

Sorption isotherms and moisture sorption hysteresis of intermediate moisture content banana

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Abstract

Sorption isotherms of dried or intermediate moisture content (IMC) products are most important to model moisture uptake during storage and distribution. Working isotherms of IMC bananas were determined at five different temperatures (10, 15, 20, 30 and 40 °C). Desorption and adsorption isotherms were determined at 10 and 40 °C. All the sorption curves were found to be Type II, with non-zero moisture content (approximately 3–9% db for different temperatures) when the water activity was zero. Different models were fitted to the data and it was found that the best results were obtained with a modified Freundlich equation (valid from 0.06 to 0.76 water activity). The dependence of the models constants on temperature was investigated and the secondary models were built accounting for the effect of a_w and temperature on the samples moisture content ($R^2 = 99\%$). Hysteresis was observed at 10 °C and 40 °C, but the effect was greater at 10 °C. The net isosteric heat of sorption (working isotherms) varied from 0.85 kJ g⁻¹ mol⁻¹ to 7.67 kJ g⁻¹ mol⁻¹, decreasing with increasing moisture content. This effect was well described by an exponential function ($R^2 > 99\%$). These results provide reliable experimental data on sorption isotherms which is important for designing an optimized packaging system.

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1. Introduction

As most microorganisms would not grow in foods with a_w below 0.6, a_w of food products would need to keep less than 0.6 to extend product shelf life. Therefore, it is essential for dried or intermediate moisture content (IMC) products to know the sorption characteristics of these products in this range of water activity (0 to 0.6), and sorption isotherms can then be used to predict the shelf life of packaged moisture-sensitive products by modelling moisture uptake during storage and distribution.

Sorption isotherms are usually classified according to their shape in five different types: I, II, III, IV and V (Brunauer et al., 1940; Basu et al., 2006). Type I curves are convex upward throughout whereas type II curves are concave upwards throughout. Types II, IV and V isotherms show

one or more inflection points. Various mathematical models have been proposed in literature to describe sorption isotherms. Some were developed with a theoretical basis to describe adsorption mechanisms (e.g. GAB and BET) (Brunauer et al., 1938; Van den Berg and Bruin, 1981), whereas others are just empirical or a simplification of more elaborate models. In some ranges of water activity, sorption isotherms can be approximated to linear equations. Dried food products usually show isotherms of Type II or III. It should be noted that in some cases these equations predict non-zero moisture content for zero water activity, e.g. Chung and Pfof equation (Chung and Pfof, 1967), Smith equation (Smith, 1947), Iglesias and Chirife equations (1) and (2) (Iglesias and Chirife, 1978, 1981), modified Chung–Pfof equation (Pfof et al., 1976), Halsey equation (Halsey, 1948) and modified Halsey equation (Iglesias and Chirife, 1976). Some of equations take into account the effect of temperature, e.g. modified Chung–Pfof equation, modified Henderson equation (Thompson

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Nomenclature

adsp	adsorption isotherm	R^2	coefficient of determination
a_w	water activity	ss	samples used in the adsorption isotherms were placed above the saturated solution of potassium hydroxide to get water activity less than 0.07
be	constant (Eq. (8))	T	temperature (K or °C)
c	constant (Eq. (7))	vd	samples used in the adsorption isotherms were dried until constant weight in a vacuum oven at 70 °C
db	dry basis	V_L	molar volume of liquid ($\text{cm}^3 \text{mol}^{-1}$)
desp	desorption isotherm	wb	wet basis
E	mean relative percentage deviation modulus	<i>Greek symbols</i>	
EMC	equilibrium moisture content (% db)	γ_s	surface tension of liquid in a pore
k	constant (Eq. (5))	θ	wetting angle
L_w	latent heat of vaporisation of pure water	<i>Subscripts</i>	
m_1	constant (Eq. (8))	pred	predicted moisture content value
MC	moisture content (% db)	obs	experimental moisture content value
m_i	experimental value	ref	reference
m_{pi}	predicted value		
ms_1	constant (Eq. (8))		
N	number of experimental data		
p	probability value		
Q_{st}	isosteric heat of sorption ($\text{J g}^{-1} \text{mol}^{-1}$)		
Q_{st}^n	net isosteric heat ($\text{J g}^{-1} \text{mol}^{-1}$)		
r	capillary radius		
R	gas constant ($8.314 \text{ J K}^{-1} \text{mol}^{-1}$)		

