Original Article

Prediction of Equilibrium Moisture Contents of Black Grape Seeds (Siah Sardasht cultivar) at Various Temperatures and Relative Humidity: Shelf-Life Criteria

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ABSTRACT

Background and Objectives: Food wastes are sometimes valuable; of which, seeds are significantly important. Grape seeds, as byproduct of grape processing, contain valuable substances for the production of advanced oils as well as feeds. Therefore, the major aim of this study was to assess moisture sorption isotherms of black grape seeds at various conditions.

Materials and Methods: Moisture sorption isotherms of black grape seeds (Siah Sardasht cultivar) were measured using static gravimetric method with saturated salt solutions at five various temperatures of 20, 30, 40, 50 and 60 °C. Water activity ranged 0.1–0.9. Five mathematical models of Guggenheim, Anderson and De Boer (three-parameter), Brunauer-Emmett-Teller (two-parameter), D'Arcy-Watt (five-parameter), Henderson (two-parameter) and Halsey (two-parameter) were used to fit data using non-linear regression analysis method.

Results: Results showed that the moisture sorption behavior of grape seed was temperature dependent as indicated by increases in equilibrium moisture contents at all levels of water activity with decreasing temperature. The best fit with experimental data in all water activities and temperatures were linked to Guggenheim, Anderson and De Boer model. D'Arcy-Watt model at 40, 50 and 60°C was adjusted well. The net isosteric heat of sorption was achieved using Clausius-Clapeyron equation and showed the maximum value (754.3 kJ/kg) at a moisture content of 0.1 (%d.b).

Conclusions: Rapid spoilage of grape seeds may occur at a water activity of 0.3 or greater for 20 °C and that of 0.6 for other temperatures. The Guggenheim, Anderson and De Boer model presented the best fitting. At highlighted temperatures, net isosteric heat increased significantly with decreases in moisture contents.

Keywords: Moisture sorption isotherm, Mathematical model, Black grape seed, Net isosteric heat

Introduction

Grapes are the most widely cultivated fruits in world. The global grape production can reach almost 60 million tons; from which, 38 million tons are processed for beverages and the rest for jellies, jams and raw consumptions. Annually, nearly 2.5 million tons of grape wastes such as skins and pips are produced by various industries (1-3). Grape seeds are the waste materials of grape processing, containing 8–20% of oils, 11% of protein, 10–20% of polyphenolic compounds such as tannin, sugars, minerals and other substances (4). The seed oils consist of high quantities of linoleic acid (70%), oleic acid (18%), palmitic acid (7%) and stearic acid (3%) (5). Quantities of monounsaturated and polyunsaturated fatty acids is much greater than saturated ones. Furthermore, the oil of grape seeds is highly popular as the gourmet oil (6).

Moisture contents of seeds should decrease to less than the critical values before processing to minimize microbial and chemical deteriorations (7). Relationships between the equilibrium moisture contents (EMC), water activity (a_w) and relative humidity (RH %) of the storage environments at constant temperatures are known as moisture sorption isotherms. These are important in physical, chemical and microbial analyses of foods and designing and optimization of drying equipment, calculation of the net heat of sorption, prediction of the necessary energy of drying processes, designing of packages, prediction of quality, stability and shelf-life and calculation of moisture changes that may occur during storage (8-10). Isotherm curves can predict the maximum moisture retention in foods during processing. Structure and composition of the food materials

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and storage temperatures play basic roles in the shape of sorption isotherms. For these reasons, sorption isotherms of various products have been considered comprehensively in literatures (11-16).

Several mathematical models have been introduced to describe moisture sorption isotherms of food materials. Based on the previous studies, the Guggenheim, Anderson and De Boer model included the best fit for the experimental data of prickly pear seeds at 40–70 ˚C (17) and pumpkin seeds at 5, 25 and 45 ˚C (18). Modified Henderson was the best model of cucumber seeds at 10–50 ˚C (19) and pea seeds at 5, 15, 25, 35 and 50˚C (20). Henderson model described sorption isotherms adequately in tomato seeds at 30–70˚C (21), grapefruit seeds at 40–70 ˚C (22) and pumpkin seeds at 5, 25 and 45 ˚C (18). The Oswin equation showed the best matching with orange seeds data at 40–70 ˚C (23). The Ferro-Fontan and Peleg model resulted in the best fit of grape seeds at 35–65 ˚C (24). Based on the importance of understanding hygroscopicity of agricultural products, the aim of this study was to investigate moisture sorption isotherms of black grape seeds at 20–60 ˚C to suggest an appropriate mathematical model for the prediction of their sorption behaviors as functions of temperatures at various relative humidity. These results provide useful information on how shelf life of black grape seeds can be prolonged with decreases in fungi contaminations until processing. The isosteric heat of sorption can be calculated using equations with the best fitting to experimental data. It describes the optimum drying condition by considering drying cost and quality of the seeds.

