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Equilibrium isotherms and isosteric heats of morel (*Morchella esculenta*)

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Abstract

Moisture desorption isotherms of *Morchella esculenta* mushrooms were determined at 5, 15, 25 and 35 °C and within the range of 0.11–0.92 water activity using a standardized conductivity hygrometer. Five models were considered to describe isotherms (GAB, Oswin modified, Henderson modified, Halsey modified and Ratti) being fitted to the experimental data by direct non-linear regression. The GAB and Ratti equations gave the best fit of the experimental data. Isosteric heats of water desorption were obtained from the experimental data by applying the Clausius–Clapeyron equation within the range of 0.04–0.50 moisture content (dry basis). © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Morel; Moisture desorption isotherm; GAB; Ratti; Henderson modified; Oswin modified; Halsey modified; Isosteric heat

1. Introduction

Wild mushrooms are a secondary source of wealth in some rural zones, and an essential ingredient of many traditional dishes from around the world. The high moisture content of wild mushrooms and their seasonal nature make their availability difficult. Knowing which are the best conditions for processing and storing is a way of increasing the availability of this kind of wild, high value products, which usually come from underprivileged areas. From such studies, drying emerges as a useful method of preservation. Traditionally, it has been carried out by exposure to sun. However, solar drying depends too much on the climate and the final product quality might not be satisfactory. Consequently, using convection dryers could be a successful decision.

Studies on mushroom dehydration are scarce in the literature, and mainly focus on drying techniques and quality aspects for cultivated mushrooms, such as *Agaricus bisporus* and *Pleurotus* sp. (Bano & Rajarathnam, 1988; Yapar, Helvaci, & Peker, 1990; Gothandapani, Parvathi, & Kennedy, 1997; Kanevce, Mitrevski, & Kanevce, 1998). Paeaekkoenen and Kurbela (1987) studied the sorption isotherms of Lactarius trivialis, and Khalloufi, Giasson, and Ratti (2000) studied the sorption isotherms of freeze-dried *Lentinus edodes, Flammulina velutipes* and *Morchella esculenta*.

In every food product there is an inherent relationship between water content and the relative humidity of the atmosphere in equilibrium with it, which is equivalent to water activity. For drying processes and storage, a sound knowledge of the relationship between moisture content and equilibrium relative humidity is essential (Gal, 1983, Chapter 2; Sun, 1999; Sanjuán, García-Pascual, Campos, García-Reverter, & Mulet, 2000), as equilibrium isotherms help establish the final moisture contents and compute energy requirements of drying (Kiranoudis, Maroulis, Tsami, & Marinos-Kouris, 1993; Mulet, García-Reverter, Sanjuán, & Bon, 1999). In this context, having a mathematical model to describe that relationship could be very useful. More than 200 equations have been proposed for modelling the equilibrium data, differing in their empirical or theoretical character, and in the number of parameters involved. Among the most frequently reported models in the literature for vegetables and fruits, those of GAB, Oswin modified, Henderson modified, Halsey modified and Ratti, have been selected to describe the experimental data.

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Nomenclature

a_w	water activity, dimensionless
A_h	Henderson constant (Eq. (5)), kg dry solid (kg water K)$^{-1}$
A_{ha}	Halsey constant (Eq. (6)), dimensionless
A_{os}	Oswin constant (Eq. (4)), kg water (kg dry solid)$^{-1}$
B_h	Henderson constant (Eq. (5)), dimensionless
B_{ha}	Halsey constant (Eq. (6)), K^{-1}
B_{os}	Oswin constant (Eq. (4)), kg water(kg dry solid K)$^{-1}$
C, C_0	GAB constants (Eqs. (1) and (2)), dimensionless
C_h	Henderson constant (Eq. (5)), K
C_{ha}	Halsey constant (Eq. (6)), dimensionless
C_{os}	Oswin constant (Eq. (4)), dimensionless
H_m	sorption heat of mono-molecular water layer, kJ kg^{-1}
H_n	sorption heat of multi-molecular water layer, kJ kg^{-1}
k_i	constants in the Ratti model ($1 \leqslant i \leqslant 5$)
K, K_0	GAB constants (Eqs. (1) and (3)), dimensionless
MRE	mean relative error, %
N	number of experimental observations
q_n^s	net isosteric heat of water, kJ kg^{-1}
Q^s	isosteric heat of water, kJ kg^{-1}
S_{yx}	standard deviation of the estimation, units of estimate
S_y	standard deviation of the sample, units of estimate
T	temperature, K
W	moisture content, kg water (kg dry solid)$^{-1}$
W_m	monolayer moisture content, kg water (kg dry solid)$^{-1}$
ΔH	enthalpy, kJ kg^{-1}
VAR	percentage of explained variance, %