et al., 1968), modified Halsey equation, modified Oswin equation (Oswin, 1946) and GAB equation. These models have been adopted as standard equations by the American Society of Agricultural Engineers for describing sorption isotherms (ASAE, 1995). The equations of BET and GAB provide the monolayer moisture content, and can be considered to be the most useful ones for determining the optimum moisture conditions for good storage stability, especially for dehydrated foods (Arslan and Toğrul, 2006). GAB, Henderson (1952), modified Henderson, modified Halsey equation and modified Oswin equation were reported to have fitted well plantain or banana's equilibrium moisture content data over 10–70% relative humidity range (plantain, a member of the banana family, a starchy and low in sugar variety) (Ajibola, 1986; Johnson and Brennan, 2000; Phoungchandang and Woods, 2000; Talla et al., 2005).

To obtain the desorption isotherm samples must be moistened to high water activity and then lose moisture in containers with the required salt solution, while for the adsorption isotherms samples must be pre-dried and then gain moisture in the closed container with the appropriate salt. Tsami et al. (1999) reported that different drying methods (freeze-drying, microwave-drying, vacuum drying and conventional drying) influenced the adsorption properties of dehydrated pectin-sugar gels in powdered form. Hubinger et al. (1992) found that sorption capacity of freeze-dried pineapple was higher than that for vacuum dried pineapple.

It has been stated that the extent of hysteresis (the difference a_w versus EMC between desorption and adsorption) is related to the nature and state of the

components in a food, reflecting their potential for structural and conformational rearrangements, which alter the accessibility of energetically favourable polar sites (Kapsalis, 1981; Al-Muhtaseb et al., 2004). The presence of capillaries in foods results in some lowering of the water activity. The Kelvin equation predicts this lowering by (Bell and Labuza, 2000):

$$a_w = \exp\left(\frac{-2\gamma_s V_L \cos \theta}{rRT}\right) \quad (1)$$

where γ_s is the surface tension of liquid in a pore, θ is the wetting angle, V_L is the molar volume of liquid ($\text{cm}^3 \text{mol}^{-1}$), r is the capillary radius, R is the gas constant ($8.314 \text{ J K}^{-1} \text{mol}^{-1}$), and T is absolute temperature (K).

The net isosteric heat of sorption can be used to estimate the energy requirements of drying and provides important information on the state of water in food products. The moisture content level of a material at which the net isosteric heat of sorption reaches the latent heat of vaporisation of water is often considered as an indication of the “bound-water” existing in the food (Duckworth, 1972; Wang and Brennan, 1991).

The effect of temperature on water activity at a constant moisture content may be described by the Clausius–Clayperon equation:

$$\left.\frac{\partial \ln(a_w)}{\partial T}\right|_M = \frac{Q_{st}^n}{RT^2} \quad (2)$$

where Q_{st}^n is the net isosteric heat of sorption ($\text{J g}^{-1} \text{mol}^{-1}$), that corresponds to:

$$Q_{st} = Q_{st}^n + L_w \quad (3)$$

where Q_{st} is the isosteric heat of sorption (the excess binding energy for removal of the water) ($\text{J g}^{-1} \text{mol}^{-1}$) and L_w the latent heat of vaporisation of pure water ($\text{J g}^{-1} \text{mol}^{-1}$).

Neglecting the effect of temperature on the net isosteric heat of sorption, the Clausius–Clayperon equation can be integrated to yield:

$$\ln(a_w/a_{w\text{ref}}) = \frac{Q_{st}^n}{R} \left(\frac{1}{T_{\text{ref}}} - \frac{1}{T} \right) \quad (4)$$

where $a_{w\text{ref}}$ is the water activity at a reference absolute temperature T_{ref} . The isosteric heat of sorption decreases with increasing moisture content and this effect can often be described by a power function or the following equation:

$$\frac{(Q_{st}^n - Q_{st\infty}^n)}{(Q_{st0}^n - Q_{st\infty}^n)} = \exp(-k \cdot MC) \quad (5)$$

where k , Q_{st0}^n and $Q_{st\infty}^n$ are the equation constants, MC is moisture content (% db).

