsorption isotherms of *Asteriscus graveolens* leaves in the range of temperature varying from 30 °C to 50 °C. The expressions and the parameters of the eight models used to fit the data are presented in Table 2. Non-linear regression analysis was used to estimate the constants of the models from the experimental results of sorption isotherms for *Asteriscus graveolens*. The goodness of fit was determined by using three statistical parameters: the standard error (S), the correlation coefficient (r) and the percent average relative deviation (P). These statistical parameters were defined as Equation 2, Equation 3 and Equation 4.

Table 2 Mathematical models used to describe desorption and adsorption isotherms of *Asteriscus graveolens*

Models names	Models equations	References
GAB	$X_e = \frac{CAB a_w}{(1 - B a_w)(1 - B a_w + A B a_w)}$	
BET	$X_e = \frac{AB a_w}{(1 - a_w)(1 + (A - 1)a_w)}$	Iglesias & Chirife (1995)
Henderson-Thompson	$X_e = \left(\frac{\ln(1 - a_w)}{-A(T + B)} \right)^{1/c}$	Brunauer, Emmett, & Teller (1938)
Modified Chung and Pfof		Henderson (1952)
Halsey	$X_e = A - B \ln(a_w) \ln(-BT)$	Chung & Pfof (1967)
Oswin	$X_e = A \left(-\frac{B}{\ln(a_w)} \right)^{1/c}$	Halsey (1948)
Peleg		Chia-Chung Chen (1990)
Adam and Shove	$X_e = A(a_w / (1 - a_w))^B$	Peleg, M. (1993)
	$X_e = A a_w^B + C a_w^D$	Chirife & Iglesias (1978)
	$X_e = A + B a_w + C a_w^2 + D a_w^2$	

$$S = \sqrt{\frac{\sum_{i=1}^{n_{\text{exp.data}}} (X_{ei} - X_{e_{\text{cali}}})^2}{n_{\text{exp.data}} - n_{\text{param}}}} \quad (2)$$

$$r = \sqrt{1 - \frac{\sum_{i=1}^{n_{\text{exp.data}}} (X_{ei} - X_{e_{\text{cali}}})^2}{\sum_{i=1}^{n_{\text{exp.data}}} (\bar{X}_e - X_{ei})^2}} \quad (3)$$

and

$$P (\%) = \frac{100}{n} \sum_{i=1}^{n_{\text{exp.data}}} \left(\frac{X_{e_{\text{cali}}} - X_{ei}}{X_{ei}} \right) \quad (4)$$

Where $X_{e_{\text{cali}}}$ is the calculated value of equilibrium moisture content by using the tested model, X_{ei} is the experimental value of equilibrium moisture content, n_{param} is the number of parameters of the particular model and $n_{\text{exp. data}}$ is the number of experimental points. The arithmetic average value of the experimental equilibrium moisture content (\bar{X}_e) is calculated as Equation 5:

$$\bar{X}_e = \frac{1}{n_{\text{exp.data}}} \sum_{i=1}^{n_{\text{exp.data}}} X_{ei} \quad (5)$$

2.3.2 Determination of the net isosteric heat of sorption

The net isosteric heat of sorption can be determined from moisture sorption data by using the following equation, which is derived from the Clausius–Clapeyron equation (Iglesias and Chirife, 1976; Kapsalis, 1987). See Equation 6:

$$\ln(a_w) = - \left(\frac{q_{st}}{R} \right) \left(\frac{1}{T_K} \right) + K \quad (6)$$

Where q_{st} is the net isosteric heat of sorption (kJ/mol); a_w the water activity (dimensionless); T_k the absolute temperature (K); R the universal gas constant (kJ/mol K); and K a constant. The sorption isotherms are plotted as $\ln(a_w)$ versus $1/T$ for fixed values of equilibrium moisture contents (the isosteres curves). The net isosteric heats of sorption could be calculated at each value of equilibrium moisture content from the slope of the isosters curves which is equal to $-(q_{st}/R)$.

Note:

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.

