Evaluation of sorption isotherm models for figs

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Pacco, H. C., Vigneault, C., Menegalli, F. C., de Castro, L. R. and Cortez, L. A. B. 2009. **Evaluation of sorption isotherm models for figs**. Canadian Biosystems Engineering/Le génie des biosystèmes au Canada. **51**: 3.77–3.83. Isotherms of moisture sorption and desorption were determined for figs at temperatures of 25, 40 and 60°C. The goodness of fits of the four most common models – GAB, linearized BET, Iglesias and Chirife (MH = modified Halsey), and Oswin models – were compared to evaluate their ability to predict the sorption data using nonlinear regression analysis. The net isosteric heat of desorption was determined using the Clausius-Clapeyron equation. A third-degree polynomial correlated well the hysteresis between the adsorption and desorption of figs and the water activity for each temperature. The results showed that the GAB equation gave the best fit to the experimental data. The isosteric heat of sorption was found to be a one-term power function of the moisture content and showed an important increase of energy required to dry figs as their moisture content decreases. The GAB equation may be applied to design more efficient drying process for figs. **Keywords:** Figs, sorption isotherms, isosteric heat, sorption, mathematic models.

Des courbes isothermiques d’absorption et de désorption de l’humidité des figues ont été déterminées pour trois températures, soit 25, 40 et 60°C. Un analyse de régression non linéaire a été utilisé pour mesurer la qualité des ajustements des quatre modèles les plus courants, c’est-à-dire: le model de GAB, le BET linéarisé, celui d’Iglesias et Chirife (ou MH pour Halsey modifié), et celui d’Oswin; et évaluer leur capacité à prédire les valeurs de sorption de l’humidité. La chaleur isostère nette de la désorption a été determinée à l’aide de l’équation de Clausius-Clapeyron. Une équation polynomiale du troisième degré a été utilisée pour prédire l’hystérèse existant entre les courbes d’adsorption et désorption de l’humidité et l’activité de l’eau des figues à chaque température. Les résultats ont démontré que l’utilisation du model de GAP produisait le meilleur des ajustements avec les données expérimentales. La chaleur isostère de la désorption a été représentée par une équation de puissance à un seul facteur en fonction de la teneur en eau et a montré une augmentation importante d’énergie requise pour sécher des figues à mesure que leur teneur en eau diminue. L’équation de GAP peut être utilisé pour augmenter l’efficacité de procédés de séchage de figues lors de leur conception. Mots clés: Figue, courbe isothermique d’absorption, chaleur isostére, sorbtion, model mathématique.

INTRODUCTION

Brazil is the second largest producer of figs in the world after Turkey, yet Brazil does not produce dried figs. The state of São Paulo supplies 80% of the national production, especially with the “Gigante de Valinhos” variety of the common type of fig (Sarria 2003). Three other types of figs are also cultivated in the world: Caprifig, Smyrna and San Pedro. However, from Florida, USA, to Northern South America and India, only the common fig is grown (Morton and Dowling 1987). The common fig is characterized by the fact that it does not require pollination, its lack of seeds, and its wide-open ostiole, which facilitates access of fungi and insects to the interior of the fruit. Furthermore, the common fig is a highly perishable variety, especially when it matures in rainy and hot seasons. Drying techniques could be used on surplus or out-of-standard fruit to reduce losses, aggregate value to production, and decrease Brazilian importation, since all dried figs consumed in this country are actually imported from the USA, Turkey, and the Middle East (Pacco 2003).

Drying is likely the oldest method of food preservation and consists of the removal of the majority of the water content in produce (Dauthy 1995). Since food water activity affects the rate of biological and chemical reactions, the drying process could inhibit the growth and reproduction of microorganisms that could lead to decay (Herringshaw 1997). It also prevents biochemical reactions that rely on moisture content and cause produce deterioration (USEPA 2005). Dehydration has been increasingly applied to some highly perishable fruits and vegetables to reduce losses and extend their shelf life. In Brazil, dried fig is a product with a high potential that should be explored for three reasons: the high nutritional value of figs, the extensive production of fresh figs in Brazil, and the benefits of supplying the domestic market (Pacco 2003).