A plot of $\ln a_w$ versus $1/T$ gives a straight line at constant moisture content. The slope of the line (Q_{st}^n/R) and then Q_{st}^n can be calculated.

As the ultimate goal of this study is to predict shelf life of IMC banana using sorption isotherm (0 to 0.6 water activity), and the aim of this study were to (i) provide reliable experimental data for the sorption characteristics of IMC banana; (ii) model the sorption isotherms using selected equations; (iii) determine their dependence on temperature; (iv) examine the hysteresis phenomena and compare the effect of different dehydration methods to prepare adsorption isotherm sample on the sorption isotherm; and (v) calculate the net isosteric heat of sorption.

2. Materials and methods

Completely ripe bananas (var. *Cavendish*) from South Africa were dried to approximately 17% db with hot air between 35 and 45 °C for 13 to 16 h, and simultaneously smoked by 150 mg of SO_2 per 100 g to enhance colour retention. The moisture content of final product was about 14% wb and soluble solids were about 50° Brix.

During storage of IMC bananas, samples can adsorb or desorb water depending on the environmental conditions. The working isotherm (Bell and Labuza, 2000) refers to the sorption isotherms using original IMC banana sample, to distinguish from desorption and adsorption isotherms. Adsorption isotherm samples were dried until constant weight in a vacuum oven at 70 °C (adsp.vd), or placed above the saturated solution of Potassium Hydroxide to get water activity less than 0.06 (adsp.ss). Desorption isotherm samples were first placed above the saturated solution of potassium sulphate to get 0.96 of water activity until constant weight. The critical water activity for shelf life of dried or IMC fruits product is 0.6 (Beuchat, 1983), therefore, the water activity range of 0.06–0.76 were selected for this study.

Adsorption, desorption and working isotherm samples (~5 g) of IMC banana prepared as described above were accurately weighed in an analytical balance (Ax200, Shimadzu Corp.), placed into Petri dishes, which in turn were placed above the saturated salt solutions in the airtight containers stored at a constant temperature (10, 15, 20, 30, 40 ± 2 °C) for working isotherm or (10 and 40 ± 2 °C) for desorption and adsorption isotherms (Table 1). This procedure was performed in triplicate.

Samples were weighed every 2 days until a weight change equal or less than 0.002 g was recorded on two consecutive weightings, when the sample was assumed to be at equilibrium. Weighing time was reduced to less than 30 s. About 15 to 30 days were required for equilibration. No visible mould was observed in the samples. After equilibration the samples' moisture content were measured following the AOAC 1980 method (drying at 70 °C, 28-mmHg vacuum until constant weight, approximately 10 h).

3. Model selection

Most commonly used isotherm equations, BET, modified BET (Weisser, 1986), GAB, modified GAB (Jayas and Mazza, 1993), Halsey and modified Halsey, were selected for fitting the experimental data for sorption isotherms of IMC banana.

Table 1
Saturated salt solutions used to establish the water activity at different levels from 0.06 to 0.76 (modified from Greenspan, 1977)

Solution	RH at 10 °C	RH at 15 °C	RH at 20 °C	RH at 30 °C	RH at 40 °C
LiBr	0.07	0.07	0.09 ^a	0.08 ^b	0.06 ^b
LiCl	0.11	0.11	0.11	0.11	0.11
$\text{C}_2\text{H}_3\text{O}_2\text{K}$	0.24	0.23	0.23	0.22	0.21
MgCl_2	0.34	0.33	0.33	0.32	0.32
K_2CO_3	0.43	0.43	0.43	0.43	0.43
$\text{Mg}(\text{NO}_3)_2$	0.57	0.56	0.54	0.52 ^c	0.50 ^c
NaNO_2	0.62 ^d	0.61 ^d	0.68	0.64	0.61
NaCl	0.76	0.76	0.75	0.75	0.75

^a KOH was used instead of LiBr.

^b NaOH was used instead of LiBr.

^c $\text{Na}_2\text{Cr}_2\text{O}_7$ was used instead of $\text{Mg}(\text{NO}_3)_2$.

^d NaBr was used instead of NaNO_2 .

