

Moisture sorption isotherms and shelf life prediction for whole dried sandfish (*Holothuria scabra*)

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Abstract: The sea cucumber commonly known as sandfish (*Holothuria scabra*) is a high-value species that is often sourced from artisanal fishers in the Philippines. The traditional drying process involves a combination of smoking and sun-drying until a moisture content of 8%-10% is reached. However, dried sea cucumber, including sandfish, is often poorly dried; resulting in spoilage and mold growth during storage. To establish conditions for proper storage of dried sandfish, this study aims to develop moisture sorption isotherms (MSI) at 30°C, 45°C and 60°C at ten levels of water activity (a_w) using an isopiestic method based on saturated salt solutions. Equilibrium moisture content (EMC) data were fitted with six mathematical models by non-linear regression analysis. Water vapor transmission rate (WVTR) of low-density polyethylene (PE) and oriented polypropylene (OPP) packaging film was also measured at 30°C and 35°C and a mass-balance model was used to predict shelf life and EMC during storage based on MSI and WVTR data. MSI of *H. scabra* followed a Type III curve, which was best fitted to a Peleg model. The best range of moisture content for storage was 0.12-0.18 g·g⁻¹ dry matter which corresponded to a_w of 0.4-0.6. Predicted shelf life for PE and OPP film packs at 30°C were 114 and 372 days, respectively; at 35°C, the estimated shelf life decreased to 83 and 141 days, respectively.

Keywords: moisture sorption isotherms, sandfish, shelf life, water activity, equilibrium moisture content, drying

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1 Introduction

Sea cucumbers are marine animals belonging to the class Holothuroidea. These animals possess a soft elongated body with a cluster of tentacles at the mouth. In commercially harvested species, the body wall is thick and is the part of the animal that is processed into food products (Purcell et al., 2012). Sea cucumber has long been used as a food tonic and folk remedy since the Ming Dynasty (1368-1644 BC). It can rightly be considered a health food due to low levels of fat, and the presence of essential amino acids and trace minerals (Chen, 2004).

The body wall also contains high levels of protein made up mostly of insoluble collagen fibers. The collagen is thought to be responsible for its unique texture when cooked (Saito et al., 2002).

Dried sea cucumber, also known as beche-de-mer or trepang, is exported to countries such as Singapore, Taiwan and Hong Kong where it is used in dishes served during special events. The species commonly known as sandfish (*Holothuria scabra*) is considered as high in value and efforts are underway on commercial aquaculture for this species (Purcell, 2014a; Purcell, 2014b).

In the Philippines, collectors of sea cucumber are mostly fishermen who sell their catch to local consolidators living in coastal villages. Consolidators process and dry the harvested sea cucumber and sell them to larger traders or exporters. For sandfish, processing

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involves removal of the internal organs, boiling, and removal of the spicule layer. Once the boiled sandfish is free of spicules, it is smoked and sun-dried for several days until the moisture content is very low (8%-10%). At this point, the texture is very firm and hard, and the dried product is referred to as trepang (Brown et al., 2010). Trepang is classified according to size based on weight or length, as well as visual appearance and moisture content. Poorly dried, misshapen or half-cooked trepang is considered less valuable and could be sold at 40% less than its maximum price (Jun, 2002).

The state of moisture within a solid material is often expressed as water activity (a_w), defined as the ratio of vapor pressure of water in the material to vapor pressure of pure water at the same pressure and temperature. Achieving and maintaining the safety and stability of a dried food or crop product is a major objective of drying; a_w is considered the most important factor affecting these two parameters. Keeping a_w below 0.7 will slow down microbial activity (mold, yeast and bacteria), and microbial activity is expected to stop completely at $a_w < 0.6$, although chemical reactions involving oxidation of lipids, non-enzymatic browning and other enzyme activities can still occur. Hence, keeping a_w at 0.3 will successfully preserve a dried material (Labuza and Altunakar, 2007).

Mathematical models of moisture sorption data describe the effect of a_w on *EMC*. Isotherms have been developed for other types of dried marine products such as sardine muscles (Djendoubi et al., 2009), squid (Ren et al., 2011) and abalone (Chun et al., 2015; Sablani et al., 2004). Trepang is known as a hygroscopic material that can reabsorb moisture, leading to decay (Nair et al., 1994); however, studies on *EMC* and *MSI* of dried sandfish are scarce in literature. The specific objectives of the study were to develop moisture adsorption isotherms for whole dried sandfish (trepang), determine the appropriate *EMC* for storing trepang, and predict shelf life of trepang stored in two types of plastic packaging materials.