The Guggenheim–Anderson–de Boer (GAB) equation, recommended by the European project COST-90 (Bizot, 1983, Chapter 4), can be considered a version of the BET model which was improved by introducing a third parameter, K, as a multiplier of the water activity. The GAB model has usually been used to fit the data of food products with satisfactory results. As a consequence of its theoretical origin, its parameters have a physical meaning, W_m being the monolayer moisture content (on a dry basis) and C and K, are constants related to temperature. From the BET and GAB models it is also possible to obtain values of the moisture content and the monolayer sorption heat.

The original Henderson model is one of the most commonly used equations and it describes well the behaviour of many hygroscopic materials, like foodstuffs and agricultural materials in the range of water activity from 0.1 to 0.75 (Leung, 1986, Chapter 6). This equation was modified in order to increase its range of application (Thompson, Peart, & Foster, 1968). The original Oswin equation (Leung, 1986, Chapter 6) is a mathematical series expansion for *S*-shaped curves, and does not consider the effect of temperature. The original Halsey equation (Leung, 1986, Chapter 6) was developed based on multilayer condensation. The Oswin and Halsey equations were derived from experimental data and are empirical expressions. They were modified later in order to increase their range of applicability. The Ratti model is a semitheoretical water sorption equilibrium expression for solid foods. It was developed on the basis of thermodynamic considerations and the effect of temperature is included through the vapour pressure of pure water (Ratti, Crapiste, & Rotsein, 1989). Those authors tested this model with sorption data of potatoes, almonds, apples, rice, wheat and corn meal. An interesting aspect of this model is that it permits to estimate the sorption heats as a function of water content.

Although *M. esculenta* isotherms were established in the literature for the freeze-dried product (Khalloufi et al., 2000), no detailed data are available for desorption. Desorption data are of great interest for convective drying. *M. esculenta* main components are (on dry basis): protein 32.7%, fat 2.0%, fibre 17.6%, ash 9.7% and carbohydrates 38.0% (Wahid, Sattar, & Khan, 1988).

The aim of this work is: (1) to provide experimental desorption isotherm data of *M. esculenta* mushroom and establish the best model to fit the data (GAB, Oswin modified, Henderson modified, Halsey modified and Ratti), and (2) determine the isosteric sorption heats.

2. Material and methods

2.1. Experimental procedure

Fresh raw material, *M. esculenta*, picked up the day before was purchased locally. The mushrooms were previously selected by visual inspection, being their initial moisture content 90.2 ± 0.1 (w.b.). Morels were slightly ground and partially dehydrated to different moisture contents by placing them in a closed container with silica gel with chemically pure indicator. In order to guarantee a homogeneous moisture content in the material, ground morels were sampled from the container where the partial dehydration proceeded, shook and kept closed during 48 h prior to measuring water content and water activity. The water activity of the samples was established by using a standardized conductivity hygrometer Novasina TH-2 (Axair Systems for Air Treatment, Pfäffikon, Switzerland), previously calibrated using the following salts: LiCl, $MgCl_2$, $Mg(NO_3)_2$, NaCl, $BaCl_2$ and $K_2Cr_2O_7$, according to the calibration procedure established for that apparatus by the manufacturer. The temperatures used for the water activity determination were 5, 15, 25 and 35 °C. The time necessary to reach equilibrium in every case was around 3 h, whichever the temperature or moisture content considered. All measurements were duplicated at least. The moisture contents at each water activity value were measured in triplicate according to the AOAC method number 934 · 06 (AOAC, 1996).