The constants of the equations were estimated by regression analysis using Statistica 5.5 software (Statsoft Inc., Tulsa, OK). To evaluate the goodness of fit of each model, the coefficient of determination (R^2), the mean relative percentage deviation modulus (E) (Eq. (6)), and the standard error of the estimates were used (Basu et al., 2006)

$$E = \frac{100}{N} \sum_{i=1}^N \frac{|m_i - m_{pi}|}{m_i} \quad (6)$$

where m_i is the experimental value, m_{pi} is the predicted value, and N is the number of experimental data. The mean relative percentage deviation modulus (E) has been widely adopted throughout the literature, with a modulus value below 10 indicative of a good fit for practical purposes.

4. Results and discussion

4.1. Working isotherm

The sorption isotherms of IMC banana showed the shape of type II, with non-zero moisture content (approximately 3–9% db for different temperatures, predicted by modified Freundlich model below) when the water activity was zero. The shape of sorption isotherms was in accordance with the work of Talla et al. (2005). The equilibrium moisture content EMC increased with increasing relative humidity at constant temperature and decreasing temperature at constant relative humidity, which might be

explained by the higher active state of water molecules at higher temperature thus the attractive forces between them decreasing (Bushuk and Winkler, 1957).

BET, modified BET, GAB, modified GAB, Halsey and modified Halsey equations were fitted to the experimental data of the sorption isotherms for IMC banana. Modified BET was the best model when fitting to the experimental data at different temperatures, but with high E value up to 26.7933. Low R^2 (<0.85), or cannot compute the standard error or great estimated constants' standard error were found for all other models (results not shown), which is different from other authors work. Moreover, on the isotherms, there was no inflection point where the extension line would go through the origin point ($a_w = 0$, MC = 0), therefore, all the models selected were not suitable to fit the data of isotherms.

Two other models (Eqs. (7) and (8)) were created to fit the experimental data of sorption isotherms at 10, 15, 20, 30 and 40 °C. Freundlich equation (Freundlich, 1926) was modified as Model 1 (Eq. (7)).

$$\text{Model 1 : MC} = a + ba_w^c \quad (7)$$

$$\text{Model 2 : MC} = m_1 + (ms_1 - m_1) \exp[(a_w/0.6)^{be} + 1] \quad (8)$$

where a_w is water activity, a , b , c , be , m_1 and ms_1 are model constants.

The coefficient of determination (R^2) of Models 1 and 2 of different sorption isotherms of IMC banana at temperature of 10, 15, 20, 30, and 40 °C were presented in Table 2.

Table 2
The coefficient of determination (R^2) and constants of Models 1 and 2 of IMC banana at temperature of 10, 15, 20, 30, and 40 °C

Temperature (°C)	Constant			R^2
	a	b	c	
<i>Model 1</i>				
10 working	8.97 ± 0.14	100.77 ± 15.92	4.62 ± 0.33	0.9913
15 working	7.76 ± 0.16	81.39 ± 10.63	3.90 ± 0.26	0.9912
20 working	7.46 ± 0.16	60.54 ± 4.40	3.66 ± 0.18	0.9897
30 working	4.12 ± 0.13	63.67 ± 3.23	3.15 ± 0.11	0.9948
40 working	3.13 ± 0.16	65.18 ± 4.42	3.08 ± 0.13	0.9913
10 adsp vd	1.09 ± 0.32	100.11 ± 10.84	3.30 ± 0.22	0.9897
40 adsp vd	1.36 ± 0.26	48.38 ± 3.21	2.23 ± 0.13	0.9911
10 adsp ss	0.026 ± 0.0028	0.96 ± 0.29	4.43 ± 0.62	0.9742
40 adsp ss	0.017 ± 0.0014	0.60 ± 0.045	3.12 ± 0.15	0.9996
10 desp	0.1 ± 0.0035	0.97 ± 0.26	4.28 ± 0.55	0.9594
40 desp	0.046 ± 0.0015	0.77 ± 0.043	3.08 ± 0.11	0.9957
	m_1	ms_1	be	
<i>Model 2</i>				
10 working	24.26 ± 0.33	18.68 ± 0.20	6.38 ± 0.59	0.9880
15 working	25.18 ± 0.34	18.82 ± 0.21	5.23 ± 0.34	0.9907
20 working	23.79 ± 0.34	17.87 ± 0.19	5.98 ± 0.39	0.9886
30 working	24.95 ± 0.35	17.37 ± 0.21	4.59 ± 0.22	0.9897
40 working	24.54 ± 0.31	16.73 ± 0.20	4.24 ± 0.15	0.9913
10 adsp vd	30.59 ± 0.55	19.81 ± 0.33	4.41 ± 0.26	0.9908
40 adsp vd	25.54 ± 0.60	16.76 ± 0.38	3.08 ± 0.22	0.9844
10 adsp ss	0.19 ± 0.0048	0.13 ± 0.0029	6.20 ± 0.69	0.9773
40 adsp ss	0.21 ± 0.0032	0.14 ± 0.0020	4.28 ± 0.18	0.9932
10 desp	0.28 ± 0.0059	0.21 ± 0.0036	6.07 ± 0.65	0.9726
40 desp	0.30 ± 0.0030	0.21 ± 0.0019	4.22 ± 0.12	0.9965

