Journal of Insects as Food and Feed, 2023; 9(8): 1017-1026

Wageningen Academic
Publishers

Dynamic vapour sorption isotherms and isosteric heats of sorption of two edible insects (*Cirina forda* and *Rhyncophorus phoenicis*)

C. Bukamba Tshanga[1,3*], P. Malumba[5], B. Kambashi Mutiaka[2], J. Bindelle[4] and F. Debaste[1]

[1]Université Libre de Bruxelles (ULB), Transfers, Interfaces and Processes (TIPs), avenue F.D. Roosevelt, 50 CP165/67, 1050 Brussels, Belgium; [2]Université de Kinshasa, Faculté des Sciences Agronomiques, Département de Zootechnie, Route Kimwenza 1, Kinshasa, Democratic Republic of the Congo; [3]Université de Kinshasa, Faculté des Sciences Agronomiques, Département de Chimie et Industries Agricoles, Route Kimwenza 1, Kinshasa, Democratic Republic of the Congo; [4]University of Liège, Gembloux Agro-Bio Tech, Terra AgricultureIsLife, Passage des Déportés 2, 5030, Gembloux, Belgium; [5]University of Liège, Gembloux Agro-Bio Tech, Terra FoodIsLife, Passage des Déportés 2, 5030, Gembloux, Belgium; celestin.bukamba.tshanga@ulb.be

Received: 1 June 2022 / Accepted: 30 December 2022

RESEARCH ARTICLE

Abstract

Cirina forda and *Rhyncophorus phoenicis* are widely consumed insects in tropical Africa. Drying is one of the main conservation techniques to improve the availability of these insects, which are harvested seasonally in the wild. The main goal of this study was to investigate the sorption isotherms and to estimate the shelf-life of these two dried insects. Sorption isotherms were determined at 25, 30, 40 and 50 °C by using dynamic vapour sorption. Amongst five isotherm sorption models that were selected to fit the experimental data, Peleg's equation was found to give the best fit for both insect species. It was observed that all insects exhibited type III sorption isotherms, indicating monolayer-multilayer behaviour with a progressively decreasing biding energy as the number of layers rises. Using the Heiss-Eichner model, dry base initial moisture contents of 8 and 7%, are proposed for *C. forda* and *R. phoenicis* respectively. These conditions ensure a shelf-life of 12 months at 30 °C when insects are packaged in polyethylene films.

Keywords: larvae, water activity, sorption model, shelf-life, drying

1. Introduction

Nowadays, insects are presented as an attractive response to the increased quantity of food protein demanded for the future (Mancini *et al.*, 2019). In comparison to conventional protein source insects can be produced at a low environmental cost par amount of food produced, with reduced greenhouse gases and ammonia emissions, and contribute to environmental sustainability through the conversion of bio-wastes into high-protein food products (Gere *et al.*, 2019; Yen, 2009). For animals and humans, insects are food ingredients that may contain a high concentration of digestible energy, fat and proteins. The development of artificial insect-rearing systems from agricultural by-products requires less land and water than raising conventional farmed animals (Mancini *et al.*, 2019).

Many insects are commonly consumed in several parts of the world. Most of these insects are caught in the wild, and their supply tends to decrease due to pressure on their natural habitats, presenting a threat to biodiversity. In the Democratic Republic of Congo (DRC), insects contribute greatly to alleviating malnutrition and food insecurity, from which a large part of the population suffers. Nsevolo *et al.* (2016) identified 14 species of insects regularly consumed in the city of Kinshasa in DRC, including Lepidoptera, Isoptera, Orthoptera, Coleoptera and Hymenoptera. Lepidoptera (caterpillars) represent the most frequently consumed insect order (46.7%). When it comes to the quantities consumed daily per individual, Coleoptera rank first (Nsevolo *et al.*, 2016). Larvae of the caterpillar *Cirina forda* (Cf), a Lepidopteran, and *Rhyncophorus phoenicis* (Rp), a Coleopteran, are among the most widely consumed insects in the DRC (Nsevolo *et al.*, 2016).

Some processing methods such us drying, ultrasound-assisted extraction, cold atmospheric pressure plasma, and dry fractionation are developed for fat, protein and chitin extraction to design insect-based ingredients (Melgar-Lalanne *et al.*, 2019). Insect-based ingredients are included in several foodstuffs in un-recognisable form to increase consumer acceptance in the West where entomophagy is limited by food neophobia and aversion to insect consumption (Hartmann and Siegrist, 2016, 2018). For people for whom entomophagy is part of the food culture, insects are usually fed in recognisable forms. Once captured in the wild, the insects undergo traditional treatments such as drying, toasting, frying, roasting or boiling, prior to consumption (Akullo *et al.*, 2017; Kinyuru *et al.*, 2010; Kröncke *et al.*, 2018; Niassy *et al.*, 2016). Drying, which is very energy-intensive, is particularly required because of the limited availability of alternative means of preservation and the need to extend the shelf-life of these insects. The ability to be stored for long periods of time that is conferred to the insects by the drying process allows them to be transported from the areas of production or capture to the consumption centres in the cities, as well as, the extension of the consumption of this food commodity when insect populations develop seasonally. Generally, insects are dried in the open air under the ray of sun. This method has several disadvantages such as: uncertainty of drying time, high labour cost, the need for large areas, infection by microorganisms and other foreign bodies (Hajar *et al.*, 2017). To optimise the drying process of insects and their storage conditions, knowledge of sorption isotherms specific to each category of insect is required.

Indeed, sorption isotherms describe the equilibrium moisture content (X_{eq}) of a specific material and the surrounding relative air humidity at a constant temperature. Sorption isotherms allow the design and optimisation of equipment and food processing for preservation, such as drying process. Sorption isotherms are also used to predict stability and quality changes during packaging and storage of dried foodstuffs (Hossain *et al.*, 2001).