Optimal design of drying and storage processes is based on knowledge of moisture sorption isotherms and water activity (Pacco 2003). Moisture sorption isotherms correlate the equilibrium moisture content with the ambient relative humidity or water activity of the vapor space surrounding a produce under constant temperature and pressure conditions. Moisture sorption isotherms added to the food isosteric heat of sorption are essential for designing efficient dryers and several other food preserving methods. These parameters are useful for predicting stability changes in produce, selecting packing material and ingredients, and estimating the energy required for the drying process (Pacco 2003).

The isosteric heat of sorption provides information on the binding energy of water in food, i.e., the interaction forces between the water vapor molecules and the absorbent surface (Khalloufi et al. 2000). The isosteric heat of sorption can be estimated using the calorimetric method and the Riedel equation (Öztekin and Soysal 2000) or, more conveniently, the Clausius-Clapeyron equation applied to moisture sorption data (Rizvi 1986; Tsami et al. 1990b).

The moisture sorption isotherms can be obtained by processes in which the moisture content is either increased (adsorption) or decreased (desorption). Desorption isotherms generally result in higher values of equilibrium moisture content than adsorption at a certain water activity. This difference between adsorption and desorption moisture content is called hysteresis (Young and Nelson 1976). This phenomenon is especially found in hygroscopic products. The equilibrium moisture content of a food at a certain water activity is expected to decrease as the temperature increases. In dried fruits, however, the inverse usually occurs for water activity above 0.65 (Ayranci et al. 1990; Tsami et al. 1990a).

The isotherm curves are composed of three distinct regions (Pacco 2003; Ouaouich 2004). The first corresponds to strongly bound water with unfreezable molecules usually not available for chemical reactions or use as plasticizers. In the second region, the water molecules bind less firmly than in the first. Finally, the properties of the water in the last region of isotherms are close to those of the free water, and no excess heat of binding can be detected.

A static gravimetric method for the experimental determination of sorption isotherms was developed and standardized by the European Cooperation Project COST 90 (Spiess and Wolf 1983). This method consists of measuring the weight of a sample before and after its dehydration and assigning the difference in weight to moisture content (Yang and Yen 1996).

Several empirical or semi-empirical equations have also been proposed for the mathematical description of the moisture sorption isotherms of food products. Their applicability and accuracy depend on the type of food and water activity range. Among them, the European Cooperation Project COST 90 recommended the GAB (Guggenheim-Anderson-de Boer) equation, which has been successfully applied to various food products (Prado et al. 1999; Khalloufi et al. 2000; Pacco 2003). The GAB equation is based on the BET (Brunauer-Emmett-Teller) theory and usually involves three physically significant parameters (Kiranoudis et al. 1993). The linearized BET equation has also shown good fit at food water activity between 0.1 and 0.5 (Labuza 1968). Ratti et al. (1989) developed a non-isothermal equilibrium expression that was proven to satisfactorily predict water sorption of food materials at a wide range of moisture levels and at different temperatures. Iglesias and Chirife (1976) modified the Halsey equation (Halsey 1948) to create the modified Halsey (MH) model and showed good fits with fruit applications as well, especially those fruits with a high sugar content such as bananas, grapefruit, peaches, pears, pineapples, and strawberries (Rizvi 1986). Sun and Byrne (1998) also found a good fit when modeling the sorption isotherm of rapeseed with the MH model. However, Ouaouich (2004) warned that the use of the MH model requires moisture content data at 0.5 water activity, which is hard to determine. The Oswin model is another example used to predict the behavior of food sorption isotherms and has been found to fit 57% of food isotherms (Ouaouich 2004).

