

STUDY OF THE MOISTURE SORPTION ISOTHERMS AND ISOSTERIC HEAT OF SORPTION OF THE MEDICINAL PLANT *Launaea Nudicaulis* FROM ALGERIAN SAHARA

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*The aim of this work is to determine the sorption isotherms of *Launaea nudicaulis*, a medicinal plant growing in south-west of Algeria. The equilibrium moisture content was measured, using the static gravimetric method, with the water activity from 6.3 to 89.8% at 30 and 40°C. It was shown that at higher water activities the moisture content increases sharply with the temperature, resulting in crossing of the isotherm curves. Four models were applied for analyzing experimental data (namely, the Peleg, GAB, Henderson–Thompson, and the BET modified ones). The desorption and adsorption data showed the best correlation with the Peleg model. The isosteric sorption heat of water was determined from the equilibrium data at different temperatures. This heat was shown to decrease as the moisture content increases and to be a polynomial function of the moisture content.*

Keywords: *Launaea nudicaulis*, equilibrium moisture content, modeling of sorption isotherm, isosteric heat of sorption.

Introduction. Water profoundly influences the product properties, such as quality and safety. Thus, it is important to know the water amount (moisture content) in the product that can be maintained at a given energy state (the water activity). The moisture sorption isotherms describe the relationship between the water activity and moisture content at constant temperature. The nature of this relationship depends on the interaction between water and other ingredients.

The amount of water vapor that can be absorbed by a product depends on its chemical composition, physical-chemical state, and physical structure. Consequently, the isotherm shape is unique for each product type due to the differences in the capillary, surface, and colligative effects. Products with low water activity are often recognized as dry, those in the activity range from 0.60 to 0.90 are intermediate-moisture products, and those with the water activities higher than 0.90 are high-water activity products [1]

Sorption Isotherms. The desorption isotherms are appropriate to the equilibrium process which enables wet samples to be equilibrated with the outer air due to moisture loss, and the adsorption isotherms correspond to the equilibrium process that begins with dry samples. When the hygroscopic equilibrium is reached, water exchange between air and product achieves equilibrium, and the water activity becomes equal to its equilibrium value. Depending on whether the adsorption or desorption process takes place, the water amount at a given water activity may be different. Thus, hysteresis is observed (Fig. 1) [2] that is characterized by the difference between the values of the moisture contents for these processes.

To control the drying process and moisture storage conditions, it is necessary to know not only the thin-layer drying equations [3], but also the relationship between the equilibrium moisture content (X_{eq}) in a herb and the relative equilibrium humidity of the drying air at a given temperature [4, 5]. This relationship is commonly described by the moisture sorption isotherm equations. Such an isotherm for food graphically relates its equilibrium moisture content in desorption or adsorption to the water activity at a definite temperature. These isotherms are extremely important quantitative measures in food preservation, storage, packaging, and drying [6–9].

Materials and Methods. *Plant description.* The *Launaea* (*L.*) *nudicaulis* is a glabrous, perennial plant 15–60-cm long with yellow juice, tufted stems, and few distant small leaves. This plant grows in Pakistan, India, and west of the Atlantic. It is often used in folk-medicine in cases of gastric burns, pain of stomach, constipation, haemorrhoids, fever in children, itch, eczema, as well as for food [10, 11].

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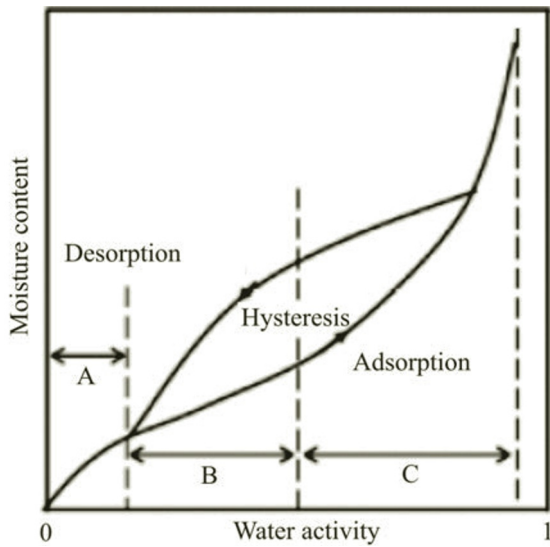


Fig. 1. Sorption isotherm for a typical food product.

