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ORIGINAL ARTICLE

Sorption isotherms of edible insect's flours: mathematical modeling and hysteresis

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Abstract

The interest in insects as food has increased in the latest years. Their use as low-moisture food ingredients has led to study their behavior during storage. The moisture sorption isotherms of Mexican edible insect's flours (cricket-*Acheta domesticus*, mealworm-*Tenebrio molitor*, superworm-*Zophoba morio*, grasshopper-*Sphenarium purpurascens*, escamol-*Liometopum apiculatum*) were determined through the dynamic method. Mathematical models were used for fitting the adsorption and desorption curves and Akaike Information Criteria (AIC_i) was used to evaluate their performance. Hysteresis was determined quantitatively. The samples presented a BET Type II behavior; for adsorption and desorption, GAB and Peleg's were the models with the best fitting according to the AIC_i, respectively. GAB ($R^2 \geq 0.991$) was used to determine M_0, with values ranging between 4.14 (superworm-adsorption) and 6.40 $gH_2O/100$ g d.s. (mealworm-desorption). Also, escamol desorption GAB C value was up to 12.6 times higher than the one observed in adsorption, being this one the less stable sample. The lowest areas of hysteresis were observed for cricket (1.32) and grasshopper (1.63), resulting in stable materials; this agreed with the C values of GAB. More studies are needed for the establishment of processing conditions of insects, information required for local producers to increase the insect market in Mexico and in the world.

Keywords Mathematical modeling · Sorption isotherms · Hysteresis · Edible insects

Introduction

The interest worldwide in edible insects has increased in the latest years due to their use as food and feed in a circular economy (van Huis et al., 2020). As food, insects are a valuable protein source, but also an interesting source of dietary fiber and fats. Additionally, they contain antimicrobial peptides, chitin and chitinase, antioxidants, and some other bioactive compounds (van Huis 2020). In Mexico, some edible insects are gaining attention due to their nutritional composition, flavor, and cultural significance. House crickets and grasshoppers have a rich nutritional profile, with high protein content (65% and 49% d.b., respectively), 12–13% (d.b.) lipids (Brena-Melendez et al. 2024; Kamau et al. 2018; Marín-Morales et al. 2022). According to (Oibiokpa et al. 2018), their true digestibility and biological value are 80.8% and 84.5%, and 93.0% and 87.4%, correspondingly. Mealworms and superworms have a protein content of around 43% (d.b.), while their lipid content can be up to 40–50% (Sete da Cruz et al. 2022). Escamoles are valued due to their protein content (up to 50% d.b.), high protein digestibility

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(93.9%) and their essential amino acid and fatty acid content (Cruz-Labana et al. 2018; Melo-Ruíz et al. 2016).

Insects have been processed as flours to be applied as ingredients for food product development such as in bread, pasta, chapatti, and even 3D printed food (Bottle et al. 2024; Bresciani et al. 2022; González et al. 2019; Khatun et al. 2021; Maldonado-Rosas et al. 2022; Reverberi 2021). These flours are also used for the obtention of protein isolates, and the extraction of peptides and lipids (Jantzen da Silva Lucas et al., 2020). This has led to an increment in the use of such flours, and determining their behavior during storage and when interacting with other low moisture ingredients in formulations is crucial to avoid the loss of their nutritional value and to assure their technological functionality as ingredients (Fogang Mba et al. 2024).

Moisture isotherm curves are the graphical expression of the water activity (a_W) and the moisture content at a certain temperature under equilibrium conditions. Hysteresis has been defined as the difference amongst the adsorption and desorption values in the curve (Al-Muhtaseb et al. 2004; Wolf et al. 1972). Mathematical models are frequently used for describing the interaction of water and food components. Theoretical, such as BET or GAB that includes the monolayer moisture value concept, semi-empirical like Iglesias & Chirife, or empirical expressions including Oswin, Khün, and Peleg, have been used for this purpose. While all of them are useful, their accuracy depends on the range of a_W studied or the type of food (Al-Muhtaseb et al. 2004; Tejada-Ortigoza et al. 2017). Standard statistical criteria (coefficient of determination R^2 or the residual sum of squares SSE) are commonly used to define if the model describes accurately the experimental data, although these do not always help with the discrimination. Akaike Information Criteria (AIC) is a statistical tool applied to assess the rightness of fit whilst punishing the use of a high quantity of parameters in a mathematical expression. Among a set of mathematical expressions, AIC defines the most appropriate one to use by giving evidence of the mode'ls strength and taking into consideration the information that one might lose when using such model to describe experimental data (Serment-Moreno et al. 2015). For instance, the importance of finding the best model to fit the hygroscopic behavior of the insect's flours would result in precise predicted moisture content of maximum stability, highly related to the shelf-life values of the materials.

