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Determination of the spreading pressure and shelf-life of spray-dried coffee powder: quality assurance of a water-soluble food

Determinación de la presión de dispersión y vida útil del café en polvo secado por aspersión: aseguramiento de la calidad de un alimento soluble en agua

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Abstract

In this work, the values of monolayer (M_o) of the GAB model, generalized GAB model, and minimum change in spreading pressure (MCSP) of the adsorbed water were compared to determine the optimal storage conditions of spray-dried coffee powder. The effect of the adsorption models on estimating the water-vapor permeance of the packaging material and the product's shelf-life was also evaluated. For this, the powder was packed in a plastic film and stored at three temperatures and two relative humidities (RH). As a result, the best physicochemical stability of the powder was achieved at water contents corresponding to MCSP, from 0.01-0.04 g water/g dry solids. It was also observed that the most extended shelf-life was 246 days, obtained at 25 °C/75 HR with a permeance of 1.45 $\times 10^{-4}$ g/m^{2}dPa, and the shortest was 8 days at 45 °C/97 HR with a permeance of 4.2 $\times 10^{-4}$ g/m^{2}dPa. Additionally, it was determined that the degradation rate tripled ($Q_{10} \approx 3$) every 10 °C with activation energies greater than 90 kJ/mol. The results show that the spreading pressure contributes to ensure the quality of water-soluble foods.

Keywords: Spreading pressure, Shelf-life, Water-vapor permeance, Monolayer, Adsorption model.

Resumen

En este trabajo, se compararon los valores de monocapa (M_o) de los modelos de GAB, GAB generalizado y mínimo cambio en la presión de dispersión (MCSP) del agua adsorbida para determinar las condiciones óptimas de almacenamiento del café en polvo secado por aspersión. También se evaluó el efecto de los modelos de adsorción sobre la estimación de la permeancia del empaque al vapor de agua y vida de anaquel del producto. Para ello, el polvo fue empacado en una película plástica y almacenado a tres temperaturas y dos humedades relativas (HR). Como resultado, se logró la mejor estabilidad fisicoquímica del polvo en contenidos de agua correspondientes a MCSP, de 0.01 a 0.04 g agua/g sólidos secos. También se observó que la mayor vida de anaquel fue de 246 días obtenida a 25 °C/75 HR con una permeancia de 1.45 $\times 10^{-4}$ g/m^{2}dPa, y la más corta fue de 8 días a 45 °C/97 HR con una permeancia de 4.2$\times 10^{-4}$ g/m^{2}dPa. Adicionalmente, se determinó que la velocidad de degradación se triplicaba ($Q_{10} \approx 3$) cada 10 °C con energías de activación superior a los 90 kJ/mol. Los resultados muestran que la presión de dispersión contribuye a asegurar la calidad de alimentos hidrosolubles.

Palabras clave: Presión de dispersión, Vida de anaquel, Permeancia al vapor de agua, Monocapa, Modelo de adsorción.

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1 Introduction

Soluble coffee is one of the most popular products because of the ease for consumers to quickly prepare a cup by pouring hot water into the instant mixture. It is obtained mainly by drying the extract of the coffee bean using techniques such as spray-drying, freeze-drying, and conductive hydro drying (Cunha *et al*., 2016; Deotale *et al*., 2022). At the industrial level, soluble coffee is generally produced by spray-drying extracts in dryers with barrels and nozzle atomizers (Brennan, 2003). The spray-dried soluble coffee uses less energy to brew a cup and has a lower environmental footprint during its production than drip filter coffee or encapsulated espresso coffee (Humbert *et al*., 2009). In addition, habitual moderate consumption of soluble coffee has been related to a low incidence of cardiovascular damage (Tsirimiagkou *et al*., 2021). Also, it has been reported to be a source of antioxidants and bioactive compounds with immunomodulation properties and potential uses in bakeries due to sugar-free colored compounds (Capek *et al*., 2014; Passos *et al*., 2017).

Spray-dried soluble coffee is a highly hygroscopic product that can immediately adsorb moisture when exposed to the environment during storage, affecting its sensory and physiological properties. For this reason, soluble coffee is usually packaged in a glass jar or a metal can under vacuum with a resealable plastic lid with or without inert gas (Robertson, 2013), which could be expensive for its distribution, marketing, and recycling. Another approach to maintaining the shelf-life of soluble coffee is using flexible packaging since they have a low environmental impact compared to non-flexible packaging (Pauer *et al*., 2020). Previous studies have shown the effect of the film type on the shelf-life at 30 °C/80% HR, achieving 112 days of storage (Alves and Borlin, 1998; Nicoli *et al*., 2010). However, how this packaging affects the water adsorption in soluble coffee has not been fully understood.

Currently, the monolayer moisture content calculated from water sorption isotherms has been proposed as the optimal storage condition for soluble coffee. Villegas-Santiago *et al*. (2020) evaluated the effect of spray-drying conditions on the water vapor adsorption isotherms of soluble coffee. They employed the BET and GAB models to determine the monolayer and found it to be about 0.06 g water/g dry solids. Noguera-Córdoba and Rivero-Barrios (2021) examined the dynamic moisture sorption isotherms and thermodynamic properties of soluble coffee and, using the BET model, estimated values around 0.027 g water/g dry solids. The discrepancies in the monolayer values may be due to the limitations of the adsorption models. These estimates should be subject to experimental validation and comparison with other parameters, such as the minimum spreading pressure that occurs when the adsorbed molecules possess a minimum tendency to contract or spread on the surface of the adsorbent (Pascual-Pineda *et al*., 2020). The minimum spreading pressure coincides with the minimum integral entropy that, similar to the monolayer, is considered a key factor for predicting storage conditions and reducing lipid oxidation, as well as preserving flavor components and structural characteristics (Osorio-Téllez *et al*., 2021).