Models to generate sorption isotherms for most tropical insects have not been established yet. To our knowledge, the moisture sorption properties of processed edible insects have only been studied by Azzollini *et al.* (2016) for the yellow mealworm (*Tenebrio molitor*) and by Kamau *et al.* (2018) for the house cricket (*Acheta domesticus* L.), the black soldier fly larvae (*Hermetia illucens* L.) and the lesser mealworm (*Alphitobius diaperinus*)(Sun *et al.*, 2021). In this work, the sorption isotherms of two edible insects, *C. forda* caterpillars and *R. phoenicis* larvae, are studied. Sorption isotherms were generated by the Dynamic Vapour Sorption (DVS) method. The experimental data were modelled using five models obtained in the literature, and the net isosteric heat was calculated by using the Clausius-Clapeyron equation. The shelf-lives at 25 and 30 °C were estimated by using the Heiss-Eichner model.

2. Materials and method

Insect samples

Two batches of edible insects, *C. forda* (Cf) caterpillars and *R. phoenicis* (Rp) larvae, were purchased on a market in the city of Kinshasa in the Democratic Republic of Congo. Samples were freeze-dried and milled before using.

Dynamic vapour method

Sorption isotherms allow the determination of characteristic values such as critical moisture content or isosteric heat. They describe the equilibrium between the water activity (a_w) and the moisture content, expressed on a dry basis (X) of a product stored at a given temperature (Dupas-Langlet *et al.*, 2016).

Water vapour sorption experiments were performed using an automatic Dynamic Vapour Sorption analyser (DVS Advantage, from Surface Measurement System Ltd, Wembley, UK) equipped with an ultra-microbalance for the accurate measurement of weight at a sensitivity of 1 µg. This instrument allows samples to be analysed in a climatic chamber where the relative humidity is conditioned by mixing a dry nitrogen gas flow with a water-saturated gas flow. A representation of such an instrument can be found in Bui *et al.* (2017). Samples of approximately 0.020 g were weighed into aluminium pans and placed on one side of the sample holder of the microbalance while an empty aluminium pan of equal mass was placed on the other side of the balance to serve as the reference pan. The first stage of analysis consisted of drying the samples at the working temperature using a dry nitrogen flow until equilibrium. The mass at the end of the initial drying step was assumed to correspond to the mass of the dried samples, which were subsequently exposed to increasing programmed relative humidity while the sample weight changed. The weight change was almost continuously measured and compared at 1-min. intervals. The range of relative humidity covered was 3 to 90% with an accuracy of ±0.5%. Equilibrium was assumed to be established when the instantaneous sample weight no longer changed by more than 0.001% for a period of 10 min. Eighteen steps at different relative humidity levels were considered for moisture content equilibration and served to build adsorption and desorption isotherms. The same experiments were performed at 20, 30, 40 and 50 °C.

Isotherm models

The experimental sorption isotherm data were fitted using five mathematical equations: GAB (Martínez-Las Heras *et al.*, 2014), Halsey (Bingol *et al.*, 2012), Oswin (Oswin, 1946), Peleg (Peleg, 1993) and Smith (Martínez-Las Heras *et al.*, 2014).

$$\text{GAB: } X_{eq} = \frac{X_0 C K a_w}{(1-a_w)(1+(C-1)a_w)} \quad (1)$$

$$\text{Halsey: } X_{eq} = \left(\frac{-b}{\ln(a_w)}\right)^{\frac{1}{a}} \quad (2)$$

$$\text{Oswin: } X_{eq} = a\left(\frac{a_w}{1-a_w}\right)^{b} \quad (3)$$

$$\text{Peleg: } X_{eq} = a.{a_w}^{b} + c.{a_w}^{d} \quad (4)$$

$$\text{Smith: } X_{eq} = a - b.\ln(1-a_w) \quad (5)$$

The models are presented in terms of equilibrium moisture content (X_{eq}) on dry basis (kg/kg d.b.), as a function of water activity a_w.

The GAB equation is an extension of the two-parameter (*X0, C*) BET model, taking into account the modified properties of the sorbate in the multilayer region through the introduction of the third K (Bingol *et al.*, 2012). For the GAB model, X_0 represents the moisture corresponding to a monolayer of water at the primary adsorption sites of the solid. C is the Guggenheim constant, characteristic of the product and related to the heat of sorption of the monolayer. K is a correction factor related to the heat of sorption of the multilayer. For the other models, A, B, a, b, c and d are empirical model parameters.

In order to select the most suitable model for describing the relationship between the equilibrium moisture content, the water activity and the temperature, two criteria are used: the coefficient of correlation (r) and the root mean square (RMS).

$$r = \sqrt{\frac{\sum_{i=1}^{N}\left(Xeq_{i,pred} - \overline{Xeq}_{i,exp}\right)^2}{\sum_{i=1}^{N}\left(Xeq_{i,exp} - \overline{Xeq}_{i,exp}\right)^2}} \quad (6)$$

$$RMS = \frac{\sqrt{\sum_{i=1}^{N}(Xeq_{i,exp} - Xeq_{i,pred})^2}}{N} \quad (7)$$

where:

$Xeq_{i,pred}$: ith predicted equilibrium moisture content (kg/kg d.b.);

$Xeq_{i,exp}$: ith experimental equilibrium moisture content (kg/kg d.b.);

N: number of experimental points.

Shelf-life estimation

The shelf-life of the insects was predicted using the Heiss-Eichner model as described by Kamau *et al.* (2018). This model predicts the shelf-life depending on the packaging used and storage conditions. The shelf-life was calculated by the expression:

$$t_s = \frac{\ln[(X_e - X_i)/(X_e - X_c)]}{K_s(A/W)(p_0/S)} \quad (8)$$

where:

X_e: the equilibrium moisture content of the product when it is left in direct contact with the ambient air (% d.b.);

X_c: the safe storage moisture content of the product (% d.b.);

X_i: the initial moisture content of the product when it is packaged (% d.b.);

K_s: permeability constant of the package to moisture vapour (kg H_2O µm/m^2/day/Pa);

p_0: vapour pressure at storage temperature at atmospheric pressure (Pa);

A: surface area of the package (m^2);

W: weight of the dry matter in the package (kg);

S: slope of the product isotherm (assumed linear over the range between X_e and X_c).