Scarce studies have described the hygroscopic behaviour of edible insects as novel ingredients or sources of proteins. In this regard, the moisture adsorption properties of edible house cricket (*Acheta domesticus*) and black soldier fly larvae (*Hermetia illucens*) has been studied with the aim of predicting shelf-life under storage conditions of up to 35 °C (Kamau et al. 2018). Authors observed that cricket flour had higher hydration capacity and was less sensitive to the effect of temperature when compared to black soldier fly larvae. GAB and BET models were the ones that provided the best data fit. Lesser mealworm (*Alphitobius diaperinus*) moisture sorption characteristics were studied by Sun et al. (2021), where GAB model performed best on the evaluated isotherms. Sorption isotherms of *Rhynchophorus phoenicis*, *Imbrasia truncate*, and *Imbrasia epimethea* have also been performed for the determination of their stability during processing, packaging, and storage (Rodrigue Fogang Mba et al. 2018). Similarly, some authors reported the hygroscopic behavior of *Tenebrio molitor* (yellow mealworm) during freezing and drying to evaluate nutritional and quality aspects (Azzollini et al. 2016; Melis et al. 2018).

While house cricket and mealworm have been deeply studied, *Zophoba morio* (superworm), *Sphenarium purpurascens* (grasshopper) and *Liometopum apiculatum* (escamol) are also edible insects reared, collected, and consumed in Mexican culture. Because of the economic importance for local producers, the evaluation of their hygroscopic properties through sorption isotherms is crucial to determine their functionality as ingredients, and their performance during processing and storage. In addition, to the author's knowledge, the hygroscopic behavior of these Mexican reared insects, their mathematical modelling and analysis, and also the comparative evaluation of their hysteresis phenomena have not been studied. This study aims to determine the moisture sorption isotherms of Mexican edible insects (*T. molitor*, *A. domesticus*, *Z. morio, S. purpurascens,* and *L. apiculatum*), to define the mathematical model that fits better adsorption and desorption curves, and to evaluate their hysteresis. The above approaches the evaluation of the hygroscopic properties of insect's flours and its relationship with the stability of such materials.

Material and methods

Edible insects and flours preparation

T. molitor (mealworm) was donated by *Zuustento* (local producer of alternative protein derived from insects in Tequisquiapan, Querétaro- https://www.zuustento.com/). *A. domesticus* (house cricket) was provided by *Griyum* (develops technology for cultivating and utilizing edible crickets to produces flours in Querétaro, Querétaro- https://www.griyum.com.mx/). *Z. morio* (superworm) were given by *Zofo* (produces gourmet snacks and salts made from worms in Benito Juárez, Quintana Roo- https://www.zofo.mx/). Zuustento, Griyum, and Zofo are local Mexican producers that specialize in the indoor farming of edible insects.

S. purpurascens (grasshopper) were obtained from maize fields in Coronango, Puebla (19°06′36'' and 19°10′42 '' North latitude and 98°14′54'' and 98°19′40''

Western longitude- 2180 m.a.s.l). *L. apiculatum* (escamol, an edible larva of ants) were acquired from local producers in Teotihuacán, Estado de México. Both grasshopper and escamol were gotten from local Mexican suppliers that collect the insects in the wild. These insects were selected for this study due to their economic importance for local producers: grasshoppers and escamoles are frequently consumed in Mexico (Escalante-Aburto et al., 2022; Pino Moreno & Reyes-Prado 2020), while mealworm, cricket, and superworm have raised commercial interest in Mexico due to entrepreneur companies such as Zuustento, Griyum and Zofo.

All insects were euthanized by freezing, transported frozen, stored at -80 °C after their reception and freeze-dried (– 50 °C, 2.0 mbar) (Labconco, Kansas City, MO). Freeze-drying was used as standard process for all insects to be consistent among the samples to be able to compare them avoiding additional processing variables. Samples were milled during 2 min at 25,000 rpm (IKA A10 basic, Wilmington, NC), sieved (mesh 40), and these flours were stored in desiccators with P_2O_5 at 25 °C for at least 5 days before their analysis to reach the lowest initial moisture content of the samples. This sample's preparation was performed according to Tejada-Ortigoza et al. (2017).