It is known that shelf-life simulations based on Fick and Henry's law depend on water vapor adsorption models. Lee and Robertson (2022) compared different adsorption models with respect to the packaged product's shelf-life and demonstrated that the linear and GAB models are the most suitable for predicting the shelf-life of a low-moisture packaged product. The GAB model is one of the most useful to describe the moisture adsorption isotherm up to water activities of 0.9 and provides information on the monolayer moisture content and the energetic interaction of the water with the surface of the food (Guadarrama-Lezama, *et al*., 2014). However, the GAB model fails to describe the isotherm at higher water activities for soluble coffee (Villegas-Santiago *et al*., 2020). A possible solution may be the generalized GAB model, whose modification assumes that the parameter of C is a polynomial function of a_w. In this context, the model could fit the adsorption data for water activities from 0 to 1 (Blahovec and Yanniotis, 2008, 2010), making it feasible to explore the effect of extreme relative humidities on the shelf-life of soluble coffee.

Simulation of the shelf-life of a food packaged in plastic films depends on several factors, including the a_w, temperature, humidity of the storage environment, and the permeance of the package to water vapor (Macedo *et al*., 2013). In general, studies are focused on simulation models to predict product shelf-life; nevertheless, these models could also be used as an alternative to determine the permeance of packing material. To date, few studies have combined the analysis of moisture-adsorption isotherms with the kinetics of changes in the moisture content of the packaged product to establish its optimal storage conditions and shelf-life (Sirpatrawan, 2009; Yu *et al*., 2013). In this sense the aim of this work is to: (1) analyze the water adsorption isotherms of spray-dried soluble coffee at different temperatures using the GAB model and the generalized GAB model, (2) evaluate the monolayer moisture content and minimum spreading pressure to predict the moisture content optimum storage of coffee powder, and (3) compare the GAB models against the linear approximation of the isotherm to determine the water vapor permeance of the packaging material, and shelf-life of the product packed.

2 Materials and methods

2.1 Materials

The 10-g soluble coffee sachet sticks (mixed soluble coffee, Premium Quality, Continental) utilized in this study were provided by Cafés Industrializados de Veracruz (CAFIVER, S.A, de C.V, Veracruz, Mexico). Soluble coffee was produced from natural coffee extract with 30% added sugars and spray-dried at 220°C. The sample's initial water activity and moisture content (M_i) were 0.258 ± 0.001 and 0.0326 ± 0.002 g water/g dry solids, respectively. The flexible package was composed of polyethylene terephthalate (PET), metallized PET, and low-density polyethylene (LDPE). The plastic film had a thickness of 56 μm, a width of 0.116 m, a length of 0.139 m, and an effective area of 0.010 m^2 in contact with the product.

2.2 Moisture-adsorption isotherms and GAB models

The adsorption of the water vapor of soluble coffee was determined by a gravimetric method at 25, 35, and 45 °C (Lang *et al*., 1981). Triplicate samples of approximately 1 g of powder were weighed and placed in desiccators with saturated salts with relative humidity values in the range of 11-97%. The required equilibrium time was 3-4 weeks based, on the change in the sample when the difference between two consecutive weightings was less than 0.001 g/g of dry solids.

The moisture-adsorption isotherm was modeled with the GAB and generalized GAB (GABg) equations in their polynomial forms (Blahovec and Yanniotis, 2008, 2010).

The GAB equation was expressed as a second-degree polynomial, Eq. (1):

$$\frac{a_w}{M} = a + ba_w + ca_w^2 \tag{1}$$

where:

$$a = \frac{1}{KM_oC} \tag{1a}$$

$$b = \frac{C-2}{M_oC} \tag{1b}$$

$$c = \frac{K(1-C)}{M_oC} \tag{1c}$$

where M is the moisture content (g water/g dry solids), M_o is monolayer moisture content (g water/g dry solids), a_w is the water activity, C is a dimensionless parameter related to heat of sorption of monolayer region, and K is related to heat of sorption of multilayer region.

The GABg model is based on the parameter C being a polynomial function of the a_w. In this manner, the GAB model can fit the adsorption data for water activities from 0 to 1.

The GABg equation was expressed as a fourth-degree polynomial, Eq. (2):

$$\frac{a_w}{M} = a' + b'a_w + c'a_w^2 + d'a_w^3 + e'a_w^4 \tag{2}$$

where:

$$a' = \frac{1}{K_gM_{og}C_o} \tag{2a}$$

$$b' = \frac{1}{M_{og}}\left(\frac{x_1}{K_gC_o} - \frac{2}{C_o} + 1\right) \tag{2b}$$

$$c' = \frac{1}{M_{og}}\left(\frac{K_g}{C_o} - \frac{2x_1}{C_o} + \frac{x_2}{K_gC_o} - K_g\right) \tag{2c}$$

$$d' = \frac{1}{M_{og}C_o}\left(K_gx_1 - 2x_2\right) \tag{2d}$$

$$e' = \frac{K_gx_2}{M_{og}C_o} \tag{2e}$$

The parameter K_g is obtained from the solution of the quartic equation, Eq. (3):

$$a'K_g^4 + b'K_g^3 + c'K_g^2 + d'K_g + e' = 0 \tag{3}$$

The parameter C_o is a function of a_w expressed in the following special polynomial form:

$$\frac{1}{C_o} = \frac{1}{C_g}(1 + x_1a_w + x_2a_w^2) \tag{4}$$

where: x_1, x_2, and C_o are empirical parameters of the polynomial in Eq. (4); C_o is a parameter related to heat of sorption of monolayer in function of a_w, C_g is a parameter related to the heat of sorption that the monolayer would have at a_w close to zero, M_{og} is monolayer moisture content (g water/g dry solids), and K_g is related to heat of sorption of multilayer region.

