Food Research International 106 (2018) 420–427

Contents lists available at ScienceDirect

Food Research International

journal homepage: www.elsevier.com/locate/foodres

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.)

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ARTICLE INFO

Keywords:
Edible insects
Processing
Storage
Water activity
Sorption isotherm
Isosteric heat

ABSTRACT

Edible insects are part of the diets of a significant proportion of rural populations in the tropics especially Africa and Asia, and their use as source of key nutrients for better nutrition is re-emerging. Indigenously, elemental methods are used to process the insects before they are consumed or sold in retail outlets. In recent years, better knowledge of processing, packaging and storage has become necessary because of commercialisation needs. A common processing approach involves drying after a brief heat-treatment step, and then milling into a powdered product which is sold to manufacturers or consumers as ingredient for processing final products. The hydration properties of dried powders of edible house cricket and black soldier fly larvae (BSFL) were studied with the aim of predicting shelf-life stability under typical packaging and storage temperatures experienced in the tropics. Moisture adsorption isotherms were determined gravimetrically at 25, 30 and 35 °C, over 0.11–0.97 water activity (a_W) range, and the data fitted to various models. Sorption isotherms were of type II according to Brunauer classification indicating monolayer-multilayer sorption behaviour. Cricket powder exhibited higher hydration capacity, and a_W of this product was less sensitive to temperature variation as compared to BSFL powder. In the two products, water exhibited transitions from bound- to free- state at ~5 g/100 g moisture content. Based on Heiss-Eichner model, a shelf-life of 7 months at 25 °C can be achieved if the cricket and BSFL powders are dried to ca. 5 g/100 g moisture content and packaged in 80 μm thick polyethylene films. At 35 °C the shelf-life of the cricket product is shortened three- to four-fold whereas the BSFL powder is unable to store.

1. Introduction

Utilisation of insects as a direct or indirect nutrient source in diets of humans has become of interest in recent years due to their diverse social, economic and nutritional benefits (Mlcek, Rop, Borkovcova, & Bednarova, 2014). The Food and Agricultural Organisation of the United Nations (FAO) reported that some 1900 edible species are known to be eaten in 80% of the world's nations (Van Huis et al., 2013). At least 2 billion people globally eat insects in over 113 countries (Barennes, Phimmasane, & Rajaonarivo, 2015). Furthermore, the use of insects for food or feed is considered as a solution to environmental pollution due to their low emission of greenhouse gases and higher feed conversion rates as compared to conventional livestock (Van Huis et al., 2013). Edible insects can therefore be used in alleviating some of the most pressing food security problems such as protein, energy and micro-nutrients malnutrition in many developing countries, and high cost of good quality animal feeds (Klunder, Wolkers-Rooijackers, Korpela, & Nout, 2012; Van Huis et al., 2013). The house cricket, *Acheta domesticus* (L.) (Orthoptera: Gryllidae) is a cosmopolitan omnivorous insect believed to be native to Southwestern Asia (Bello, Naroka, Abubakar, Helmina, & Olusanya, 2013; Parajulee, DeFoliart, & Hogg, 1993), but has spread worldwide (Ayieko, Ogola, & Ayieko, 2016; Van Huis et al., 2013). The black soldier fly, *Hermetia illucens* L. (Diptera: Stratiomyidae) is native to the Neotropics, but is now found in every

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https://doi.org/10.1016/j.foodres.2018.01.012
Received 22 August 2017; Received in revised form 7 January 2018; Accepted 9 January 2018
Available online 11 January 2018

zoogeographic region following decades of spreading throughout the warmer parts of the world (Marshall, Skevington, Kelso, & Zhou, 2015). There is considerable interest in using *H. illucens* larvae for organic waste control and composting, but the larvae also are highly viewed as nutritionally rich alternative protein source in dairy, pig, poultry and aquaculture feeds as well as pet foods (Oliveira, Doelle, List, & O'Reilly, 2015).

As nutrient sources, fresh adult crickets and black soldier fly larvae are highly perishable and require prompt processing for shelf-life extension. To understand the stability during storage, the relationship between moisture content and water activity needs to be established. Moisture sorption isotherms describe the relationship between the moisture content of a material and the moisture level of the surrounding at a constant temperature (Klofutar & Abramovic, 2006). Hence moisture sorption isotherms help to identify critical moisture contents of storage, that can assist to improve shelf-life of stored products; they are fundamental in making packaging and storage decisions (Jha, Dahiya, & Singh, 2014). Moreover, the sorption energy e.g. isosteric heat of sorption that is derived from the moisture sorption data and provide information concerning the state of water in a food (Aktas, Ulger, Daglioglu, & Sahin, 2014). Over 200 models have been proposed by different researchers for simulation of sorption behaviours of different food matrices (Goneli, Ju, & Corre, 2016). Generally, it is difficult to find mathematical models that describe accurately sorption isotherms over the entire range of water activity for the various types of foods (Akoy, Von Horsten, & Ismail, 2013) because every substrate has its own unique interaction with moisture depending on its composition. However, fitting carefully selected models can help to describe sorption behaviour, thus provide useful information on the state of water and its interaction with a substrate.