Moisture sorption isotherms

A dynamic instrumental method using an Aquasorp Isotherm Generator (Decagon Devices Inc., Pullman, WA) was used for the determination of adsorption and desorption isotherms of each insect flour, in duplicate. The isotherms were determined in a range of a_w values of– 0.10–0.93 at 25 °C following the manufacturer's instructions.

Isotherm modeling

Semi-empirical (Chirife & Iglesias, 1978), empirical (Oswin 1946), Peleg (1993), and Kühn (1967), and theoretical (GAB, (van den Berg 1985)) models with different number of parameters were used to describe the data obtained by the dynamic method. Table 1 shows the expressions used for the moisture content calculations:

The evaluation of the model fitting was based on R^2 (coefficient of determination, Eq. 6), and SSE (residual sum of squares, Eq. 7) calculated as follows:

$$R^2 = 1 - \frac{\sum \left(y_i - \hat{y}_i\right)^2}{\sum \left(y_i - \bar{y}_i\right)^2} \tag{6}$$

$$SSE = \sum \left(y_i - \hat{y}_i\right)^2 \tag{7}$$

where y_i and $\hat{y}_i$ are the experimental observation and the model determined value, correspondingly.

The model parameters were obtained by non-linear regression minimizing the SSE through Microsoft Excel 365 Solver tool.

For the selection of the best fitting, AIC was performed. The first term in Eq. 8 assesses the goodness of fit, while the second penalises the high number of parameters use for the fitting. Here, $\hat{\sigma}^2$ is the variance's maximum probability estimator and p is the number of parameters in the evaluated model.

$$AIC = -\frac{n}{2} \ln \hat{\sigma}^2 + 2p \tag{8}$$

Finally, AIC values vary with the data set used and differences regarding to the model with the minimum AIC values are employed (ΔAIC_{i}, Eq. 9). Values of $\Delta AIC_{\mathrm{i}} \leq 2$ indicate

Table 1 Mathematical models used to describe moisture sorption isotherms†

Model	Mathematical expression	Parameters	
Semi-empirical			
Iglesias & Chirife	$M = \left[\frac{-A}{\ln a_w}\right]^{\frac{1}{B}}$	A and B are constants	(1)
Empirical			
Oswin	$M = A\left[\frac{a_w}{1-a_w}\right]^{B}$	A and B are constants	(2)
Peleg	$M = k_1 a_w^{n_1} + k_2 a_w^{n_2}$	k_1, n_1, k_2, and n_2 are dimensionless parameters	(3)
Khün	$M = \frac{A}{\ln \frac{1}{a_w}} + B$	A and B are constants	(4)
Theoretical			
GAB	$M = \frac{M_0 C K a_w}{(1-Ka_w)(1-Ka_w+CKa_w)}$	M_0 is the monolayer moisture content (g H_2O/100 g dry solids), C and K are dimensionless parameters related to heat of sorption in the monolayer (C) and the multilayer (K)	(5)

†M is the moisture content at equilibrium (g H_2O/100 g dry solids)

strong model support evidence, values between $2 < \Delta AIC_i \le 4$ indicate moderate support, and values $\Delta AIC_i > 4$ indicate a slight support (Burnham & Anderson 2002). This means that, among the tested models, the $\Delta AIC_i = 0$ is the one with the best fit.

$$\Delta AIC_i = AIC_i - AIC_{min} \tag{9}$$

Sorption surface area

The sorption surface area (S_0) has been estimated according to Moraes & Pinto (2012), where the monolayer moisture value (M_0) is used:

$$S_0 = 35.3M_0 \tag{10}$$

Sorption hysteresis

The change between the desorption (M_{des}) and adsorption (M_{ads}) equilibrium moisture content values is the hysteresis (Hys). This was determined using the Peleg model as a function of a_w for each insect as follows:

$$Hys = M_{des} - M_{ads} \tag{11}$$

As reported by Tejada-Ortigoza et al. (2017), the integral of the Hys changes as a function of a_w changes, or area of hysteresis (A_H), can be used to quantify this phenomenon. This has been calculated at defined a_w intervals in increments of 0.02 a_w units as in Eq. 12, in a a_w range of 0.15 to 0.93:

$$A_H = \int Hys \, da_w \tag{12}$$

Results and discussion

Sorption isotherms of edible insects

All the studied edible insects presented a typical BET Type II behavior as observed in Fig. 1. This agrees with the results found in cricket and black soldier fly powders (Kamau et al. 2018), and for mealworm (Azzollini et al. 2016), where the samples exhibited a Type II sorption isotherm. As stated by Azzollini et al. (2016), hygroscopic food products rich in protein content might exhibit sigmoid Type II isotherms behavior, which had also been reported for lean beef (Trujillo et al. 2003) and fish fillet (Martins et al. 2015). For both adsorption and desorption isotherms, the highest moisture contents within the evaluated range were observed for mealworm and grasshopper (Fig. 1).