2 Materials and methods

2.1 Experimental samples

For *EMC* determination, live sandfish (*H. scabra*)

samples with an average individual weight of 800 g were gathered by divers from the sea ranching site of the Palawan Aquaculture Corporation (PAC) in Coron, Palawan (Philippines). The live samples were kept in aerated tanks before being processed. Processing methods for sandfish vary between countries, provinces and even municipalities; however, the general process involves degutting, boiling, removal of the spicule layer, a second and/or third boiling, smoking and sun-drying (Purcell, 2014a; Nair et al., 1994; Ram et al., 2010). Consultations with researchers of the National Fisheries Research & Development Institute (NFRDI) of the Philippine Department of Agriculture, as well as preliminary trials showed that boiling twice for one hour was necessary to soften samples with sizes of 800 g or more. For this study, samples were degutted by slitting the posterior end and squeezing out the internal organs, boiling for 1 hour in fresh water, manual brushing to remove spicules, and second boiling for 1 hour in fresh water. To minimize decay, samples were partially dried on-site for 12 hours at an average temperature of 60°C using a wood-fired hot-air dryer; final drying was completed at the Institute of Agricultural Engineering (University of the Philippines Los Baños) using a convection oven set at 60°C. Drying was stopped when samples reached an average moisture content of 25% wet basis; samples were then stored for two weeks in a desiccator.

2.2 Determination of equilibrium moisture content

The *EMC* of whole dried samples was determined at three temperatures (30°C, 45°C and 60°C) and ten levels of a_w using saturated solutions of different salts as shown in Table 1. A gravimetric method with closed-loop air circulation was devised to pump air through each salt solution and into a re-sealable plastic container (Komax Biokips, South Korea) holding dried samples (Figure 1). Saturation of each salt solution was ensured by maintaining undissolved crystals in the solution; salt crystals were added as needed during the experiment. Constant temperature was maintained using a Daesung Fox 2002 digital thermostat (Busan, South Korea; accurate to 0.1°C) to activate a nichrome wire heater. Air temperature was checked using a Gemini Tinytag Plus 2 data logger (West Sussex, UK). For each temperature

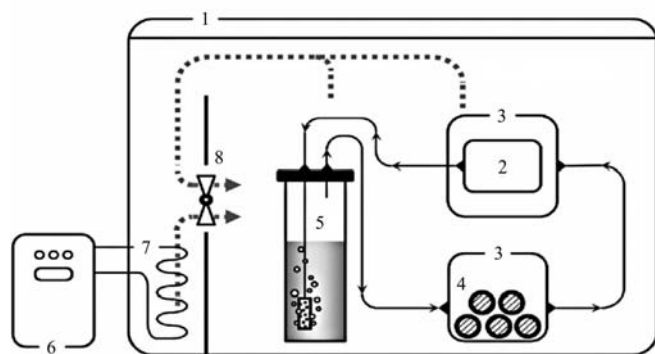
setting, two replicate samples were weighed at intervals until three consecutive readings showed a change of 0.01 g or less; upon reaching this state, the samples were subjected to the next higher a_w . An Ohaus Adventurer analytical balance (New Jersey, USA) readable to 0.001 g was used for measuring sample weight. After all samples had reached equilibrium, dry matter content was determined by oven drying to a constant weight at 105°C (Bai et al., 2013). EMC on a dry basis (EMC_{db}) of each sample at each a_w was calculated using Equation (1).

$$EMC_{db} = \frac{W_i - W_{dm}}{W_{dm}} \quad (1)$$

where: W_i = sample weight at the i^{th} a_w , ($i = 1$ to 10), and W_{dm} = corresponding weight of dry matter of the sample.

Table 1 Published water activities of various saturated salt solutions at different temperatures

Saturated salt solution	Temperature, °C	Source		
		30	45	60
Sodium hydroxide NaOH	0.0758 0.056 0.0361	Greenspan (1977)		
Lithium chloride LiCl	0.1128 0.1116 0.1095	Greenspan (1977)		
Potassium acetate $KC_2H_3O_2$	0.2257 0.1974 0.1747	Labuza et al. (1985)		
Magnesium chloride $MgCl_2$	0.3244 0.311 0.2926	Greenspan (1977)		
Potassium carbonate K_2CO_3	0.4397 0.4299 0.4212	Labuza et al. (1985)		
Sodium dichromate $Na_2Cr_2O_7$	0.525 0.485 0.47	Wexler and Hasegawa (1954), TETB (undated)		
Sodium nitrite $NaNO_2$	0.6408 0.5938 0.5647	Apelblat and Korin (1998)		
Sodium chloride NaCl	0.7509 0.7468 0.7441	Greenspan (1977)		
Potassium chloride KCl	0.8362 0.8174 0.8025	Greenspan (1977)		
Potassium sulfate K_2SO_4	0.97 0.9612 0.931	Greenspan (1977); Foxworthy (1988)		



1. Sealed and insulated plastic box and lid 2. Air pump 3. Sealed plastic container containing 4. The test material 5. Saturated salt solution and a temperature control system with a 6. digital thermostat 7. Electric heater 8. A circulating fan

Figure 1 Schematic diagram of the isopiestic apparatus with closed-loop air circulation composed

2.3 Moisture Sorption Isotherm Models

EMC_{db} of pre-dried sandfish versus a_w was fitted to six mathematical models (Equation (2) to (7), Table 2) using the Solver add-in of Microsoft Excel 2010 spreadsheet software to determine equation coefficients. The software was used to calculate the maximum value of the coefficient of determination (R^2) by iteration using Equation (8).

