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MODELLING MOISTURE SORPTION CHARACTERISTICS OF SIKKIM HIMALAYAN SEABUCKTHORN (*Hippophae salicifolia* D.) BERRIES

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Abstract: Seabuckthorn berries are edible and very rich in nutrients. The shelf life of fresh sea buckthorn does not exceed one week. Hence, the knowledge and understanding of moisture sorption isotherms for foods is of great importance in the design and optimization of packaging studies and prediction of shelf life. In this context, the present study was undertaken with an objective to develop moisture sorption isotherms of seabuckthorn and to suggest the best fit model for predicting the its sorption behaviour. This experiment was conducted using static gravimetric method using eight different saturated salt solutions corresponding to different relative humidities in the range of 11.31 - 85.11 % at 20°C. The experimental data obtained was fitted to six selected models viz., GAB, BET, Henderson, Oswin, Halsey and Modified BET. Desorption equilibrium moisture content values ranged from 7.76 – 50.30 % dry basis. The range of statistical parameters for the six tested models were: coefficients of regression R^2: 0.962 - 0.972, average percent error E: 10.20 % – 20.73 %, and χ^2: 0.0007 – 0.0012 and *RMSE*: 0.024 -0.03. Out of the six models tested, Halsey model showed the lowest values of χ^2 (0.0007), *RMSE* (0.024), second lowest value of E (10.7%) and highest value of R^2 (0.972) which indicated that this model can be can be regarded as the best fit model to predict the sorption behavior of seabuckthorn.
Keywords: Seabuckthorn, Moisture sorption, Modelling, relative humidity.

INTRODUCTION

Seabuckthorn (*Hippophae rhamnoides* L.) is a winter hardy deciduous shrub with yellow or orange berries (Bailey and Bailey, 1978). Seabuckthorn is mostly grown in the regions ranging from the Atlantic coasts of Europe to north-western Mongolia and north-western China. In India, Seabuckthorn is found in the states of Himachal Pradesh, Ladakh in Jammu and Kashmir, Uttarakhand, Arunachal Pradesh and Sikkim (Acharya et. al. 2010). In Sikkim, seabuckthorn (*Hippophae salicifolia* D. Don) grows generally at altitudes ranging from 2377–3093 m (Basistha et. al. 2009). India's agricultural departments are now taking note and pledging to cultivate one million hectares of sea buckthorn by 2020 as part of Green India Mission.

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Sea buckthorn berries are edible and very rich in nutrients. The seabuckthorn berries contain 8.44% oil in fruit pulp, 34.6 % protein in berry and 10.37% oil in seeds. The vitamin C, vitamin A, carotenoid and total soluble solid content in fruit juice are 1161.1–1302.5 mg/100g, 0.75 mg/100g, 7.2– 7.4 mg/100g, and 15.92– 17.66 respectively in fruit juice where as vitamin E in seed oil is 101.5–277.6 mg/100g, vitamin E in fruit pulp oil is about 255-435mg/100g, and total flavone in fresh fruit 354mg/100g (Li, 1999). Due to such high nutritional content, Jaiswal et. al (2017) has termed the Seabuckthorn as the “next major health food”. Despite so many health benefits, it is very difficult to store the berry for more than one week in ambient condition due to its delicate nature and high moisture content (Araya- Farias et. al. 2011). However, it can be extended up to 30 days without any quality changes by packaging in suitable containers with high barrier properties and air ambiance.

A moisture sorption isotherm describes the relationship between the water activity (a_w) and equilibrium moisture content for a food product at a constant pressure and temperature. The knowledge and understanding of moisture sorption isotherms for foods is of great importance in food science and technology for many problems such as the design and optimization of processing operations especially packaging problems, and for predicting shelf life.

Several researchers have investigated moisture sorption characteristics of different fruits, vegetables, and spices (Bazardeh and Esmaiili 2014; Seid and. Hensel 2012). These moisture sorption data can be modelled using many empirical and semi empirical equations that have been proposed in the literature (Kaymak-Ertekin and Sultanoglu 2001; Alakali et. al. 2009; Bazardeh and Esmaiili 2014). Although several mathematical models exist to describe water sorption isotherms of food materials, no unique or single model gives accurate results throughout the whole range of water activities, and for all types of foods (Lomauro et al., 1985). Moreover, there is no published information on sorption characteristics for Seabuckthorn till date. Therefore, the present study was taken up with the aim of developing moisture sorption isotherms and finding the best fit model to predict the moisture sorption characteristics of seabuckthorn.

