

Sorption isotherms for oat flakes (*Avena sativa* L.)

Isotermas de adsorción para hojuelas de avena (*Avena sativa* L.)

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ABSTRACT

Moisture sorption isotherms of oat flakes were determined at temperatures of 5, 25 and 37°C, using a gravimetric technique in an a_w range of between 0.107 and 0.855. These curves were modeled using six equations commonly applied in food. The quality of the fit was assessed with the regression coefficient (r^2) and the mean relative percentage error (MRPE). The best fit were obtained with the Caurie model with r^2 of 0.996, 0.901 and 0.870, and MRPE of 7.190, 17.878 and 16.206, at 5, 25 and 37°C, respectively. The equilibrium moisture presented a dependence on temperature in the studied a_w range, as did the security moisture (X_s). These results suggest that the recommended storage conditions of oat flakes include: a relative air humidity of 50% between 5 and 25°C and of 38% up to 37°C.

Key words: water activity, simulation, diffusion models, storage.

RESUMEN

Se determinaron las isotermas de sorción de humedad de la avena en hojuelas a temperaturas de 5, 25 y 37°C, utilizando una técnica gravimétrica en el rango de a_w entre 0,107 y 0,855. Estas curvas se modelaron utilizando seis ecuaciones comúnmente aplicadas en alimentos. La calidad del ajuste se evaluó con el coeficiente de regresión (r^2) y el porcentaje de error medio relativo (PEMR). Los resultados mostraron que el modelo de Caurie fue el que mejor se ajustó a los datos experimentales, con r^2 de 0.996, 0.901 y 0.870, y %PEMR de 7.190, 17.878 y 16.206, para las temperaturas de 5, 25 y 37°C, respectivamente. La humedad de equilibrio presentó dependencia con la temperatura en el rango de a_w estudiado, al igual que la humedad de seguridad (X_s). Se obtuvo para este último parámetro valores de 0.053, 0.041 y 0.063 para 5, 25 y 37°C, respectivamente. Los resultados también permiten concluir que las condiciones de almacenamiento recomendadas para la avena en hojuelas son humedad relativa a 50% a 5 y 25°C, y de 38% para 37°C.

Palabras clave: actividad acuosa, simulación, modelos de difusión, almacenamiento.

Introduction

Oat (*Avena sativa* L.) is a cereal that belongs to the gramineae family, widely used worldwide for human consumption, animal food and cosmetic products (Gates, 2007). Compared with other cereals, oat is considered a source of proteins, carbohydrates, fiber, vitamins and minerals (Gates, 2007). It has components of minor proportion, such as unsaturated fat and β -glucans, which contribute to the health and well-being of human beings (Peterson, 2004).

Oat flakes are an important raw material in the food industry (Gates, 2007). One of the most important aspects concerning oat quality is its moisture content, since many of its physical properties depend on this property (Prieto *et al.*, 2006). But its water activity (a_w) is much more important, which indicates the water quantity in a food that may be used for developmental biochemical reactions (*i.e.*, lipid oxidation processes, enzyme reactions, Maillard reaction and microbial development) (Gálvez *et al.*, 2006).

Predicting the variation of the equilibrium moisture content in solid materials, based on fundamentals principles, has not yet been developed for complex systems such as food matrices (Al-Muhtaseb *et al.*, 2004). A common practice is to correlate the experimental data through mathematical models, which render satisfactory results in certain a_w ranges and certain food types (Zhang *et al.*, 1996). This adjustment is obtained by graphing the experimental moisture data in the product equilibrium at a pressure and temperature (Corzo and Fuentes, 2004) with the thermodynamic activity of water. The water activity in equilibrium equals the relative moisture of the air that surrounds it (Zhang *et al.*, 1996). The most used strategy for studies on sorption desorption phenomena is sorption isotherms, which are important in the analysis and design of several food processes and can be used to predict changes in food stability and to select appropriate packaging materials and ingredients (Zhang *et al.*, 1996). The sorption-desorption behavior, as well as the isosteric heat of food products and the influence of temperature over

Received for publication: 2 November, 2013. Accepted for publication: 19 March, 2014.

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the moisture content in equilibrium, provides very useful information for the processes of drying, preservation and conditioning of the food materials (Badui, 1993). Besides, it defines the storing conditions that guarantee food stability (Correa *et al.*, 2004).