The R^2 of all sorption isotherms were higher than 0.95 and E less than 13, and it can be concluded that in terms of R^2 and E , both Models 1 and 2 are suitable for describing the relationship between EMC and water activity at each temperature (10, 15, 20, 30, and 40 °C), under the conditions tested (0.06–0.76) water activity (or from 3.21% to 38.16% db moisture content). The constants a , b , b_e and ms_1 in Models 1 and 2 for working isotherms can be correlated with temperatures and then were fitted to average EMC at each water activity and different temperatures (Fig. 1a and b). R^2 of two new models were 0.9907 and 0.9924, respectively, E were 5.3919 and 10.5135 for Models 1 and 2, respectively, in which the former was at quite a low level. Moreover, p -levels of all the constants of the two new models estimated were less than 0.01, and the distribution of residuals obtained by the two models was normal and the trend was not biased, which showed the two new models fitted well all the data at different temperatures (0.06–0.76 water activity). Lower E value and better fit at high water activity showed that Model 1 (modified Freundlich) was better than Model 2 to fit the experimental data of

sorption isotherms. The constants estimated were integrated into Eqs. (7) and (8) and then specific equations for this IMC banana's sorption isotherm are shown in Eq. (9) and (10).

$$MC = (10.83 - 0.20 T) + 61.98a_w^{(3.90-0.0234T)} \tag{9}$$

$$MC = 24.53 - (4.93 + 0.07T) \times \exp[-(a_w/0.6)^{(6.69-0.06T)} + 1] \tag{10}$$

where T is temperature (°C).

4.2. Moisture sorption hysteresis

The comparisons between working isotherms, adsorption isotherms and desorption isotherms of IMC banana at 10 °C and 40 °C are shown in Figs. 2 and 3, respectively. At a constant water activity, the EMC of the desorption

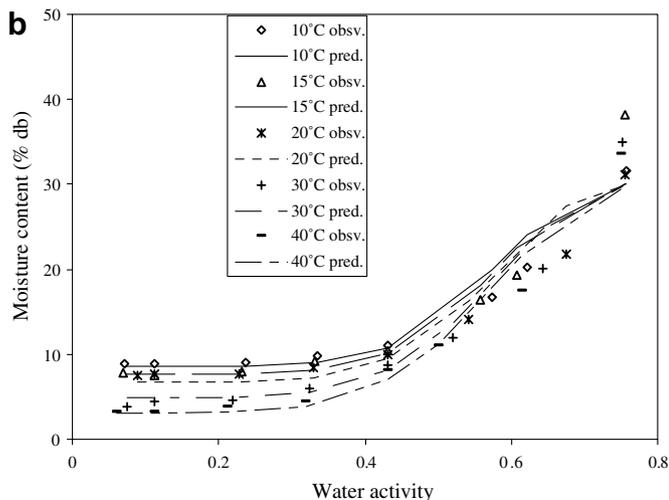
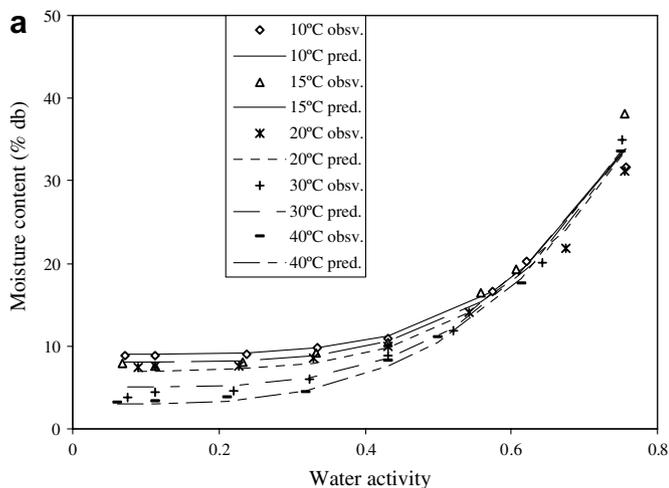