The aim of this study was to compare the most common equations applied to correlate the equilibrium moisture content with the water activity of fig fruit. The comparison was based on the ability of the equations to describe the sorption isotherm of a specific variety of figs.

MATERIALS and METHODS

Sample preparation

Figs of the ''Gigante de Valinhos'' variety (*Ficus carica* L.) that had been freshly harvested in the district of Valinhos, São Paulo state, Brazil, were initially sorted for maturity level, color, and size. Fruits free of mechanical injury and disease were washed and surface air dried.

Sorption isotherms

The adsorption and desorption isotherms were determined by the static gravimetric method (Spiess and Wolf 1983) at 25, 40 and 60°C over a water activity range of 0.045 to 0.90. Ten saturated salt solutions were used to determine the equilibrium humidity for dried figs following the specifications described in Labuza et al. (1985) and Young and Nelson (1976). These solutions and their relative humidities at 25, 40 and 60°C are presented in Table 1.

Desorption and adsorption curves were determined using a 2 g sample of fresh fig or freeze-dried fig, respectively. The produce was ground to accelerate the mass transfer process. Three drops of formol were added to each sample to avoid any microorganism growth. Each sample of either fresh or freeze-dried fig was weighed and handled in a 12 mL cup (38.2 mm in diameter and 10.5 mm in height). The cup was placed in 150 mL Petri dish (99.5 mm in diameter and 19.2 mm in height) already containing 40 mL of one of the 10 required saturated salt solutions (Table 1). A short support was used in the Petri dish to maintain the cup above the saturated salt solution.

Table 1. Salt solutions and their equilibrium relative humidities at 25, 40 and 60°C.

	Relative humidity (%)		
Salt	25°C	40°C	60°C
NaOH	7.00	6.50	4.50
LiCl	11.30	11.21	10.95
$MgCl_2$	32.78	31.60	29.76
K_2CO_3	43.16	31.60	43.20
$Mg(NO_3)_2$	52.89	43.20	47.30
NaBr	57.57	53.17	49.66
$NaNO_2$	65.70	61.50	59.00
NaCl	75.10	74.68	74.50
KCl	84.20	82.00	80.00
$BaCl_2$	90.03	89.30	87.50

Each Petri dish was closed to be air tight and placed in a small temperature-controlled chamber maintained at one of the three following temperatures: 25, 40, and 60°C. The samples were weighed on a 0 to 200 g ± 0.0001 g scale (Sartorius CP224S, Sartorius, Germany) every week until a constant mass was reached (AOAC 1997). At this point, the samples reached the equilibrium humidity for the fig pulp, which was calculated on a mass basis. The equilibrium was reached within 10 week for both processes, adsorption and desorption. The fig moisture value content obtained was then related to the temperature and relative humidity of the air at which the saturated salt used for each test normally equilibrates (Table 1) to form the sorption isotherms. After the equilibrium was reached, the samples were placed in an oven dryer kept at 60°C for 48 h to determine the fig pulp dry mass, in accordance with the methodology described by the AOAC (1997). All data were obtained in triplicate for each of the 10 salts and three temperatures.

The moisture sorption isotherm data were correlated to the water activity using the following mathematical models (Table 2): GAB, linearized BET, Iglesias and Chirife (MH), and Oswin. The parameters of these equations were determined by correlating the mathema-

Table 2. Parameter values and regression coefficients for each model calculated from adjustment of desorption isotherm data for fig pulp at different temperatures (T, °C).