To the best of our knowledge, moisture sorption properties of processed edible insects have not been studied. For convenience of packaging, storage and utilisation in various product systems, edible insects are likely to be dried and milled into powders, which are then packaged and sold to manufacturers or consumers as ingredients for processing final products. Understanding moisture sorption is important for appropriate packaging and storage as package selection and storage recommendations can be made based on empirical knowledge of the hydration behaviour of products. Furthermore, the thermodynamics of sorption give important information for drying operations such as energy requirements, which is vital in designing drying equipment. The objective of this study was to investigate hydration properties of dried powders of *A. domesticus* and *H. illucens*, and to model the shelf-life under typical handling conditions in Africa. The two insects were selected based on their high protein content. They have also been identified as suitable species for mass production based on ease of rearing, biomass supply, environmental implications (European Food & Scientific Committee, 2015).

2. Materials and methods

2.1. Experimental materials

Adult crickets and black soldier fly larvae (BSFL) harvested at the pre-pupae stage were obtained from stock colonies maintained at the Animal Rearing and Containment Unit of the International Centre of Insect Physiology and Ecology (*icipe*), in Nairobi, Kenya. The insects were blanched in boiling water (98 °C) for 5 min and dried in a solar tent to constant weight. The design of the solar tent consisted of a clear plastic sheet stretched over a wooden box (0.6 m wide × 1.2 m long × 0.2 m high), which was placed longitudinally on a slanting metal frame constructed to a height of 1 m on the air inlet end, and 1.2 m on the air exit end. The inside of the box was painted black and the air entry and exit ends were drilled with closely space holes of 1 cm diameter. The temperature and relative humidity in the solar tent before introducing the samples ranged between 50 and 60 °C and 15–25%, respectively, as determined using an El-USB-2 data logger (Lascar electronics Inc. Pennsylvania, USA). After drying to constant weight, the insect materials were milled using a kitchen blender (Philips HR, 2850) and then passed through a 1 mm sieve. Samples of the sieved material were then taken and analysed for proximate composition according to AOAC (1996) methods.

Table 1
Water activity of saturated salts.

Salt	Water activity (a_W)		
	25 °C	30 °C	35 °C
Lithium chloride (LiCl)	0.11	0.11	0.11
Potassium acetate (KCH_3COO)	0.23	0.22	0.22
Magnesium chloride ($MgCl_2$)	0.33	0.32	0.32
Potassium carbonate (K_2CO_3)	0.43	0.43	0.43
Magnesium nitrate ($Mg(NO_3)_2$)	0.53	0.51	0.50
Potassium iodide (KI)	0.69	0.68	0.67
Sodium chloride (NaCl)	0.75	0.75	0.75
Potassium chloride (KCl)	0.84	0.84	0.83
Potassium sulphate (K_2SO_4)	0.97	0.97	0.97

Source: Greenspan (1977).

2.2. Obtaining moisture adsorption isotherms

The static gravimetric method was used. Samples were equilibrated under saturated salt solutions with water activity (a_W) of 0.11–0.97 (Table 1). All salts were reagent grade. Each saturated salt solution was contained in separate airtight glass desiccators (25 cm diameter) complete with lid and plate (23 cm diameter porcelain plate with 7 holes of 3 cm in diameter, and with feet to elevate plate 16 cm above the floor of the desiccator). Each desiccator was labelled with the respective a_W. Approximately 0.5 g samples of the insect powders were weighed into pre-weighed aluminium dishes (8 cm long × 5 cm wide × 2 cm deep). These were first pre-dried for 7 days over phosphorous pentoxide in a desiccator to dehydrate fully, and the weight of the dehydrated samples (w_1) was taken. Triplicates of these samples were stored over each of the saturated salts in the airtight desiccators, which were then placed in incubators maintained at 25 °C, 30 °C or 35 °C throughout the equilibration period. At (a_W) > 0.6, crystalline thymol (2-isopropyl-5-methylphenol) was placed inside desiccators to inhibit fungal growth. The samples were weighed after every 48 h until change in weight was negligible ($\leq$ 0.001 g/day). The new weight of each of the equilibrated samples was taken (w_2). Equilibrium moisture contents (M_{eq}) were determined as $(w_2 - w_1)/w_1$, and expressed in g/100 g dry basis.

2.3. Mathematical modelling of sorption isotherms

Experimental data were fitted to Guggenheim-Anderson-de Boer (GAB), Brunauer-Emmet-Teller (BET), Caurie, Smith, and Khun models. Mathematical expressions of these models are presented in Table 2. Model parameters were obtained by nonlinear regression analysis using Origin Scientific Graphing and Analysis software version 8 (OriginLab Corporation, Northampton, MA, USA).

2.4. Data analysis

Goodness of fit of each model was evaluated using coefficient of determination (R^2) and percent root mean square error (RMSE %). The R^2 indicates how well a given model explains the variability of the predicted values, whereas RMSE (%) provides a measure of overall model error; RMSE < 10% indicates a good fit (Igbabul, Ariahu, & Umeh, 2013). The RMSE (%) was computed using the expression:

Table 2
Mathematical expressions of models applied to experimental data.