2.2. Mathematical description of isotherms

The list of equations used to fit experimental isotherms are in Eqs. (1)–(7). The first four models have temperature and water activity as independent variables, while the Ratti model has temperature and the equilibrium moisture content as independent variables. The GAB and Ratti equations allow to estimate sorption heats.

GAB (Samaniego-Esguerra, Boag, & Robertson, 1991; Lim, Tang, & He, 1995; Myhara, Sablani, Al-Alawi, & Taylor, 1998):

$$W = \frac{W_m C K a_w}{(1 - K a_w)[1 + (C - 1) K a_w]}, \tag{1}$$

where

$$C = C_0 \exp\left(\frac{\Delta H_C}{RT}\right), \quad \Delta H_C = H_m - H_n, \tag{2}$$

$$K = K_0 \exp\left(\frac{\Delta H_k}{RT}\right), \quad \Delta H_k = L_r - H_n. \tag{3}$$

Oswin modified (Lim et al., 1995; Sun, 1999):

$$W = (A_{os} + B_{os} T)\left[\frac{a_w}{1 - a_w}\right]^{C_{os}}. \tag{4}$$

Henderson modified (Lim et al., 1995; Sun, 1999):

$$W = \left[-\frac{1}{A_h (T + C_h)} \ln(1 - a_w)\right]^{1/B_h}. \tag{5}$$

Halsey modified (Lim et al., 1995; Sun, 1999):

$$W = \left[\frac{-\exp(C_{ha} + B_{ha} T)}{\ln(a_w)}\right]^{1/A_{ha}}. \tag{6}$$

Ratti (Khalloufi et al., 2000):

$$\ln(a_w) = -k_1 W^{k_2} + k_3 \exp(-k_4 W) \ln(P_s) W^{k_5}. \tag{7}$$

The saturation vapour pressure (P_s) can be estimated by the equation proposed by ASAE (2000).

2.3. Isosteric sorption heat

The isosteric sorption heat, Q^s, is the required energy to remove water from the mass unit of a solid matrix. Hence, it is an assessment of the binding energy between water molecules and the solid matter of a food product. The net isosteric sorption heat, q_n^s, represents the difference between the isosteric heat, Q^s, and pure water vaporization energy, L_r.

$$\left[\frac{d \ln(a_w)}{dT}\right]_w = \frac{q_n^s}{RT^2}. \tag{8}$$

Through the Clausius–Clapeyron equation (Eq. 8), the net isosteric sorption heat can be established by plotting $\ln(a_w)$ at a specified moisture content versus $1/T$ and measuring the slope (Sánchez, Sanjuán, Simal, & Rosselló, 1997; Mulet et al., 1999). Eq. (8) is based on the assumption that q_n^s is independent of temperature; although this is not always true, it has often been accepted (Iglesias, Chirife, & Ferro Fontán, 1989). The application of this method requires the measurement of sorption isotherms, at least, at three temperatures. On the other hand, the integration of Eq. (8) allows calculation of q_n^s by taking measurements at only two temperatures (Iglesias & Chirife, 1976a,b):

$$q_n^s = R\left[\frac{T_1 T_2}{T_2 - T_1} \ln \frac{a_{w2}}{a_{w1}}\right], \tag{9}$$

where a_{wi} is the measurement of water activity at temperature T_i and R is the universal gas constant (8.314 J $K^{-1}mol^{-1}$). It was found (Riedel, 1977) that the next equation could adequately describe the influence of temperature on water activity:

$$\left[\ln \frac{a_{w2}}{a_{w1}}\right]_W = A e^{-b_w}\left[\frac{1}{T_1} - \frac{1}{T_2}\right], \tag{10}$$

where A and b are two characteristic constants.