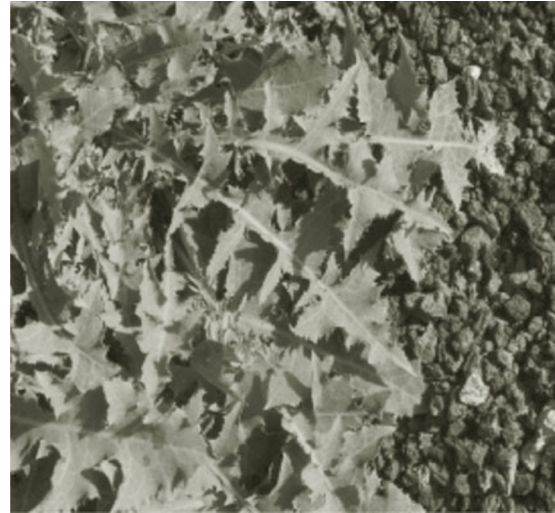


Fig. 2. *Launea nudicaulis*.



Fig. 3. Experimental apparatus.

Due to the widespread use of *L. nudicaulis* (Fig. 2) in folk-medicine and its various biological properties, including insecticidal, cytotoxic, antimicrobial, hypoglycaemic, and anti-inflammatory [12–14], this plant has been extensively investigated and reported to be a rich source of various classes of compounds, such as flavonoids, terpenoids, steroids, and their glycosides [13–15].

Equilibrium moisture content determination. The desorption equilibrium moisture content of *Launea nudicaulis* was determined at 30 and 40°C over the water activity range 0.063–0.89, using the static gravimetric method. This method involves the use of saturated salt solutions to maintain a constant relative humidity in enclosed, still moist air at a certain temperature for obtaining the complete sorption isotherms. Mass transfer between the product and ambient atmosphere is assured by natural diffusion of water vapor, and the water activity of the product is equal to the relative humidity of the atmosphere under equilibrium conditions. Seven saturated salt solutions (KOH, KF, K₂CO₃, NaNO₃, NaCl, KCl, and BaCl₂) were used (Fig. 3).

The equilibrium moisture content is determined as

$$X_{\text{eq}} = \frac{M_h - M_s}{M_s}, \quad (1)$$

where M_h and M_s are the masses with and without moisture.

TABLE 1. Mathematical Formulas for Different Models

Model	Formula
Peleg	$X_{eq} = Aa_w^C + Ba_w^D$
GAB	$X_{eq} = \frac{ABCa_w}{(1 - Ca_w)(1 - Ca_w + C Ba_w^D)}$
Henderson–Thompson	$X_{eq} = \left[-\frac{\ln(1 - a_w)}{A(T + C)} \right]^{1/b}$
BET modified	$X_{eq} = \frac{(A + BT)Ca_w}{(1 - a_w)(1 - a_w + Ca_w)}$

TABLE 2. Water Activity of Saturated Salt Solutions and Equilibrium Moisture Content

T, °C	30			40			
	Salt	a _w	X _{eq}		a _w	X _{eq}	
Desorption			Adsorption	Desorption		Adsorption	
	KOH	7.38	0.0589	0.052	6.3	0.0422	0.040
	KF	20.8	0.1013	0.0852	22.7	0.0951	0.0789
	K ₂ CO ₃	43.11	0.1485	0.0987	42.3	0.1266	0.1054
	NaNO ₃	72.75	0.2873	0.246	71	0.2196	0.1986
	NaCl	75.1	0.3649	0.314	74.7	0.2980	0.234
	KCl	83.62	0.5374	0.4612	82.3	0.4433	0.399
	BaCl ₂	89.8	0.7301	0.6284	89.1	0.8729	0.435

Modeling of the sorption isotherms. Several models for the dependence of the value of X_{eq} on the air moisture content have been suggested in the literature [16]. Some models take into account the temperature effect; among them are the BET (Brunauer, Emmett, and Teller) and Henderson ones. The American Society of Agricultural Engineers has adopted these models for describing the sorption isotherms [17].

The sorption isotherm describes the interaction between water and a food product. Equations for fitting these data are of special interest in many aspects of food preservation by dehydration. Numerous mathematical equations have been reported in the literature for describing the water sorption isotherms of food materials. They are very different in character (empirical, semiempirical or theoretical) and area of application.