Model and [applicable a_W range]	Expression	Reference
Brunauer-Emmet-Teller (BET) [$a_W < 0.43$]	$M_{eq} = \frac{M_O \times C_b \times a_W}{(1-a_W)(1+(C_b-1)a_W)}$	(Brunauer, Emmet, & Teller, 1938)
Guggenheim-Anderson-de Boer (GAB) [$a_W < 0.75$]	$M_{eq} = \frac{M_O \times C_g \times k_g \times a_W}{(1-k_g a_W)(1+(C_g-1)k_g a_W)}$	(Anderson, 1940)
Smith [$a_W > 0.5$]	$M_{eq} = A + B \ln(1 - a_W)$	(Smith, 1947)
Khun [$a_W < 0.75$]	$M_{eq} = \frac{A}{\ln a_W} + B$	(Khun, 1967)
Caurie [$a_W < 0.98$]	$\frac{1}{M_{eq}} = \frac{1}{C_c M_O}\left[\frac{1-a_W}{a_W}\right]^{(2C_c/M_O)}$	(Caurie, 1981)

M_{eq}: equilibrium moisture content (g/100 g); a_W: water activity; M_o: monolayer moisture content (BET, GAB and Caurie models); : BET constant (BET model); and K_g: GAB constants (GAB model); A and B: constants in Smith and Khun models;C_c: Caurie constant (Caurie model).

$$\mathrm{RMSE}\,(\%) = 100 \times \left[\frac{\sum_{i=1}^{n}((M_{e(i)} - M_{p(i)})/M_{e(i)})^2}{n}\right]^{1/2} \tag{1}$$

where M_e and M_p are the equilibrium moisture contents determined experimentally and predicted from the sorption models, respectively, at $a_{w(i)}$, and n is the number of data points. Experimental data were also analysed using Stata version 12 for variance and where necessary, means were separated using Bonferroni adjustment at $p = .05$.

2.5. Modelling of isosteric heats of sorption

The Clausius-Clapeyron equation was used to estimate the net isosteric heat of sorption by plotting the $\ln a_W$ against the inverse of absolute temperature ($1/T$) at constant equilibrium moisture content according to the expression:

$$\ln(a_W) = -(q_{st}/R)/(1/T) + K \tag{2}$$

where q_{st} is net isosteric heat of sorption (kJ/mol K), a_W is the water activity (dimensionless), T is absolute temperature (in Kelvin, K), R is the universal gas constant (8.314 J/mol K) and K is a constant. This relationship is based on the assumptions that moisture content of the system is constant, and heat of vaporisation of pure water and excess heat of sorption are invariant with temperature. The slope of the least squares regression fit ($-q_{st}/R$) was used to obtain q_{st} because R is known. This procedure was repeated for a number of equilibrium moisture contents so as to deduce the dependency of isosteric heat of sorption on moisture content.

3. Results and discussion

3.1. Proximate composition

The moisture contents of the dried cricket and black soldier fly larvae powders were 4.52 ± 0.16 and 6.41 ± 0.73, respectively. Protein, fat, fibre, ash, and carbohydrate contents of adult cricket and BSFL powders are presented in Table 3 on dry matter basis. The composition agrees with previous values reported in the literature for the two insects (Finke, 2002; Tran, Gnaedinger, & Mélin, 2015). The cricket powder contained significantly higher ($p < .001$) crude protein, which is comparable to the crude protein content of fresh African palm weevil *Rhynchophorus phoenicis* (Fabricius) (Coleoptera: Curculionidae) reported to be 66.1% (Ekpo, 2011). Crude protein content of BSFL compared well to that of traditionally processed (boiled in salty water for 1 h and sun-dried) *Hemijana variegata* Rothschild (Lepidoptera: Euptoritidae) caterpillar (44.5%) (Egan, Toms, Minter, Masoko, & Olivier, 2014). The BSFL powder contained significantly ($p < .001$) higher levels of fat, fibre, and ash. Fat content of BSFL was comparable to that of *R. phoenicis* (21.2%) while that of cricket was lower than that reported for traditionally processed *H. variegata* caterpillar (19.8%) (Egan et al., 2014). The ash contents were higher than those reported for yellow mealworm (*Tenebrio molitor* L. (Coleoptera: Tenebrionidae)) (2.5%) and *H. variegata* caterpillar (2.4%) (Egan et al., 2014). The fibre contents compared well with those recorded for edible termites (6%) (Raksakantong, Meeso, Kubola, & Siriamornpun, 2010). The available carbohydrate content of both insect meals was higher than that reported for the beetle *Tenebrio molitor* (0.01–3.86%) and the stink bug *Euschistus strennus* (0.01%) (Rumpold & Schluter, 2013) showing that when consumed a higher amount of carbohydrate would be digested and absorbed.

Table 3
Proximate composition (g/100 g dry basis) of cricket and BSFL powders.

Parameter	Cricket	BSFL
Crude protein	65.85 ± 4.80^a	44.04 ± 0.40^b
Crude fat	12.16 ± 0.24^b	24.95 ± 0.50^a
Crude fibre	6.83 ± 0.09^b	9.97 ± 0.43^a
Crude ash	4.81 ± 0.16^b	9.50 ± 0.04^a
Available carbohydrate[1]	10.34 ± 5.02^a	11.54 ± 1.45^a

Means ± standard deviation (n = 3) followed by same superscript letters in the same row are not significantly different ($p > .05$).

[1] Available carbohydrate was calculated as 100 − (crude protein + crude fat + crude fibre + ash).