Model	Equations	Constants	T (°C)		
			25	40	60
GABh (Spiess and Wolf 1983)	$X_{eq} = \frac{X_m\ C\ K\ a_w}{(1 - k\ a_w)(1 - K\ a_w + C\ K\ a_w)}$	X_m	0.09	0.10	0.06
		C	21.63	12.18	282.94
		K	0.96	0.90	1.00
		R^2	0.9805	0.9778	0.9814
		χ^2	0.0146	0.0099	0.0074
Linearized BET (Kiranoudis et al. 1993)	$X_{eq} = \frac{X_m\ C\ a_w}{(1 - a_w)\ (1 + (C - 1)a_w)}$	X_m	0.60	0.60	0.60
		C	210.31	5928.3	100.86
		R^2	0.9696	0.9330	0.9813
		χ^2	0.0227	0.0283	0.0074
MH (Iglesias and Chirife 1976)	$X_{eq} = \left(\frac{k_1}{\mathrm{In}(k_2/a_w)}\right)^{k_3}$	k_1	4.22	3.38	3.72
		k_2	99.20	100.99	99.35
		k_3	2.78	1.89	2.44
		R^2	0.9624	0.9625	0.9258
		χ^2	0.0280	0.0161	0.0285
Oswin (Ouaouich 2004)	$X_{eq} = A.(\frac{a_w}{1 - a_w})^B$	A	0.1598	0.1610	0.1256
		B	0.6352	0.5209	0.6782
		R^2	0.9772	0.9747	0.9672
		χ^2	0.0815	0.0520	0.0615

X_{eq} = equilibrium material moisture content (kg kg^{-1}, dry basis).
X_m = monolayer moisture content (kg kg^{-1}, dry basis).
a_w = water activity.
A, B, C, K, k_1, k_2 = constants.
R^2 = coefficient of determination.
χ^2 = goodness of fit between the model and the experimental data.

tical models to the experimental data using the method of direct nonlinear regression on the Statistica 5.0 software (Statsoft 2005) at a 5% significance level.

The chi-square (χ^2) and coefficient of determination (R^2) values were calculated using the Statistica 5.0 software to identify the model that provides the highest goodness of fit with the experimental data. According to different authors (Costa Neto 1977; Steel and Torrie 1980), χ^2 is a method for comparing models based on their residuals. $\chi^2 \approx SS/\sigma^2$, which is the sum of the squared residuals between the model and the data obtained divided by the standard deviation of their residuals. Thus, comparing different models, the one presenting the smaller χ^2 is likely representing better the experimental data assuming that the residues are normally distributed in each compared model. Likewise Montgomery (2001), R^2 indicates how well a regression line represents the data. After plotting the residues against the experimental values to identify any abnormal distribution, the model presenting the lowest χ^2 and the highest R^2 was identified as the most suitable model to describe the isotherms of figs.

Isosteric heat of sorption

The net isosteric sorption heat (q_{st}) and equilibrium moisture content (X_{eq}, kg water kg^{-1} produce, dry basis) were correlated using Eq. 1 and the Statistica software (Statsoft 2005), where q_0 = net isosteric heat of sorption of the first molecule of water in the food (J/mol); X_0 = characteristic moisture content of the food material (kg water kg^{-1} produce, dry basis). The sorption heat was calculated from desorption isotherms using the Clausius-Clapeyron equation (Eq. 2) (Rizvi 1986; Tsami et al. 1990b), where a_{w1} and a_{w2} = water activity corresponding to T_1 and T_2, the absolute temperature at conditions 1 and 2, respectively. This sorption heat corresponds to the angular coefficient of this linear regression multiplied by the Universal Gas Constant (R). The water activity was determined using the GAB regression equation (Table 2) over the humidity range of 0.05 to 0.30.

$$q_{st} = q_0 \, e^{-\frac{X_{eq}}{X_0}} \tag{1}$$

$$\mathrm{In}\left(\frac{a_{w2}}{a_{w1}}\right)_{x=cons\quad\tan\quad t} = \frac{q_{st}}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \tag{2}$$

RESULTS

The experimental results were analyzed to compare the water activity and water content at equilibrium for the three different temperatures tested. The four existing models presented were compared. The adsorption hysteresis, the desorption hysteresis and the water sorption isosteric heat were calculated.