By combining and rearranging Eqs. (9) and (10) an expression for estimating isosteric sorption heats is obtained (Mulet et al., 1999):

$$Q^s = q_n^s + L_r = C e^{-b_w} + L_r \tag{11}$$

being C the product AR. Tsami, Maroulis, Marinos-Kouris, and Saravacos (1990b) proposed an empirical function similar to Eq. (11) to compute Q^s. From the Ratti model, given by Eq. (7), the sorption heat can also be obtained as:

$$\frac{Q^s}{L_r} = k_3 \exp(-k_4 W) W^{k_5} + 1. \tag{12}$$

2.4. Parameter estimation

The parameters of Eqs. (1)–(7) were estimated by non-linear regression analysis using the generalized reduced gradient method available in Solver (Microsoft

Excel 2000) for minimization of the sum of the squared residuals (Berthouex & Brown, 1994). All experimental data were used to fit each model by direct non-linear regression, since this procedure presents advantages over indirect non-linear regression (Kiranoudis et al., 1993). From the literature were obtained the parameter values for different products used to start the computational procedure since numerical optimisation methods could only locate local optima (Tsami, Marinos-Kouris, & Maroulis, 1990a; Lim et al., 1995; Mulet et al., 1999; Khalloufi et al., 2000).

In order to assess how accurate each model was in representing experimental data, two criteria were used: the percentage of explained variance (VAR) (Sanjuán, Simal, Bon, & Mulet, 1999), and the mean relative error (MRE) (Lim et al., 1995; García, Kobylañski, & Pilosof, 2000):

$$\mathrm{VAR} = \left[1 - \frac{S_{yx}^2}{S_y^2}\right] 100, \tag{13}$$

$$\mathrm{MRE} = \frac{100}{N} \sum_{i=1}^{N} \left| \frac{(W_{i\,\mathrm{exp}} - W_{i\,\mathrm{calc}})}{W_{i\,\mathrm{exp}}} \right|. \tag{14}$$

The VAR represents the relative variance explained by the model with respect to the total variance, and it varies from 0 to 100. The MRE indicates the relative error of the predictions and it is a criterion used to assess the fitting of a model to experimental data. Values below 10% are indicative of a reasonably good fit for most practical purposes.

3. Results and discussion

3.1. Desorption isotherms

Desorption isotherms for *M. esculenta* were determined at 5, 15, 25 and 35°C (Table 1). As expected, there was a slight increase in water activity with temperature; however, this influence was not noteworthy as it occurs with many food materials. Data shown in Fig. 1 looks like a type III pattern isotherm, characteristic of high sugar content foods, holding relatively small amounts of water at low water activities and large amounts at high relative humidities (Samaniego-Esguerra et al., 1991). According to BET theory, a type III isotherm appears when the binding energy of the first layer is lower than the binding energy between water molecules (Leung, 1986, Chapter 6). Nevertheless, as the lower experimental water activity is 0.11, it is difficult to conclude that the isotherms follow a type III pattern.

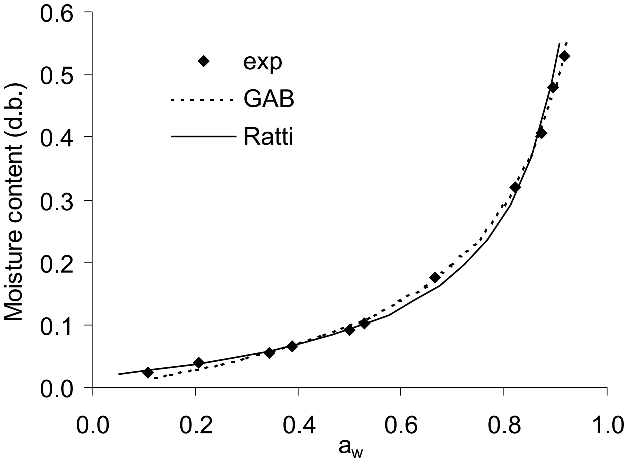

Fig. 1. Experimental data and desorption isotherms at 5 °C.

Table 2 shows the identified parameters of the five models analysed. According to the obtained figures for both assessing criteria, VAR and MRE, all models can be grouped into three sets: the GAB, Ratti and Oswin modified models could be considered which best represent experimental data, since all of them reached high values of VAR and MRE lower than 10%; the Halsey modified model yielded the most inappropriate results according to the two criteria chosen; the Henderson modified model would be in the middle, since it had a good figure for VAR but a worse value for MRE (13.8%).