MATERIAL AND METHODS

The fresh seabuckthorn (*Hippophae salicifolia* D.) berries were procured from local village (Lachung, India). The berries were washed, wiped dry and stored in sealed polyethylene packets in the refrigerator till further analysis. The samples were brought to room temperature for measurement. The samples were weighed using a digital balance (Contech, India) with an accuracy of ±0.001 g. Seabuckthorn berries of relatively uniform size and weight were used in the experiments. All the chemicals were procured from Merck, India.

Experimental Procedure

The clean seabuckthorn samples were taken for moisture sorption studies. Moisture sorption experiments were conducted using static gravimetric method (Labuza, 1984). Eight saturated salt solutions were used to give different corresponding relative humidity in the range of 11.31-85.11%. The relative humidities of the saturated salt solutions at the studied temperature are presented in Table 1. Representative samples of seabuckthorn berries were weighed and placed on small petri dishes, which were then kept inside eight sealed desiccators containing saturated salt solutions. Samples were kept in triplicate inside the desiccators. The desiccators were stored under controlled conditions at a temperature of 20 ± 1.1°C. Samples were weighed at 7 days interval. Samples were equilibrated for three to five weeks to reach a constant weight (0±0.001 g). The initial and equilibrium moisture content of the samples were determined using hot air oven drying method at 70°C for 48 h.

Table 1. Saturated salt solutions with corresponding equilibrium relative humidity

Salt	Equilibrium relative humidity at 20˚C, %
Lithium Chloride	11.31
Potassium acetate	23.2
Magnesium Chloride	33.6
Potassium Carbonate	43.16
Sodium Bromide	59.2
Sodium Nitrite	65.3
Sodium Chloride	75.5
Potassium Chloride	85.11
Ref: Singh and Sahay (2001)	

Modelling of Experimental Data

Out of the large number of models available in the literature to model moisture sorption characteristics, some of those more commonly used for vegetables and peppers were best described by the Guggenheim-Anderson-de Boer (GAB) model, Modified Brunauer, Emmett and Teller (BET) model, Oswin equation and Halsey equation which were shown in Table 2 (Kaymak-Ertekin and Gedik 2004; Kaymak-Ertekin and Sultanoglu 2001). These models were selected for the present study and experimental data of all samples were fitted to these six sorption models. The equilibrium relative humidity values were converted to water activity (a_w) by dividing with 100.

Table 2: Equations describing the sorption isotherms

Name of the models	Equations	References
Modified BET	$M = \frac{A}{1 - Ba_w}$	Modified BET (1996)
Oswin	$M = K\left(\frac{a_w}{1 - a_w}\right)^n$	Oswin (1946)
GAB	$M = \frac{M_o CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)}$	Van den Berg (1985)
Halsey	$a_w = \exp\left(-K/M^n\right)$	Halsey (1948)
Henderson	$1 - a_w = \exp\left(-CT_{ab}M^n)\right)$	Henderson (1952)
Brunauer-Emmett & Teller (BET)	$M = \frac{M_o CKa_w}{(1 - a_w) + (1 + (C - 1)a_w)}$	Brunauer et. al (1940)

Where *C, K, A, B* and *n* are constants in sorption isotherm models, a_w is water activity, *M* the equilibrium moisture content (dry basis) and M_o is the monolayer moisture content (dry basis).

For determination of model coefficients, these models were fitted to the experimental data using Lab fit curve fitting software V7.2.48. The statistical criteria used to evaluate the goodness-of-fit of each model were coefficient of determination (R^2), reduced chi square ($\chi 2$), root mean square error (RMSE) and average percentage error (E). For quality fit, R^2 value of the selected model should be highest and $\chi 2$, RMSE and E values should be lowest. The above parameters were calculated using the following equations (Kumar et. al. 2006).

$$\chi 2 = \frac{1}{N - z}\sum_{i=1}^{N}\left(M_{\exp,i} - M_{pre,i}\right)^2 \qquad \ldots (1)$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(M_{\exp,i} - M_{pre,i}\right)^2\right]^{1/2} \qquad \ldots (2)$$

$$E = \frac{100}{N}\sum_{i=1}^{N}\frac{\left|M_{\exp,i} - M_{pre,i}\right|}{M_{\exp,i}} \quad \ldots (3)$$

where, $M_{exp,i}$ is the experimental equilibrium moisture content, $M_{pre,i}$ is the predicted equilibrium moisture content, $\chi 2$ is reduced chi square, *RMSE* is root mean square error, *E* is average percentage error, *N* is number of readings and z is number of constants in the model.