As expected, a comparison of the ln a_w (water activity) with the inverse of the absolute temperature (Fig. 1) showed a significant decrease ($P < 0.01$) of the effect of the temperature on the water activity as the fig moisture contents (X_{db}) increased. In fact, the temperature did not show any significant effect on the water activity when the $X_{db} \leq 0.20$ (R^2 0.68, $P > 0.1$). This result is similar to the results obtained by Ayranci et al. (1990) with apricots, grapes and figs.

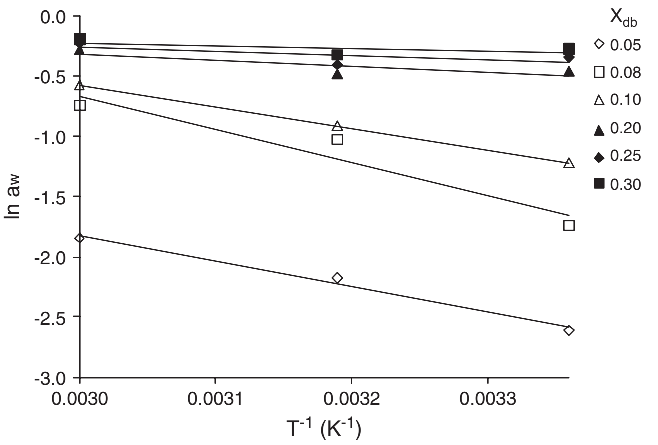

Fig. 1. Water activity (ln a_w) plotted with the inverse absolute temperatures (T^{-1}) for the fig pulp for six moisture contents (X_{db}) from 0.05 to 0.3 g/g measured on a dry basis.

In a comparison of the water content at equilibrium (X_{eq}) obtained during the sorption and desorption test as a function of the water activity of fig for three different temperatures (25, 40, and 60°C) using isotherm conditions, the results showed similar patterns for all the temperatures (Fig. 2). The results of isotherm sorption obtained during this work are similar to the results published by Ayranci et al. (1990), who evaluated the isotherm sorption of grapes, apricots and figs. As presented by Ouaouich (2004), the X_{eq} isotherm pattern formed three distinct regions. A strong increase in X_{eq} was encountered at lower water activity values (< 0.2), corresponding to strongly

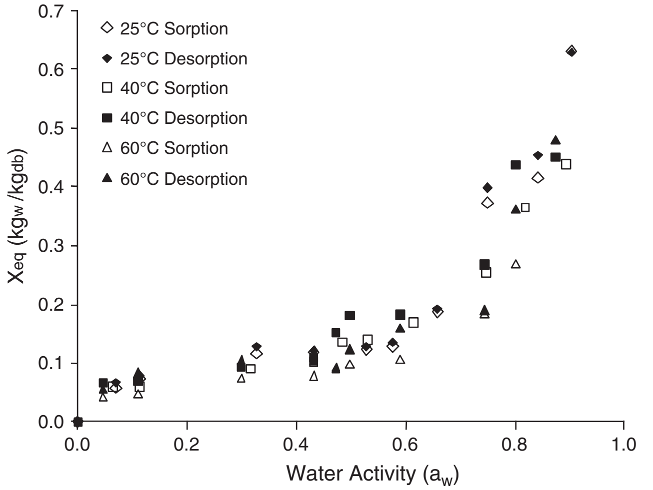

Fig. 2. Water content at equilibrium (X_{eq}) obtained during the sorption and desorption test as a function of the water activity (a_w) of figs using 25°C, 40°C and 60°C isothermal conditions.

bound water with unfreezable molecules. For intermediate values ($0.2 < a_w < 0.65$, approximately), the water molecules bound less firmly and generated more stable X_{eq} values, evidence of results that are more independent from a_w variations. In the higher region ($a_w > 0.65$), the properties of water are close to those of the free water, and the X_{eq} was proportional to the water activity.