3.2. Moisture sorption isotherms

Equilibrium moisture contents (M_{eq}) of cricket and BSFL powders at different relative humidity levels of are presented in Fig. 1. The two substrates exhibited type II sorption, which is due to monolayer-multilayer sorption mechanism (Brunauer et al., 1938). At low a_W, moisture is adsorbed to active binding sites. As the a_W increases, more water molecules become covalently associated to the bound water creating multilayer moisture that is less strongly bound. Further increase in multilayer sorption at higher a_W levels causes dissolution of soluble low molecular weight constituents, which brings about a sharp increase in the equilibrium moisture content at $a_W > 0.8$. Type II isotherms have also been reported for mealworm larvae (*T. molitor*) (Azzollini, Derossi, & Severini, 2016) and other high protein foods such as Pirarucu (*Arapaima gigas* (Schinz) (Osteoglossiformes: Arapaimidae)) fillet (Martins, Martins, & Pena, 2015) and lean beef (Trujillo, Yeow, & Pham, 2003). Cricket powder adsorbed significantly higher amount moisture than BSFL powder across all the water activity levels at the three temperature regimes (25 °C: $p < .003$; 35 °C: $p < .001$; 35 °C: $p < .001$). This observation can be attributed to compositional and physicochemical differences. From the proximate composition, BSFL powder contained less protein and more fat, and would therefore have fewer hydrophilic sites as compared to cricket powder. For both substrates, the adsorption decreased significantly with increasing temperature at all water activity level (cricket powder: $p < .001$; BSFL: $p < .001$) as expected. The change was more evident in the BSFL powder. The decrease in adsorbed moisture with increasing equilibration temperature can be attributed to the excitation of water molecules to higher energy levels, thus causing them to break away from their binding sites (Al-Mahasneh, Alkoaik, Khalil, Fulleros, & El-Waziry, 2014). Proteins are also known to have more water binding capacities at the lower temperature (Ko, Hong, Min, & Choi, 2008). At a constant M_{eq}, a_W of the two substrates increased with increase in equilibration temperature, but the BSFL powder was evidently more sensitive to this effect. For instance, at 5 g/100 g M_{eq}, increasing the equilibration temperature from 25 to 35 °C increased the a_W of BSFL powder from 0.49 to 0.67. A water activity as low as 0.60 can support the growth of yeasts and moulds (Sperber, 1983). The BSFL powder exhibited a higher sensitivity due to its higher

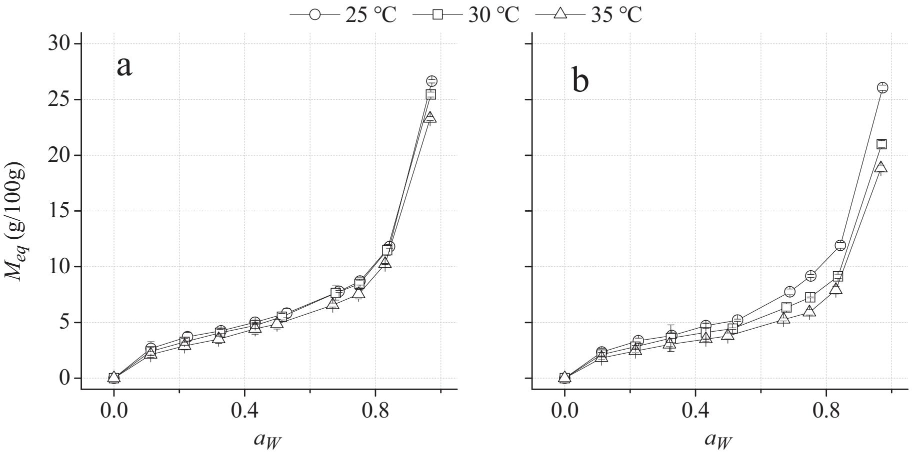

Fig. 1. Moisture adsorption isotherms of cricket (a) and black soldier fly larvae (b) powders.

fat content and lower protein content meaning that it had fewer active bonding sites. It would therefore be easier to excite water molecules to higher energy levels in BSFL than in the cricket powder causing them to break away and increase the water activity. Moreover, an increase in a_W by 0.1 can decrease shelf-life of a food or feed by a factor of two to three (Labuza, 1984). Thus for safe storage in warmer environments, drying to much lower moisture contents would be needed for BSFL powder as compared to cricket powder.

3.3. Fitting of sorption models

Fig. 2 and Fig. 3 show the experimental data fitted to GAB, BET, Caurie, Smith and Khun models over the theoretically applicable a_W ranges (Table 2). Model parameters alongside the measures of fit are presented in Table 4. All the models fitted the experimental data well with RMSE of ≤10% except the smith model. The GAB and BET models provided the best fits judging from the RMSE (≤3%) and R^2 values (≥0.995). The GAB simulation fitted a broader a_W range compared to BET model. Although the two models provided the best fits to experimental data, the BET model overestimated the sorption when a_W exceeded 0.43 and could therefore be used to characterise monolayer sorption only. The GAB model underestimated the sorption when a_W exceed 0.75. The Caurie model provided good descriptions to experimental data over the entire water activity range with RMSE values of < 10% and $R^2 > 0.99$. It could therefore be used in characterising monolayer, multilayer and free water (capillary) sorption. The Smith model did not give adequate fit (RMSE > 10%) to the experimental data. Furthermore, the model underestimated sorption at $a_W < 0.6$. The Khun ($a_W < 0.75$) model described the sorption data quite well with $R^2 > 0.97$ and RMSE < 10% but overestimated sorption at $a_W < 0.2$ and > 0.75. Thus unlike the GAB model whose upper limit is theoretically the same ($a_W = 0.75$), the Khun model was not appropriate in the monolayer region.

3.4. Properties of sorbed water

The BET, GAB and Caurie models are superior because the magnitudes of their parameters have physical meaning (Rao et al., 2006; Timmermann, 2003), unlike those of the Khun and Smith models, which at best, provided mathematical compensation for curve fitting. Theoretically, the parameters C_b and C_g of the BET and GAB equations relate to the net enthalpies of sorption of the monolayer whereas k_g from GAB model relates to the enthalpy of sorption of water molecules that build above the monolayer (Mutungi et al., 2011). For the two substrates C_b and C_g decreased with increasing equilibration temperature, indicating that monolayer binding energies decreased. The parameter k_g also decreased with increasing equilibration temperature particularly for BSFL powder, pointing to a less pronounced multilayer sorption and subsequent liquefaction of water molecules in the void spaces of the material (Timmermann, 2003).