3 Results and discussion

3.1 Sorption isotherm of *Asteriscus graveolens*

The experimental results of the equilibrium moisture content of *Agraveolens* at each water activity (a_w) for the three temperatures are given in Figure 1, Figure 2, and Figure 3. Sorption isotherms of *A. graveolens* leaves showed type II behavior according to the BET classification. These curves are typical of plant products as reported by (Saad et al., 2014; Touati et al., 2008; Ait-Mohammed et al., 2004). From the Figures, it is seen that the equilibrium moisture content increased with water activity at constant temperature. The curve is divided into three regions. In region I (water activity between 0.05 and 0.2), water is tightly bound to the product. In region II (water activity between 0.2 and 0.6) the water is less tightly held and usually present in small capillaries. In region III (water activity between 0.6 and 0.9), the water is held loosely in large capillaries, or is free (Heldman et al., 2006).

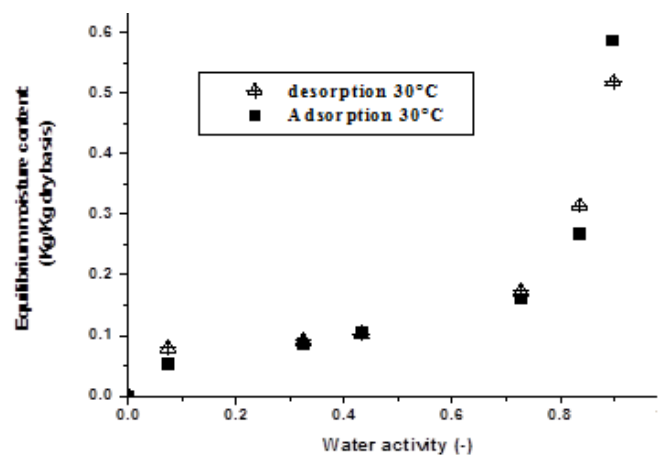


Figure1 Desorption and adsorption isotherms of *Asteriscus. g* leaves obtained at 30°C.

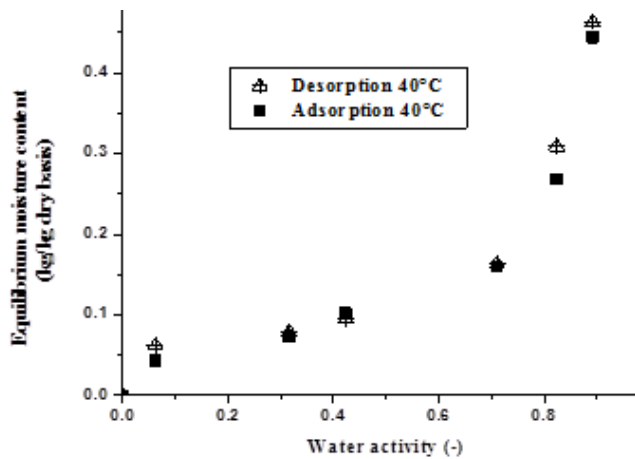


Figure 2 Desorption and adsorption isotherms of *A. graveolens* leaves obtained at 40°C.

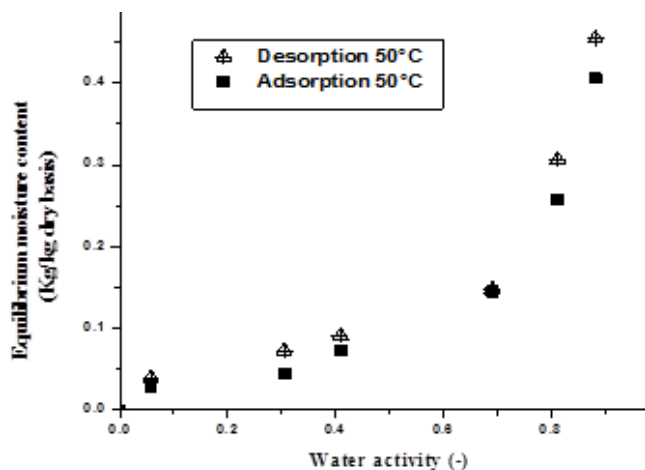


Figure 3 Desorption and adsorption isotherms of *A. graveolens* leaves obtained at 50°C.