Table 2 Moisture adsorption isotherm models

Equation number	Sorption isotherm models ^a
(2)	GAB ^z $EMC_{db} = \frac{M_o C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)}$
(3)	Modified Henderson ^y $EMC_{db} = \left[\frac{\ln(1 - a_w)}{-A(T + B)} \right]^{1/C}$
(4)	Modified Halsey ^x $EMC_{db} = A \left[\frac{B}{\ln(a_w)} \right]^{1/C}$
(5)	Modified Oswin ^v $EMC_{db} = (A + BT) \left[\frac{a_w}{1 - a_w} \right]^{1/C}$
(6)	Smith ^w $EMC_{db} = A - BT - C \cdot \ln(1 - a_w)$
(7)	Peleg ^v $EMC_{db} = A(a_w)^B + C(a_w)^D$

Sources: ^z Labuza and Altunakar (2007); ^y Raji and Ojediran (2011); ^x Djendoubi, Boudhrioua, Bonazzi, Kechaou (2009); ^w Boeri, Neto da Silva, Ferreira, Saraiva, Moreira (2013); ^v Peleg (1992) where $B < 1$, $D > 1$.

^a EMC_{db} = equilibrium moisture content dry basis (kg kg⁻¹ dry matter), A , B , C , D , K and M_o are equation parameters, T = absolute temperature.

$$R^2 = 1 - \frac{\sum (m_j - \hat{m}_j)^2}{\sum (m_j - \bar{m})^2} \quad (8)$$

where, $m_j = j^{\text{th}}$ experimental EMC_{db} ; $\hat{m}_j = j^{\text{th}}$ predicted EMC_{db} using an isotherm model, and \bar{m} = mean of experimental EMC_{db} .

Each model was also evaluated using the standard error of estimation (SEE) (Equation (9)) and the mean relative percentage deviation modulus (P_e) (Equation (10)).

$$SEE = \sqrt{\frac{\sum (m_j - \hat{m}_j)^2}{n}} \quad (9)$$

$$P_e = \frac{100}{n} \sum \frac{|m_j - \hat{m}_j|}{m_j} \quad (10)$$

where, n = number of experimental data points.

A model is generally considered a good fit for practical purposes when P_e values are below 10% (Peng et al., 2007); R^2 values close to unity and low values of SEE indicate a good fit of the model to the experimental data (Ceylan et al., 2007).

From a plot of EMC versus a_w using the best MSI

model, the most stable portion of the isotherm could be determined.

2.4 Determination of water vapor transmission rate of flexible plastic films

The whole-bag method described by Moyls (1998) was used for measuring the water vapor transmission rate (WVTR) of commercially available polyethylene (PE) and oriented polypropylene (OPP) plastic films. Film thickness of PE and OPP film samples were measured with a digital micrometer (Fowler High Precision Inc, Massachusetts, USA). Plastic packs with dimensions of 10.2 cm × 10.2 cm were fabricated using a heat sealer; a paper towel wetted with 2.5 mL of distilled water was inserted inside to generate an internal humidity of 100% when the pack was sealed. Any internal air bubbles were carefully pressed out by hand before final sealing of the pack.

WVTR was measured at 30°C and 35°C using the same apparatus used for measuring EMC of the dried sandfish (Figure 1). Saturated NaCl solution was used to generate a constant relative humidity of 75%. For each temperature, the weight of 3-5 replicate plastic packs was monitored every three days until enough readings were obtained (minimum of five). The bags were weighed individually on a Scaltec SBC 31 analytical balance (Scaltec Instruments, Germany) to the nearest 0.1 mg. WVTR (g cm⁻² day⁻¹) of PE and OPP bags was computed using Equation 11 (ASTM E96-95 1995). ANOVA and Tukey’s HSD test were conducted using Statistica 8.0 (StatSoftInc, Oklahoma, USA) to determine significance of differences between mean WVTR of PE and OPP films at 30°C and 35°C.

$$WVTR = \frac{G}{tA} \tag{11}$$

where, *G* = weight change (g); *t* = time (days), *Gt*⁻¹ = slope of the linear regression line for *G* versus *t* (g day⁻¹); and *A* = effective film area for mass transfer (cm²).

Permeability (*P*, g mm m⁻² d⁻¹ atm⁻¹) of each film was determined from WVTR and film thickness (*L*) using Equation (12) from Hernandez (1997).

$$P = \frac{(WVTR)(L)}{p_{sat} \left(\frac{Y_i - Y_o}{100} \right)} \tag{12}$$

where, *Y_i* = relative humidity in the package headspace, and *Y_o* = external relative humidity.