Fig. 1. The plot of fit of Models 1 (a) and 2 (b) to working isotherm of IMC banana at temperature of 10, 15, 20, 30, and 40 °C.

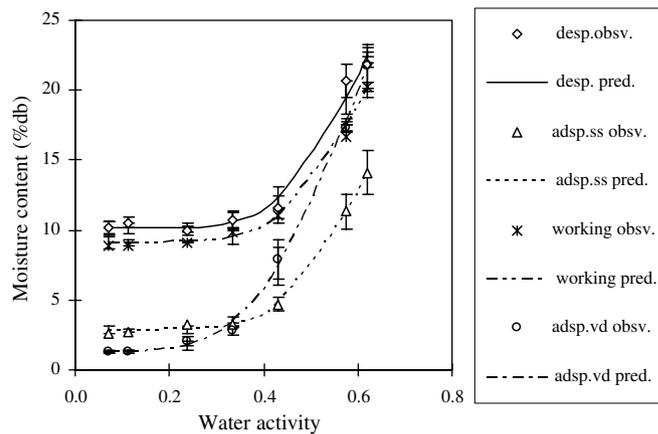


Fig. 2. The plot of fit of Model 1 to working, desorption and adsorption isotherms of IMC banana at temperature of 10 °C. The symbols show the experimental data, bars the standard deviation and lines the values predicted by modified Freundlich equation.

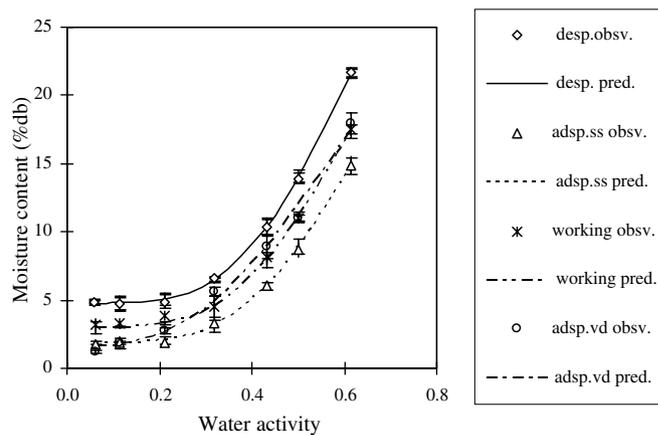


Fig. 3. The plot of fit of Model 1 to working, desorption and adsorption isotherms of IMC banana at temperature of 40 °C. The symbols show the experimental data, bars the standard deviation and lines the values predicted by modified Freundlich equation.

isotherm and adsorption isotherm (adsp.ss) are highest and lowest, respectively.

Hysteresis has been related to the nature and state of the components in a food, reflecting their potential for structural and conformational rearrangements (Eq. (1)). When water moves out from the capillaries of banana, during moisture desorption, the narrow ends of surface pores trapped and held water internally below the water activity where the water should have been released, thus there was greater moisture content at a low range of water activity. During adsorption, the pure water would dissolve solutes present in the dried banana and the dissolution of solutes increased the surface tension (γ_s), resulting in lower water activity at given moisture content. The swelling of polymeric materials during moisture adsorption can also lead to hysteresis. As protein and carbohydrates in dried banana become swollen, polar sites, once obscured during drying, may now be available to interact with water by forming hydrogen bonds. During drying or desorption, some solutes may supersaturate below their crystallization water activity resulting in more water being held as a_w is lowered. Bell and Labuza (2000) reported that foods with high sugar content frequently exhibit this phenomenon.