The Figures also show the hysteresis phenomenon between adsorption and desorption, in which water content on the desorption isotherm is higher than that on the adsorption side at the same water activity. One of the hypotheses used to explain hysteresis is to consider a

pore connected to its surroundings by a small capillary. During adsorption, as the relative humidity rises, the capillary begins to fill while the pore is empty. Only when the partial pressure of the vapor in air is greater than the vapor pressure of the liquid in the capillary will the moisture move into the pore. Starting from saturation the pore is full of liquid. This fluid can only escape when the partial pressure of the surrounding air falls below the vapor pressure of the liquid in the capillary. Since the system of pores has generally a large range of capillary diameters, it follows that differences between adsorption and desorption will be observed (Mujumdar, 2014). A pronounced temperature dependence over the whole relative humidity range can also be seen. The binding forces decreased with increasing temperature, that is, less moisture is absorbed at higher temperatures at the same relative humidity (Mujumdar, 2014; Labuza et al., 1985), similar trends for many medicinal plants have been reported (Touati et al., 2007; Saad et al., 2014; Lamharrar et al., 2007).

3.2 Modeling of sorption isotherms

Table 2 and Table 3 show, respectively, the results of the non-linear regression analysis of desorption and adsorption isotherms of *A. graveolens* leaves obtained at 30 °C, 40 °C and 50 °C. The values of constants of the eight models, i.e., GAB, BET, Handerson-Thompson, modified Chung and Pfoest, Halsey, Oswin, Peleg, and Adam and Shove fitted to the desorption and adsorption data along with their standard error (S), the correlation coefficient (r) and the percent average relative deviation (P) for the studied temperatures are given.

Table 3 Results of fitting of the desorption isotherms of *Asteriscus graveolens* leaves

Models names	T(°C)	Parameters					r	S	P(%)
		A	B	C	D				
GAB	30	21.93	2.360	0.2435	-	0.878	0.103	37.54	
	40	0.281	6.907	13.515	-	0.921	0.073	29.62	
	50	0.278	6.876	13.062	-	0.920	0.078	36.18	
BET	30	0.279	0.579	-	-	0.98	0.038	27.24	
	40	0.253	0.663	-	-	0.98	0.028	23.33	
	50	0.306	0.509	-	-	0.99	0.023	24.14	
Henderson	30	4.643	0.699	-28.77	-	0.96	0.058	38.51	
	40	6.247	0.756	-28.47	-	0.97	0.043	33.10	
	50	6.264	0.718	-28.37	-	0.98	0.037	34.43	
Chung and pfo	30	6.542	-28.75	10.458	-	0.87	0.105	38.84	
	40	6.622	-28.71	11.040	-	0.88	0.092	38.89	
	50	6.286	-28.69	10.856	-	0.88	0.094	43.06	
Halsey	30	-7.66	0.153	1.209	-	0.98	0.031	4.13	
	40	-7.637	0.150	1.230	-	0.99	0.02	3.76	
	50	-5.542	0.085	1143.	-	0.99	0.01	3.97	
Oswin	30	0.1050	0.712	-	-	0.98	0.038	26.61	
	40	0.1066	0.688	-	-	0.98	0.027	21.04	
	50	0.1017	0.739	-	-	0.99	0.022	22.38	
Peleg	30	0.126	0.229	1.121	9.749	0.99	0.009	4.13	
	40	0.109	0.238	0.839	7.396	0.99	0.007	3.76	
	50	0.113	0.355	0.863	7.220	0.99	0.009	3.97	
Adam and Shove	30	0.052	-0.243	0.367	0.367	0.93	0.092	33.33	
	40	0.041	-0.207	0.342	0.342	0.95	0.069	30.09	
	50	0.032	-0.197	0.346	0.346	0.95	0.066	31.52	

The higher the value of (r) and the lower the values of (S) were chosen as the criteria for goodness of fit. The results indicate that all the models are acceptable for predicting the equilibrium moisture content. However, the Peleg and Halsey models gave the best fitting of adsorption and desorption isotherms for the three temperatures, with lowest standard error and the highest coefficient of correlation.