The BET, GAB and the Caurie models also estimated the monolayer moisture loading (M_o), that is, the moisture content when all the ionic and polar groups of the adsorbent have been occupied by water molecules (Valdez-Niebla, Paredes-Lopez, Vargas-Lopez, & Hernandez-Lopez, 1993). The M_o is the moisture content for utmost stability of a food/feed material (Igbabul et al., 2013). The BET model predicted lower M_o values (2.1–3.0 g/100 g) compared to GAB (2.6–3.6 g/100 g) and Caurie (4.0–5.1 g/100 g) model. The GAB model is a refinement of the BET model which covers a wider a_W range and always gives a higher monolayer value (Timmermann, 2003). Also, the Caurie model's higher monolayer values could be attributed to a wider a_W range of applicability of < 0.98 as compared to both the GAB (< 0.75) and BET (< 0.43) models. The M_o values, nevertheless, compared well with those reported for dried milled fish (3.24–5.12%) at 25–50 °C where the

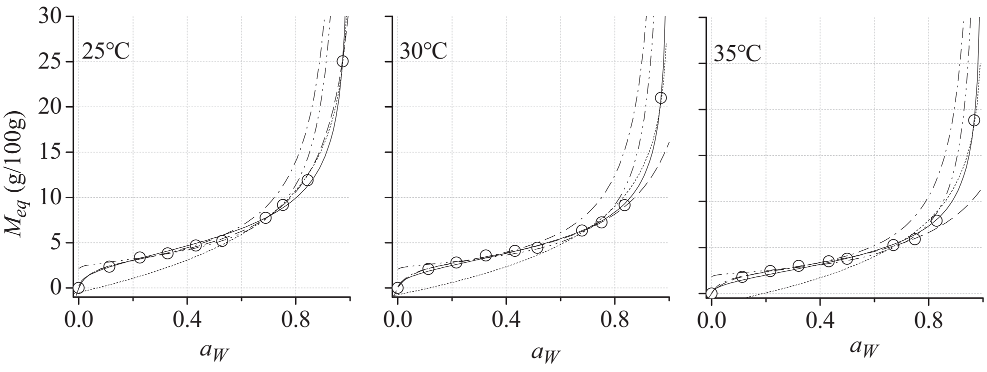

Fig. 2. Moisture adsorption experimental data (circle) of cricket powder fitted with BET (dash-dot), GAB (dash-dash), Caurie (solid line), Smith (dotted) and Khun (dash-dot-dot) models.

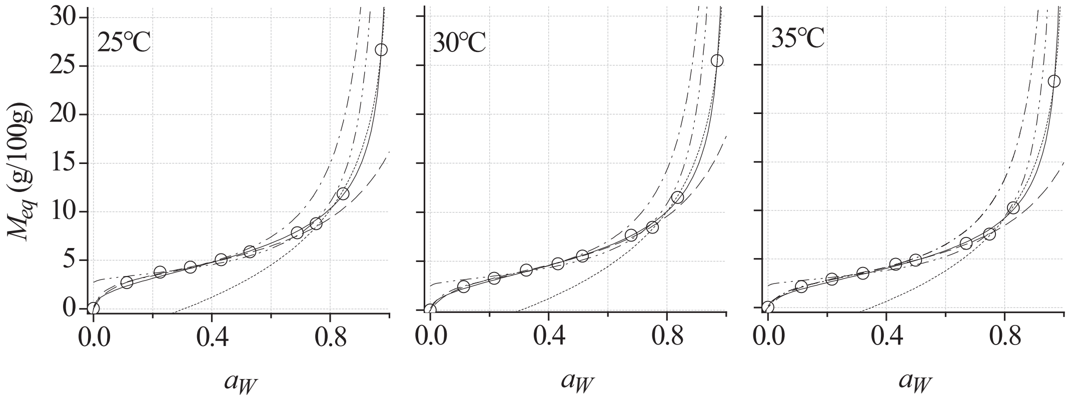

Fig. 3. Moisture adsorption experimental data (circle) of black soldier fly larvae powder fitted with BET (dash-dot), GAB (dash-dash), Caurie (solid line), Smith (dotted) and Khun (dash-dot-dot) models.

Table 4
Model parameters and measures of fit at the various equilibration temperatures.