A large number of the developed sorption equations results in the difficulties associated with choosing a unique mathematical model for describing the sorption data in the whole range of the water activity a_w for different food products. This is due to the following reasons:

1. Depression of the water activity in foods occurs due to a combination of factors, each being predominant in a given range of the water activity. In general, insoluble macromolecules govern sorption behavior at low a_w, and soluble components manifest their effect principally through the colligative properties over the range of high a_w.
2. The moisture sorption isotherms of foods are determined by the integrated hygroscopic properties of numerous constituents, whose sorption properties may change as a consequence of the physical and/or chemical interactions induced by heating or other pretreatments.

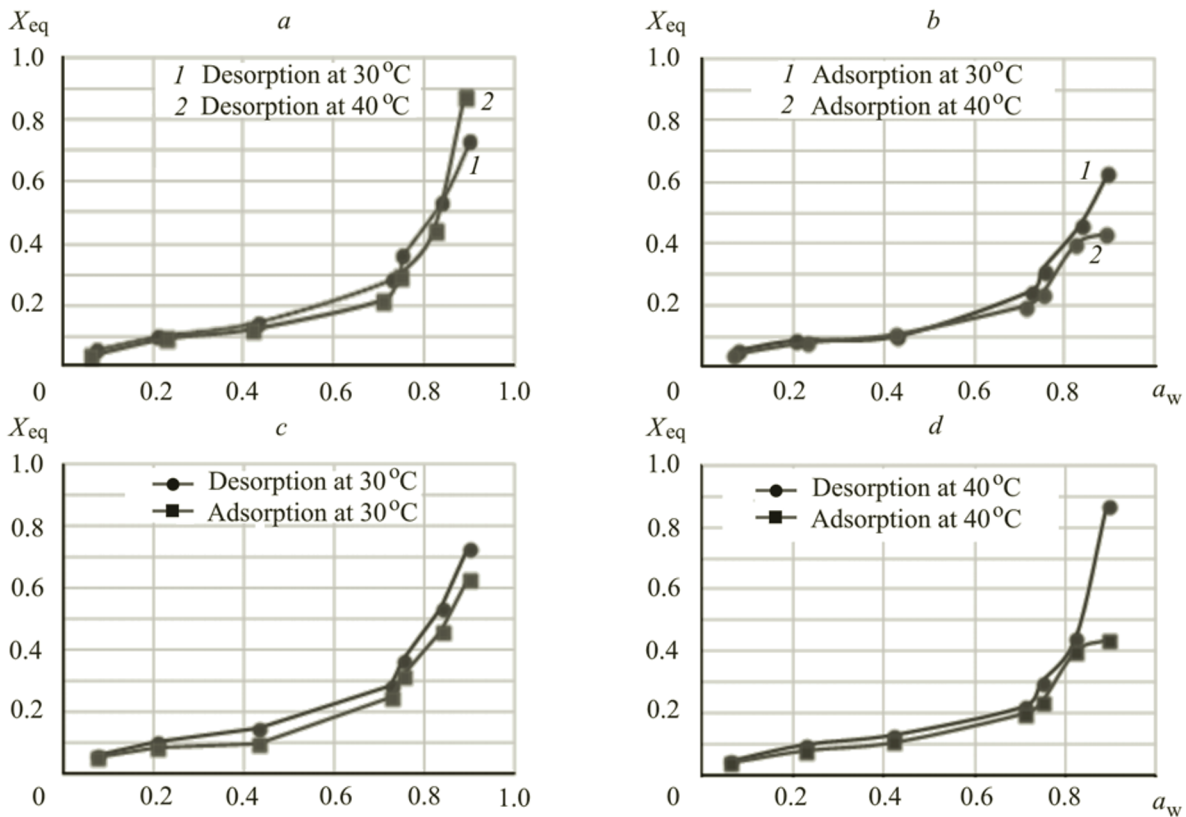


Fig. 4. Desorption and adsorption isotherms (experiment).

As food sorbs water, it usually undergoes changes in the constitution, dimensions, and other properties; water sorption also leads to the phase transformations of sugars contained in the food. When this sugar contained, say, in a fruit, is freeze dried, it can be converted into an amorphous form. When adsorption takes place, sugar reconverts into a crystalline form.

The choice of an equation to fit the sorption data should come from different factors: i) agreement between the sorption data and model results; ii) range of application; iii) theoretical justification of the parameters; iv) simplicity; v) desired objectives.

All biological materials are characterized by a wide range of the relative humidity and temperature. Therefore, there is a need to select the most appropriate moisture sorption equation for a specific product and given ranges of the humidity and temperatures [18].