The nutritional composition of these insects has been highly reported in the literature (Kamau et al. 2018; Marín-Morales et al. 2022; Melo-Ruíz et al. 2016; Sete da Cruz et al. 2022). While it is not the main objective of this study, a discussion regarding a possible relation with the sorption behavior of these materials might be appropriate for a deeper understanding of the hygroscopic properties of these materials. Table 2 shows the reported proximate composition of the evaluated insect's flours.

The highest protein and lipid content is observed for cricket and superworm, respectively. It has been reported that less protein and more fat content might result in fewer hydrophilic sites in the matrix (Kamau et al. 2018). This behavior can be also observed in Fig. 1A, where superworm and escamol had the lower water adsorption capacity. On the contrary, grasshopper, perhaps due to its high carbohydrate and protein content as well to its low lipid content, resulted in the matrix with the highest water adsorption capacity. Maidannyk et al. (2019) reported that in carbohydrate-protein-oil systems, oil might cover particles with a free fat layer that impacts their water sorption profile. For drying purposes, desorption curves are very useful. Among the evaluated samples, for the same a_W value, it was observed that cricket and mealworm had the lowest and the highest moisture content. In this case, the sample with the reported highest protein content (cricket) retained less water during desorption when compared to the other samples.

It is relevant to note that, while the protein content in the studied samples might range between 42–65 g/100 g of dry solids (ds) as reported by some other authors (Table 2), these flours cannot be considered protein isolates. The above because they have not passed through a protein extraction/separation/fractionation process. Thus, the effect on their hygroscopic behavior caused by their fat (20–33.6 g/100 g ds) and carbohydrate content must be considered as well (Azzollini et al. 2016; Kamau et al. 2018).

Isotherm model fit

GAB, BET, Smith, Khün, and Caurie, are models that have been previously applied to fit the experimental data of edible house cricket, black soldier fly larvae, mealworm, and lesser mealworm (*Alphitobius diaperinus*) (Azzollini et al. 2016; Kamau et al. 2018; Sun et al. 2021). While R^2 and SSE are criteria commonly used to define the fitting behavior of models for sorption isotherms, it is frequently observed that it can be challenging to discriminate the best one by only using these values. As noticed in Table 3 R^2 values were ≥ 0.95, being ≥ 0.99 a common value obtained among the models fitting making it difficult to define the best option. Due to this, AIC_i was used, where the minimum value indicates the best fitting condition (Serment-Moreno et al. 2015).

Regarding the isotherm model fit, it can be observed (Table 3) that, for adsorption, GAB, followed by Iglesias & Chirife, were the models with the lowest AIC_i values among