Materials and Methods

Raw materials and phase separation

Black grapes (Siah Sardasht cultivar) were purchased from vines of Sardasht in Western Azerbaijan Province, Iran. The freshly-harvested black grapes were stored at 4–5 °C until use. Separation of seeds from the black grape pulps was carried out manually before use.

Moisture sorption isotherms

After weighing 1 g of the grape seeds in Petri dishes, grapes were transferred into air-tight glass jars containing saturated salt solutions. Static gravimetric method was used according to the instructions COST 90 (25, 26) at 20, 30, 40, 50 and 60 °C for measuring moisture sorption isotherm of the black grape seeds. Nine saturated salt solutions were selected to produce various relative humidity values of 0.1–0.9. Regression equations for the calculation of a_w of the salt solutions at various temperatures are presented in Table 1. A small quantity of toluene was used in jars to prevent fungal activity in samples at the relative humidity above 50%. Experiments were repeated three times.

Table 1. Regression equations of water activity of salt solutions at various temperatures (14, 50)

salts	Regression equations	predicted a_w
LiCl	Ln a_w= 500.95 / T- 3.85	0.1
CH3COOK	Ln a_w= 861.39/ T- 4.33	0.2
MgCl2	Ln a_w= 303.35 / T- 2.13	0.3
K2CO3	Ln a_w= 145.00 / T- 1.30	0.4
Mg(NO3)2	Ln a_w= 356.60 / T- 1.82	0.5
NaNO2	Ln a_w= 435.96 / T- 1.88	0.6
NaCl	Ln a_w= 228.92 / T- 1.04	0.7
KCl	Ln a_w= 367.58 / T- 1.39	0.8
BaCl2	Ln a_w= 96.955/ T- 0.426	0.9

a_w: water activity, T: absolute temperatures (K)

Samples were transferred into jars and set to equilibrate with surrounding air at selected temperatures using incubator (B30, Germany) until there was not more than a 0.001-g difference between two consecutive measurements (±0.001 g) (AX623, Sartorius, Germany). Temperatures were checked and controlled within ±1 °C. The EMC of the seeds were assessed by drying samples in vacuum oven (General Electronic 5KH33GG, USA) at 50 ˚C for 48 h using the following equation.

$$m_e = \frac{m_w - m_d}{m_d} \qquad (1)$$

Where, m_e was the EMC (% dry basis) and m_w and m_d were weights of the wet (moisture equilibrated samples at incubator) and dry samples, respectively.

Modeling of moisture sorption isotherms

The GAB, BET, D'Arcy-Watt, Henderson and Halsey equations (Table 2) were fitted to experimental sorption data using regression analysis. The efficiency of fit was evaluated with $p < 10\%$ (27), the least RMSE (Root-mean-square error), χ^2 (chi-squared) and maximum (R^2) (Coefficient of determination) (28) (Table 3).

Assessment of isosteric heat

Net isosteric heat is an important thermodynamic parameter derived from moisture sorption isotherms. Clausius-Clapeyron equation is often used to assess net isosteric heat by plotting ln a_w against 1/T at a specific moisture content of the materials and plotting the slope (29).

$$q_{st} = -R \frac{\partial (Lna_w)}{\partial \left(\frac{1}{T}\right)} \qquad (2)$$

Where, a_w was the water activity, q_{st} was the net isosteric heat of sorption (kJ/kg), R was the universal gas constant (kJ/kg K) and T was the absolute temperature (K).

Table 2. Models fitted to the experimental data of grape seeds

Name of the equation	Equation	Reference
GAB	$m_e = m_m \dfrac{k_1 k_2 a_w}{(1 - k_1 a_w)(1 - k_1 a_w + k_1 k_2 a_w)}$	(3)
BET	$m_e = m_m \dfrac{k a_w}{(1 - a_w)(1 - a_w + k a_w)}$	(4)
D'Arcy- Watt	$m_e = \dfrac{k_1 k_2 a_w}{1 + k_1 a_w} + k_5 a_w + \dfrac{k_3 k_4 a_w}{1 - k_3 a_w}$	(5)
Henderson	$m_e = (-\dfrac{ln(1 - a_w)}{k_1 T})^{\frac{1}{k_2}}$	(6)
Halsey	$m_e = (\dfrac{-k_1}{ln(a_w)})^{\frac{1}{k_2}}$	(7)

Where m_m is monolayer moisture content, m_e is the equilibrium moisture content (% d.b), k_1, k_2, k_3, k_4, k_5 are sorption constants, a_w is water activity, T is temperature.