The *Peleg model* involves a purely empirical equation without a theoretical background. It is of the same or even better suitability than the *GAB* (Guggenheim, Anderson, and de Boer) *model* [18] which has the advantage of perfectly describing the isotherms of desorption and adsorption for $a_w < 0.90$ [19].

The *Henderson model* was applied successfully to various products [20]. To take into account the influence of temperature on the hygroscopic equilibrium and to perform interpolations, this model is useful due to the advantage of describing the whole sorption isotherm range for different temperatures. The *BET model* allows one to examine the phenomenon of fixation and liberation of water molecules.

The formulas for all the models are presented in Table 1. Hereafter, the water activity a_w is given in kg/kg db, and A , B , C , and D are constants specific to individual equations [21].

Results and Discussion. The hygroscopic equilibrium of *Launeae nudicaulis* is determined after 13 days for desorption and 9 days for adsorption. The values of the water activity and equilibrium water content for desorption and adsorption are given in Table 2. The sorption isotherms obtained at 30 and 40°C are of type II and characterized by a sigmoid form, being widespread [22]. The mentioned isotherms are given in Fig. 4. The first parts of the curves correspond to monolayer adsorption, then it becomes multimolecular. It is seen that at higher water activities, the moisture content increases sharply with the temperature, resulting in intersection of the isotherm curves. This behavior may be explained by considering excitation states of water molecules. At increased temperatures, molecules are in the state of increased excitation,

TABLE 3. Parameters of Different Models for Desorption

Coefficient	$T, ^\circ\text{C}$	Peleg	GAB	Henderson–Thompson	BET modified
A	30	1.0886	0.0993	-0.0988	-1.5096E+002
	40	0.2392	0.0644	3.3586E+003	-1.5594E+002
B	30	6.4949	14.4897	0.7758	0.4848
	40	0.6524	41.1147	18.479	0.4985
C	30	0.2031	0.9656	-3.3304E+002	59.152
	40	2.6776	1.0396	5.7616E+005	1.0844
D	30	0.4547	-	-	-
	40	12.283	-	-	-
r	30	0.9984	0.9964	0.9842	0.9893
	40	0.9989	0.9986	0.3174	0.9884
$P, \%$	30	3.8692	5.1439	27.9680	9.3447
	40	5.1454	9.4461	147.34	31.3084
SEE	30	0.0824	0.0021	0.0516	0.0243
	40	0.0011	0.0052	0.0297	0.0454

TABLE 4. Parameters of Different Models for Adsorption

Coefficient	$T, ^\circ\text{C}$	Peleg	GAB	Henderson–Thompson	BET modified
A	30	0.1172	0.0835	-0.1030	-1.5096E+002
	40	0.59703	8.2562E-002	-1.5345	-1.5597E+002
B	30	0.2754	11.461	0.7276	0.4984
	40	4.8766	9.2799	4.63816	0.4984
C	30	0.9493	0.9699	-3.3432E+002	4.2910E+001
	40	0.1216	0.923848	-8.0265E+002	1.0195E+002
D	30	5.6427	-	-	-
	40	0.3547	-	-	-
r	30	0.9985	0.9947	0.9866	0.9896
	40	0.984	0.978	0.705	0.952
$P, \%$	30	5.3454	11.635	26.4498	9.3751
	40	9.7274	11.6566	80.36	14.76
SEE	30	5.774E-005	0.02285	0.0445	0.01255
	40	0.0019	0.0011	0.046	0.03192

thus increasing their distance apart and decreasing the attractive forces between them. This leads to a decrease in the degree of water sorption with increasing temperature at a given water activity [23]. Such behavior is due to the thermodynamic considerations. Similar results have been reported earlier [24–27].

The hysteresis effect was observed for *Launeae nudicaulis* (Fig. 4c and d). This phenomenon is not fully understood, although there is good reason to consider that some properties of the thermodynamically irreversible processes must occur during desorption or adsorption or both of them. Many explanations for this phenomenon have been reported [23, 28]. One theory used to explain hysteresis suggests that in wet conditions the polar sites onto which water is adsorbed are not entirely saturated. After water has dried, the holding sites are drawn close enough, together with shrinkage, to saturate each other. This results in a reduction of the water binding capacity during adsorption [29].