RESULTS AND DISCUSSION

The average initial moisture content of seabuckthorn berries was found to be 83.78 ± 0.6 % wet basis (516.53±1.5 % dry basis). The experimental results of equilibrium moisture contents (*EMC*) for seabuckthorn berries at various water activity (a_w) values are given in Table 3. The equilibrium moisture content represents the average moisture contents of three replications. From Table 4.1, it can be observed that the *EMC* values ranged from 7.76 – 50.30% dry basis for water activity range of 0.1132–0.8511. Fig. 1 shows the developed desorption isotherm for seabuckthorn berries at 20 ± 1.1°C.

Table 3: Equilibrium moisture content (% dry basis) of seabuckthorn at 20 ±1.1°C

Water activity, aw	Average Equilibrium moisture content, % db
0.1131	7.7669
0.232	9.0090
0.336	10.4761
0.4316	11.8811
0.592	16.8316
0.653	25.7856
0.755	38.0952
0.8511	50.3034

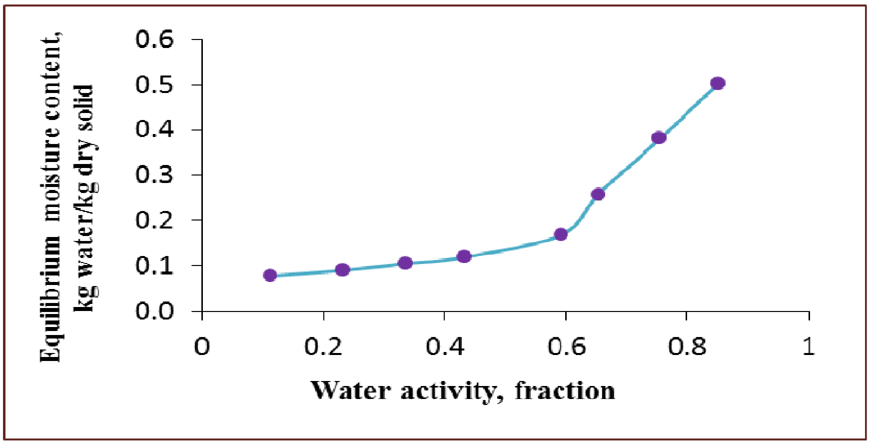

Fig 1: Desorption Isotherm of seabuckthorn berries at 20±1.1°C

The desorption isotherm developed from the experimental data represents the characteristic sigmoidal curve, typical of the many sorption isotherms of food (Iglesias and Chirife, 1982). According to the BET classification (Brunauer et al., 1940), this desorption curve is a type II curve. Similar type of sorption isotherms have been developed for raisins, currants (Tsami et al., 1990), and chilli pepper (Seid and Hensel, 2012). From fig. 1, it can be observed that the equilibrium moisture content decreased with a decrease in water activity at constant temperature. This may be due to the tendency of the food material to lower vapour pressure when decreasing the relative humidity of air (Moreira et. al. 2008). Similar effect has been reported by Kaymak-Ertekin and Sultanoğlu (2001) during sorption studies of green and red peppers. No reported literature was available for comparison of sorption data of seabuckthorn. Hence, the results were compared with sorption data of chilli peppers at 30°C as reported by Seid and Hensel (2012). The equilibrium moisture contents reported by these scientists chilli pepper were found to be lower than the observed experimental data due to higher temperature.

Modelling of Desorption Data

In the present study, six EMC models were fitted to the experimental desorption data. The coefficients of these models were obtained from the Labfit software and are reported in Table 4. From Table 4, it can be observed that the values of statistical parameters of the models viz., coefficients of regression R^2 in the range of 0.962 - 0.972, average percent error E in the range of 10.2 % – 20.73%, and χ^2 in the range of 0.0007 – 0.0012 and *RMSE* in the range of 0.024 -0.03. Almost all the models showed coefficients of regression of R^2 >0.96. A value of average percent error of 10 or less was considered as a good fit (Wang & Brennan, 1991). Out of the six models tested, Halsey (10.7%) and BET (10.2%) models showed an average percent error of about 10% which indicated that these two models can be considered as acceptable for predicting the equilibrium moisture content (EMC) of seabuckthorn. However, out of all the tested models, Halsey model showed the lowest values of χ^2 (0.0007), *RMSE* (0.024), second lowest value of E (10.7%) and highest value of R^2 (0.972) and hence, can be regarded as the best fit model to predict the desorption behaviour of seabuckthorn.