Model	Cricket			BSFL		
	25 °C	30 °C	35 °C	25 °C	30 °C	35 °C
BET [a_W < 0.43]						
M_o (g/100 g)	3.017	2.899	2.682	2.833	2.519	2.132
C_b (−)	33.071	23.594	18.334	26.811	24.450	23.998
R^2	0.996	0.997	0.999	0.996	0.995	0.996
RMSE (%)	3.13	2.28	0.88	2.91	2.72	2.52
GAB [a_W < 0.75]						
M_o (g/100 g)	3.593	3.556	3.288	2.943	2.852	2.561
C_g (−)	21.463	15.214	13.653	24.948	20.534	18.809
k_g (−)	0.799	0.803	0.784	0.909	0.825	0.783
R^2	0.999	0.997	0.999	0.997	0.997	0.998
RMSE (%)	1.62	1.76	1.78	3.07	2.49	1.69
Smith [a_W > 0.5]						
A(−)	−1.015	−0.884	−1.216	−0.485	−0.738	−1.201
B(−)	7.495	7.352	6.999	7.001	6.055	5.700
R^2	0.983	0.983	0.979	0.997	0.98	0.973
RMSE (%)	11.77	11.09	13.97	4.96	10.75	15.56
Khun [a_W < 0.75]						
A(−)	−1.898	−1.929	−1.756	−2.121	−1.635	−1.323
B(−)	2.441	2.228	1.898	1.891	1.862	1.671
R^2	0.980	0.974	0.971	0.990	0.980	0.974
RMSE (%)	4.28	5.50	6.02	3.87	4.91	5.27
Caurie [a_W < 0.98]						
C_c(−)	1.104	1.099	1.059	1.094	0.999	0.948
M_o(−)	5.111	4.966	4.569	5.026	4.571	4.046
R^2	0.998	0.999	0.998	0.997	0.998	0.997
RMSE (%)	6.23	3.46	6.13	4.17	6.368	11.61

C_b, C_g, k_g, C_c, $A$and B: model constants; M_o: Monolayer moisture content (g/100 g); R^2: coefficient of determination; RMSE: root mean square error.

major component, like in dried milled crickets and BSFL, is protein (Sablani, Myhara, Mahgoub, Al-ttabi, & Al-Mugheiry, 2001). Azzollini et al. (2016) estimated GAB M_o of 5 g/100 g for *T. molitor* powder. The M_o could be regarded as the tightly bound water. At this very low moisture content, chemical reactions that depend on solvation are expected to be rather slow but deteriorations arising from lipid phase reactions such as oxidative rancidification may be enhanced (Rao et al., 2006). Generally, BSFL powder exhibited lower monolayer loading which can be attributed the higher fat content. As determined in the proximate composition, about a quarter of dry BSFL powder comprised fat, thus lipid phase deteriorative reactions may be considerable at such low moisture content. Furthermore, the M_o of both substrates decreased with increase in equilibration temperature, which correlated well with the decrease monolayer energy constants C_g and C_b predicted by the BET and GAB models, respectively. Similar trends have been reported in numerous foods (Akoy et al., 2013; Al-Mahasneh et al., 2014; Chalermchat & Owasit, 2011; Goneli et al., 2016; Kiranoudis, 1993; Seid & Hensel, 2012).

The Caurie equation further allows evaluation of a number of properties of sorbed water, namely number of binding sites, density of sorbed water, surface area of absorbent and proportion of bound water (Rao et al., 2006). The bound water interacts tightly with the solids in a food and is not available for microbial growth or chemical reactions whereas free water does not have any interaction with the material and is able to freeze. It is therefore crucial to generate information regarding bound water as most of the unit operations involving food processing and preservation depend on the removal of water or its immobilisation in food (Singh, 2006). Experimentally, free and bound water can be determined using thermal analysis technique such as the differential scanning calorimetry in which either the melting or evaporation phenomenon is monitored (Chen, Wei, & Zhang, 2010). Other researchers have used nuclear magnetic resonance spectroscopy (Chen et al., 2010; Le Botlan, Rugraff, Martin, & Colonna, 1998) and dieletric techniques (François, Gaudillat, Costa, Lakkis, & F., 2003). Where such methods are lacking, specific models can generate useful information. The number of adsorbed monolayers (n_m) may be calculated as the ratio of the Caurie monolayer moisture content to the Caurie constant, M_o/C_c. Also the Caurie constant C_c is assumed to be equivalent to the density of adsorbed water in the monolayer, thus allowing estimation of the surface area of the adsorbent (A) using the expression: $A = M_o/(C_c \times d \times 10^8)$, where d is the diameter of water molecule ($d = 3.673 \times 10^{-10}$m). The per cent bound water is estimated as $M_o \times n_m$. From Table 4, the density of adsorbed water (C_c) was higher in the cricket powder than in BSFL powder, and decreased with increasing equilibration temperature in both substrates. Table 5 presents the computed values for the additional derived parameters. Number of monolayers, surface areas of adsorbent, and percent bound water decreased with temperature. Percent bound water was higher in the cricket powder at all temperatures. The decline in bound water with increasing temperature implies a decrease in sorption activity at the low a_W range, and a decrease in surface area reflects a decline in the exposure of charged polar groups and carboxyl groups of peptides that

Table 5
Properties of sorbed water according to Caurie model.

Equilibration temperature	Cricket			BSFL		
	n_m	A (m2/g)	W_b(%)	n_m	A (m2/g)	W_b(%)
25 °C	4.63	126.00	23.65	4.60	125.11	23.10
30 °C	4.52	123.01	22.44	4.57	124.50	20.90
35 °C	4.31	117.43	19.71	4.27	116.24	17.28

n_m: number of monolayers; A: area of adsorbent; W_b: bound water.