Table 1
Experimental data of water activity

W_e (kg water/kg d s)	Water activity			
	5 °C	15 °C	25 °C	35 °C
0.025 ± 0.001	0.109 ± 0.001	0.122 ± 0.003	0.127 ± 0.001	0.144 ± 0.001
0.040 ± 0.023	0.207 ± 0.011	0.238 ± 0.013	0.258 ± 0.014	0.273 ± 0.012
0.049 ± 0.002	0.289 ± 0.001	0.308 ± 0.001	0.305 ± 0.002	0.314 ± 0.005
0.055 ± 0.001	0.344 ± 0.010	0.329 ± 0.019	0.355 ± 0.009	0.379 ± 0.002
0.065 ± 0.001	0.388 ± 0.007	0.409 ± 0.005	0.448 ± 0.001	0.470 ± 0.001
0.093 ± 0.004	0.499 ± 0.003	0.514 ± 0.001	0.526 ± 0.001	0.541 ± 0.002
0.103 ± 0.002	0.529 ± 0.013	0.544 ± 0.010	0.556 ± 0.010	0.570 ± 0.011
0.175 ± 0.004	0.667 ± 0.013	0.672 ± 0.010	0.673 ± 0.008	0.681 ± 0.007
0.320 ± 0.002	0.823 ± 0.002	0.822 ± 0.004	0.822 ± 0.003	0.827 ± 0.002
0.407 ± 0.002	0.872 ± 0.001	0.878 ± 0.002	0.879 ± 0.002	0.882 ± 0.002
0.479 ± 0.005	0.896 ± 0.002	0.904 ± 0.001	0.905 ± 0.001	0.908 ± 0.002
0.530 ± 0.001	0.919 ± 0.003	0.906 ± 0.001	0.905 ± 0.001	0.923 ± 0.001

Table 2
Estimated parameters for the different isotherm models

Model	Parameters					VAR (%)	MRE (%)
GAB	W_m (d.b.) 0.0978	C_0 0.0876	ΔH_c(kJ/kg) 357.95	K_0 0.914	ΔH_k (kJ/kg) 0.301	99.6	9.0
Oswin modified		A_{os} 0.155	B_{os} -1.95×10^{-4}	C_{os} 0.717		99.2	9.0
Henderson modified		A_h 4.41×10^{-3}	B_h 0.728	C_h 606.70		99.5	13.8
Halsey modified		A_{ha} 1.198	B_{ha} -2.53×10^{-3}	C_{ha} -2.402		98.6	18.8
Ratti	k_1 4.62×10^{-2}	k_2 -1.237	k_3 5.90×10^{-3}	k_4 12.675	k_5 -1.144	99.7	3.1

Thus, the Ratti, GAB and Oswin modified models, in that order, appear to be the most suitable to describe the relationship between water content, water activity and temperature throughout the whole considered intervals ($5\ °C \leqslant T \leqslant 35\ °C, 0.11 \leqslant a_w \leqslant 0.92$).

However, if other criteria were used in order to choose one model, such as the least number of parameters and the physical meaning of them, the final classification will be different. For instance, as it is easier to fit experimental data to an equation with more parameters, the Oswin model could appear superior to the two others, since it has only three parameters instead five as in the case of GAB and Ratti. On the other hand, a model whose parameters have a physical meaning is highly interesting because provides a link with physical phenomena and allows comparisons with other materials. Thus, the GAB model emerges as the best model, followed by the Ratti model, which is a semi theoretical expression. The value of monolayer moisture for morels from the GAB model (0.098 kg water, d.b.) was similar to that obtained for *L. edodes* (0.12 kg water, d.b.) and slightly higher than the figure obtained for adsorption on freeze-dried morel (0.043 kg water, d.b.) (Khalloufi et al., 2000). As an example, in Fig. 1 are shown estimations of data using GAB and Ratti models at 5 °C.

3.2. Isosteric sorption heat

There are basically two methods of determining water sorption heats: the calorimetric method and the direct use of moisture sorption isotherms (Tsami, 1991). The first needs careful handling of the samples and an accurate control of experimental conditions (Mulet et al., 1999). The second one can be subjected to important experimental errors (Chirife & Iglesias, 1992). Choosing a method depends mostly on available information and equipment. When equilibrium isotherms are available, the isosteric sorption heats are easily derived from the Clausius–Clapeyron equation Eq. (8), providing a rapid computational procedure. As can be observed in Table 3, the isosteric sorption heats for *M. esculenta* at different moisture contents obtained from isotherms through the differential method Eq. (8) and by the integral method Eq. (9) are very close.