Isothermic heat in the desorption processes represents the energy required for breaking inter-molecular forces between the water vapor and the adsorbent surface, while in sorption processes it corresponds to the energy delivered by the molecules as they are being adsorbed (Gálvez *et al.*, 2006; Rizvi, 1995).

Models that are available in the literature for describing moisture sorption isotherms can be divided into several categories: kinetic models based on a mono-layer (Mod-BET model), kinetic models based on a multi-layer and condensed film (Guggenheim, Anderson and de Boer – GAB, model), semi-empirical (Ferro-Fontan, Henderson and Halsey models) and empirical models (Smith and Oswin models) (Al-Muhtaseb *et al.*, 2004). The BET model represents a fundamental milestone in the interpretation of multi-layer sorption isotherms, particularly Types II and III (Timmermann, 1989). The GAB model is considered to be the most versatile sorption model available in the literature (Al-Muhtaseb *et al.*, 2004). The Smith model (Smith, 1947) is useful in describing the sorption isotherm of biological materials such as starch and cellulose. Henderson (1952) proposed a semi-empirical model for the equilibrium moisture content of cereal grains. The use of Caurie's equation is very important in modeling dehydrated food isotherms since it provides the parameter called "security moisture content" (X_s), which indicates the moisture content that results in the maximum stability of food during its storage (Gálvez *et al.*, 2006).

In more recent years, the GAB isotherm equation has been widely used to describe the sorption behavior of foods (Bizot, 1983; Weisser, 1985; Iglesias *et al.*, 1995). Having a reasonably small number of parameters (three), the GAB equation has been found to adequately represent the experimental data in the range of water activity of the most practical interest in foods, *i.e.*, 0.10-0.90 (Timmermann, 2001). The GAB equation has been recommended by the European Project Group COST 90 on Physical Properties of Foods (Wolf *et al.*, 1985) as the fundamental equation for the characterization of water sorption of food materials (Timmermann, 2001). This model has been used with protein, rice, turkey, chicken, tomato, potato starch, and wheat starch (Timmermann, 2001).

On the other hand, the Oswin model has been used with meat and fruits (Boquet *et al.*, 1978) and the Henderson

model with Starchy food, Proteins, Meats, and Fruits (Chirife *et al.*, 1978).

The purpose of this study was to experimentally determine the equilibrium moisture curves of oat flakes (*Avena sativa* L.) at three temperatures (5, 25 and 37°C) and to fit them by means of the mathematical models: GAB, BET, Oswin, Caurie, Smith and Henderson. Furthermore, the present study aimed to establish the best storage and packaging conditions for oat flakes.

Materials and methods

Determining sorption isotherms

Avena sativa oat flakes were provided by the Gelatinas y Refrescos de Colombia Company, located in Envigado (Colombia). The oat product (*Avena sativa* or *A. byzantine* L.) was previously cleaned, dried, stabilized, shelled, cut transversely and crushed to form flakes, scales or flakes, and had rounded, soft edges, uniform size, characteristic color, 15% moisture, 2.0% ash, and 1.8-2.0% crude fiber. The method consisted of putting a previously known mass of sample (triplicate) in tightly closed containers at a constant temperature (5, 25 and 37°C) with a saturated dilution of salt of known water activity (Tab. 1). From then on, the weight of the sample was controlled every 10 d until it achieved a constant weight (equilibrium condition). The containers with relative moisture solutions (%RH) higher than 75% had *Thymol* added to the structure supporting the sample in order to prevent developing microorganisms, especially fungi. Before doing the equilibrium moisture trials, the initial moisture of the oat flakes was determined by triplicate, following the methods of the AOAC No. 925.10, for which an analytic scale (Acculab LA-60, New Jersey), of 0.001 g precision, and a forced-convection heat drying chamber (Binder, Tuttlingen) were used.

Modeling of the isotherm sorption

The equilibrium moisture content on a dry basis was plotted against the a_w , to obtain the equilibrium moisture curves. The experimental data were fit to the mathematics models

TABLE 1. Salts and equations used to obtain a_w at a determined temperature (K).

Nomenclature	Sal	Regression equation	r^2
KCl	Potassium chloride	$\ln a_w = (367.58 \times 1/T) - 1.39$	0.97
MgCl ₂	Magnesium chloride	$\ln a_w = (176.9 \times 1/T) - 1.711$	0.99
NaCl	Sodium chloride	$\ln a_w = (228.92 \times 1/T) - 1.040$	0.96
MgNO ₃	Magnesium nitrate	$\ln a_w = (356.60 \times 1/T) - 1.82$	0.99
LiCl	Lithium chloride	$\ln a_w = (500.95 \times 1/T) - 3.85$	0.98

Source: Labuza *et al.* (1985).