However, the application of such a model requires definition of a safe storage moisture content of the product corresponding to the safe storage borderline a_w, which can be determined experimentally. At the present state of knowledge, the borderline a_w of the two insects and criteria for their definition have not been determined yet. The classical safe storage borderline a_w used by several authors for the shelf-life of biological products in tropical regions is set to 0.6 or 0.7 (Chuzel and Zakhia, 2007; Ikhu-Ornoregbe and Chen, 2005; Kamau *et al.*, 2018; Lima and Cal-Vidal, 1988). In this work, we consider the safe storage borderline a_w of 0.6.

The conditions used to estimate shelf-life are based on the assumption of 10 kg of initial product wrapped in polyethylene bags (thickness: 80 µm; surface area: 0.1474 m^2; water vapour permeability constant: 1.55×10^{-4} kg µm/m^2 /day/Pa) (Kamau *et al.*, 2018; Lima and Cal-Vidal, 1988). Climatic conditions were chosen from the available data for the Region of Kinshasa: extremum average air relative humidity between 0.66 and 0.85 and extremum average ambient temperatures varying between 19.8 and 30 °C.

Determination of net isosteric heat of sorption

The net isosteric heat of sorption is a thermodynamic parameter that estimates the biding energy of the force between the water vapour molecules and the solid. It provides information that aids to understand the sorption mechanism and detecting the type of water binding that is occurring at a given moisture content (Quirijns *et al.*, 2005). The net isosteric heat of sorption (ΔH_{is}) is defined as the difference between the amount of energy required to remove water from the material (Q_{st}) and the amount of energy required for normal water vaporisation (ΔH_v):

$$\Delta H_{is} = Q_{st} - \Delta H_v \tag{9}$$

The net isosteric heat was calculated from the sorption isotherms at several temperatures using the Clausius-Clapeyron equation (Quirijns *et al.*, 2005; Mghazli *et al.*, 2016).

$$\Delta \mathrm{H_{is}} = -\mathrm{R}\left[\frac{\mathrm{d}(\ln(\mathrm{a_w}))}{\mathrm{d}\left(\frac{1}{\mathrm{T}}\right)}\right]_{\mathrm{X_{eq}}} \tag{10}$$

a_w values at constant X_{eq} were estimated using the Peleg model. The value of $\ln(a_w)$ was plotted as function of 1/T. The isosteric heat is obtained by dividing the slope of the line obtained after linear regression by -R. In this paper, only desorption characteristics are considered to calculate the net isosteric heat of sorption, as drying is the targeted practical application (Quirijns *et al.*, 2005).

3. Results and discussion

Sorption isotherms and moisture sorption hysteresis

According to the classification of Brunauer *et al.* (1940) moisture isotherms of Cf and Rp are of type III expressing a multilayer adsorption. Similar results was observed for sorption isotherms of red tilapia (Camaño *et al.*, 2021) and ham (Leonardo *et al.*, 2020). This class is manly about products with hight salt or protein content (Erbaş *et al.*, 2016). The same observation was done for lesser mealworm ingredients (powder, protein concentrate, whey protein concentrate) (Sun *et al.*, 2021). The results obtained for other insects showed type II isotherms such as the yellow mealworm (Azzollini *et al.*, 2016), the house cricket and the black soldier fly larvae (Kamau *et al.*, 2018). The influence of temperature on the isotherm of Cf is really limited, being only significant above 80% of relative humidity. For Rf, the influence temperature is more pronounced but remains small, below 80% of relative humidity.

A negligible hysteresis is observed for Cf for all tested temperatures. The largest difference between adsorption and desorption is at 25 °C and is illustrated in Figure 1 by continuous lines. The difference in equilibrium moisture content between the adsorption and desorption always remains below 2% d.b. A small hysteresis is observed for Rp, illustrated at 25 °C by continuous lines in Figure 2. For all temperatures, the desorption isotherm is located above the adsorption isotherm creating a hysteresis loop which amplitude is more important at low temperatures than at high temperatures.

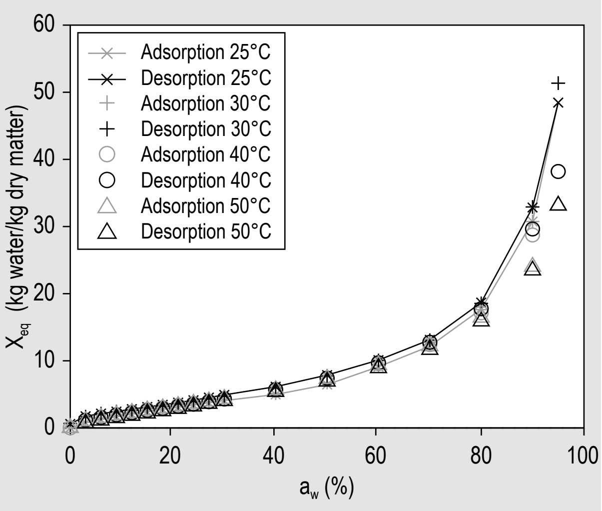

Figure 1. Moisture sorption hysteresis of *Cirina forda*: equilibrium moisture content as a function of the equilibrium relative air humidity in adsorption (grey data) and desorption (black data), at four different temperatures.