Peleg equation was found to be satisfactory for many other plant species (Saad et al., 2014; Moreira et al.,

2008). The Halsey equation is suitable for materials of types I,II and III, it have been used to describe 220 experimental sorption isotherms of 69 different foods (Mujumdar, 2014; Rao et al., 2014). Comparisons were done between experimental and calculated (Peleg and Halsey models) data of desorption and adsorption isotherms obtained for *A. graveolens* leaves at the three temperatures and are shown, respectively, in Figure 4, Figure 5 and Table 4.

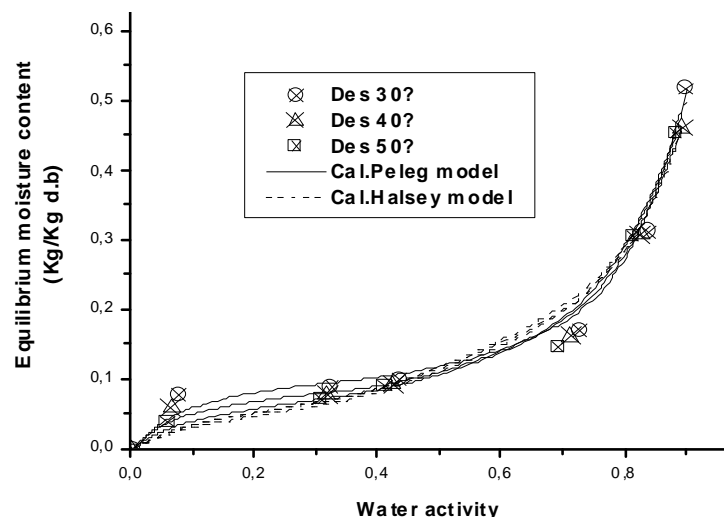


Figure 4 Comparison between experimental and calculated (Peleg and Halsey models) data of desorption isotherms of *A. graveolens* leaves obtained at 30 °C, 40 °C, and 50 °C. Cal.: calculated

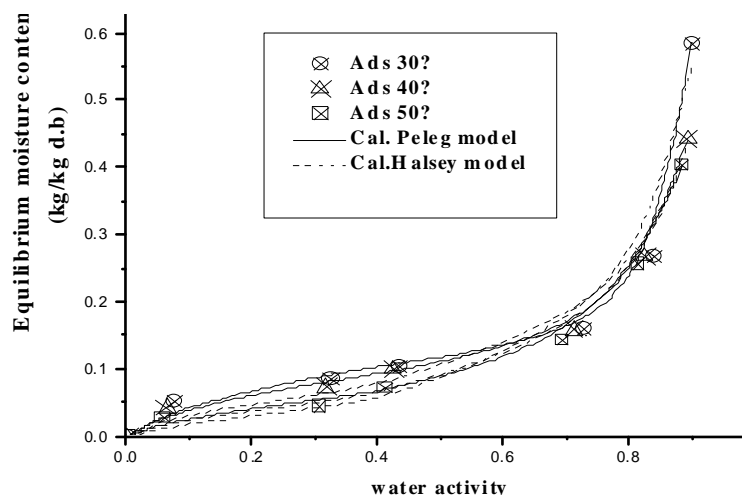


Figure 5 Comparison between experimental and calculated (Peleg and Halsey models) data of adsorption isotherms of *A. graveolens* leaves obtained at 30 °C, 40 °C, and 50 °C. Cal.: calculate

Table 4 Results of fitting of the adsorption isotherms of *Asteriscus graveolens* leaves