TABLE 2. Models of sorption isotherms for equilibrium moisture.

Model	Equation	Parameters
GAB	$X_w = \frac{X_m C K a_w}{(1 - K a_w)(1 + (C-1) K a_w)}$	X_m , is the moisture of product corresponding the situation in which the point of primary adsorption are saturated by water molecules C, is the Guggenheim's constant, characteristic of the product and related to the adsorption heat of the single layer K, is the correction factor related to adsorption heat of the multilayer layer
BET	$X_w = \frac{X_m C a_w}{(1 - a_w)(1 + (C-1) a_w)}$	X_m , is the moisture of product corresponding to the monomolecular layer of the adsorbed water C, characteristic constant of the material related to the heat released in the sorption process
Henderson	$X_w = 0.01 \left[\frac{-\log(1 - a_w)}{10^f} \right]^{1/n}$	n y f , are characteristic parameter of the product
Caurie	$X_w = \exp[(a_w) \ln(V)] - \left[\frac{1}{4.5 X_s} \right]^{1/n}$	V, characteristic constant of the material X_s , security moisture content that give maximum stability to dehydrated food during the storage
Smith Oswin	$X_w = B + A \log(1 - a_w)$ $X_w = A \left[\frac{a_w}{1 - a_w} \right]^B$	A and B are constant of model and they are characteristics for each food

Source: Gálvez *et al.* (2006).

(Gálvez *et al.*, 2006): GAB (Guggenheim, Anderson and de Boer) (Ocampo, 2006), BET (Brunauer, Emmett and Teller), Henderson, Caurie, Smith and Oswin, which are shown in Tab. 2.

The quality of the fit of the proposed models was assessed with a linear correlation coefficient (r^2) and the percentage of the mean relative error (MRPE) (Eq. 1); both are statistic parameters widely used in food isotherms (Tolaba, 2004). The r^2 must be higher than 0.85 to get a good fit in the experimental data.

$$MRPE = \frac{100}{n} \sum_{i=1}^n \left(\frac{|X_{ei} - X_{ci}|}{X_{ci}} \right) \quad (1)$$

where,

X_{ei} , experimental moisture content (g water/g sample)

X_{ci} , estimated moisture content from every model (g water/g sample)

n , number of observations

Results and discussion

Equilibrium moisture content

The oat flake sample initial moisture was 0.137 ± 0.001 g water/g sample sorption isotherms for the working temperatures are shown in Fig. 1 (5, 25 and 37°C), which correspond to type II isotherms according to Vander Waals' classification (Debnath *et al.*, 2002). These types of isotherms are the most frequently found in foods such as

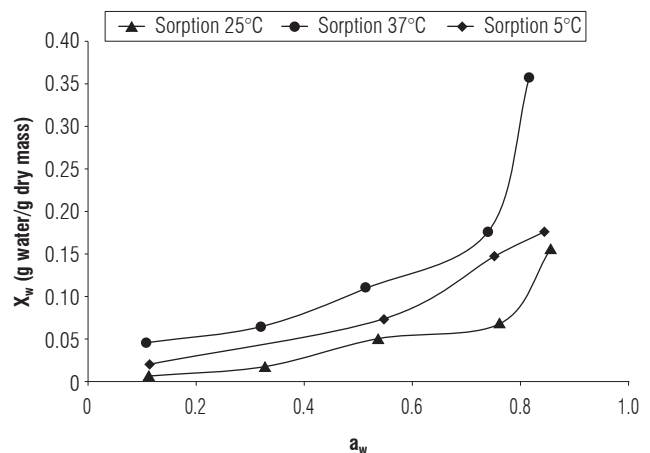


FIGURE 1. Experimental equilibrium moisture content of oat flakes for the working temperatures (5, 25 and 37°C).

fruits and vegetables (Martínez *et al.*, 1998; Kiranoudis *et al.*, 1993). According to Fig. 1, the equilibrium moisture curve slope above a particular value of a_w increases drastically for the three working temperatures. This behavior can be explained by the endothermic dissolution of sugars at raised values of a_w (Brett *et al.*, 2009).

Figure 1 shows that, between 5 and 25°C, as the temperature rose, the equilibrium moisture content for a given a_w decreased, which is a common phenomenon in different types of food (Johnson *et al.*, 2000; Gálvez *et al.*, 2006). However, at 25 and 37°C, for a given a_w , the equilibrium moisture increased with the temperature over the studied range of water activity, which has already been observed in other types of foods (Foster *et al.*, 2005).