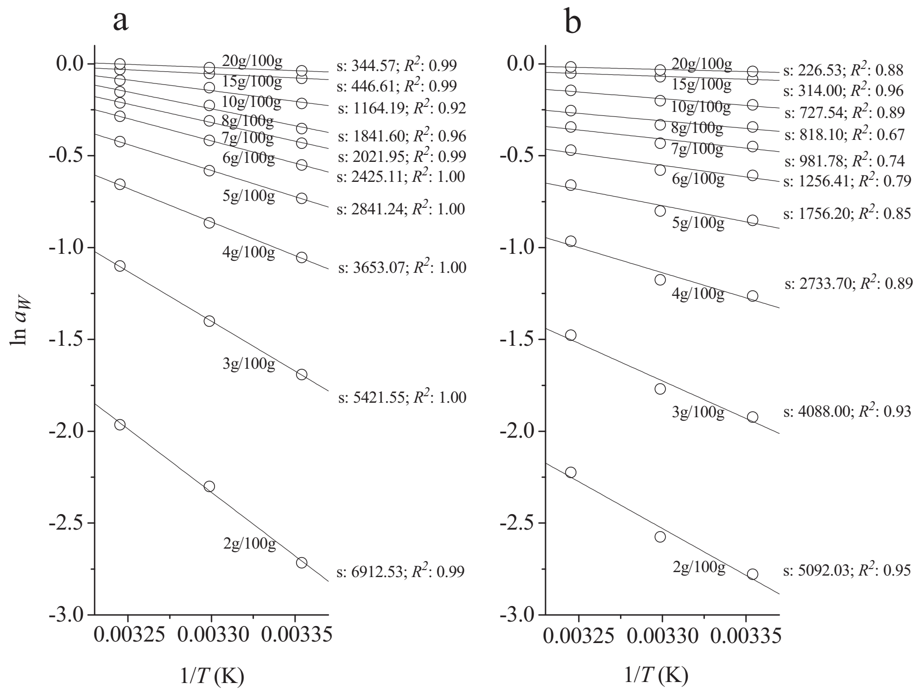

Fig. 4. Clausius-Clapeyron relationships between water activity and temperature in black soldier fly larvae (a) and cricket (b) powders at various equilibrium moisture contents. The slope (s) which is q_{st}/R, and the coefficient of determination (R^2) for each linear regression equation are shown.

bind water, as proteins generally tend to shrink at higher temperature (Rao et al., 2006). Thus some functional aspects may also be lost if the substrates are stored under the warmer conditions for a protracted period.

3.5. Isosteric heat of sorption

Adsorption processes emit heat, and q_{st} is a measure of the amount of energy released. Fig. 4 shows the plots of lna_W as a function of $1/T$ for equilibrium moisture contents. The corresponding regression parameters: (slope, (s) and coefficient of determination (R^2)) of the linear fits at equilibrium moisture contents between 2 and 20 g/100 g are presented. Regression parameters for other moisture contents between 20 and 40 g/100 were similarly calculated but have not been shown on Fig. 4. Net isosteric heats of sorption (q_{st}) are presented in Fig. 5 for equilibrium moisture contents spanning 2–40 g/100 g. For both substrates, equilibrium moisture contents were those predicted by the Caurie model. Isosteric heats of sorption of the BSFL powder were higher than those of the cricket powder indicating that binding of water in sorption sites was more exothermic in the BSFL than in the cricket powder. In the two substrates, q_{st} was high at low equilibrium moisture content. Below ca. 5 g/100 g moisture content, q_{st} decreased rapidly as more water became adsorbed. The high q_{st} values at low moisture contents indicate high water binding energy, which is characteristic of monolayer sorption. A transition followed at 5–20 g/100 g moisture content whereby net isosteric heat of sorption decreased at a decreasing rate. This is explained by the fact that most sites with high water binding energies were already occupied but sorption continued on sites with lower water binding energies (Chalermchat & Owasit, 2011). Furthermore, declining isosteric heats indicate progressive weakening of water-solid interactions, subsequently leading to free moisture (Toujani, Hassini, Azzouz, & Belghith, 2011). At equilibrium moisture content of about 20 g/100 g, q_{st} approached zero meaning that total isosteric heat of sorption approximated latent heat of vaporisation of water. Thus above 20 g/100 g equilibrium moisture, water existed in free liquefied form. Such water can support profuse microbial, chemical and biochemical deterioration.

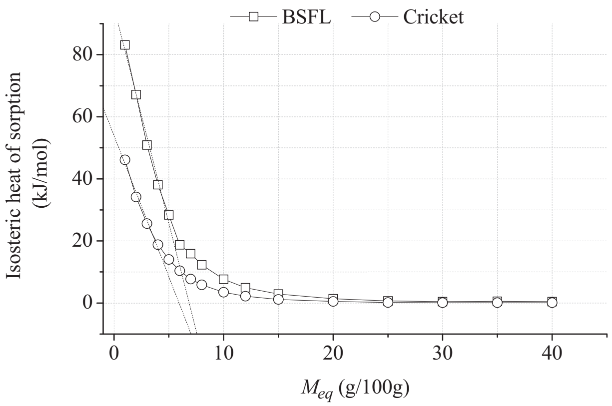

Fig. 5. Relationship between isosteric heats of sorption and equilibrium moisture contents of cricket and black soldier fly larvae (BSFL) powders.