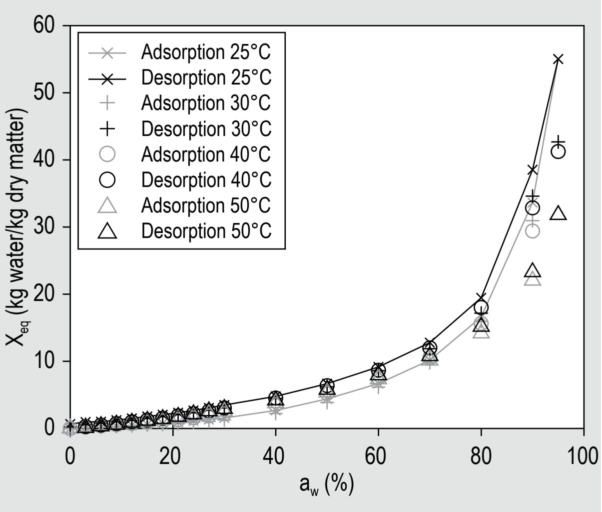

Figure 2. Moisture sorption hysteresis of *Rhyncophorus phoenicis*: equilibrium moisture content as a function of the equilibrium relative air humidity in adsorption (grey data) and desorption (black data), at four different temperatures.

The hysteresis which traduces the dependence of the sorption equilibrium on its sorption history may be seen as a recurrent characteristic of most biological materials (Van den Berg, 1981). To explain the significant hysteresis for Rp and not for Cf, several qualitative explanations can be called. As for materials many biomaterials, affinity for water and polarity of compounds, swelling phenomena, the importance of barriers of diffusion and difference on capillarity on and within these dried larvae may be called. Rp being richer in fat than Cf, it can also be assumed that the difference of the amplitude of the observed hysteresis can be due to the fact that during adsorption, the compounds constituting Rp micropores, having a low affinity for water, are less likely to return to their initial structures. While the Cf micropores would consist of the compounds having a greater affinity for water, the latter dissolving during adsorption tend more to recover their initial structures, hence the difference in amplitude observed for the two insects.

Mathematical modelling

Five sorption isotherm models were used for the analysis of the relationship between insect water content and relative humidity at equilibrium for Cf and Rp. The coefficients and statistics of these two insects are given in Table 1 and 2. The coefficient of correlation r was used as the primary criterion. The RMS was used as a secondary criterion.

Figure 3, presenting a desorption curve at 50 °C, confirms that, as apparent from Table 1 and 2, all models except the GAB model are suitable for simulating the sorption isotherm for both insects. The GAB model gives low-quality fitting mostly at 40 and 50 °C. The Peleg equation gives the best fit for both insects and can be used for predicting the water activity of the insects studied in this work. The same observation was done for others products such as melon seeds (*Cucumis melo*) (Mallek-Ayadi *et al.*, 2020), argan leaves (*Argania spinosa* (L)) (Ennoukh *et al.*, 2022), grapes seeds (*Vitis vinifera*) (Majd *et al.*, 2014), rice (Bingol *et al.*, 2012) and red tilapia viscera (*Oreochromis* spp.) (Camaño *et al.*, 2021). The ability of the Peleg model to give a better fit than the others can be attributed to its four adjustable parameters, whereas all the other suitable models only depend on two adjustable parameters. For fitting with limited experimental data, the Oswin model is recommended.

As the GAB model is also suitable at 25 and 30 °C, its parameter values, which have a physical meaning, can be analysed. The monolayer moisture content X_0 is a value used for predicting the storage condition that will result in the minimum quality loss over a maximum period. According to Quirijns *et al.* (2005), the magnitude of GAB parameters for both insects (C>>1, and K<<1) illustrated the presence of a monolayer with water strongly bound and a multilayer in which the water molecules differ from bulk liquid molecules. At all temperatures, Cf exhibited a higher

Table 1. Coefficients and statistics of mathematical models for sorption isotherms of *Cirina forda* at different temperatures.[1]

Model	Parameter	25 °C		30 °C		40 °C		50 °C	
		Ads.	Des.	Ads.	Des.	Ads.	Des.	Ads.	Des.
GAB	X_0	6.713	6.797	6.851	6.924	6.177	6.210	5.783	5.948
	C	38.44	346.1	37.65	126.2	387.4	307.2	240.3	210.8
	K	0.394	0.399	0.402	0.406	0.362	0.364	0.340	0.325
	RMS	0.5031	0.6407	0.4347	0.5538	0.7366	0.7871	0.7398	0.6820
	r	0.9859	0.9792	0.9904	0.9854	0.9593	0.9537	0.9498	0.9532
Halsey	a	1.355	1.412	1.286	1.286	1.514	1.521	1.610	1.548
	b	10.99	12.86	8.292	8.292	13.93	14.52	15.62	12.48
	RMS	0.1929	0.0582	0.1682	0.1682	0.3448	0.3993	0.3103	0.3166
	r	0.9984	0.9971	0.9988	0.9988	0.9911	0.9884	0.9902	0.9901
Oswin	a	7.091	8.264	7.073	7.963	7.700	7.848	7.419	6.924
	b	0.655	0.605	0.672	0.634	0.557	0.554	0.518	0.541
	RMS	0.0699	0.1319	0.0593	0.1089	0.1796	0.2272	0.1287	0.1361
	r	0.9996	0.9990	0.9998	0.9995	0.9971	0.9956	0.9981	0.9981
Peleg	a	60.72	56.69	71.69	64.72	38.36	37.37	29.67	30.94
	b	11.64	9.694	13.84	11.34	7.685	7.063	8.678	10.39
	c	15.50	14.25	16.74	15.58	12.80	12.76	14.37	15.51
	d	1.065	0.818	1.099	0.918	0.841	0.853	0.933	1.086
	RMS	0.1248	0.1122	0.1404	0.1392	0.0659	0.0667	0.0918	0.0837
	r	0.9990	0.9993	0.9989	0.9990	0.9996	0.9996	0.9990	0.9992
Smith	a	0.000	0.000	0.000	0.000	0.070	0.004	0.544	0.114
	b	14.42	14.33	14.23	14.72	12.05	12.26	10.41	10.45
	RMS	1.0840	0.5230	0.7055	0.6285	0.2629	0.2406	0.1318	0.1539
	r	0.9662	0.9828	0.9735	0.9780	0.9935	0.9947	0.9979	0.9972

[1] Ads. = adsorption, Des. = desorption, RMS = root mean square.