In the first range of temperatures (5 to 25°C), the moisture decreased with increasing temperatures, indicating that the oat flakes became less hygroscopic. This fact was attributed to the higher energy levels and lower stability of water molecules as temperature increases, promoting their separation from the binding sites within the food matrix (Brett *et al.*, 2009).

The system behavior between 25 and 37°C has been widely studied in foods rich in carbohydrates, such as beets (Iglesias *et al.*, 1975) raisins (Saravacos *et al.*, 1986) barley malt (Barreiro *et al.*, 2003) and soy texturized proteins with a 20% sugar content (Cassini *et al.*, 2006). In order to explain this behavior, it must be considered that oat flakes have about 1% sugar, mainly constituted by starch (Aaman *et al.*, 1984; Brett *et al.*, 2009), which may associate them to a greater exposition of active sites or hydroxyl groups as both the a_w and the temperature increase (Brett *et al.*, 2009). Under such conditions, the molecules in the glass stage may approximate to the glass transition region and become plasticized, thereby increasing their mobility and availability for sorption (Brett *et al.*, 2009; Perdomo *et al.*, 2009). The role of the glass transition in starch-rich products and how it affects their sorption mechanism has been studied by Al-Muhtaseb *et al.* (2004) and Perdomo *et al.* (2009).

The results obtained in this study confirm the premise of Van den Berg (1981) in relation to the modification of the sorption capacity around the transition region where the amorphous region of starch begins to be plasticized, increasing their availability of hydroxyl groups for the sorption of water molecules (Brett *et al.*, 2009).

On the other hand, starch, quantitatively the main component of cereals such as oat, contains two polymers that contribute to its molecular structure: amylose, mainly a linear molecule, and amylopectin, a nonlinear and highly branched molecule (Damodaran *et al.*, 2007; Brett *et al.*, 2009). Both molecules form semicrystalline superstructures (with crystalline and amorphous layers arranged in an onion-like structure) where most of the crystalline regions are formed by amylopectin although part of the amylose molecules are also present in them (Zobel, 1988; Brett *et al.*, 2009). Within such a system, amorphous zones are more water-accessible than crystalline regions in which the intermolecular interactions between the chains are too strong to allow solvent penetration. Consequently, water influences the structure by acting as a plasticizer of the amorphous regions (Al-Muhtaseb *et al.*, 2004; Brett *et al.*, 2009). This effect causes a depression of the glass transition

temperature (T_g). The plasticizing effect is very small at low a_w values; therefore, the T_g associated with the amorphous regions in samples will be typically high and the material will be glassy at room temperature. Water access will be higher in the rubbery state (at $T > T_g$) than in the glassy state, since the free volume increases above T_g . Increasing water activity causes an increase in the availability of the polar sites to the water molecules (Al-Muhtaseb *et al.*, 2004; Brett *et al.*, 2009), such as shown in the curve for 37°C (Fig. 1).

Modeling isotherms

Table 4 presents a summary of the results obtained for the proposed models with their respective constants and r^2 and MRPE values. Based on the values of r^2 and MRPE, the equation proposed by Caurie was seen to be the most useful among those evaluated in this study. The Henderson model also presented a good behavior in respect to the r^2 but produced a high MRPE (superior to 19.8%). The GAB, BET and Smith models did not satisfactorily describe the behavior of the oat flake sorption isotherms. On the other hand, the GAB, Oswin and Smith models presented some very high MRPE while BET'S produced a very low r^2 . This result does not agree with Brett *et al.*, 2009, who reported a good fit with GAB and BET models to oat flour (Brett *et al.*, 2009), nor with Ayala (2011), who reported good fit in cassava flour with the GAB, Oswin and Smith models (Ayala, 2011), or with Gálvez *et al.* (2006), who observed good fit in corn flour with the GAB and Oswin models (Gálvez *et al.*, 2006).

In this study, (X_s) values equal to 0.053, 0.041 and 0.063 g water/g sample were obtained for working temperatures of 5, 25 and 37°C, respectively, with which the dependence of this parameter on temperature can be observed. These values correspond to X_s values that have been reported for foods rich in starch (Gálvez *et al.*, 2006; Montes *et al.*, 2009).