It has been known that sorption hysteresis is affected by temperature, the properties of components, methods of equilibration, and the history of materials. Hysteresis was observed in this study within the whole range of water activity tested at 10 °C and 40 °C. The magnitude of hysteresis was smaller at 40 °C than at 10 °C, probably due to increased elasticity of capillary walls, and greater capability of forming hydrogen bond between protein/carbohydrate and water.

There were crossovers between adsp.vd and adsp.ss at low water activity, adsp.vd and working isotherm at high water activity. Comparing adsp.ss to adsp.vd, the latter had lower EMC at low water activity range, because during vacuum drying of banana, the microporus structure of banana was partly damaged, and the phenomenon of case-hardening occurred at the banana surface which limited moisture migration through the surface layers from the environment, resulting in adsorbing and holding less moisture content than adsp.ss at a low water activity range. However, at high water activity range, adsp.vd held more water than adsp.ss, because starch gelatinization normally occurs between 58 °C and 75 °C and so, during vacuum drying, starch of banana gelatinized and improved hydration properties (water-binding capacity). After the hard case softening, more moisture content than adsp.ss was absorbed into banana samples.

4.3. Net isosteric heat of sorption

The relationships between the net isosteric heat of sorption and moisture content of IMC banana are shown in Fig. 4. The net isosteric heat of sorption of the IMC banana ranged from 846 J g⁻¹ mol⁻¹ at 20% moisture content (dry basis) to 7669 J g⁻¹ mol⁻¹ at 10% moisture content (dry

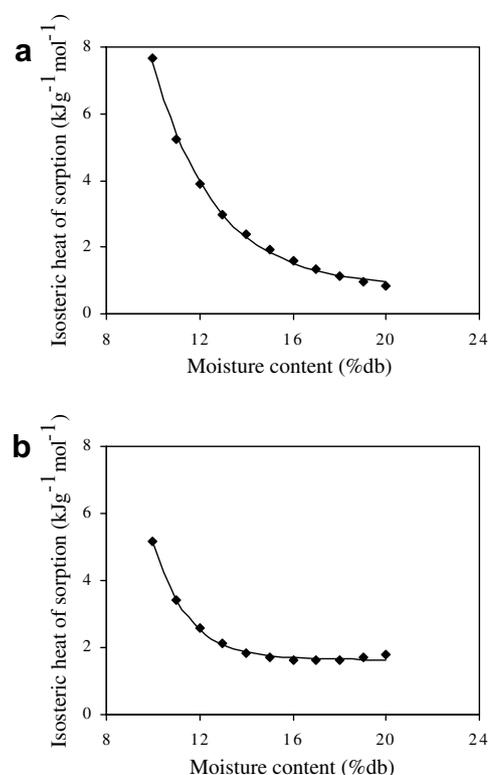


Fig. 4. Net isosteric heat of sorption of IMC banana for Models 1(a) and 2 (b). The symbols show the experimental data, and lines the values predicted by the exponential equation.

Table 3

Exponential regression of isosteric heat against moisture content (dry basis)

	R^2	Exponential equation
Model 1	0.9978	$Q_{st}^n = 304.85 - 304.03 \exp(-0.4MC)$
Model 2	0.9969	$Q_{st}^n = 3053.75 - 3052.11 \exp(-MC)$

basis) for Model 1, and from 1782 J g⁻¹ mol⁻¹ at 20% moisture content (dry basis) to 5147 J g⁻¹ mol⁻¹ at 10% moisture content (dry basis) for Model 2. As the moisture content increased, the isosteric heat of sorption of IMC banana decreased. When the net isosteric heat of sorption was regressed against moisture content (dry basis) using an exponential function, the results showed a good fit (Table 3).

5. Conclusions

IMC banana exhibited Type II isotherms. The equilibrium moisture content increased with decreasing temperature at constant water activity. The magnitude of the hysteresis at 40 °C was smaller than at 10 °C. The IMC banana sample dehydrated by vacuum oven and placed on saturated solutions showed different desorption isotherms. Among the sorption isotherm models chosen to fit isotherm data, Models 1 (modified Freundlich) described better the working, desorption and adsorption isotherms (0.06–0.76 water activity). The isosteric heat of

sorption of IMC banana decreased with an increase in moisture content and was found to be an exponential function of moisture content. These results provide reliable experimental data on sorption isotherms which is important for designing an optimized packaging system.

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