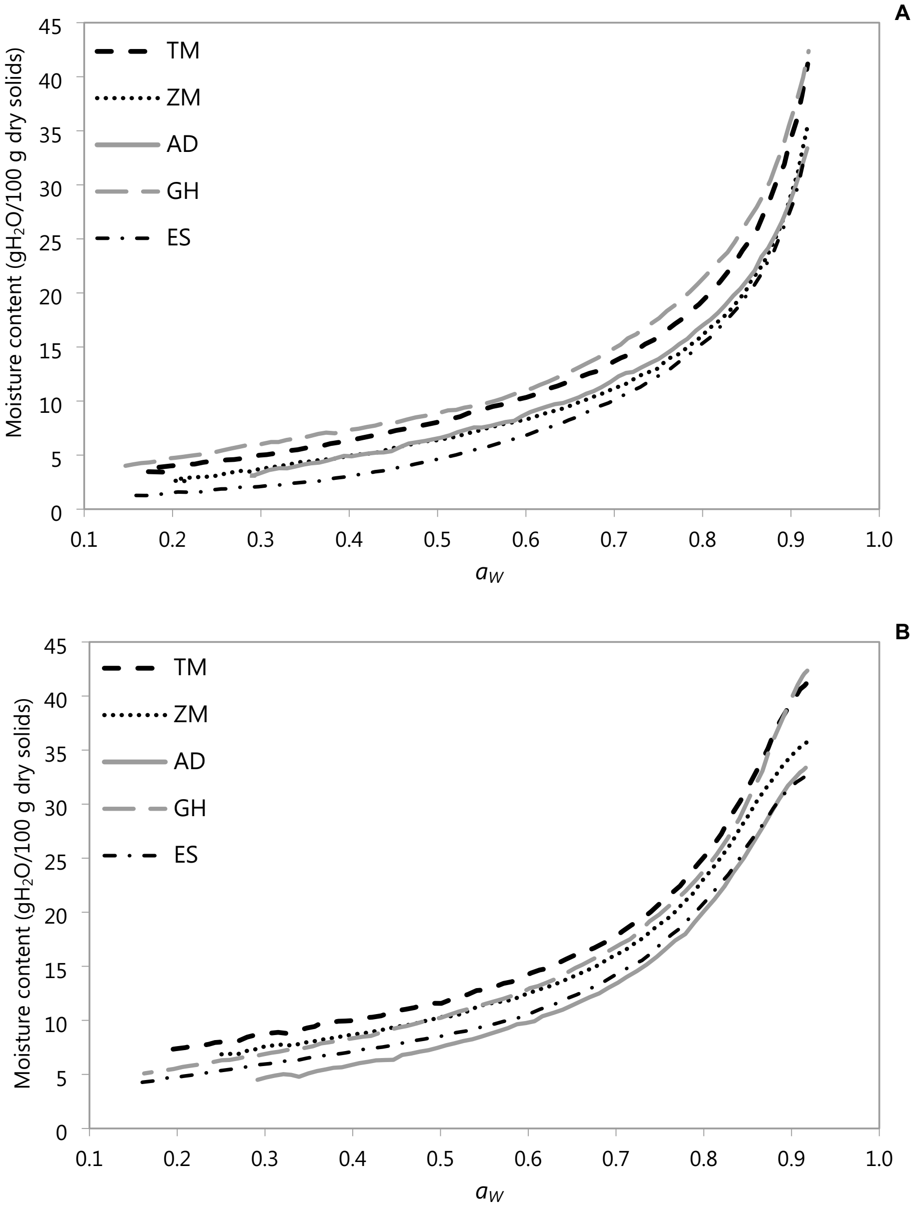

Fig. 1 **A** Water adsorption and **B** desorption isotherms of edible insect flours at 25 °C. *Tenebrio molitor* (mealworm, TM), *Zophoba morio* (ZM), *Acheta domesticus* (cricket, AD), *Sphenarium purpurascens* (grasshopper, GH), and *Liometopum apiculatum* (escamol, ES)

Table 2 Reported proximate composition of the evaluated insects' flours in dry basis

	Mealworm	Superworm	Cricket	Grasshopper	Escamol
Protein (%)	42.9	43.3	65.9	48.9	42.1 – 50.6
Lipids (%)	41.9	51.5	12.2	13.1	30.3 – 34.9
Carbohydrates (%)	11.5	2.8	17.1[1]	27.4[2]	8.7–20.8[3]
Ash/ Minerals (%)	3.6	2.4	4.8	7.9	6.5 – 7.9
References	(Sete da Cruz et al. 2022)	(Sete da Cruz et al. 2022)	(Kamau et al. 2018)	(Marín-Morales et al. 2022)	(Melo-Ruíz et al. 2016)

Reported as [1]crude fibre + available carbohydrate, [2]chitin + carbohydrates, [3]fibres + soluble carbohydrates

the used ones. GAB has been reported as well as the best fitting model using AIC_i criteria for lesser mealworm powder (Sun et al. 2021). With reference to desorption, Peleg's model was the one with the lowest AIC_i values within the evaluated samples.