Table 2. Coefficients and statistics of mathematical models for sorption isotherms of *Rhyncophorus phoenicis* at different temperatures.[1]

Model	Parameter	25 °C		30 °C		40 °C		50 °C	
		Ads.	Des.	Ads.	Des.	Ads.	Des.	Ads.	Des.
GAB	X_0	7.100	7.214	6.404	6.516	6.288	6.417	5.562	5.625
	C	3.299	13.675	4.772	18.77	9.74	17.34	23.40	27.84
	K	0.416	0.423	0.376	0.382	0.369	0.376	0.326	0.330
	RMS	0.3978	0.6537	0.5923	0.7794	0.5686	0.7816	0.5441	0.6351
	r	0.9927	0.9814	0.9759	0.9606	0.9757	0.9581	0.9643	0.9539
Halsey	a	1.087	1.252	1.183	1.342	1.259	1.358	1.386	1.436
	b	4.156	8.263	4.723	8.885	5.976	9.016	6.685	8.134
	RMS	0.3876	0.4517	0.5488	0.5857	0.4460	0.5765	0.3507	0.4077
	r	0.9947	0.9922	0.9848	0.9818	0.9888	0.9817	0.9879	0.9840
Oswin	a	5.156	7.416	5.173	6.980	5.734	6.944	5.432	5.918
	b	0.812	0.693	0.736	0.639	0.686	0.629	0.612	0.588
	RMS	0.2354	0.3023	0.3877	0.4176	0.2773	0.3904	0.1903	0.2365
	r	0.9980	0.9962	0.9913	0.9892	0.9950	0.9904	0.9962	0.9943
Peleg	a	73.54	65.80	45.51	46.50	44.26	41.52	31.00	27.31
	b	13.74	9.11	8.46	7.05	9.793	7.109	12.90	10.13
	c	20.82	14.73	14.71	11.78	15.79	13.78	17.02	16.63
	d	2.173	1.152	1.967	1.086	1.588	1.251	1.580	1.456
	RMS	0.0453	0.0837	0.0421	0.1601	0.0439	0.0900	0.0525	0.0403
	r	0.9999	0.9998	0.9999	0.9983	0.9999	0.9994	0.9997	0.9998
Smith	a	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
	b	14.43	15.74	12.16	13.16	12.16	12.16	9.658	9.990
	RMS	1.0839	0.8088	0.7493	0.5432	0.7493	0.7493	0.2566	0.1926
	r	0.9662	0.9786	0.9802	0.9861	0.9802	0.9802	0.9951	0.9978

[1] Ads. = adsorption, Des. = desorption, RMS = root mean square.

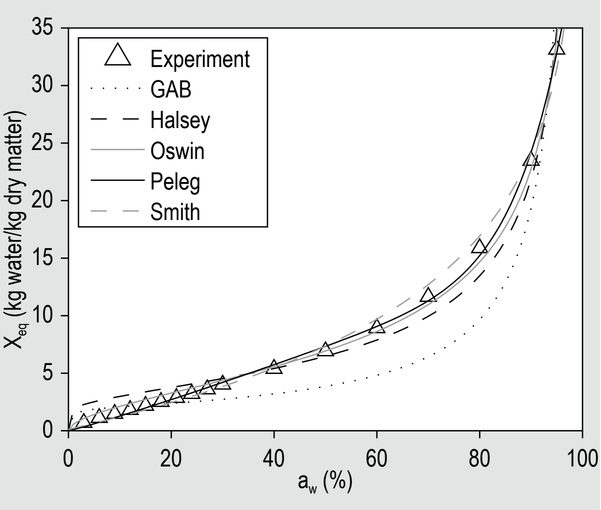

Figure 3. Comparison of the experimental data and the best fit of the five models for the desorption of Cf at 50 °C.

C value than Rp, which indicates that Cf has a monolayer more strongly bound to the material than Rp. Higher X_0 values were obtained for Rp than for Cf. The combination of those elements indicates that it is more expensive to dry Cf than Rp to reach the ideal storage conditions.

Shelf-life estimation

According to the sorption isotherms, to ensure safe storage of Rp at 25 and 30 °C, the larvae must be dried at 8.81 and 8.04% d.b., respectively. For Cf, the caterpillars must be dried to 9.53 and 9.42% d.b. for storage at 25 and 30 °C, respectively. These values are higher than those obtained for dried powders of house cricket and black soldier fly larvae, which vary, respectively, from 5.85 to 6.723% d.b. and from 4.64 to 6.58% d.b. in the temperature range of 25 to 35 °C (Kamau *et al.*, 2018).

Table 3 summarises the equilibrium moisture content that would be reached when the two insects are left in contact with the air without special packaging in the average weather condition of Kinshasa.

Table 3. Equilibrium moisture content of unpacked insects in Kinshasa.

Insect	25 °C	30 °C
Cirina forda	11.2-24.7% d.b.	11.1-24.7% d.b.
Rhyncophorus phoenicis	10.6-27.2% d.b.	10.0-24.7% d.b.

These estimated moisture values show that storage exposing these dried insects to direct contact with the air can cause the moisture content to evolve to values above those that guarantee safe storage. This suggests that for better preservation, dried insects should be packed in airtight packages.

Using the Heiss and Eichner model, as described by Kamau *et al.* (2018), the shelf-life of the dried insect powders can be predicted in relation to possible packaging and storage conditions. The application of this model made it possible to estimate the shelf-life of insects dried at 25 and 30 °C as a function of the initial humidity of conservation. Figure 4 shows, for the two insects studied, that drying at a humidity lower than X_c increases the shelf-life very quickly. Storage at 9% d.b. allows Cf to be kept between 4 and 7 months depending on the temperature, whereas storage at 8% d.b. allows *Rhyncophorus* larvae to be kept from 3 to 10 months depending on the temperature.