The four existing models presented (GAB, BET, Oswin, and MH) were compared for the prediction of moisture content (dry basis) at equilibrium (X_{eq}) based on the water activity (a_w). Analyzing the residues of each of the models proposed, the plots (not presented here) of the residues against the experimental values did not indicate abnormal distribution for any models. The regression coefficient (R^2) and the value of χ^2 were then used to identify the most suitable model for predicting X_{eq}, and the results of this comparison are presented at Table 2. At all temperatures, the GAB equation showed the best fit based on the lower χ^2 goodness of fit statistical results and also resulted in the highest R^2. In fact, this model is able to explain 97.8% or more of the variation of X_{eq} when the experimental conditions and water activity are known.

At 60°C, the BET equation showed values of χ^2 and R^2 very similar to those in the GAB model, giving 0.0074 and 0.9813 compared with 0.0074 and 0.9814, respectively. These results are comparable with those obtained by Prado et al. (1999), who showed that sorption data at $\geq$ 60°C were better fitted using the GAB and BET models than the other models. However, the BET model did not perform as well as the GAB model at lower temperature presenting higher χ^2 and lower R^2 (Table 2).

In general, the modified Halsey (MH) model presented χ^2 and R^2 comparable with the BET model. In fact, as shown in Table 2, the MH model sometimes presented better χ^2 or better R^2 than the BET model depending on the temperature used. The performances of these two models could be considered equivalent in term of performance of predicting the dry base water content at equilibrium of dried product for a temperature range of 25 to 60°C.

Among these four models, the Oswin model presented the lowest performance in term of χ^2 values, but comparable R^2. Even though the Oswin model has been found to fit 57% of food isotherms (Ouaouich 2004), its performance with figs is not the best, but the R^2 values are very interesting −0.9772, 0.9747 and 0.9672 for 25, 40 and 60°C, respectively. These results contradict the performance of this model, presenting the highest capacity to predict the moisture content (dry basis) at equilibrium of dried product since it performed very well with pears dried "in natura" at 80°C (Ouaouich 2004). From these results, it may be concluded that there is not one best model to predict the moisture content (dry basis) at equilibrium of dried product, but that the performance of each model depends on the nature of the dried product and the temperature used.

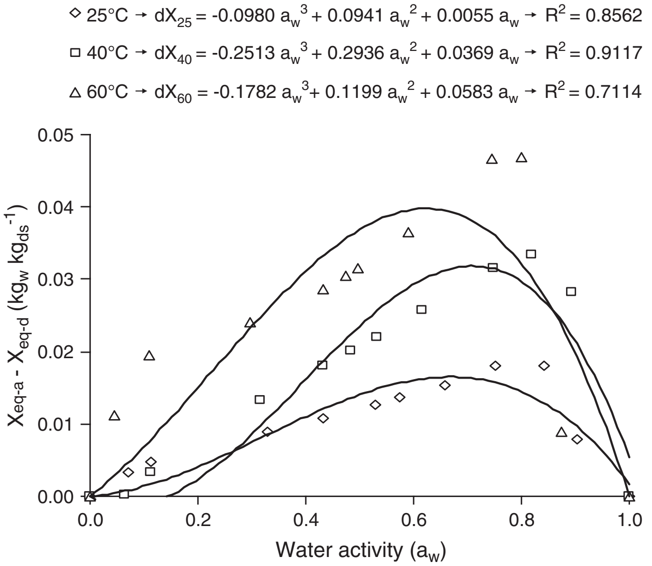

Fig. 3. Hysteresis ($dX = X_{eqd} - X_{eqa}$, $kg_w\ kg_{ds}$) of fig pulp at three temperatures (25, 40 and 60°C) adjusted by the GAB equation; where X_{eqd} and X_{eqa} are the moisture content during desorption and adsorption processes, respectively.