Table 3 Statistical criteria summary of the adsorption (ads) and desorption (des) isotherms of edible insect's flours

Model	Criteria	Mealworm		Superworm		Cricket		Grasshopper		Escamol	
		Ads	Des	Ads	Des	Ads	Des	Ads	Des	Ads	Des
Iglesias & Chirife	R^2	0.999	0.994	0.998	0.984	0.988	0.975	0.998	0.994	0.968	0.985
	SSE	13.6	72.6	22.5	100.9	116.9	411.2	17.0	135.7	732.5	161.1
	AIC	−163.3	−1.6	−106.5	29.0	36.7	124.7	−170.7	39.7	194.9	55.5
	AIC_i	**0.0** †	148.2	**0.0** †	137.9	192.4	269.0	139.7	129.6	446.9	220.5
GAB	R^2	0.999	0.997	0.996	0.991	0.998	0.993	1.000	0.997	0.999	0.993
	SSE	13.3	27.0	27.5	57.3	9.1	49.1	4.1	74.5	5.6	48.3
	AIC	−163.2	−76.7	−87.8	−9.3	−155.7	−13.5	−310.4	−13.5	−252.0	−53.2
	AIC_i	0.0	73.0	18.7	99.7	**0.0** †	130.8	**0.0** †	76.5	**0.0** †	111.7
Oswin	R^2	0.995	0.992	0.997	0.989	0.999	0.990	0.992	0.993	0.995	0.990
	SSE	80.2	80.1	33.1	88.1	14.2	65.1	111.8	219.7	54.0	127.2
	AIC	−5.3	6.0	−74.2	19.3	−123.4	3.1	16.1	84.1	−45.0	33.8
	AIC_i	158.0	155.8	32.3	128.3	32.3	147.4	326.4	174.0	207.0	198.8
Peleg	R^2	0.989	0.999	0.990	0.998	0.998	0.999	0.994	0.998	0.987	0.999
	SSE	117.4	10.3	72.3	13.7	10.3	6.6	66.7	25.4	106.2	9.5
	AIC	32.7	−149.8	-4.6	−109.0	−144.1	−144.3	−22.0	−89.9	18.9	−164.9
	AIC_i	196.0	**0.0**†	101.9	**0.0**†	11.5	**0.0**†	288.3	**0.0**†	270.9	**0.0**†
Khun	R^2	0.989	0.967	0.991	0.950	0.978	0.959	0.982	0.972	0.986	0.955
	SSE	416.4	1320.0	266.1	1518.4	465.6	1195.8	935.4	2143.3	395.4	1536.6
	AIC	141.3	224.6	100.9	221.5	141.8	195.2	174.7	243.3	114.8	218.0
	AIC_i	304.6	374.4	207.4	330.4	297.4	339.5	485.1	333.2	366.8	382.9

†Substantial evidence supporting model fit ($\Delta AIC \leq 2$)

The parameters of the models used are presented in Table 4. While GAB was the model with the best fitting only for adsorption, this model yielded $R^2 \geq 0.991$ for both, adsorption and desorption curves, and its parameters might be useful for the physical interpretation of the data (Basu et al. 2006). For instance, *k* values are related to the multilayer heat sorption, and for the studied insects the estimated values ranged between 0.93 and 0.96 (Table 4). GAB C values are related to the monolayer heat sorption, and high values suggest that a high amount of energy is needed for the removal of water, because of its strong linkage to the matrix (Velázquez-Gutiérrez et al. 2015). Among the

Table 4 Estimated sorption model parameters for the adsorption (ads) and desorption (des) isotherms of edible insect's flours

Model	Parameter	Mealworm		Superworm		Cricket		Grasshopper		Escamol	
		Ads	Des	Ads	Des	Ads	Des	Ads	Des	Ads	Des
Iglesias & Chirife	A	9.29	41.73	5.99	27.28	5.65	7.76	12.50	16.94	2.29	13.72
	B	1.26	1.65	1.18	1.57	1.15	1.20	1.32	1.37	0.85	1.38
GAB	M_0†	4.77	6.40	4.14	5.63	5.01	5.04	5.07	5.70	4.88	4.97
	C	7.92	74.04	4.62	46.99	2.65	4.34	12.73	17.19	1.17	14.70
	k	0.96	0.93	0.95	0.93	0.93	0.95	0.96	0.96	0.94	0.94
Oswin	K	8.20	12.24	6.44	10.53	6.45	7.65	9.48	10.82	4.58	9.30
	N	0.61	0.51	0.66	0.52	0.70	0.66	0.56	0.53	0.86	0.52
Peleg	k_1	13.65	15.38	12.64	13.22	16.75	12.78	12.55	12.59	7.79	11.46
	k_2	37.61	45.69	34.97	39.33	35.16	38.52	41.49	48.40	29.18	38.62
	n_1	0.83	0.47	1.02	0.48	1.35	0.86	0.61	0.49	1.11	0.55
	n_2	6.75	6.24	7.68	5.76	9.12	6.30	6.11	5.53	4.97	5.83
Khun	A	−4.15	−4.42	−3.52	−4.16	−3.73	−4.12	−4.65	−4.96	−3.81	−4.17
	B	1.56	5.17	0.95	4.14	0.75	1.45	1.87	2.67	−1.04	2.36