Figures 2, 3, and 4 present the experimental and predicted curves for the models at 5, 25 and 37°C, respectively, using the six proposed equations. Here, one can see that the curve predicted by the Caurie model is the best fit to the experimental data, consistent with those predicted by the r^2 and MRPE.

Additionally, based on the results from Fig. 1 and Tab. 4, the recommended storage conditions of oat flakes include: relative air humidity of 50% between 5 and 25°C and of 38% up to 37°C, which will prevent the deterioration of enzymatic reactions of carbohydrates, proteins, vitamins etc.

TABLE 4. Estimated values for the adjusted sorption models.

Model	Parameter	5°C	25°C	37°C
GAB	K	0.930	0.931	1.040
	C	3.621	-0.090	37.552
	X_m	0.073	-0.645	0.050
	r^2	0.955	0.833	0.892
	MRPE	51.858	420.395	9.482
BET	X_m	0.036	0.022	0.056
	C	10.350	2.936	11.764
	r^2	0.952	0.669	0.872
	MRPE	11.921	14.420	13.040
Henderson	N	1.2384	0.881	1.243
	F	-1.6554	-1.052	-1.910
	r^2	0.991	0.968	0.869
	MRPE	9.784	19.678	26.937

Model	Parameter	5°C	25°C	37°C
Caurie	V	22.094	53.517	14.820
	X_s	0.053	0.041	0.063
	r^2	0.999	0.966	0.939
	MRPE	5.752	17.878	16.206
Smith	A	-0.2335	-0.174	-0.412
	B	0.0063	-0.010	-0.001
	r^2	0.996	0.901	0.870
	MRPE	9.645	37.665	26.282
Oswin	A	0.734	0.034	0.118
	B	0.6128	0.804	0.542
	r^2	0.998	0.975	0.922
	MRPE	987.006	13.854	18.900

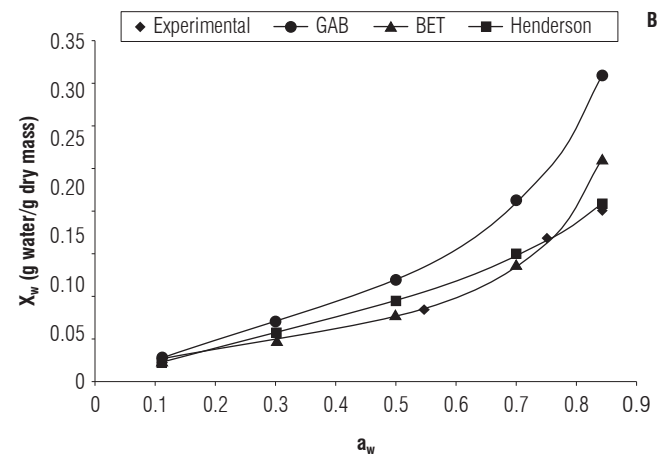
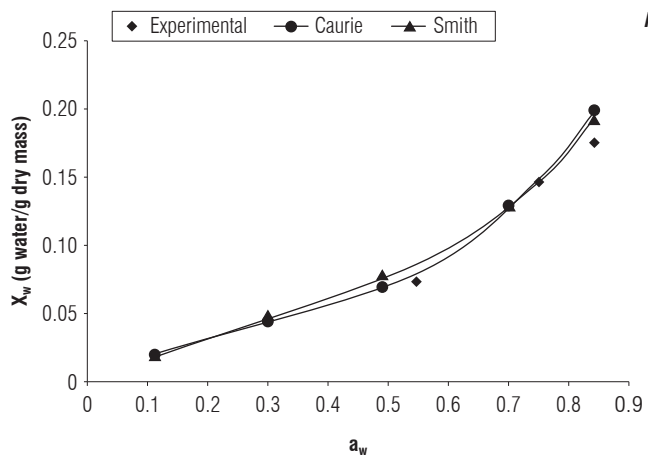


FIGURE 2. Modeling of oat flake sorption isotherms at 5°C.