2.5 Estimation of shelf life

Shelf life was adapted from the procedure described by Hernandez (1997); the procedure is as follows:

- 1) Determine the effect of temperature on the coefficients of the selected MSI model (from Section 2.3) by linear regression of each coefficient (A, B, C and D from Table 2) with absolute temperature.
- 2) Estimate the values of A, B, C and D at 30°C and 35°C by using the derived regression equation.
- 3) Calculate *a_w* for a specified EMC and temperature by substituting the estimated values for A, B, C and D in the selected MSI model. For this step, an EMC range was selected such that the dried sandfish was not brittle (over-dried) or contained excessive moisture (under-dried). This meant specifying minimum, intermediate and maximum values for EMC (on a dry basis) and calculating the corresponding *a_w*.
- 4) Fit a linear regression equation (Equation (13)) for *a_w* versus EMC at 30°C and 35°C. Equation (13) represents the linearized region of interest in the selected MSI model, where *a* = y-intercept and *b* = slope (g dry matter g⁻¹ moisture).

$$a_w = a + b \cdot EMC \tag{13}$$

1) Calculate equilibrium relative humidity (ERH) and *a_w* at *t* = 0 and *t* = *t_s*, where *t_s* represents the time needed to reach the end of the product shelf life. When *t* = *t_s*, it is assumed that the product has reached its maximum allowable EMC in the region of interest.

2) Calculate shelf life (*t*, days) using Equation (14) from Hernandez (1997) where *W* = dry weight of dried sandfish (g), *P* = water vapor permeability of plastic film (g-μm m⁻² d⁻¹ kPa⁻¹), *A* = effective surface area of the package (m²) for gas exchange, *p_{sat}* = vapor pressure at saturation for a given temperature (kPa), *Y_o* = external relative humidity, and *Y_i* = relative humidity of the package headspace. For this equation, moisture gained or lost by the product is assumed to be equal to water vapor transmitted through the packaging film.

$$t = \frac{LW}{PAbp_{sat}} \ln \left(\frac{Y_o - Y_{i,t=0}}{Y_o - Y_{i,t}} \right) \tag{14}$$

The parameter *Y_{i,t=0}* represents the equilibrium relative

humidity (ERH, %) in the package headspace at the start of the trial. On the other hand, $Y_{i,t}$ represents ERH when the *EMC* of the sample has reached its maximum value. External relative humidity was set at 75%, which is the mean RH of the Philippines.

2.6 Prediction of moisture content during storage

A storage trial was conducted using sandfish samples gathered in April 2014 from Morong, Bataan (Philippines); the samples were provided by the Department of Agriculture - Bureau of Fisheries and Aquatic Resources (Region III). Due to limited volume available, only three specimens were processed and pre-dried using the procedure discussed previously. Average sample weight and moisture content at the start of the storage trial was 44.5 g and 0.154 g g⁻¹ dry matter, respectively. Each specimen was sealed in a low-density PE bag with a mean film thickness of 31.4 μm and dimensions of 13 cm × 10 cm. Packs were kept for 45 days at room conditions (temperature = 31.4°C±1.3°C, relative humidity = 60.0%±6.1%) and weighed at regular intervals; dry matter content of each sample was determined by oven-drying for three days at 105°C. Moisture content of each sample during the storage trial was then estimated.

The procedure described in Section 2.5 was followed until the fourth step for predicting moisture content of dried sandfish packed in PE and kept at room temperature. Region of interest for each sample was based on the minimum, median and maximum MC recorded during the trial. As the final step, another form of Equation (14) to determine *t* as a function of moisture content (MC) was used as shown in Equation (15).

$$t = \frac{LW}{PAbp_{sat}} \ln \left(\frac{p_o - a \cdot p_{sat} - b \cdot p_{sat} \cdot MC_i}{p_o - a \cdot p_{sat} - b \cdot p_{sat} \cdot MC} \right) \quad (15)$$

where, p_o = water vapor pressure outside the package (kPa), and MC_i = initial moisture content.

The latter equation was used to predict *MC* during the storage period; refer to the Addendum for the derivation of Equation (15).

3 Results and discussion

3.1 Moisture sorption isotherms

Mean air temperature (±SD) during the experiment

was 29.8°C ± 0.1°C, 44.5°C ± 0.4°C, and 60.5°C ± 1.1°C. The time required for dried samples to reach a stable weight varied with temperature and a_w ; some treatments reached this state after 1 week, while others needed 52 days to stabilize.