The parameters were estimated by non-linear regression using Kaleidagraph ver. 4.5 software (Synergy Software, Reading, PA, USA). Goodness-of-fit was evaluated using the coefficient of determination (R^2) and the mean-relative deviation modulus (E), Eq. (5) (Lomauro *et al*., 1985).

$$E\% = \frac{100}{N}\sum_{l=1}^{N}\frac{|M - M_{pl}|}{M} \tag{5}$$

where: M is the moisture-content experimental, M_{pl} is the predicted moisture content at the observation l, and N is the number of observations. It is generally assumed that a good fit is obtained when E <10%.

2.3 Minimum change in spreading pressure

The optimal moisture content for storing coffee powder was determined from the minimum change in the slope of spreading pressure (π) concerning to a_w, or minimum change in spreading pressure (MCSP), using Eq. (6) (Pascual-Pineda *et al*., 2021).

$$\frac{d\pi}{da_w} = \frac{RT}{M_wS_B}\left(\frac{M}{a_w}\right) \tag{6}$$

where π is the spreading pressure (J/m^2), S_B is the specific surface area of GAB (m^2/g dry solids) at 25 °C, M_w is the molecular weight of water (18.015 g/mol), R is the gas constant (8.314 J/mol K), and T is the absolute temperature (K).

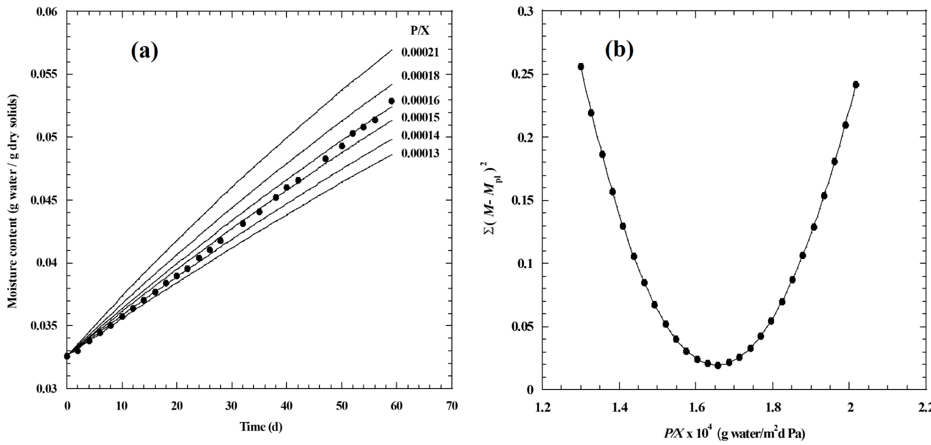

Fig. 1. Numerical solution of the permeation equation for moisture transport through a thin film using the GAB model. Numerical solution of the Eq. (9) at different permeance values (a), and the minimum point of the sum of squares (b).

2.4 Shelf-life assessment

Eighteen, 10-g sticks of freshly packed soluble coffee, with an initial moisture content of 0.0326 ± 0.002 g water/g dry solids, were weighed and placed in conditioned chambers with solutions saturated with sodium chloride (~75% RH) and potassium sulfate (~97% RH) and incubated at 25, 35, and 45 °C. The samples were taken from each chamber at 2-days intervals until the critical moisture content (M_c) was reached. Each stick was weighed and returned to the chamber to continue its shelf-life assessment. Weights of samples as a function of time were measured, and the moisture content was obtained by a material balance, which consisted of dividing the increases in water mass by the dry mass and adding this to the initial moisture content of the product. Shelf-life (t_s) was defined as the time necessary for the product to reach the M_c.

The Q_{10} parameter that decreases shelf-life for a 10 °C increase of temperature, was calculated using the Eq. (7) (Labuza, 1984).

$$\ln Q_{10} = 10B = \frac{10E_A}{R(T)(T+10)} \tag{7}$$

where: B was obtained by Eq. (8).

$$\ln t_s = \ln t_{s0} - BT \tag{8}$$

where: t_{s0} is the shelf-life at 0 °C, and E_A is the activation energy (J/mol K) related to product degradation.

2.5 Shelf-life model and permeance of packaging film

The shelf-life of the soluble coffee was assumed to be controlled by the water-vapor diffusion through the packaging film. Therefore, the mass balance and permeation of the package system are described in Eq. (9).

$$\frac{dM}{dt} = \frac{P}{X}\frac{A}{W_s}P^o(a_{wext} - a_{wint}) \tag{9}$$

where P/X is the permeance (the permeability constant P divided by the thickness of the film X) (g water/m^{2}dPa), dM/dt is the rate of moisture (g water) per unit dry weight (g dry solids) transferred per day (d), A is the surface area of the package (m^2), P^o is the saturation vapor-pressure (Pa), W_s is the food's total dry weight in the package (g dry solids), a_{wext} is the water activity equilibrated at an external relative-humidity (RH) condition, and a_{wint} is the water activity of the food that is equal to the RH inside the package.

The GAB models (Eqs. (1) and (2)) were used to establish the relationship between a_{wint} and the moisture content gained by the soluble coffee stick. The a_{wint} was calculated by the Newton-Rapson method within the a_w range-of-interest determined by the product's initial (M_i) and critical (M_c) moisture content. Subsequently, the differential Eq. (9) was solved numerically by the fourth-order Runge-Kutta method (Kiusalaas, 2013) using different permeance values. Fig. 1a illustrates the procedure mentioned previously, where a simulation of the change in water content is obtained at each permeance value of the plastic film. Subsequently, each simulated curve was compared with the experimental data to find the minimum sum-of-squares value (Fig. 1b). This work considered that the water-vapor permeance of the plastic film corresponded to the closest prediction of the experimental kinetic data.

On the other hand, the isotherm was also treated as a linear function of a_w:

$$M = b'' a_w + c'' \tag{10}$$

where: b'' is the slope of the isotherm and c'' is the intercept.