Drying allows caterpillars to be kept longer than *Rhyncophorus* larvae. An initial storage humidity of 3% extends the shelf-life above 50 months for both insects at both temperatures. Such a long conservation period is not useful, as drying should mostly allow sufficient conservation to fill the gap between successive harvest seasons. Moreover, reaching such low moisture contents is costly and could be detrimental to the nutritional value of the product due to excessively prolonged heat treatment. Under the studied conditions for storage at 30 °C, which is the average maximum temperature observed in the marketing region studied, we suggest drying the insects for storage at an initial humidity of 7% d.b. for Rp and 8% d.b. for Cf to obtain a shelf-life of at least 1 year. Considering that the insects are harvested annually, a storage period of 12 months would be sufficient to ensure their availability locally until the next harvest.

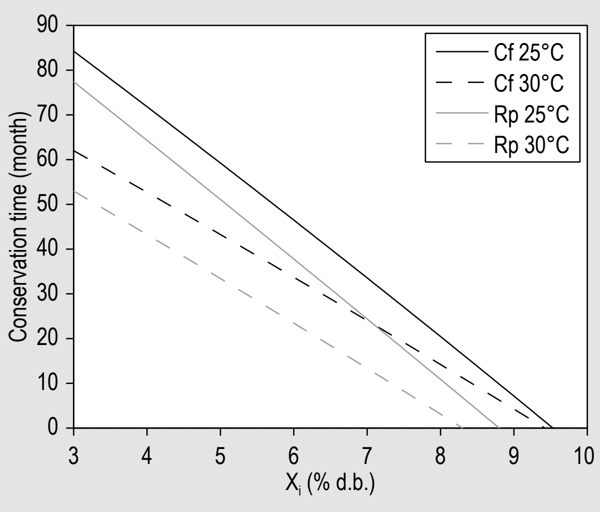

Figure 4. Relationship between shelf-life and initial moisture of storage of *Cirina forda* (black curves) and *Rhyncophorus phoenicis* (grey curves) at 25 °C (continuous lines) and 30 °C (dashed lines).

The shelf-life of the insects predicted using the Heiss-Eichner model is based on the packaging used and storage conditions, manly the equilibrium and initial moisture content of dried insect. This model does not include other parameters, such as lipid oxidation, discoloration or microbial growth (Larouche *et al.*, 2019), whose completion kinetics may render stored insects unsuitable for consumption or for trading.

While extensive drying decreases the initial moisture content of the insects and allows for longer storage times according to Heiss-Eichner's model, very low water contents in lipid-rich materials, such as the two larvae used in this work, can facilitate autocatalytic lipid oxidation (Ssepuuya *et al.*, 2016) and generate undesirable peroxides in these materials. Indeed, fat oxidation quality of insects, as it directly influences the palatability, and health of consumers can become a limiting factor for consumers and trade (Mouithys-Mickalad *et al.*, 2021). It is therefore important to integrate, in future work on the evaluation of drying on the storage capacities of these larvae, a determination of the lipid oxidation products in the estimation of the product lifetimes.

As observed for others foodstuffs processing and storage methods can affect microbial load, colouration and lipid oxidation which must be taken into account to extend the shelf-life of product. Some works have shown a very varied initial microbial load in insect larvae and the presence of some pathogenic bacteria such as *Salmonella* spp., *Staphylococcus aureus*, *Escherichia coli* and *Bacillus cereus* (Dooshima, 2014; Gold *et al.*, 2018; Kashiri *et al.*, 2018; Megido *et al.*, 2017; Wynants *et al.*, 2019). Dooshima (2014) studied the microbial quality of *C. forda* caterpillar from nigerian market and found a total viable count of microorganisms ranges from 2.0 to 3.7 log cfu/g. Megido *et al.* (2017) found total aerobic count of 7.63 log cfu in smoked *C. forda* caterpillar bought in traditional market in Belgium. Techniques such as blanching permit to minimise microbial contamination and also lipid oxidation and colour alteration (Larouche *et al.*, 2019; Megido *et al.*, 2017). However, the application of such a model requires definition of a safe storage moisture content of the product corresponding to the safe storage borderline a_W, which can be determined experimentally.

Isosteric heat of sorption

The net isosteric heats of sorption for different moisture content presented in Figure 5 were shown to decrease when the moisture content increases for both insects. The isosteric heat evolution with the moisture content globally follows a hyperbolic law (drawn on Figure 5) that expresses a regular reduction of the interaction of the adsorbed water as the number of layers rises. This behaviour is coherent with the calculated parameters of the GAB model (C>>1, and K<<1). As this analysis is based on a series of mathematical transformations on a very limited set of data, further interpretation should be considered with care.

Above 40% d.b., the values of isosteric heat are below 20 kJ/mol, which is less than 5% of the latent heat of vaporisation of water. This suggests that the water added above this moisture content is comparable to free water. Kamau *et al.* (2018) observed for both house cricket and black soldier fly larvae above 20 g/100 g equilibrium moisture water existed in free liquefied form.

4. Conclusions

The study of the equilibrium behaviour of *C. forda* and *R. phoenicis* larvae highlights similar behaviour for both insects: a type III isotherm expressing a multilayer adsorption well described by the GAB model below 40 °C. Above this temperature, the use of empirical models allows a continued good fit of the isotherm. The four-parameter Peleg model gives the best fit to the experimental sorption data of Cf and Rp but could be replaced by the two-parameter Oswin model if limited experimental data are available.