The initial moisture contents of pre-dried samples ranged from 0.091-0.137 g g⁻¹ dry matter, with a mean (±SD) of 0.106 ± 0.013 g g⁻¹ dry matter. Average initial weight (± SD) of samples was 3.354 ± 0.873 g. Since a_w used for the study had a range of 0.0361-0.97 (Table 1), samples reached equilibrium through both moisture loss and gain. Hence, the isotherms fitted to the *EMC* vs a_w data are a combination of adsorption and desorption; the coefficients of the sorption isotherms obtained at 30°C, 45°C and 60°C are shown in Table 3. Based on the values for R^2 , *SEE* and P_e , the Peleg model had the highest R^2 and lowest *SEE* values; values of P_e for the Peleg model were also less than 10% except for the isotherm at 30°C. The Peleg model was therefore selected as the best fit to the *EMC* data of dried sandfish. This model is purely empirical and assumes that sigmoid moisture isotherms are generated by two sorption mechanisms, and it is not dependent on the presence of a well-defined monolayer (although it does not exclude this possibility). The Peleg model can be used for both sigmoid and non-sigmoid isotherms (Peleg, 1992; Labuza and Altunakar, 2007).

Moisture sorption isotherms of most food materials can be classified as Type I, II or III based on the shape of the curve. For whole dried sandfish, sorption isotherms followed a Type III curve regardless of temperature (Figure 2). *EMC* was fairly stable at 0.1 g g⁻¹ dry matter for $a_w < 0.4$; beyond this threshold, *EMC* started to increase gradually up to a_w of 0.6. At $a_w > 0.6$, dried sandfish rapidly gained moisture. Previous studies on isotherms of pure collagen have been limited to bovine hide as the source of material; results showed that these isotherms were sigmoid and were classified as Type II (Green, 1948; Boki and Kawasaki, 1994). On the other hand, materials that contain mainly crystalline compounds such as sugar or salt typically display Type III isotherms (Labuza and Altunakar, 2007). Although sea cucumber is unlikely to contain significant levels of sugar, salt content of dried *H. scabra* can reach 5% wet basis (Yaptenco and Pangan, 2015).

Table 3 Coefficients of moisture adsorption isotherm models for *Holothuria scabra* at different temperatures

Isotherm model	Model coefficients	Temperature, °C		
		30	45	60
GAB	M_o	0.0915	0.0714	0.0670
	C	1.6587E+04	4.6024E+04	2.8567E+04
	K	0.8463	0.9133	0.9640
	R^2	0.9105	0.9795	0.9935
	SEE	0.0419	0.0207	0.0137
	P_e	19.69	13.78	10.96
Modified Henderson	A	0.0341	0.0193	0.0117
	B	0.1010	0.0100	0.0010
	C	1.5395	1.0838	0.8088
	R^2	0.8622	0.8705	0.9196
	SEE	0.0520	0.0520	0.0482
	P_e	30.80	32.96	33.55
Modified Halsey	A	0.2863	0.7048	0.3758
	B	0.1572	0.0271	0.1581
	C	2.7366	2.0248	1.4490
	R^2	0.8437	0.9709	0.9830
	SEE	0.0553	0.0247	0.0222
	P_e	23.98	16.84	16.97
Modified Oswin	A	0.1559	0.1414	0.0176
	B	5.4226E-05	1.0132E-05	3.5156E-04
	C	3.1744	2.3237	1.6670
	R^2	0.8575	0.9416	0.9605
	SEE	0.0528	0.0349	0.0338
	P_e	27.42	22.59	24.25
Smith	A	9.8598E-03	1.0032E-02	1.0003E-02
	B	-2.1335E-04	-1.2292E-04	-2.4400E-05
	C	0.1264	0.1498	0.2054
	R^2	0.8910	0.9253	0.9070
	SEE	0.0462	0.0395	0.0518
	P_e	22.34	25.22	33.36
Peleg	A	0.0598	0.0981	0.1003
	B	-0.2544	-0.0577	0.0367
	C	0.4946	0.6151	0.8641
	R^2	3.1302	5.8724	6.2974
	SEE	0.9557	0.9993	0.9956
	P_e	0.0295	0.0037	0.0113
	A	12.33	1.59	7.08

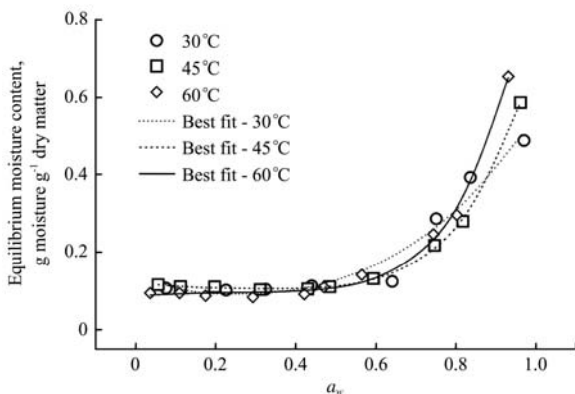


Figure 2 Fitting of moisture adsorption isotherms at different temperatures to the Peleg model (Each data point represents the mean of two samples)

In theory, Type III isotherms must trend toward zero as a_w reaches zero (Blahovec and Yanniotis, 2009). However, Trondillo and Yaptenco (2015) observed the formation of a hardened surface layer in smoke-dried sandfish using scanning electron microscopy. Bai et al. (2013) compared oven-dried specimens of *Stichopus japonicus* to air-dried and electro-hydrodynamically dried samples. A similar surface-hardened structure was observed in oven-dried samples which could restrict the diffusion of moisture from the inner regions, resulting in a higher EMC at reduced a_w .