Table 4: Estimated sorption model constants and statistical parameters for seabuckthorn berries during desorption

Sl. No.	Model	
1.	**Oswin**	
	K	0.166
	n	0.646
	E, %	14.71
	R^2	0.970
	RMSE	0.0252
	χ^2	0.00085
2.	**GAB**	
	M_o	0.0988
	C	7.681
	K	0.956
	E, %	13.11
	R^2	0.971
	RMSE	0.025
	χ^2	0.00081
3.	**Modified BET**	
	A	0.082
	B	0.99
	E, %	14.96
	R^2	0.966
	RMSE	0.028
	χ^2	0.0011
4.	**Halsey**	
	K	0.071
	n	1.25
	E, %	10.70
	R^2	0.972
	RMSE	0.024
	χ^2	0.00078
5.	**BET**	

	M_0	0.0802
	C	30.87
	E, %	10.20
	R^2	0.964
	$RMSE$	0.027
	χ^2	0.0010
6.	**Henderson**	
	C	0.0122
	n	0.916
	E, %	20.72
	R^2	0.962
	$RMSE$	0.03
	χ^2	0.0012

Fig. 4 shows the comparison of the experimental data with the predicted equilibrium moisture contents by Halsey model. From this figure, it can be observed that Halsey model is fitting closely to the experimental data in the studied water activity range. Similar type of modelling for desorption behaviour of red chili pepper has been carried out by Seid and Hensel (2012) in the temperature range of 30°-70°C. According to them, GAB model was found to be the best fit model with an *E* value of 5.95% and R^2 value of 0.99 for the water activity range of 0.11–0.92. Kaymak-Ertekin and Sultanoğlu (2001) modeled the desorption behaviour of green and red peppers in the temperature ranges 30°- 60°C. They reported Halsey model to be the best fit model for their experimental water activity range of 0.11-0.9 with *E* value of 3.716 %. Hence, it can be inferred that the best fit desorption model varies with the temperature and water activity range studied even for similar type of food product. It may be attributed to variation in shape of the products studied.

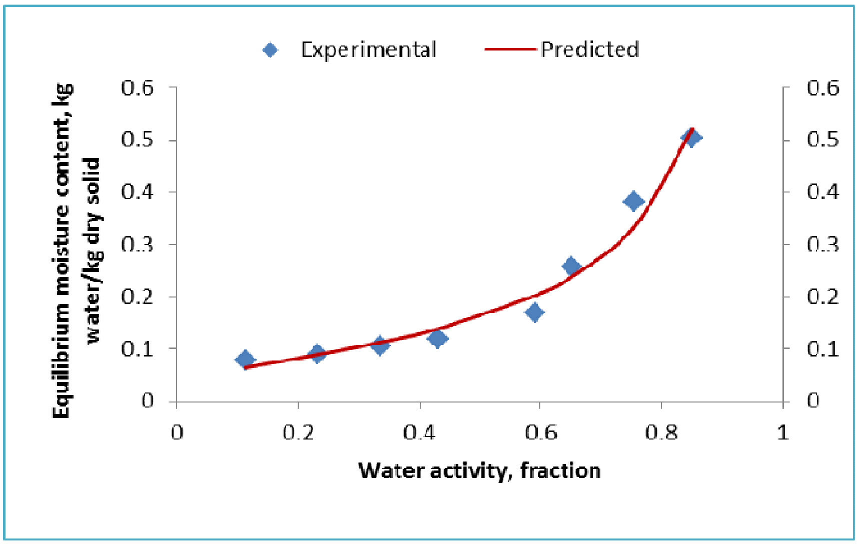

Fig 4: Comparison of the experimental EMC with the predicted EMC by Halsey model

CONCLUSIONS

In the present study, seabuckthorn (*Hippophae salicifolia* D. Don) berries collected from Sikkim were investigated for their moisture sorption characteristics. The study revealed a type II sigmoid desorption isotherm for seabuckthorn berries. Out of the six models selected, Halsey model was found to be the best fit model for predicting the desorption characteristics of seabuckthorn berries. The findings of the present study would be useful in selection of suitable packaging material for enhancing the shelf life of seabuckthorn berries.

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