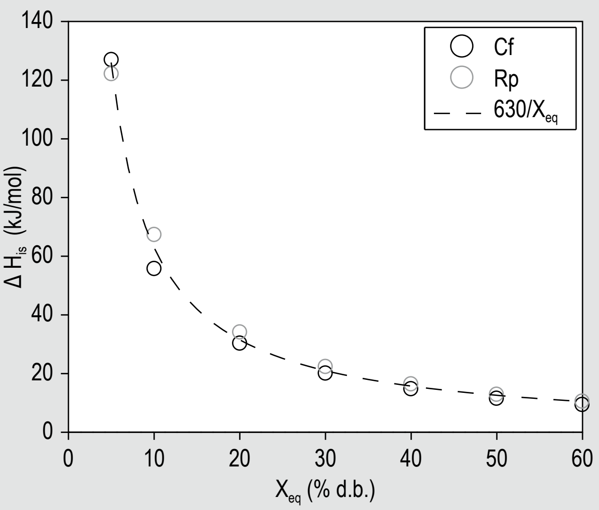

Figure 5. Net isosteric heat of desorption of *Cirina forda* (black circles) and *Rhyncophorus phoenicis* (grey circles).

The data provided by the isotherm can be used to propose conservation strategies for the larvae of these two insects. Considering storage in an airtight polyethylene package, drying to moisture contents of 7 and 8% d.b., respectively, for Rp and Cf are proposed to ensure a preservation of 12 months at 30 °C. The results obtained in this work do not take into account the alterations due to factors such as the growth of microorganisms, lipids oxidation and colour alteration during storage which should also be taken into account for a better estimation of the product's shelf-life.

Conflict of interest

The authors declare no conflict of interest.

References

Akullo, J., Obaa, B.B., Acai, J.O., Nakimbugwe, D. and Agea, J.G., 2017. Knowledge, attitudes and practices on edible insects in Lango sub-region, northern Uganda. Journal of Insects as Food and Feed 3: 73-81.

Azzollini, D., Derossi, A. and Severini, C., 2016. Understanding the drying kinetic and hygroscopic behaviour of larvae of yellow mealworm (*Tenebrio molitor*) and the effects on their quality. Journal of Insects as Food and Feed 2: 233-243.

Bingol, G., Prakash, B. and Pan, Z., 2012. Dynamic vapor sorption isotherms of medium grain rice varieties. LWT – Food Science and Technology 48: 156-163.

Brunauer, S., Deming, L.S., Deming, W.E. and Teller, E., 1940. On a theory of the Van der Waals adsorption of gases. Journal of the American Chemical Society 62(7): 1723-1732.

Bui, R., Labat, M. and Aubert, J.E., 2017. Comparison of the saturated salt solution and the dynamic vapor sorption techniques based on the measured sorption isotherm of barley straw. Construction and Building Materials 141: 140-151.

Camaño Echavarria, J.A., Rivera Torres, A.M. and Zapata Montoya, J.E., 2021. Sorption isotherms and thermodynamic properties of the dry silage of red tilapia viscera (*Oreochromis* spp.) obtained in a direct solar dryer. Heliyon 7(4): e06798. https://doi.org/10.1016/j.heliyon.2021.e06798

Chuzel, G. and Zakhia, N., 2007. Adsorption isotherms of gari for estimation of packaged shelf-life. International Journal of Food Science and Technology 26: 583-593.

Dooshima Igbabul, B., 2014. Nutritional and microbial quality of dried larva of *Cirina forda*. International Journal of Nutrition and Food Sciences 3: 602.

Dupas-Langlet, M., Dupas, J., Samain, S., Giardiello, M.I., Meunier, V. and Forny, L., 2016. A new method to determine 'equilibrated' water activity and establish sorption isotherm by erasing surface history of the samples. Journal of Food Engineering 184: 53-62.

Ennoukh, F., Sghaier, K. and Bchitou, R., 2022. Efficacy moisture sorption isotherms and net isosteric heat of sorption for argan leaves (*Argania spinosa* (L) Skeels). Journal of Food Processing and Technology 13: 942.

Erbaş, M., Aykın, E., Arslan, S. and Durak, A.N., 2016. Adsorption behaviour of bulgur. Food Chemistry 195: 87-90.

Gere, A., Radványi, D. and Héberger, K., 2019. Which insect species can best be proposed for human consumption? Innovative Food Science and Emerging Technologies 52: 358-367.

Gold, M., Tomberlin, J.K., Diener, S., Zurbrügg, C. and Mathys, A., 2018. Decomposition of biowaste macronutrients, microbes, and chemicals in black soldier fly larval treatment: a review. Waste Management 82: 302-318.

Hajar, E., Rachid, T. and Najib, B.M., 2017. Conception of a solar air collector for an indirect solar dryer. Pear drying test. Energy Procedia 141: 29-33.

Hartmann, C. and Siegrist, M., 2016. Becoming an insectivore: results of an experiment. Food Quality and Preference 51: 118-122.

Hartmann, C. and Siegrist, M., 2018. Development and validation of the food disgust scale. Food Quality and Preference 63: 38-50.

Hossain, M.D., Bala, B.K., Hossain, M.A. and Mondol, M.R.A., 2001. Sorption isotherms and heat of sorption of pineapple. Journal of Food Engineering 48: 103-107.

Ikhu-Ornoregbe', D.I.O. and Chen, X.D., 2005. Use of sorption isotherms for the estimation of shelf life of two Zimbabwean flours. Developments in Chemical Engineering and Mineral Processing 13: 79-90.

Kamau, E., Mutungi, C., Kinyuru, J., Imathiu, S., Tanga, C., Affognon, H., Ekesi, S., Nakimbugwe, D. and Fiaboe, K.K.M., 2018. Moisture adsorption properties and shelf-life estimation of dried and pulverised edible house cricket *Acheta domesticus* (L.) and black soldier fly larvae *Hermetia illucens* (L.). Food Research International 106: 420-427.

Kashiri, M., Marin, C., Garzón, R., Rosell, C.M., Rodrigo, D. and Martínez, A., 2018. Use of high hydrostatic pressure to inactivate natural contaminating microorganisms and inoculated *E. coli* O157:H7 on *Hermetia illucens* larvae. PLOS ONE 13: e0194477.