Sorption isotherms are generally known to approach zero levels of moisture content as a_w approaches zero. However, the sorption isotherms at 30°C, 45°C and 60°C stabilized at 0.1 g g⁻¹ dry matter and did not shown any trending towards zero. This could be attributed to the formation of a case-hardened sample due to rapid evaporation of moisture from the surface combined with migration of inorganic salts to the surface (Bai et al., 2013). The hardened layer would then restrict the diffusion and evaporation of moisture from the surface of the product.

By inspection of Figure 2, the region of interest for estimating shelf life was taken for an EMC range of 0.12-0.18 g g⁻¹ dry matter; the intermediate value for EMC was taken at the midpoint of the range, or 0.15 g g⁻¹ dry matter.

Most food materials will hold less moisture at higher temperatures for the same a_w . However, other changes in the material such as dissociation of water or an increase in the water solubility of solutes can result in shifts in a_w (Labuza et al., 1985). Although temperature had a visible effect on coefficients B, C and D of the Peleg model, (Figure 3), it had no obvious effect on the adsorption isotherms of dried *H. scabra* (Figure 2). Furthermore, the isotherm at 30°C crossed over the isotherms at 45°C and 60°C at a_w of 0.80 and 0.85, respectively. In products containing high levels of sugars, this inversion effect is attributed to the increased solubility of sugars and the consequent reduction in mobility of water at higher temperatures (Bhandari and Adhikari, 2008). In other food products, the lack of any significant effect of temperature on EMC has also been

observed. Sorption isotherms of dried fruit slices, buckwheat seeds and pineapple pulp powder were not affected by temperature, possibly due to physical and/or chemical changes occurring during the sorption process (Hubinger et al., 1992; Maza and Campbell, 1985; Gabas et al., 2007). Changes in shape and size due to shrinkage may also affect isotherms. For the sea cucumber *Stichopus japonicus*, shrinkage of 26.4% has been observed when oven-dried at 80°C to a final moisture content of 0.12 g g⁻¹ dry matter (Bai et al., 2013). Shrinkage was also observed during drying of sandfish (data not shown) which could also contribute to shifts in the sorption isotherm.

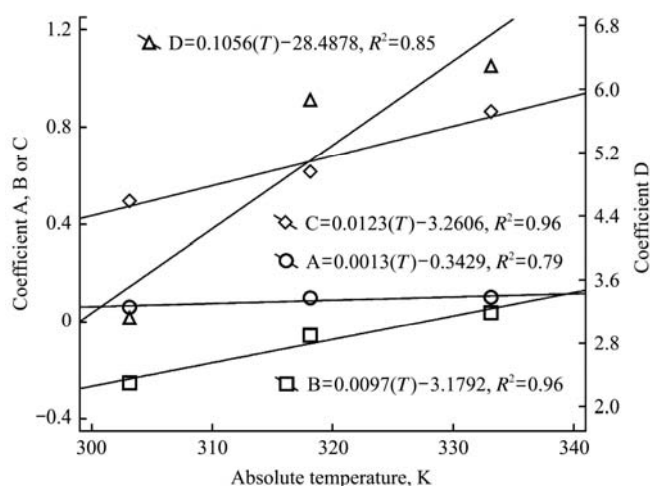


Figure 3 Effect of absolute temperature on Peleg model coefficients. Lines represent the best-fit curve by linear regression

3.2 Water vapor transmission rate of polyethylene and oriented polypropylene films

Average film thickness of PE and OPP film samples was 43.6 μm and 35.4 μm , respectively. Moisture loss through plastic film under constant temperature and RH was linear with time (Figure 4); R^2 values were in the range of 0.992-1.000. ANOVA showed that packaging film and temperature had a significant effect on WVTR; however, the interaction of these two factors was not significantly different (Table 4).

Table 5 shows the mean values for WVTR and permeability of PE and OPP film samples; although differences exist between experimental values and WVTR reported in published studies, the values are generally within the same order of magnitude. Moyls et al. (1998) reported differences in WVTR between

manufacturers of PE films, and even between film samples from the same bag. For the present study, WVTR of OPP film was significantly less compared to PE film for both 30°C and 35°C. However, temperature had a greater effect on WVTR of OPP film; when temperature was increased from 30°C to 35°C, WVTR of PE and OPP film increased by 47.6% and 186.7%, respectively.