Models names	T, °C	Parameters				r	S	P, %
		A	B	C	D			
GAB	30	0.373	5.778	11.231	-	0.84	0.130	43.65
	40	0.281	6.477	11.999	-	0.91	0.075	29.20
	50	0.289	6.061	12.414	-	0.93	0.065	39.33
BET	30	0.589	0.167	-	-	0.97	0.049	37.49
	40	0.255	0.615	-	-	0.99	0.023	24.26
	50	0.351	0.357	-	-	0.99	0.011	16.07
Henderson	30	14.867	0.439	-24.96	-	0.95	0.070	49.54
	40	6.213	0.763	-28.56	-	0.97	0.041	34.77
	50	6.329	0.678	-28.43	-	0.99	0.023	30.35
Chung and ppost	30	6.285	-28.63	9.866	-	0.82	0.136	43.85
	40	6.522	-28.69	11.650	-	0.88	0.086	26.43
	50	6.199	-28.61	12.137	-	0.87	0.086	53.84
Halsey	30	-7.466	0.156	0.933	-	0.98	0.047	31.58
	40	-7.564	0.146	1.208	-	0.99	0.017	12.77
	50	-5.628	0.085	1.087	-	0.99	0.007	8.55
Oswin	30	0.0664	0.976	-	-	0.97	0.050	39.84
	40	0.0989	0.695	-	-	0.98	0.023	22.55
	50	0.0834	0.779	-	-	0.99	0.011	15.84
Peleg	30	0.169	0.515	3.064	18.34	0.99	0.008	5.91
	40	0.146	0.482	0.968	10.02	0.99	0.007	5.60
	50	0.105	0.521	0.748	7.173	0.99	0.009	10.05
Adam and Shove	30	0.046	-0.306	0.415	0.415	0.89	0.127	44.60
	40	0.031	-0.146	0.296	0.296	0.94	0.069	30.31
	50	0.024	-0.202	0.326	0.326	0.96	0.052	30.91

3.3 Isostatic heats of sorption

The net isosteric heats of sorption of *A. graveolens* leaves obtained for different moisture contents were determined by using Equation 6 and the best-fit Peleg model. Figure 6 represents the variation of isosteric heat

of sorption both for adsorption and desorption processes with equilibrium moisture contents as is shown in the figure, the isosteric heats of desorption and adsorption were higher than the latent heat of vaporization of pure water. This observation indicates that the energy of

binding between the water molecules and sorption sites was higher than the energy which holds the water

molecules together in the liquid phase, similar findings have been reported by [Choudhury et al., \(2011\)](#).

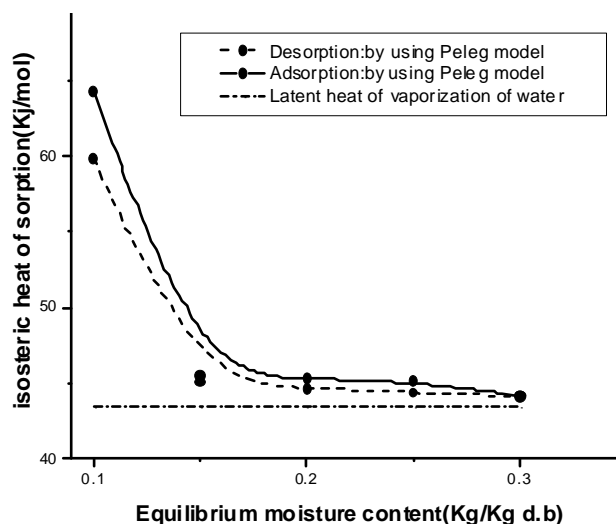


Figure 6 Values of desorption and adsorption isosteric heats of *A. graveolens* leaves versus equilibrium moisture content.

At low moisture contents, the isosteric heat of sorption is high and then decreased at high moisture contents. The high heat of sorption at lower moisture contents might be due to the fact that the water is tightly bound to the material, corresponding with high interaction energy. At increasing moisture content, the most active sites become occupied and sorption occurs on the less active sites giving lower heats of sorption (Tsami et al., 1990; Iglesias and Chirife, 1976).

The heat of adsorption has been observed to present a higher magnitude than the corresponding heat of desorption. According to Rao et al. (2014), chain rupture or irreversible swelling may be the cause for a heat of desorption being smaller than a heat of adsorption.

4 Conclusions

The moisture sorption isotherms of *A. graveolens* leaves at 30 °C, 40 °C and 50 °C have been established. The desorption and adsorption isotherms are sigmoidal in shape and showed a marked temperature effect. Both Peleg and Halsey equations were the best models for prediction of desorption and adsorption phenomena among eight commonly used models investigated. The desorption and adsorption isotherms show the occurrence

of moisture sorption hysteresis. The net isosteric heat of desorption and adsorption were calculated using the Clausius-Clapeyron equation. The net isosteric heats of sorption of *A. graveolens* leaves were found to increase with the decrease in moisture content and it approached the value of heat of vaporization of free water at higher moisture content.

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