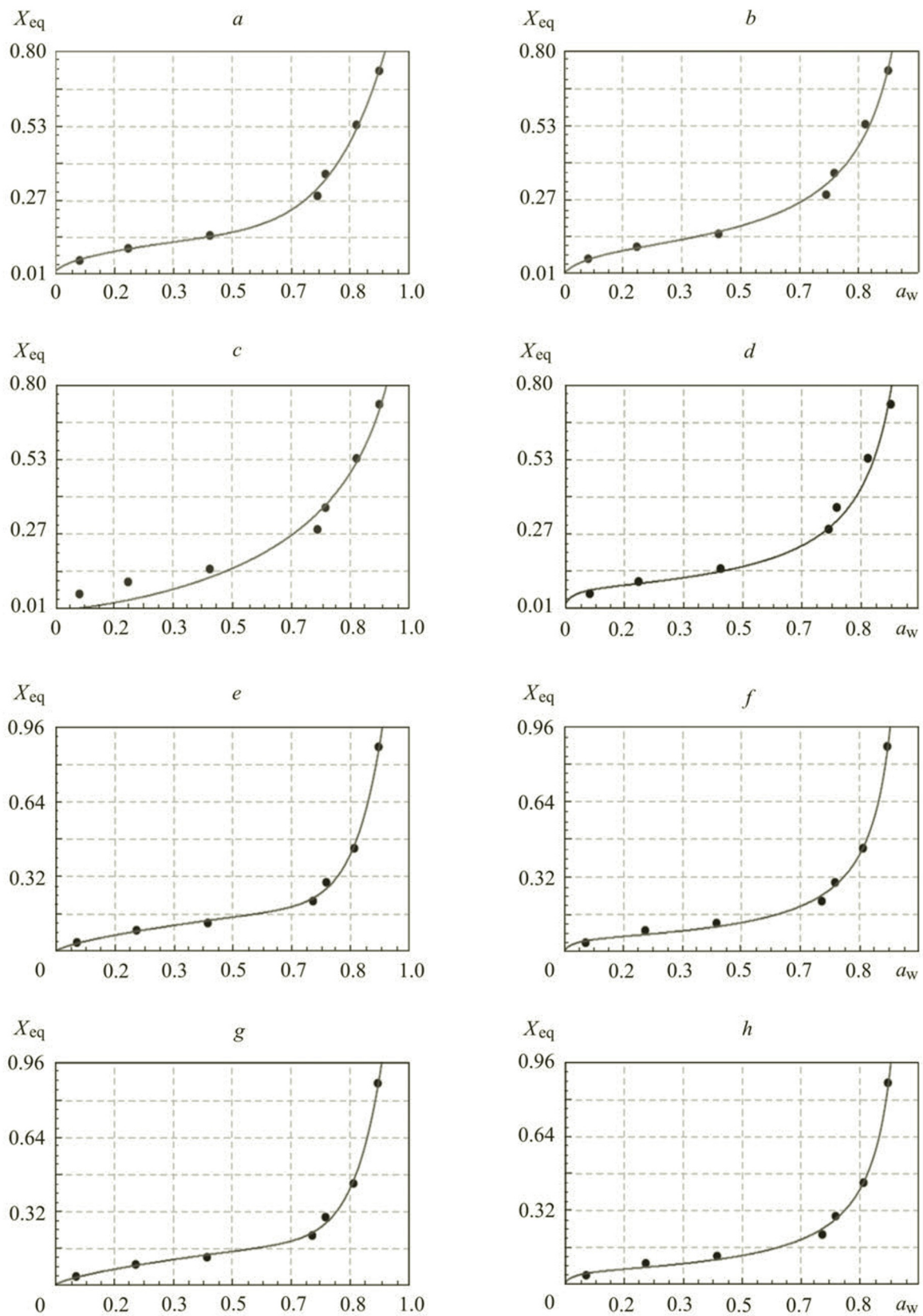


Fig. 5. Desorption isotherms at 30°C (a–d) and 40°C (e–h) for different models: Peleg (a and e), GAB (b and f), Henderson–Thompson (c and g), and BET (d and h).