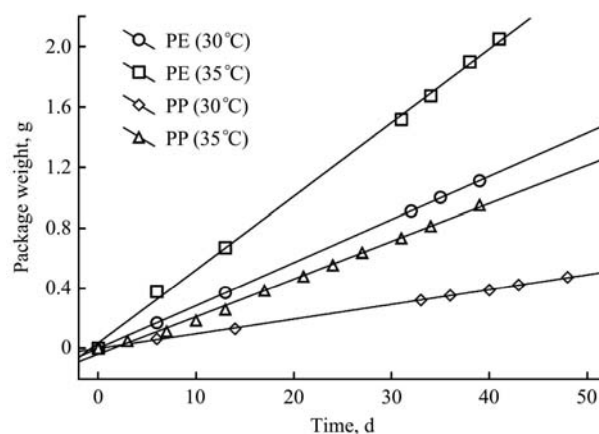


Figure 4 Time course of weight change of specimen plastic packs containing wet paper under constant temperature and relative humidity (75%) conditions. Each plot is a representative of 5 specimen packs; solid lines represent the best-fit equation for package weight (G) versus time (t) by linear regression. The slope ($G t^{-1}$) of the best-fit lines is used for estimating water vapor transmission rate of the specimen packs using Equation (11)

Table 4 ANOVA of water vapour transmission rate with respect to plastic film and temperature (ANOVA)

Effect	SS	DF	MS	F	Prob. F
Intercept	30.88	1	30.88	3645.99	0**
Film	3.86	1	3.86	455.18	0**
Temperature	2.53	1	2.53	298.28	0**
Film x Temp	0.019	1	0.019	2.26	0.155
Error	0.118	14	0.008		

3.3 Estimation of Shelf Life and Prediction of Moisture Content during Storage

The estimated shelf life for dried sandfish when packed in PE and OPP film is shown in Table 6. Keeping packs at higher temperature is predicted to reduce shelf life due to the increased transmission rate of water vapor. Regardless of temperature, the lower vapor transmission rate of OPP was predicted to give a longer shelf life. However, since OPP film is more sensitive to temperature changes than PE film, the reduction in shelf life is higher for OPP (62.2%) when compared to PE (26.9%).

Table 5 Experimental and published water vapor transmission rates of polyethylene and polypropylene plastic film

Material	L^z , μm	T , $^\circ\text{C}$	$WVTR$, $\text{g m}^{-2} \text{d}^{-1}$ ^y			Permeability (P), $\text{g mm m}^{-2} \text{d}^{-1} \text{atm}^{-1}$	Source	
Polyethylene	43.6	30	1.47	±	0.13	b	6.12	This study
		35	2.17	±	0.11	a	6.81	
Polypropylene	35.4	30	0.45	±	0.03	d	1.52	This study
		35	1.29	±	0.03	bc	3.28	
Polyethylene	32	23	3.71				4.89	Moyls et al., (1998) ^x
	44	23	3.00				5.40	
	61	23	5.22				13.02	
Polypropylene	25	38	5-7					Kirwan and Strawbridge (2003) ^w

Note: ^z L = film thickness, T = temperature, $WVTR$ = water vapor transmission rate; ^yFor this study, values shown the mean ±SD of at least three replicate samples measured for internal and external relative humidities of 100% and 75%, respectively. Means of $WVTR$ with a common letter are not significantly different by Tukey's HSD test at the 5% level of significance. ^xFor low-density polyethylene at internal and external relative humidity of 100% and 12.9%, respectively. ^wFor oriented polypropylene at external relative humidity of 90%.

Table 6 Estimated shelf life of dried sandfish as a function of packaging material and temperature

Packaging material (thickness)	Temperature, $^\circ\text{C}$	Shelf life, d
Polyethylene (43.6 m)	30	114.0
	35	83.3
Polypropylene (35.4 m)	30	372.3
	35	140.7

Mean (±SD) air temperature and RH during the storage trial were $31.4^\circ\text{C} \pm 1.3^\circ\text{C}$ and $60.0\% \pm 6.1\%$, respectively. Under these conditions, the water vapor partial pressure was 2.76 kPa; at saturation, the water vapor pressure was 4.60 kPa. Based on the Peleg model, a_w for Pack 1 was 0.72; the corresponding water vapor pressure in the product was 3.31 kPa. Since the vapor pressure in the product was greater than the vapor pressure in the surrounding atmosphere, the product in Pack 1 was expected to lose moisture over time. On the other hand, a_w for Pack 2 and 3 was 0.56 and 0.43, respectively; the corresponding product vapor pressure was 1.79 and 1.18 kPa, respectively. Hence, Pack 2 and 3 were expected to gain moisture during storage. This is confirmed by a plot of MC versus time (Figure 5); superimposing the mathematical model of Equation (14) on the data points shows that the model was able to predict moisture content closely. It should be noted that the MSI model is based on adsorption data while the case of Pack 1 involves desorption. The close agreement of the predicted MC to the actual MC may indicate that hysteresis is minimal for dried sandfish.