Kinyuru, J.N., Kenji, G.M., Njoroge, S.M. and Ayieko, M., 2010. Effect of processing methods on the *in vitro* protein digestibility and vitamin content of edible winged termite (*Macrotermes subhylanus*) and grasshopper (*Ruspolia differens*). Food and Bioprocess Technology 3: 778-782.

Kröncke, N., Böschen, V., Woyzichovski, J., Demtröder, S. and Benning, R., 2018. Comparison of suitable drying processes for mealworms (*Tenebrio molitor*). Innovative Food Science and Emerging Technologies 50: 20-25.

Larouche, J., Deschamps, M.-H., Saucier, L., Lebeuf, Y., Doyen, A. and Vandenberg, G.W., 2019. Effects of killing methods on lipid oxidation, colour and microbial load of black soldier fly. Animals 9: 182.

Leonardo Betiol, L.F., Evangelista, R.R., Ribeiro Sanches, M.A., Basso, R.C., Gullón, B., Lorenzo, J.M., Carla da Silva Barretto, A. and Romero, J.T., 2020. Influence of temperature and chemical composition on water sorption isotherms for dry-cured ham. LWT – Food Science and Technology 123: 109112.

Lima, A.W. and Cal-vidal, J., 1988. estimation of shelf life of film-packaged freeze-dried banana. Journal of Stored Products Research 24: 73-78.

Majd, K.M., Karparvarfard, S.H., Farahnaky, A. and Ansari, S., 2014. Thermodynamic properties of water sorption isotherms of grape seed. International Agrophysics 28: 63-71.

Mallek-Ayadi, S., Bahloul, N. and Kechaou, N., 2020. Mathematical modelling of water sorption isotherms and thermodynamic properties of *Cucumis melo* L. seeds. LWT – Food Science and Technology 131: 109727.

Mancini, S., Moruzzo, R., Riccioli, F. and Paci, G., 2019. European consumers' readiness to adopt insects as food – a review. Food Research International 122: 661-678.

Martínez-Las Heras, R., Heredia, A., Castelló, M.L. and Andrés, A., 2014. Moisture sorption isotherms and isosteric heat of sorption of dry persimmon leaves. Food Bioscience 7: 88-94.

Megido, R.C., Desmedt, S., Blecker, C., Béra, F., Haubruge, É., Alabi, T. and Francis, F., 2017. Microbiological load of edible insects found in Belgium. Insects 8: 12.

Melgar-Lalanne, G., Hernández-Álvarez, A.J. and Salinas-Castro, A., 2019. Edible insects processing: traditional and innovative technologies. Comprehensive Reviews in Food Science and Food Safety 18: 1166-1191.

Mghazli, S., Idlimam, A., Mahrouz, M., Lahnine, L., Hidar, N., Ouhammou, M., Mouhib, M., Zantar, S. and Bouchdoug, M., 2016. Comparative moisture sorption isotherms, modelling and isosteric heat of sorption of controlled and irradiated Moroccan rosemary leaves. Industrial Crops and Products 88: 28-35.

Mouithys-Mickalad, A., Tome, N.M., Boogaard, T., Serteyn, D., Schmitt, E. and Paul, A., 2021. Evaluation of the fat oxidation quality of commercial *Hermetia illucens* meal. Journal of Insects as Food and Feed 7: 965-974.

Niassy, S., Fiaboe, K.K.M., Affognon, H.D., Akutse, K.S., Tanga, M.C. and Ekesi, S., 2016. African indigenous knowledge on edible insects to guide research and policy. Journal of Insects as Food and Feed, 2: 161-170.

Nsevolo, P., Taofic, A., Caparros, R., Sablon, L., Haubruge, É. and Francis, F., 2016. Entomological biodiversity as a food in Kinshasa (Democratic Republic of Congo). Annales de la Societe Entomologique de France 52: 57-64.

Oswin, C.R., 1946. The kinetics of package life. III. The isotherm. Journal of the Society of Chemical Industry 65: 419-421.

Peleg M., 1993. Assessment of a semi-empirical four parameter general model of sigmoid moisture sorption isotherms. Journal of Food Process Engineering 16: 21-37.

Quirijns, E.J., Van Boxtel, A.J.B., Van Loon, W.K.P. and Van Straten, G., 2005. Sorption isotherms, GAB parameters and isosteric heat of sorption. Journal of the Science of Food and Agriculture 85: 1805-1814.

Ssepuuya, G., Aringo, R.O., Mukisa, I.M. and Nakimbugwe, D., 2016. Effect of processing, packaging and storage-temperature based hurdles on the shelf stability of sautéed ready-to-eat *Ruspolia nitidula*. Journal of Insects as Food and Feed 2: 245-253.

Sun, H., Necochea Velazco, O., Lakemond, C., Dekker, M., Cadesky, L. and Mishyna, M., 2021. Differences in moisture sorption characteristics and browning of lesser mealworm (*Alphitobius diaperinus*) ingredients. LWT – Food Science and Technology 142: 110989.

Van Den Berg, C., 1981. Vapor sorption equilibria and other water starch interactions: a physico-chemical approch. PhD Thesis, Wageningen University, Wageningen, the Netherlands.

Wynants, E., Frooninckx, L., Crauwels, S., Verreth, C., De Smet, J., Sandrock, C., Wohlfahrt, J., Van Schelt, J., Depraetere, S., Lievens, B., Van Miert, S., Claes, J. and Van Campenhout, L., 2019. Assessing the microbiota of black soldier fly larvae (*Hermetia illucens*) reared on organic waste streams on four different locations at laboratory and large scale. Microbial Ecology 77: 913-930.

Yen, A.L., 2009. Edible insects: traditional knowledge or western phobia? Entomological Research 39: 289-298.