On rearranging and defining a_w:

$$a_w = \frac{M - c''}{b''} \tag{11}$$

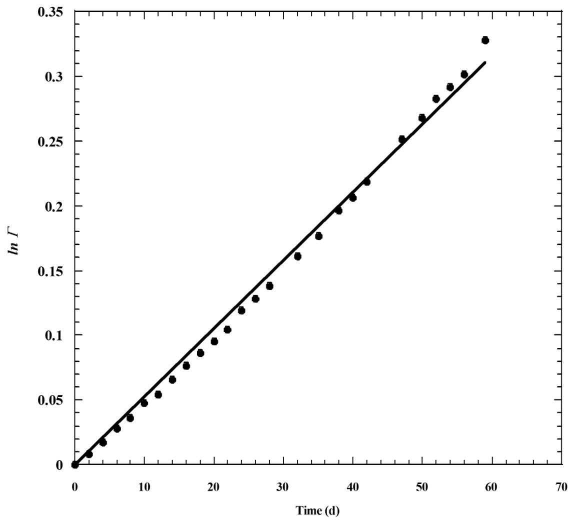

Fig. 2. Analytical solution of the permeation equation for moisture transport through a thin film using a linear approximation of the isotherm.

Substituting the above relationship in Eq. (9) and integrating, the following analytical solution is obtained:

$$\ln \Gamma = \Phi t \quad (12)$$

where:

$$\Gamma = \ln \frac{M_e - M_i}{M_e - M} \quad (12a)$$

$$\Phi = \frac{P}{X} \frac{A}{W_s} \frac{P^o}{b''} \quad (12b)$$

where Γ is the unaccomplished moisture fraction, Φ is the overall permeance of the system, M_e is the equilibrium moisture content of the food at a_{wext}, and t is the storage time. Fig. 2 depicts an example of the analytical solution (Eq. (12)) from which the plastic film's water-vapor permeance was also calculated using the values of Φ in combination with b'' values.

An apparent activation energy (E_p) (J/mol K) was calculated for each relative humidity condition from the water-vapor permeance using Eq. (13).

$$\ln\left(\frac{P}{X}\right) = \ln\left(\frac{P}{X}\right)_0 - \frac{E_p}{RT} \quad (13)$$

Estimations were performed by Python (ver. 3.7.9), Pandas (ver. 1.3.3), and SciPy. Optimize (ver. 1.7.1) software.

2.6 *Statistical analysis*

A full factorial design was used to determine significant effects produced by the relative humidities and temperature of storage on the permeance of the packaging film. Statistical calculations were carried out using the Design Expert ver. 8 software program. Analysis of variance and the Fisher minimum significant difference (LSD) method were utilized to define the significance of the results at $p < 0.05$.

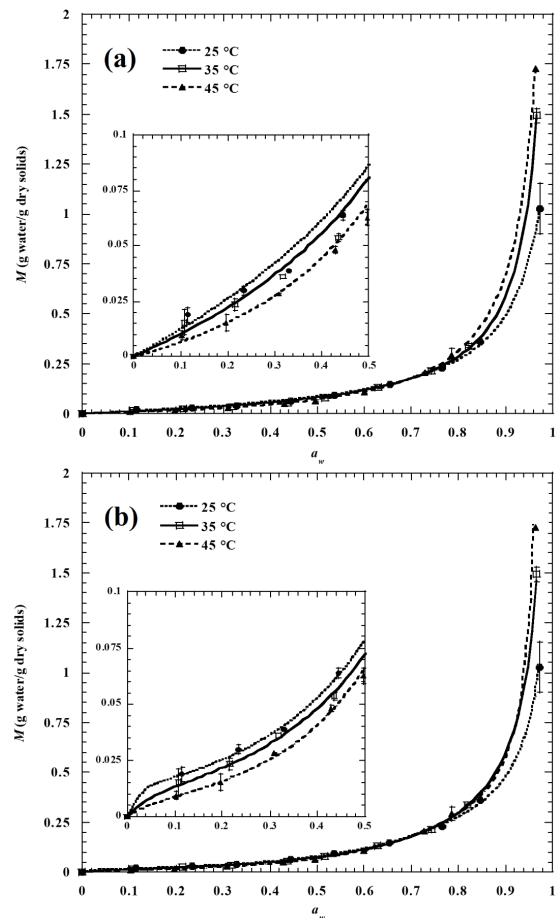

Fig. 3. Moisture adsorption isotherms of soluble coffee at 25, 35 and 45 °C. Symbols (●, □, ▲) are the experimental points, and the lines are the fitted points using the GAB model (a), and generalized GAB model (b).

3 Results and discussion

3.1 *Adsorption isotherms*

The water vapor-adsorption isotherms of soluble coffee at different temperatures are presented in Fig. 3. The isotherms of soluble coffee show that the moisture content increased exponentially with the increasing a_w following the form of a type-III isotherm, according to the Brunauer's classification (Brunauer *et al*., 1938). Type-III isotherms are typical of food products rich in soluble components, such as powdered honey (Mutlu *et al*., 2020), the borojó fruit (Rodríguez-Bernal *et al*., 2015), roasted coffee beans (Collazos-Escobar *et al*., 2019), and soluble coffee (Noguera-Córdoba

Table 1. Parameters obtained from the adjustment of the GAB models for spray-dried soluble coffee adsorption data.