Table 3
Isosteric heats of sorption obtained from isotherms Eqs. (11) and (12)

Moisture (kg water/kg d.s.)	Isosteric heat (kJ/kg)	
	Eq. (11)	Eq. (12)
0.530	2468	2470
0.479	2466	2467
0.407	2466	2466
0.320	2477	2480
0.175	2504	2503
0.103	2548	2549
0.093	2558	2560
0.065	2714	2704
0.055	2742	2742
0.040	2815	2818

The values of isosteric heat for silica gel dried morel were similar to those obtained for the freeze-dried morel (Khalloufi et al., 2000). Khalloufi et al. (2000) equation was established from four data points; the results are shown in Fig. 2 along with data obtained from experimental isotherms. Eq. (11) was used to describe the relationship between the isosteric heat (obtained by

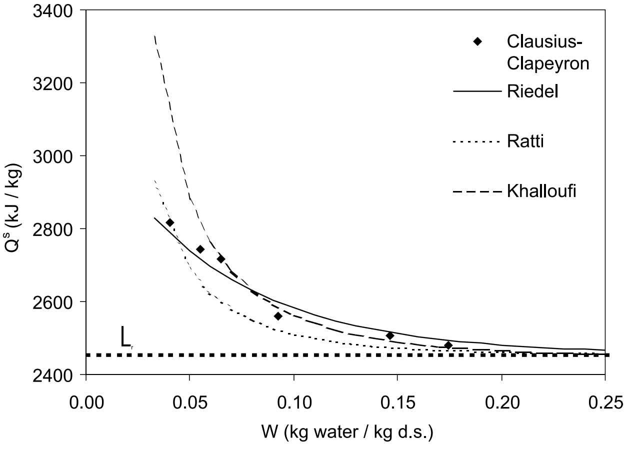

Fig. 2. Heat of sorption vs. moisture content (d.b.).

differential method) and the experimental moisture content. The parameter estimation yielded values for C of 766.79 kJ kg^{-1} and b of 18.47 kg dry solid/kg water, the VAR being 92.6% and MRE 0.82%, respectively. Fig. 2 shows the isosteric heats from isotherms as well as the estimations using the Riedel model Eq. (11) and the Ratti equation Eq. (12). It can be observed that both models could describe the experimental isosteric heat data well. However, the main difference between them lies in the fact that the Riedel equation derives from the Clausius–Clapeyron equation, while the Ratti model estimates the isosteric heats directly from the former experimental data (water activity versus equilibrium moisture content). As the isosteric heats derived from isotherms through the Clausius–Clapeyron equation can be subjected to important experimental errors (Chirife & Iglesias, 1992), it will be interesting to test these models by using sorption heat data obtained by calorimetric methods.

Finally, from the GAB model the monolayer moisture content and the energy associated to that water content, could be obtained by applying Eqs. (2) and (3). The estimated figures were 0.0978 kg water (kg dry solid)$^{-1}$ and 2812.5 kJ kg^{-1}, respectively. These figures differ from the ones derived from the Clausius–Clapeyron equation probably due to the high influence of errors associated with experimental data (Chirife & Iglesias, 1992).

4. Conclusions

The equilibrium desorption isotherm for *M. esculenta* mushrooms has been obtained at 5, 15, 25 and 35 °C. Temperature only slightly influences water activity. From the five models used, the Ratti, GAB and Oswin models provided the best fit to experimental data according to the two criteria chosen previously. The Henderson modified model could be a reasonable alternative to the precedent equations, though it reached an MRE value slightly higher than those (13.8%), while the Halsey modified equation yielded the worst fit for describing desorption isotherms of morels. Desorption heats were calculated through the direct use of moisture sorption isotherms by applying the Clausius–Clapeyron equation. The figures were similar when applying the differential or integral method. An equation developed from the Riedel and Clausius–Clapeyron equations was fitted to those data; they were also estimated by applying the expression proposed by Ratti et al. (1989). Both procedures showed a close agreement.

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