†M_0 is expressed in g water/100 g dry solids

studied insect samples, the highest values were obtained for desorption curves, being escamol desorption GAB C value up to 12.6 times higher than the one observed in adsorption. For mealworm and superworm, these values were 9.3 and 10.2 times higher, respectively. Among the insect's flours, the lowest C values were observed for cricket, followed by escamol and grasshopper. Low C values suggest that the drying process for these materials may be less demanding and fast under specific time and temperature conditions, as less energy is required to remove the water.

M_0 is the moisture content at which all polar and ionic groups of the food matrix are occupied by water, and at which the material is the most (Kamau et al. 2018). Regarding M_0 estimated values, for adsorption, all samples resulted with lower values for adsorption curves, when compared to their desorption curve. Low values reflect less available binding sites due to physico-chemical and structural characteristics of the sample, mainly occurring at low a_w values (Velázquez-Gutiérrez et al. 2015). For adsorption, these values ranged between 4.14 and 5.07 g water/100 g dry solids, while for desorption these values were among 4.97 and 6.40 g water/100 g dry solids. Thus, drying these samples to around these monolayer moisture contents would be appropriate in terms of stability and powdering operations (Azzollini et al. 2016). Although, it is important to highlight that at these low moisture contents, while chemical reactions that depend on solvation are expected to slow down, some other reactions due to the lipid content (oxidative rancidification), might be promoted (Jayaraj Rao et al. 2006). Similar values of k and M_0 in GAB model parameter were estimated also for mealworm (*Tenebrio molitor*) and for *Rhynchophorus phoenicis*, *Imbrasia truncata* and *Imbrasia epimethea* (Azzollini et al. 2016; Rodrigue Fogang Mba et al. 2018).

S_0 indicates the number and size of pores in the food matrix, which might be associated to the water binding capacity of the materials. Because of their proportionality, S_0 and M_0 behave alike. In general, these values were higher for desorption (146.3–225.9 m^2/g), when compared to adsorption (146.3–168.3 m^2/g). Among samples, mealworm had the highest S_0 values in desorption (225.9 m^2/g). While no S_0 values have been published for insects, fruit peels values ranged between 251 and 357 m^2/g for adsorption and among 308 and 531 m^2/g for desorption. These high values are common of materials with a microporous structure (Velázquez-Gutiérrez et al. 2015). Similar values of S_0 at 30 °C have been reported for texturized soy protein (191 m^2/g) (Cassini et al. 2006).

Hysteresis

Figure 2 shows this phenomena behavior and also its quantification through A_H values, which were obtained as the area under the presented curves. Regarding this phenomena, clear differences are observed among the studied samples. For instance, both cricket and grasshopper appeared to be the most stable materials with the lowest values of A_H (1.32 and 1.63, respectively). Within the evaluated a_w range, superworm was the material with the highest A_H value (3.60), followed by mealworm (3.42), and escamol (3.39). The highest hysteresis values among the samples were obtained at the highest a_W values evaluated. For escamol, this value reached up to 8.78 g H_2O/100 g dry solids at a $a_W = 0.93$. In addition, it can be observed that all materials reach the highest hysteresis values at around $a_W \geq 0.7$.