Temperature (°C)	GAB					GABg						
	M_o (g water / g dry solids)	C	K	R^2	E (%)	M_{og} (g water / g dry solids)	C_g	K_g	x_1	x_2	R^2	E(%)
25	0.076	1.646	0.955	0.998	8.227	0.05	32.472	0.973	78.435	-132.261	0.993	2.461
35	0.073	1.329	0.985	0.999	8.22	0.063	4.651	0.988	13.535	-18.028	0.997	2.939
45	0.097	0.601	0.984	0.999	5.72	0.043	3.520	1.012	9.662	-18.834	0.993	3.998

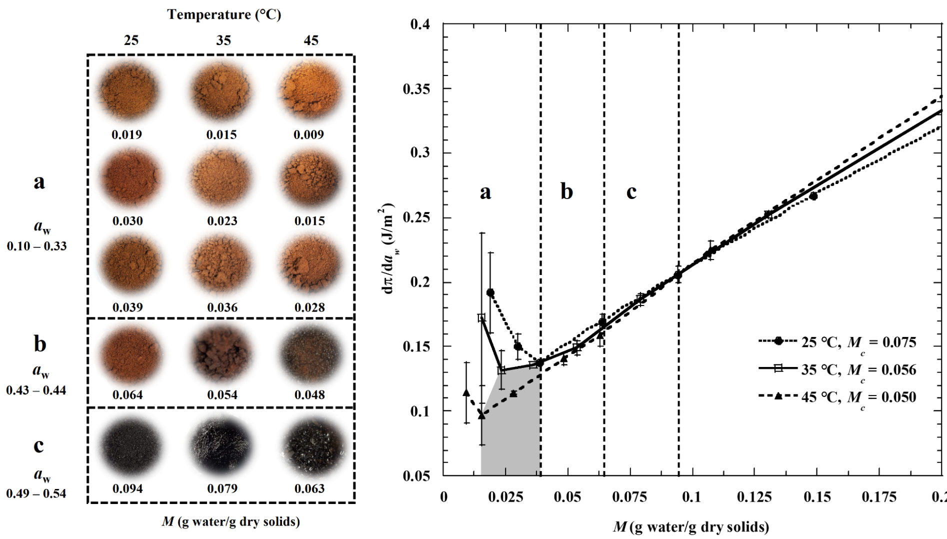

Fig. 4. Effect of the water content and temperature on the visual appearance of soluble coffee after 70 days of storage and change in spreading pressure, ($d\pi/da_w$), at 25, 35, and 45 °C. The intervals of a_w corresponding to the water contents are: 0.1-0.33 (a), 0.43-0.44 (b), and 0.49-0.54 (c).

and Rivero-Barrios, 2021). The isotherms of soluble coffee demonstrated that, at a_w below 0.6, the moisture content increased with a decreasing storage temperature due to the adsorption process' exothermic nature. The decrease in temperature causes the water molecules to bind the adsorption sites with greater energy and in more significant quantities, causing an increase in moisture content (Collazos-Escobar *et al*., 2022). Subsequently, as the water adsorption progressed to a_w above 0.6, it was observed that the increase in temperature led to higher moisture contents with the concomitant solubilization of the coffee powder. This effect of temperature has been reported for foods rich in sugars or for low-molecular-weight compounds, such as amaranth-sorghum grains (Gichau *et al*., 2020), roasted coffee beans (Collazos-Escobar *et al*., 2022), pistachio-nut paste (Hayoglu and Gamli, 2007), and for chestnut and wheat flours (Moreira *et al*., 2010).

The adsorption data were fitted to the GAB models, and the estimated parameters are presented in Table 1. The GAB and GABg models had a good quality-of-fit since the R^2 values were close to unity and the percentages of E were less than 10 (Lomauro *et al*., 1985). The lowest values of E showed that the GABg model gives the best fit (Fig. 3b) in the entire studied range of a_w compared to the GAB model (Fig. 3a). This is because the GABg model considers that parameter $1/C_o$ changes with the a_w (Eq. (4)), increasing with the positive values of x_1 and decreasing with the negative values of x_2 (Table 1). This makes sense in that the surface or structure of powder changes as water is adsorbed and dilutes some parts of the coffee solids. The parameters C and C_g are related to the heat of sorption of the monolayer and are of an enthalpic nature that decreases with an increasing temperature. For the GAB model, the values of C were less than 2, indicating that the water-water interaction controls the water adsorption, causing multilayers of water to build on the powder surface while the monolayer is still incomplete (Gregg and Sing, 1982). The multilayer adsorption is also suggested by K and K_g values close to unity, implying that multilayers behave like liquid water with the ability to dissolve the water-soluble food components (Rodríguez-Bernal *et al*., 2015).

The monolayer moisture content, M_o, of the GAB model was between 0.073-0.097 g water/g dry solids and resulted higher than the M_{og} of the GABg model (0.043-0.063 g water/g dry solids) (Table 1). Comparing the fits of the GAB models in Fig. 3, it can be observed that the generalized GABg model precisely fitted the isotherm's concave region at a low a_w, immediately before the inflection point, which is a requirement for a reasonable estimation of the monolayer capacity of a solid adsorbent (Brunauer *et al*., 1938; Gregg

Table 2. Linear model parameters obtained by linear regression of spray-dried soluble coffee adsorption data.

Temperature (°C)	b'' (g water /g dry solids)	c'' (g water /g dry solids)	R^2	M_e (g water/g dry solids) at 75% RH	M_e (g water/g dry solids) at 97% RH
25	0.213	-0.0255	0.952	0.137	0.188
35	0.1787	-0.0182	0.953	0.114	0.154
45	0.183	-0.0289	0.995	0.104	0.147

Table 3. Water vapor permeances (P/X) (g/ m^2 d Pa) ($\times 10^4$) values obtained from Eq. 9 in combination with the GAB models and from the linear model of the isotherm.