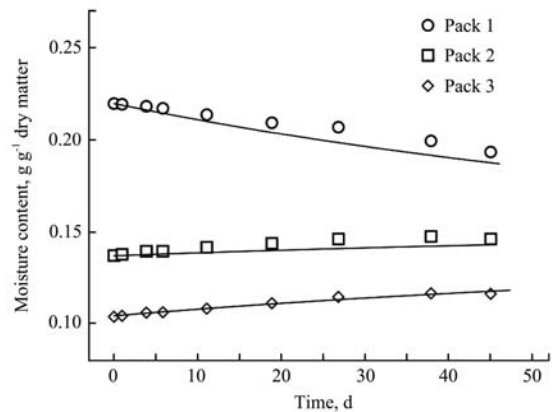


Figure 5 Time course of moisture content of dried sandfish packed in polyethylene plastic and stored at room temperature

4 Conclusions

The study showed that the moisture adsorption isotherm for dried sandfish follows a Type III isotherm. The Peleg model gave the best fit to the experimental data, with a coefficient of determination above 95%. Although absolute temperature was linearly related to most of the Peleg coefficients, the predicted EMC for a constant a_w did not follow the expected trend (i.e. lower EMC at higher temperatures). This could be due to changes in the physical structure of the material during drying, or chemical properties altered during the boiling process. Moreover, the sorption isotherms did not approach zero as a_w was reduced. This could be due to the presence of a hardened surface layer that restricted moisture diffusion at low a_w . Further investigation on dried sandfish isotherms is needed to determine the causes of cross-overs of sorption isotherms at different

temperatures, as well as the effect of surface hardening on equilibrium moisture content at low a_w .

Water vapor transmission rate of oriented polypropylene film was significantly lower compared to low-density polyethylene; this was predicted to give a longer shelf life for dried sandfish. Even though PE film is more easily procured, the transparency of OPP film which makes the package more attractive together with the possibility of lower WVTR may make OPP film a better choice, especially for individually packed products. However, further storage trials should be conducted to determine shelf life of dried sandfish packed in bulk (250-500 g) to better simulate the actual practice of retailers.

The use of moisture sorption isotherms of whole dried sandfish and vapor transmission properties of plastic film as inputs to mass balance equations allowed shelf life and moisture content during storage to be predicted. Although the sorption isotherms deviated from convention, the predicted *EMC* agreed closely with measured *EMC* in a storage trial, serving as a validation of the mathematical models that were developed.

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Addendum: Derivation of Equation (15)

For products that can absorb or release moisture, the moisture content changes with the relative humidity of the surrounding environment. When packed in semi-permeable film, it can be assumed that the rate of moisture loss or gain by the product (m_p) is equal to the rate of water vapor passing through the package material (m_f). Hernandez (1997) claimed these two mass flows can be expressed as shown in Equation (A.1) and (A.2), respectively.

$$m_p = \frac{Wdm}{dt} \quad (\text{A.1})$$

$$m_f = \frac{PA}{L}[p_o - p_i(MC)] \quad (\text{A.2})$$

where, W = dry weight of the product (g); MC = moisture content of the product (g g^{-1} dry matter); t = time; P = permeability of the packaging film; A = surface area of the package for gas exchange; L = thickness of the film; p_o = water vapor pressure outside the package, and $p_i(MC)$ = water vapor pressure in the package headspace as a function of MC .

Equating Equation (A.1) and (A.2) and rearranging

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terms, we get a differential equation which can be integrated to give Equation (A.3); the limits MC_1 and MC_2 denote the initial and final moisture content of the product during the storage period t (i.e. the portion of interest in the isotherm).

$$t = \frac{LW}{PA} \int_{MC_1}^{MC_2} \frac{dMC}{p_o - p_i(MC)} \quad (\text{A.3})$$

To derive an expression for $p_i(MC)$, we assume that the isotherm during the storage period is linear; i.e. EMC is low and fairly stable for any change in a_w . Using Equation (13), and noting that $a_w = p_i/p_{sat}$ under equilibrium conditions, $p_i(MC)$ is expressed as shown in Equation (A.4).

$$p_i(M) = a \cdot p_{sat} + b \cdot p_{sat} \cdot MC \quad (\text{A.4})$$

where, a and b are linear regression constants.

Substituting Equation (A.4) in Equation (A.3) and integrating the resulting expression, we get an equation for calculating the time required to reach a moisture level MC from an initial moisture level MC_i (Equation (A.5)).

$$t = \frac{LW}{PA b p_{sat}} \ln \left(\frac{p_o - a \cdot p_{sat} - b \cdot p_{sat} \cdot MC_i}{p_o - a \cdot p_{sat} - b \cdot p_{sat} \cdot MC} \right) \quad (\text{A.5})$$