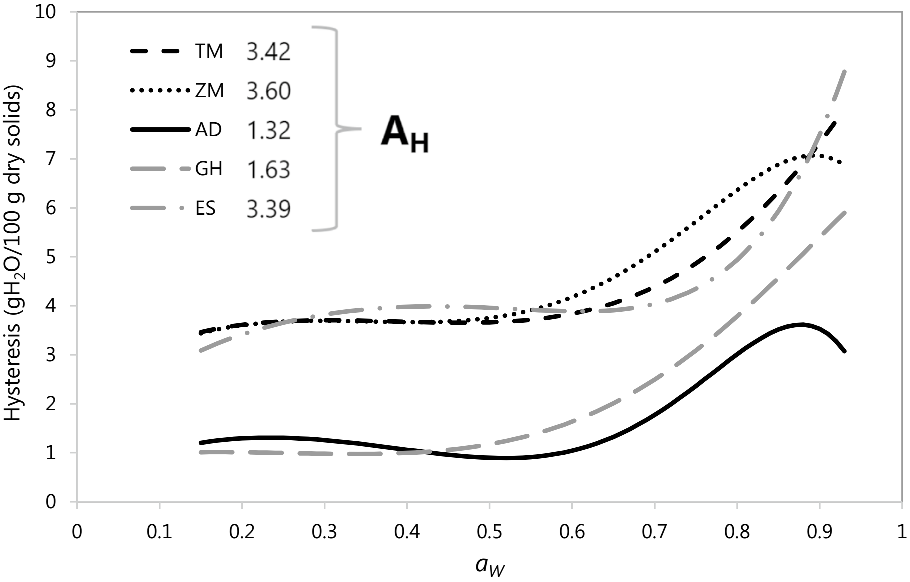

Fig. 2 Hysteresis phenomena of *Tenebrio molitor* (mealworm, TM), *Zophoba morio* (ZM), *Acheta domesticus* (cricket, AD), *Sphenarium purpurascens* (grasshopper, GH), and *Liometopum apiculatum* (escamol, ES) modelled through Peleg's equation as function of water activity

Regarding hysteresis, chemisorption and structural effects of the sorbent in interaction with the sorbate are possible causes of this phenomena. During adsorption, hysteresis is explained as the phenomenon occurring when the capillaries that form the porous region start to swell up because of the increase in relative humidity. Then, water moves to the pore interior when the partial pressure of the water vapor of the air turns higher than the vapor pressure of the capillaries. While during desorption the pore is saturated when the process starts, so water diffusion occurs from the boundary to the surface of the material (Lahsasni et al. 2004). In addition, hysteresis values are associated with the energy required for the emptying or filling of binding sites and capillaries of the food matrix (Al-Muhtaseb et al. 2002).

Food composition is an important feature that affects the water sorption behavior, and its importance relies on its possible use as quality index during storage (Maidannyk et al. 2019). It can be observed from Fig. 2, that the magnitude of hysteresis is smaller for cricket when compared to the other flours. According to Table 2, along with the samples, cricket has a high protein and low carbohydrate content. While a relation can be stablished, it is important to note that is not only the composition but also the structure, the responsible for the sorption capability of materials (Maidannyk et al. 2019). As far as the author's knowledge, no hysteresis values have been previously reported for edible insects. However, the study of this phenomenon is crucial in the determination of these material's stability as food ingredients.

Conclusions

The determination and understanding of hygroscopic properties of edible insects is still a fairly studied area. Sorption isotherms and hysteresis are powerful tools to evaluate storage stability of dehydrated products, such as edible insect flours. In this study, adsorption and desorption curves were determined through the dynamic method and then mathematically modelled. For all the studied insect flours, a BET Type II behavior was observed. In terms of hygroscopicity, superworm and escamol were the ones with the lowest adsorption capacity according to their isotherm. However, these matrices are the ones with the highest content of lipids, which might cause some other undesirable reactions during storage. During desorption, cricket retained less water when compared to the rest of the samples. Cricket was the sample with the highest protein content reported by other authors.

AIC_i was used to evaluate the best model fitting. For adsorption and desorption isotherms, GAB and Peleg models were the most accurate. The GAB model parameters were also used due to their physical meaning used to interpret the obtained data. Escamol desorption GAB C value was up to 12.6 times higher than the one observed in adsorption, being this one the less stable sample. Being able to forecast the behavior of the samples through mathematical models will reduce the number of iterations to find optimal processing parameters and will estimate results within the evaluated range avoiding expending additional time/resources. Moreover, finding the best model will increase the acceptance from the industry/producers to use these results and to apply them to their processes.

Finally, hysteresis and the area under the hysteresis curve were also determined. Hysteresis values reflected that cricket and grasshopper might be the most stable materials in terms of hygroscopicity, as previously defined by relating the isotherms and the reported composition of these materials. The interpretation of hysteresis phenomena has not been reported previously in the literature, and its importance relies on the stability, processing behavior, and shelf-life determination of edible insect's flours as food ingredients. In this study, the highest area under the hysteresis curve was observed for superworm, the sample with the highest lipid content.

More studies are needed for the establishment of processing conditions of insects as food and feed. Further studies must be focused on the evaluation of the stability and shelf-life of the samples and how composition might be affected by the storage. For instance, the future determination of isotherms of defatted samples might be useful for the complete understanding of these flours under storage and under drying.

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Declarations

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