Temperature (°C)	Approximate external RH (%)							
	75				97			
	RH (%)	Linear	GAB	GABg	RH (%)	Linear	GAB	GABg
25	76.5	1.45 ± 0.03^{a}	1.38 ± 0.03^{a}	1.50 ± 0.03^{b}	97.3	2.14 ± 0.07^{g}	2.17 ± 0.07^{g}, m	$2.31 \pm 0.07^{h,o}$
35	74.3	$1.65 \pm 0.10^{c,d}$	$1.59 \pm 0.10^{b,c}$	1.74 ± 0.11^{d}	96.7	$2.50 \pm 0.05^{i,j}$	2.45 ± 0.02^{i}	2.60 ± 0.02^{j}
45	72.7	$2.30 \pm 0.04^{e,f}$	$2.27 \pm 0.03^{e,m}$	$2.39 \pm 0.04^{f,o}$	96.2	$4.25 \pm 0.11^{k,l}$	4.11 ± 0.09^{k}	4.23 ± 0.10^{l}

Mean values ± standard deviation ($n = 3$) with different letters between rows denote significant differences between temperatures ($P < 0.05$). Different letters between columns denote significant differences between models ($P < 0.05$).

and Sing, 1982). The monolayer value provides information on the amount of water strongly adsorbed to active sites, such as ionic, polar groups, or micropores in the food matrix (Flores-Andrade *et al*., 2017; Iglesias *et al*., 2022; Pavón-García *et al*., 2015). Nevertheless, the fit of the GAB models may be affected by sample dissolution at an a_w greater than 0.6, thus the estimation of the monolayer could be imprecise. Therefore, to corroborate this estimate, the minimum change in spreading pressure regarding a_w (MCSP) was determined, which has been shown to coincide with the monolayer value (Pascual-Pineda *et al*., 2021).

3.2 Minimum change in spreading pressure

The MCSP of the spray-dried soluble coffee at 25, 35, and 45 °C are listed in Fig. 4. The plot reveals the change $d\pi/da_w$ as a function of the moisture content of soluble coffee. The minimum value of $d\pi/da_w$ (MCSP) is reached when the spreading-pressure isotherm increases linearly with increasing a_w (data not shown), meaning that π is less sensitive to changes in the relative humidity of the environment. At this point, the water transfer from the vapor state to the solid surface is carried out with low free energy and; thus, optimal food stability is obtained (Pavón-García *et al*., 2015; Rockland, 1969). For soluble coffee, the MCSP was within the range of 0.015-0.039 g water/g dry solids (gray zone in Fig. 4) and indicated the condensed state of the water adsorbed on the surface of the food. In this state, the water molecules were more ordered, with low mobility and strong water interactions with the solid surface, rendering it less available to participate in deterioration reactions (Osorio-Téllez *et al*., 2021). The MCSP values were similar to the monolayer values reported for freeze-dried, spray-dried, and ground coffee products, with a range of 0.019-0.045 g water/g dry solids (Noguera-Córdoba and Rivero-Barrios, 2021; Villegas-Santiago *et al*., 2020).

The monolayer and MCSP values, as optimal water-storage contents, were experimentally supported by the visual appearance of soluble coffee. Fig. 4 reveals the visual appearance of soluble coffee at different intervals of a_w and storage temperatures. The a-frame corresponds to the conditions of MCSP and encompasses the best form of soluble coffee with moisture contents of 0.009 to 0.039 g water/g dry solids (a_w of 0.1-0.33). These values were lower than those estimated by the GAB models (Table 1). However, this result agreed with that reported by Harris *et al*. (1974) to preserve the color, odor, flavor, or appearance of lyophilized soluble coffee packed in hermetically sealed cans and stored for one year at 21°C/20% RH. The b-frame shows that at 35 and 45 °C, the incipient solubilization of the coffee powder begins with moisture contents of 0.048 and 0.054 g water/g dry solids (a_w of 0.43-0.44), and the c-frame indicates the conditions for a complete dissolution of the solids, with moisture contents of 0.063-0.094 g water/g solids (a_w of 0.49-0.54). The beginning of the c-frame was taken as the critical moisture content (M_c) of the coffee powder, which was 0.075, 0.056, and 0.050 g water/g dry solids at 25, 35, and 45 °C, respectively. Based on the previous results, it can be observed in Table 1 that the GAB model overestimated the M_o values, because they fall under the conditions of complete dissolution (c-frame in Fig. 4). In contrast, the M_{og} values of the generalized GABg model were better estimated, but still fell very near to incipient solubilization (b-frame in Fig. 4). Consequently, the optimal moisture content for storing the soluble coffee corresponded to MCSP values of 0.015-0.039 g water/g dry solids; therefore, the soluble coffee was produced with a moisture content of 0.032 g water/g solids and was immediately packed inside a plastic film for its shelf-life study.

3.3 Water-vapor permeance

Water-vapor permeance was determined from the permeation equation (Eq. (9)), combined with changes in the moisture content of the product packaged in the plastic film during storage and the moisture-adsorption isotherms of the soluble coffee. Table 2 shows the isotherm's linear model parameters in the interval ranging from M_i to M_c. The M_e values were used to obtain the analytical solution (Eq. (12)) of the permeation equation, and the values of the slope of the isotherm (b'') were employed to calculate the water-vapor permeance. The linear model parameters were similar to those reported for onion flakes, green beans, rice cakes, and dry tea (Lee and Robertson, 2022; Robertson and Lee, 2021). Table 3 presents the water-vapor permeances (P/X) values under different storage conditions, calculated using the linear and GAB model of the isotherm. In general, the permeance values estimated with the linear model were not significantly different from the GAB models. The water-vapor-permeances values of the packaging material were lower than those reported for PET/LDPE laminates, of 7-11×10^{-4} g/ m^2dPa in a range of 20-40 °C and 55-90% RH (Samaniego-Esguerra and Robertson, 1991), and similar to those pf PETmet/LDPE films, with values around 1.8-2.64 ×10^{-4} g/ m^2dPa at 38 °C/90% RH (Alves and Bordin, 1998; Marangoni *et al*., 2018). Water-vapor permeances are dependent on storage temperature and relative humidity. As the RH increased, the water permeance increased. The same tendency was observed for storage temperatures ranging from 25 to 45 °C. The relationship between the ln(P/X), and the inverse of temperature showed straight lines. At 75% RH, the Arrhenius parameters were of $\ln(P/X)_0 = -1.535$, E_p = 18.18 kJ/mol, and R^2 = 0.968; while at 97% RH, these were of $\ln(P/X)_0$ = 2.205, E_p = 26.53 kJ/mol, and R^2 = 0.905. The apparent activation energies of the permeance, E_p, increased with the RH due to the partial pressure difference between the interior and exterior of the packing. This behavior can be attributed to that the water molecules outside of packing required more energy to be transported through the film. The high E_p values were probably because the flexible packing employed has a dense structure with a low fractional free volume, which represents the amount of free space between the polymer chains that are available for gas diffusion. The E_p values were in the same order as those reported for the permeation of nitrogen and methane in a glassy polymer with a low free volume fraction (Yampolskii *et al*., 1998). In addition, the activation energies obtained in the present study were similar to those published by Kulchan *et al*. (2010), of 21-22 kJ/mol, for LDPE and OPP films, and higher than that reported by Samaniego-Esguerra and Robertson (1991), of 11-12 kJ/mol, for PET/LDPE laminates.