3.6. Shelf-life estimation

High ambient relative humidity and temperature in the tropics presents a challenge for storage of dried products. Using the Heiss-Eichner model (Heiss & Eichner, 1971) the shelf-life of the dried insect powders was predicted in relation to possible packaging and storage conditions: polyethylene is a common packaging material; ambient temperatures range between 23 and 35 °C; and relative humidity could approach 90%. According to the model, when the critical a_W for a particular system under given storage conditions and the moisture sorption behaviour are known, the potential shelf-life of the packaged product in days (t_s) is given by the equation:

$$t_s = \frac{\ln[(x_e - x_i)/(x_e - x_c)]}{k_S(A/W)(p_o/s)} \tag{3}$$

where x_e is the equilibrium moisture content (g/g d·b) of the product if it is left in contact with the atmosphere outside the package (depends on temperature, relative humidity and the product's hydration properties); x_c is safe storage moisture content of the product (g/g d·b) i.e. the moisture content corresponding to the safe storage borderline a_W; $x_i$is initial moisture content of the product when it is packaged (g/g d.b); k_s is permeability constant of the package to moisture vapour (kg H_20 μm/

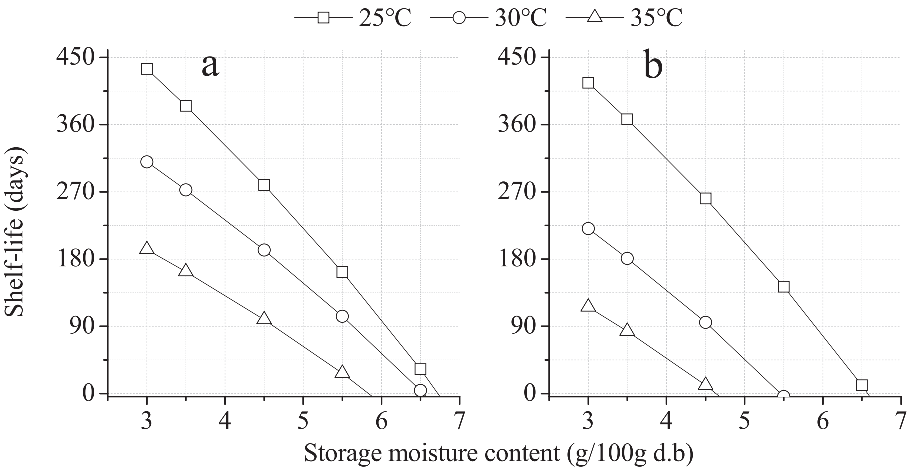

Fig. 6. Estimated shelf-life of cricket (a) and black soldier fly larvae (b) powders packaged in 80 μm-thick polyethylene bags and stored at different temperatures.

m²/day/Pa); p_o is vapour pressure at storage temperature at atmospheric pressure (Pa); A is surface area of the package (m²); W is weight of the product (dry matter) in the package (kg); and s is slope of the product isotherm (assumed linear over the range between x_e and x_c). This model was also used by other researchers (Chuzel & Zakhia, 1991; Ikhu-Omoregbe & Chen, 2005; Lima & Cal-Vidal, 1988). A safe storage borderline a_W of 0.6, which is the classical a_W level that ensures microbiological stability for a food product was applied. A 90% relative humidity (a_W 0.9) was used as the as ambient storage relative humidity, representing the highest ambient air humidity that may be experienced in many tropical areas. From the sorption isotherms, the safe storage moisture contents (x_c) of cricket and BSFL powder were 0.06723 and 0.06579 g/g at 25 °C, 0.0654 and 0.0546 g/g at 30 °C and 0.0585 and 0.0464 g/g 35 °C, respectively. The equilibrium moisture contents (x_e) were 0.1466 and 0.1410 g/g at 25 °C, 0.1452 and 0.1201 g/g at 30 °C, and 0.1349 and 0.1080 g/g 35 °C, respectively. The estimated shelf-lives of cricket and BSFL powders packaged in 10-kg polyethylene bags (thickness: 80 μm; surface area: 0.1474 m²; water vapour permeability constant: 1.55×10^{-4} kg μm/m²/day/Pa (Lima & Cal-Vidal, 1988) are presented in Fig. 6. As expected, shelf-life would decrease with increasing initial storage moisture and storage temperature. A shelf-life of 200–220 days is possible if the cricket and BSFL powders are dried to 5 g/100 g moisture content and stored at 25 °C. However, at this moisture content, the cricket powder will store for only 63 days at 35 °C whereas at the same temperature the BSFL powder will not be able to store. At 30 °C, the cricket powder will store for 180 days if dried to 4.5 g/100 g moisture content whereas the BSFL powder will require to be dried to a moisture content of ~3.5 g/100 g to be able to store for the same period. A limitation of this shelf-life estimation, however, is that it focused on the permeability of the package to water vapour but permeability dynamics to gases, that is, oxygen and carbon dioxide, which may cause oxidation need to be investigated. Another interesting packaging material in terms of permeability to water vapour and oxygen is polypropylene. This material has better barrier properties and may therefore be of interest for combating oxidative deterioration. We also did not consider a specific target quality change, which could influence the choice of the critical water activity value.

4. Conclusions

Moisture sorption of cricket and BSFL powders involve monolayer-multilayer mechanisms and belong to type II of Brunauer classifications. The BSFL powder has lower sorption capacity compared to the cricket powder. The BSLF product is also more sensitive to a_W variation arising from temperature changes, which has impact on shelf-life. Considering possible packaging and storage conditions in the tropics including ambient temperature of 23–35 °C, relative humidity as high as 90%, and use of polyethylene as a common packaging material, the shelf-life of the two products was modelled. It is possible to store the cricket and BSFL powders for 7 months at 25 °C if the products are dried to ~5 g/100 g moisture content. Nevertheless, storage experiments should be carried out to validate this predictions taking into account target microbial levels and other quality parameters.

Acknowledgements

This work was supported by the INSFEED project –"Insect feed for poultry and fish production in Kenya and Uganda (CultiAF Grant No: 107839-001) co-funded by International Development Research Centre (IDRC) and Australia Centre for International Agricultural Research (ACIAR). Authors acknowledge the institutional support provided by *icipe*.

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