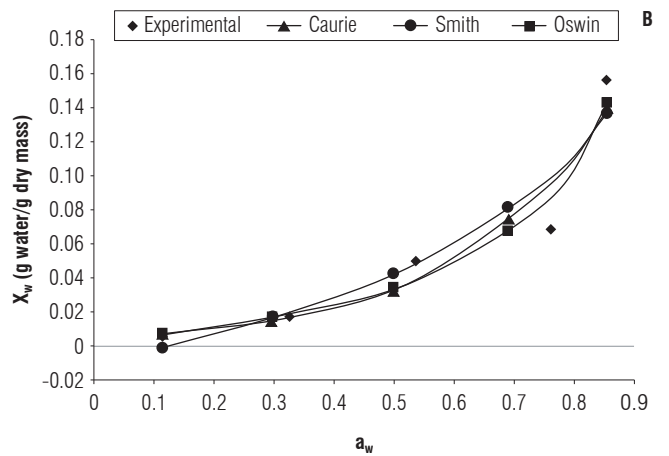
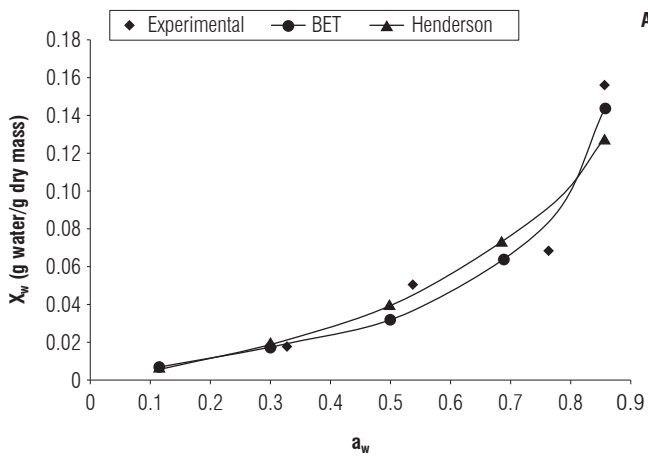


FIGURE 3. Modeling of oat flake sorption isotherms at 25°C.

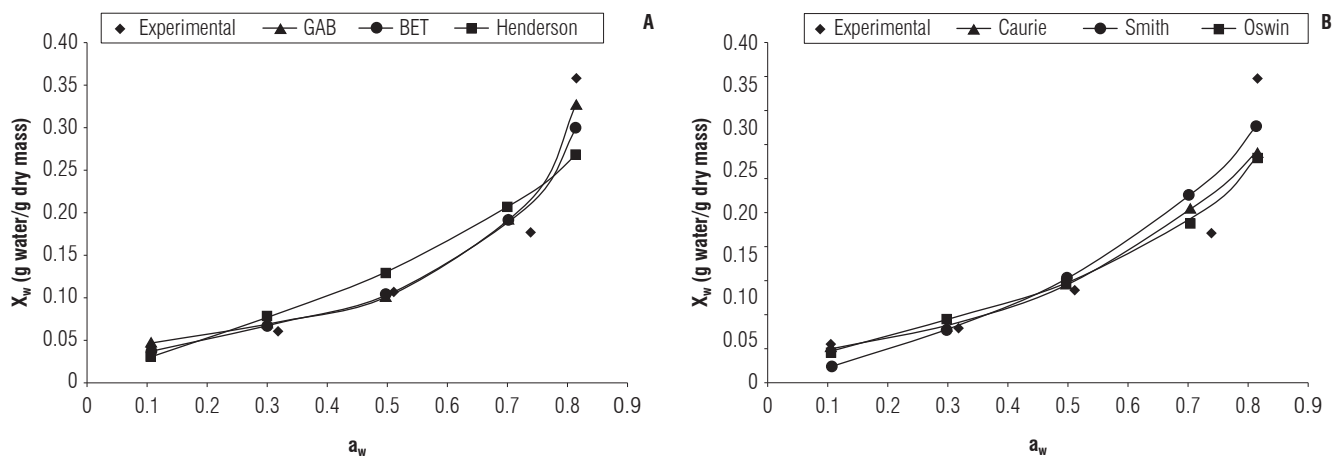


FIGURE 4. Modeling of oat flake sorption isotherms at 37°C.

Conclusions

The behavior of the equilibrium moisture of oat flakes at 5, 25, and 37°C may be satisfactorily described by means of the sorption isotherm Caurie model.

The oat flake moisture sorption isotherms showed a clear dependence on temperature for the working values in this study.

Between 5 and 25°C, the equilibrium moisture decreased as the temperature increased, but, between 25 and 37°C, the increases in temperature caused a rise in the equilibrium moisture.

The recommended storage conditions of oat flakes include: a relative air humidity of 50% between 5 and 25°C and of 38% up to 37°C.

Acknowledgements

This study had financial support from the Sustainability Program 2011/2012 of the Committee for Research Development (CODI) of the Universidad de Antioquia, Colombia.

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