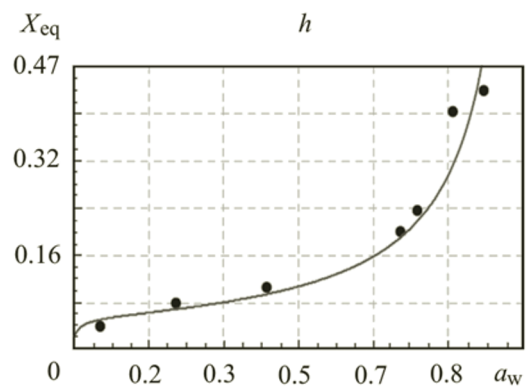
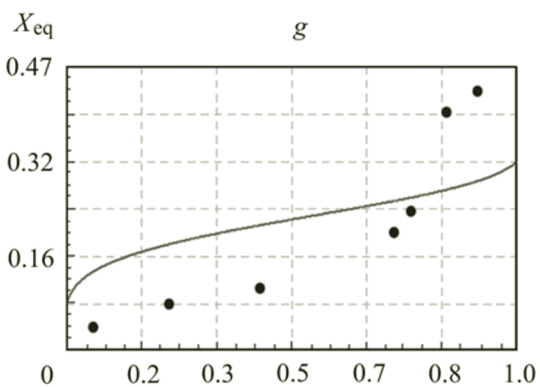
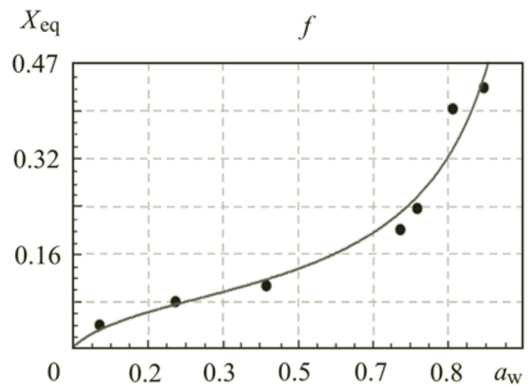
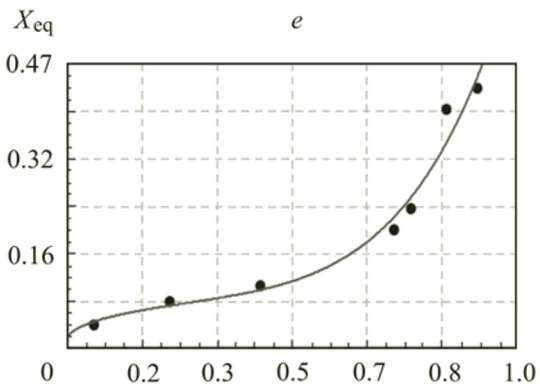
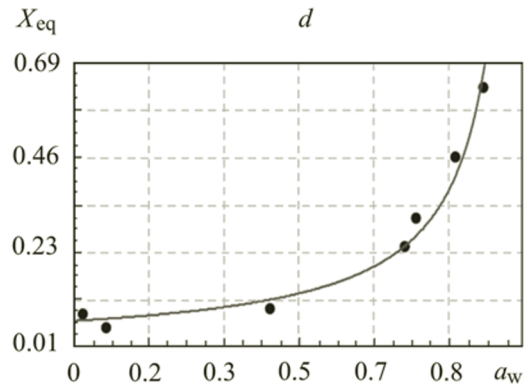
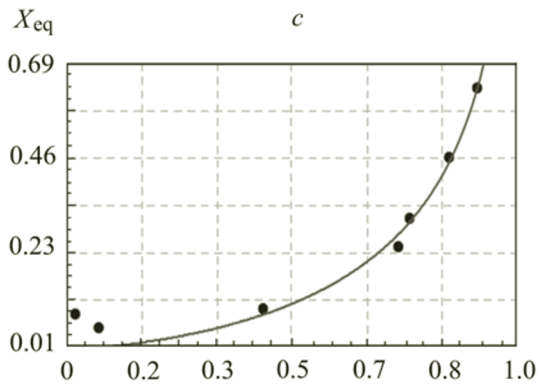
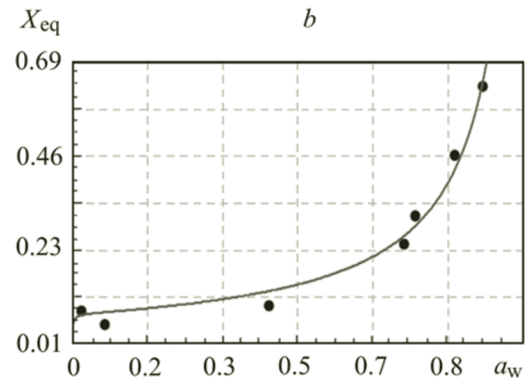
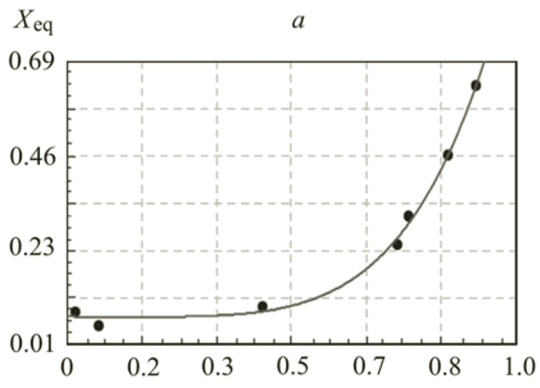