The water-vapor-permeance values were utilized to simulate changes in the moisture content of the product packaged during storage, and these were compared with experimental kinetic data. In Fig. 5, the dotted lines were drawn using the linear sorption model, and the solid lines were obtained employing the GAB models. The predictions of the three models coincided with each other; therefore, the complexity of the GAB models did not contribute to yielding perceptible differences in the simulations of moisture-content gain. In general, the moisture content increased linearly with storage time, demonstrating the water-vapor migration from the environment into the package. At the same temperatures, in the samples stored at 75% RH, the water-vapor migrations were slower than at 97% RH, due to that a low relative humidity provides a minor moisture gradient between the inside of the sticks and the storage environment. On the other hand, the increase in storage temperature increased the moisture gain inside the package, which is attributed to the fact that it relaxes the polymers of the packaging material, giving rise to more spaces, which increase permeation (Siracusa, 2012). The same trends have been reported for the kinetics of increasing the moisture content in onions flakes and crackers packaged in LDPE and CPP films, respectively (Hao *et al*., 2016; Robertson and Lee, 2021).

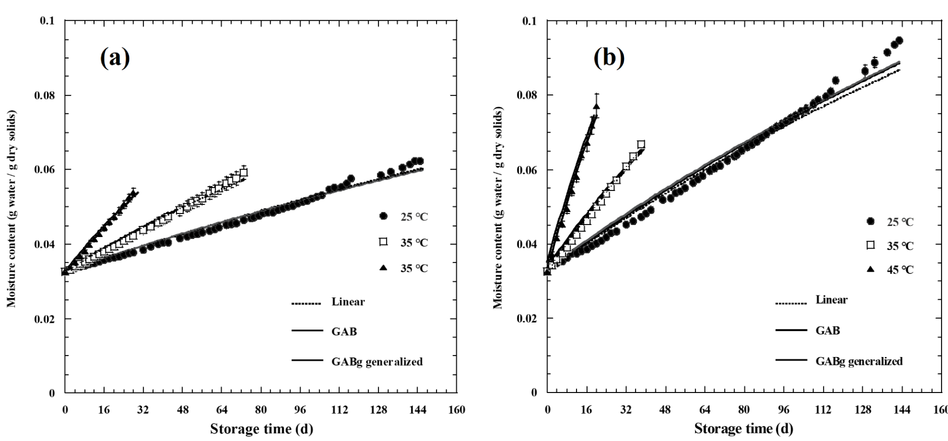

Fig. 5. Changes in moisture content of the soluble coffee packaged in a plastic film during storage at 25, 35, and 45 °C with an approximation relative humidity of 75% (a) and 97 % (b). Symbols (●, □, ▲) are the experimental points, the solid lines are based on the GAB models, and dotted lines are based on a linear model.

Table 4. Simulated and actual shelf life in days for spray-dried soluble coffee packed in the plastic film and stored at different temperatures and relatives humidities to reach critical moisture content.

Temperature (°C)	M_c (g water / g dry solids)	Approximate external RH (%) [a]							
		75				97			
		Experimental	Linear	GAB	GABg	Experimental	Linear	GAB	GABg
25	0.075	243 ± 5	246	250	253	138 ± 2	107	103	103
35	0.056	70 ± 2	68	69	69	32 ± 1	27	27	27
45	0.050	24 ± 1	24	24	24	10 ± 1	8	8	8

[a]See Table 3 for actual values.

Table 5. Parameters of the equation for the shelf-life plot of spray-dried soluble coffee packed in a plastic film.

Approximate external RH (%)[a]	$\ln t_{s0}$	B	Q_{10}	E_a (kJ/mol)	R^2
75	39.975	0.116	3.182	91.334	0.998
97	44.005	0.131	3.715	103.589	0.996

[a]See Table 3 for actual values.

3.4 *Shelf-life determination*

The times to reach the critical moisture content (M_c) of packaged soluble coffee have already been given as the shelf-life in Table 4. As can be observed, the linear and GAB models predicted practically the same shelf-life under the same storage conditions. The best shelf-life estimates were presented at 75% RH, in that since they agreed with the experimental data. However, a slight overestimation was observed at 25 °C/75% RH, being 3% for GAB and 4% for the GABg generalized. In contrast, at 97% RH, all models underestimated the experimental data by 10-22%, which may be due to the transfer of water between the package headspace and the soluble coffee beginning to generate significant internal resistance (Delgado and Da-Silva, 2010; Hao *et al*., 2016; Taoukis *et al*., 1988). Although the permeation model requires considering the diffusion of water vapor inside the package, it still approximates shelf-life under extreme relative-humidity conditions. Macedo *et al*. (2013) found that the shelf-life of granola packed in BOPP films could be satisfactorily predicted even with a 20% underestimation. In contrast, Sirpatrawan (2009) simulated the shelf-life of rice crackers packed in PE and PP films, and found that the simulation model could not be applied with overestimates greater than 30%. Based on the latter, the linear model of the isotherm and the analytical solution (Eq. (12)) of the permeation equation resulted in being to be the simplest for estimating the shelf-life of soluble coffee.