The adsorption and desorption hysteresis of figs (Fig. 3) was calculated using the adjusted values based on the GAB model. A significant third-degree polynomial correlation between the adsorption and desorption hysteresis of figs and the water activity was shown for each temperature. Although those correlations were not very high ($0.71 < R^2 < 0.91$), the result showed a significant increase in the adsorption and desorption hysteresis of figs as the isotherm temperature increased ($P < 0.01$). These hysteresis values were relatively lower than the ones found for onions by Mazza and Lemaguer (1980). In fact, these authors obtained a decrease in hysteresis with increasing temperature, whereas the opposite result was obtained with figs in this study. This phenomenon could be due to different inter-cell capillarity potential for water condensation and evaporation during the sorption and desorption process, although this would not explain why onions and figs react so differently. It is also possible that the changes in molecular structure occur differently from one type of produce to another during the sorption and desorption processes.

The isosteric heat of sorption (q_{is}, kJ kg^{-1}) was also calculated from sorption isotherms and the Clausius-Clapeyron equation (Rizvi 1986) and plotted against the different dry basis moisture contents (X_{db}) (Fig. 4). The water activity was determined using the GAB regression equations (Spiess and Wolf 1983). The water content showed a significant effect ($P > 0.001$) on the sorption heat. A one-term power regression was calculated and showed a very good correlation between sorption heat and the water content, resulting in a R^2 of 0.9987 (Eq. 3). This result was similar to the correlations obtained in the literature for similar types of produce such as grapes, figs and apricots (Ayranci et al. 1990). Even though Tsami et al. (1990b) proposed using an exponential expression to correlate these two parameters, using a one-term power regression resulted in a much better coefficient of

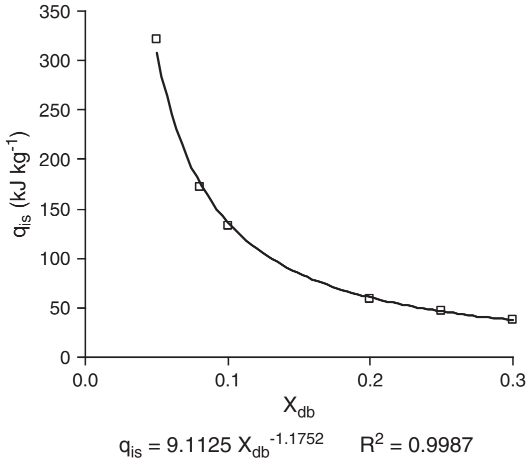

Fig. 4. Net isosteric sorption heat (q_{is}, kJ kg^{-1} produce) at different moisture contents measured on a dry basis (X_{db}).

determination; 0.9440 compared with 0.9987, respectively.

$$q_{is} = 9.1125 X_{db}^{-1.1752} \qquad R^2 = 0.9987 \tag{3}$$

CONCLUSION

The equilibrium moisture contents of figs were measured under isotherm conditions consisting of three temperature levels (25, 40, 60°C). These equilibrium moisture contents were correlated to the water activity of the fruit. The moisture content of the figs did not equilibrate at the same level during the adsorption and desorption isothermal process, showing that hysteresis increased considerably with the temperature. The water activity increased as the temperature increased. However, the importance of this temperature effect on water activity decreased with the increasing water content of the figs and disappeared when the fig water content reached 0.20.

Four existing models (GAB, BET, Oswin, and MH) were applied to correlate the equilibrium moisture content (X_{eq}) with the water activity (a_w) of figs. The comparison was based on their ability to describe the X_{eq} of figs using the coefficient of determination (R^2) and Chi-square (χ^2) from the regression analysis. The GAB equation exhibited the best ability to predict the moisture content at equilibrium for all the tested temperatures, explaining 97.8% or more of the variation of X_{eq} when the temperature and water activity (a_w) are known. The water content showed a significant effect on the isosteric heat of sorption (q_{is}) calculated from sorption isotherms. The isosteric heat of sorption was found to be a one-term power function of the moisture content, representing an important increase of energy required to dry figs per unit of water extracted as the moisture content of the produce decreases during the drying process. These results are similar to those presented in the literature for similar types of produce.

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