Fig. 6. Adsorption isotherms at 30°C (a–d) and 40°C (e–h). The remaining capture is same as in Fig. 5.

Fitting of the sorption models to experimental data. The experimental data on the desorption and adsorption curves of *Launeae nudicaulis* were fitted to four sorption models (Peleg, GAB, Henderson–Thompson, and BET modified ones). The results of the nonlinear regression analysis of the experimental data are presented in Tables 3 and 4. In the tables, P is the average relative error, SEE is the standard error estimation, and r is the correlation coefficient. The values of P and SEE were obtained from the following equations:

$$P = \frac{100}{N} \sum_{i=1}^N \frac{X_{eqi}^{exp} - X_{eqi}^{pre}}{X_{eqi}^{exp}}, \quad (2)$$

$$SEE = \sqrt{\sum_{i=1}^N \frac{(X_{eqi}^{exp} - X_{eqi}^{pre})^2}{N - n_p}}. \quad (3)$$

For all the models, the parameters A , B , C , and D were found. The Peleg model was shown to give the best agreement with the experimental data for the desorption and adsorption isotherms with the average relative error from 3.8692 to 5.1454% for desorption and from 5.3454 to 9.7274% for adsorption. This model gives the correlation coefficient $r = 0.9984$ and 0.9989 for desorption at 30 and 40°C, respectively. For adsorption r is equal to 0.9985 and 0.984 at 30 and 40°C.

The Henderson–Thompson model gives the lowest estimators for predicting. The parameters of this model can be recommended for prediction of the desorption and adsorption isotherms of *Launeae nudicaulis* in the temperature range 30–40°C at the water activity from 0.063 to 0.898. These isotherms are given in Figs. 5 and 6.

Determination of the Isostatic Heat of Sorption. The net isosteric heat of sorption is used as a thermodynamic function for analysis of the sorption isotherms. The net isosteric heat of sorption, or the differential enthalpy, representing the energy required for removing moisture from a food material (i.e., the water–solid binding strength), is of practical use in complete drying calculations and modeling [30]. This characteristic is obtained by subtraction of the heat of water vaporization from the total heat of sorption Q_{st} . The value of q_{st} can be determined from the experimental data by using the Clausius–Clapeyron equation in the form given in [30–35]:

$$\frac{\partial \ln a_w}{\partial \left(\frac{1}{T}\right)} = -\frac{Q_{st} - \lambda}{R} = -\frac{q_{st}}{R}, \quad (4)$$

where λ is the latent heat of vaporization of pure water and R is the gas constant equal to 0.4618 kJ/(kg · K). Integrating Eq. (4) on the assumption that the net isosteric heat of sorption is temperature independent gives the following equation:

$$\ln a_w = -\frac{q_{st}}{R} \frac{1}{T} + K, \quad (5)$$

where K is an integration constant. The vaporization heat of pure water can be calculated as

$$\lambda = R(6887 - 5.31T). \quad (6)$$

The isosteric heat of sorption Q_{st} was determined from the slope of the $\ln a_w$ versus $1/T$ curve at constant moisture content (see Fig. 7). The values of a_w at different moisture contents were calculated from the equilibrium data with the use of Eq. (5).