On the other hand, the dependence of shelf-life on temperature was expressed in terms of the Q_{10} and E_a values at 75 and 97% RH (Table 5). At 97% RH, the E_a was 103.58 kJ/mol and was higher than at 75%, with E_a of 91.33 kJ/mol. The increase in activation energy implies that the shelf-life of soluble coffee, stored at high relative humidity, decreases considerably with increasing temperature (Manzocco *et al*., 2019). At 97% RH, the deterioration of soluble coffee nearly quadrupled (Q_{10} =3.7) for every 10 degrees Celsius, while at 75%, it was tripled (Q_{10} =3.1). In this study, the Q_{10} values were obtained under extreme conditions of temperature and relative humidity, and they were higher than usually reported, $Q_{10} \approx 1.2$, for food products packed in glass containers, such as roasted and ground coffee (Cardelli and Labuza, 2001), vitamin C in emulsions (Al-Haushey and Moussa, 2015), and for green tea packed in aluminum foil with a zip-lock vacuum (Xiao *et al*., 2022).

Conclusions

Moisture-sorption isotherms of spray-dried soluble coffee powder showed a curve typical of type III BET classification shape. In contrast to GAB model, the generalized GAB model described the adsorption isotherm of soluble coffee up to water activities of 0.97 and accurately estimated the water content of the monolayer. The minimum change in the spreading pressure (MCSP) of the adsorbed water proved to be an adequate parameter to establish the optimum water content for packing the soluble coffee from 0.015 to 0.03. The water-vapor permeance of the packaging material was estimated from the steady-state permeation equation, utilizing the moisture-adsorption isotherms, and comparing it with the experimental moisture-adsorption kinetics of the packaged product. The linear function of the isotherm yielded the same estimate of the packaged product's water-vapor permeance and shelf-life compared to the GAB model. Under accelerated conditions at 75% RH, the soluble coffee's predicted shelf-life was 246, 68, and 24 days at 25, 35, and 45 °C, respectively, showing a good agreement with the experimental shelf-life. Similarly, at 97% RH, a good approximation of shelf-life was obtained for 107, 27, and 8 days at 25, 35, and 45 °C, respectively. These results demonstrated that no mathematical complexity is required in describing the water-vapor adsorption isotherm to effectively estimate the shelf-life of soluble products under different storage temperatures and relative humidity conditions. This is a practical benefit in terms of simplifying the calculation process for determining the shelf-life of low moisture foods packaged in plastic films.

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Nomenclature

A	surface area of the package (m^2)
a_w	water activity
a_{wext}	water activity equilibrated at an external relative humidity
a_{wint}	water activity of the food which is equal to the relative humidity inside the package
a	constant parameter in the quadratic polynomial expression of the a_w/M ratio (Eq. (1))
a'	constant parameter in the biquadratic polynomial expression of the a_w/M ratio (Eq. (2))
B	slope in the linear expression of $\ln t_s$ (Eq. (8))
BOPP	Bi-axially oriented polypropylene
b	parameter at linear member in the quadratic polynomial expression of the a_w/M ratio (Eq. (1))
b'	parameter at linear member in the biquadratic polynomial expression of the a_w/M ratio (Eq. (2))
b''	slope in the linear model of the isotherm (Eq. (10)) (g water/g dry solids)
C	constant related to the heat of sorption of monolayer of the GAB model
C_o	constant related to the heat of sorption of monolayer of the generalized GAB model
C_g	constant related to the heat of sorption of monolayer of the generalized GAB model at $a_w \approx 0$
CPP	Cast polypropylene
c	parameter at quadratic member in the quadratic polynomial expression of the a_w/M ratio (Eq. (1))
c'	parameter at quadratic member in the biquadratic polynomial expression of the a_w/M ratio (Eq. (2))
c''	intersection in the linear model of the isotherm (Eq. (10)) (g water/g dry solids)
d'	parameter at cubic member in the biquadratic polynomial expression of the a_w/M ratio (Eq. (2))
E	mean relative-deviation modulus (%)
E_A	activation energy related to product degradation (kJ/mol)
E_p	activation energy of permeation (kJ/mol)
e'	parameter at biquadratic member in the polynomial expression of the a_w/M ratio (Eq. (2))
K	constant related to the heat of sorption of multilayer of the GAB model
K_g	constant related to the heat of sorption of multilayer of the generalized GAB model
l	index or observation
LDPE	Low density polyethylene
M	moisture content (g water/g dry solids)
M_c	critical moisture content (g water/g dry solids)
M_e	equilibrium moisture content of the food at a_{wext} (g water/g dry solids)
M_i	initial moisture content of the sample (g water/g dry solids)
M_o	monolayer moisture content of the GAB model (g water/g dry solids)
M_{og}	monolayer moisture content of the generalized GAB model (g water/g dry solids)
M_{pl}	predicted equilibrium moisture content at the observation l (g water/g dry solids)
M_w	molecular weight of water (18.015 g/mol)
N	number of observations
OPP	Oriented polypropylene
P^o	permeability of the plastic film (g μm/ m^2 d Pa)
PE	Polyethylene
PET	Polyethylene terephthalate
PP	Polypropylene
P/X	water vapor permeance of the plastic film (g / m^2 d Pa)
P	saturation vapor pressure (Pa)
Q_{10}	ratio of the shelf-life at two temperatures (10 °C apart) on shelf-life plots
R	universal gas constant (8.314 J/mol K)
R^2	coefficient of determination
S_B	specific surface area of GAB (m^2/g dry solids)
T	temperature (K)
t	storage time (d)
t_s	shelf-life of the product (d)
t_{s0}	shelf-life of the product (d) at 0 °C
W_s	food's total dry weight in the package (g dry solids)
X	thickness of the film (μm)
x_l	($l = 1,2$) parameters in polynomial set for C_o (Eq. (4))
Γ	unaccomplished moisture fraction
Φ	overall permeance of the system (1/d)
π	spreading pressure (J/m^2)

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