The variation of the heat of sorption of *Launeae nudicaulis* with the moisture content is given in Fig. 8. The net isosteric heat of sorption is shown to increase with decreasing moisture content. At low moisture content, the isosteric heat of sorption is high, suggesting that the binding energy for water removal is the highest. An increase in the moisture content decreases the isosteric heat of sorption due to reduced water interactions. The higher isosteric heat of sorption at lower moisture contents might be due to the greater resistance to water movement from the interior of the sample to its surface. These trends are similar in both food and pharmaceutical products [28, 36]. The isosteric heats of desorption and adsorption of water in the *Launeae nudicaulis* leaves can be expressed mathematically as power functions of the moisture content:

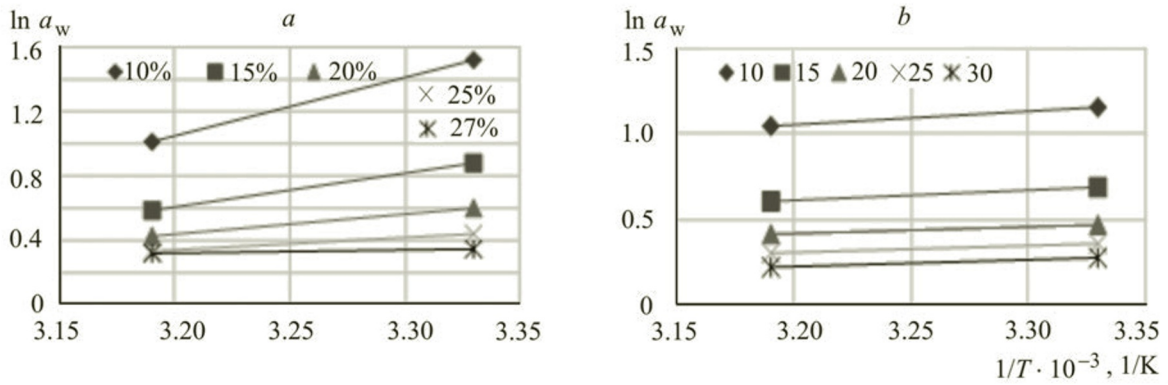


Fig. 7. Dependence of the water activity on $1/T$ for desorption (a) and adsorption (b).

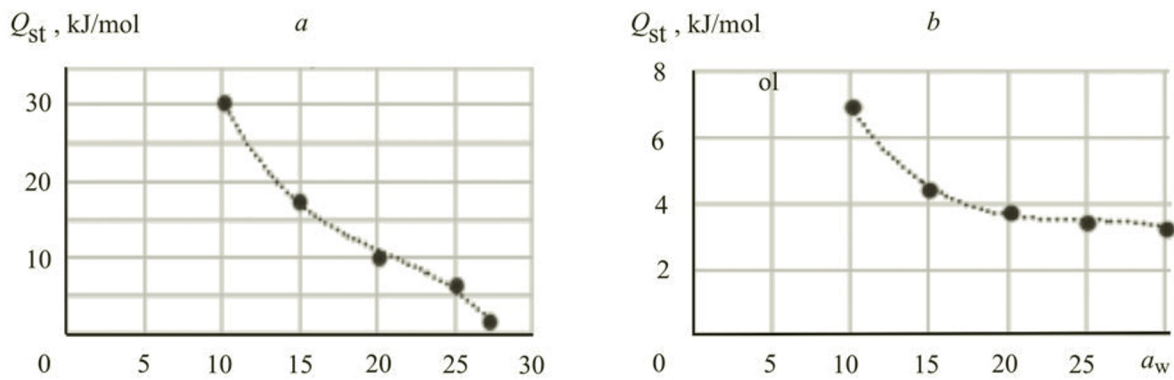


Fig. 8. Dependence of the isosteric heat on the moisture content for desorption (a) and adsorption (b).

$$Q_{st,des} = 109.21 - 12.648X_{eq} + 0.5735X_{eq}^2 - 0.0093X_{eq}^3, \quad r = 0.9969; \quad (7)$$

$$Q_{st,ads} = 19.523 - 1.9402X_{eq} + 0.0787X_{eq}^2 - 0.0011X_{eq}^3, \quad r = 0.9983. \quad (8)$$

Conclusions. The moisture sorption isotherms of *Launaea nudicaulis* at 30 and 40°C have been determined. The desorption and adsorption isotherms are sigmoidal in shape and show marked temperature effect. The Peleg model properly explains the variation in the adsorption and desorption equilibrium moisture contents, and is therefore the best model for describing sorption phenomena among four commonly used models investigated. The adsorption and desorption curves exhibit the hysteresis effects for adsorption and desorption. The isosteric heat of sorption is shown to increase with decrease in the moisture content, and the heat of desorption is higher than that of adsorption.

NOTATION

a_w , water activity; M , mass; P , average relative error; Q , total heat of sorption; q , net isosteric heat of sorption; r , correlation coefficient; T , temperature; X , moisture content; λ , latent heat of vaporization of pure water. Indices: eq, equilibrium.

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