Table 3. Evaluation criteria used by the models

Name of the equation	Equation	Reference	
Chi-squared	$\chi^2 = \dfrac{\sum_{i=1}^{n} \left(MR_{\exp i} - MR_{pre,i}\right)^2}{N - n}$	(44)	(8)
Root-mean-square error	$RMSE = \sqrt{\dfrac{\sum_{i=1}^{n} \left(MR_{pre,i} - MR_{\exp i}\right)^2}{N}}$	(51)	(9)
Mean relative modulus	$P\% = \dfrac{100}{N} \sum \dfrac{abs\left(M_{\exp} - M_{pre}\right)}{M_{\exp}}$	(52)	(10)
Least square	$R^2 = 1 - (\text{Residual SS})/(\text{Corrected total SS})$	(53)	(11)

Results

Sorption isotherms

Moisture sorption isotherms for the black grape seeds are presented in Fig. 1. The EMC of the seeds increased by increasing a_w at constant temperatures. In the present study, slopes of the isotherms were gentle at $a_w < 0.3$ for 20 ˚C and $a_w < 0.6$ for 30, 40, 50 and 60 ˚C; where, relatively low moistures were absorbed for high increases in a_w. Above this level, however, high quantities of water were absorbed for small increases in a_w, especially at 20 ˚C, showing that rapid spoilage of grape seeds might be expected at $a_w > 0.3$ for 20 ˚C and $a_w > 0.6$ for other temperatures. It can be concluded that as the temperature increased at a constant a_w, the EMC decreased. Therefore, at any a_w, grape seeds became less hygroscopic with increasing temperatures. At fixed moisture contents, increases in a_w were observed with increasing temperatures, possibly making the products more susceptible to microbial spoilage.

Model use

The GAB, BET, D'Arcy-Watt, Henderson and Halsey equations were used to sorption data. Based on Table 4, GAB model produced the lowest P% values ranging 4.664–8.268%, compared to that the other models. Did. Furthermore, the smallest RMSE (0.005–0.014) and χ^2

(0.000) and the highest R^2 (92.9–99.8%) at all temperatures were achieved by the GAB equation. Based on Table 4 and Fig. 2, the D'Arcy-Watt model at 40, 50 and 60 ˚C was acceptable for the prediction of EMC while the GAB model showed the best fitting performance. The BET, Henderson and Halsey models were not appropriate to predict moisture sorption of the black grape seeds because they presented values higher than 10% of P and negative values of R^2. Parameters estimated from the five models are listed in Table 5.
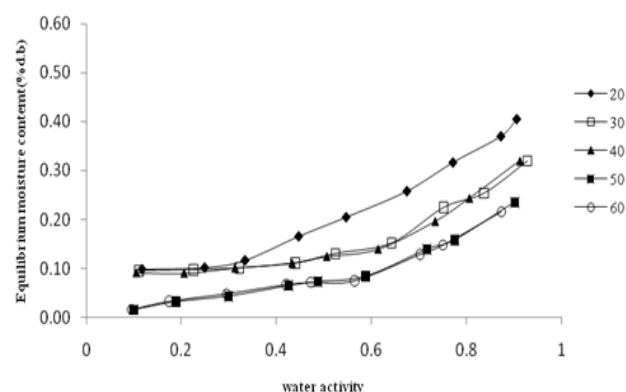

Figure 1. Moisture sorption isotherms of black grape seeds at various temperatures

Table 4. Evaluation of the prediction models

Temperature	model	P%	RMSE	χ^2	R^2
20	GAB	8.268	0.014	0.000	0.990
20	BET	32.612	0.083	0.009	0.002
20	D'Arcy –Watt	11.261	0.022	0.001	0.969
20	Henderson	41.003	0.103	0.014	0.360
20	Halsey	82.470	0.189	0.046	0.198
30	GAB	6.513	0.012	0.000	0.978
30	BET	42.967	0.075	0.007	*
30	D'Arcy –Watt	13.338	0.022	0.001	0.978
30	Henderson	39.882	0.072	0.007	0.401
30	Halsey	42.267	0.074	0.007	0.163
40	GAB	7.050	0.011	0.000	0.929
40	BET	36.957	0.060	0.005	*
40	D'Arcy –Watt	2.723	0.004	0.000	0.997
40	Henderson	43.310	0.074	0.007	0.385
40	Halsey	45.994	0.080	0.008	*
50	GAB	4.664	0.005	0.000	0.998
50	BET	20.370	0.028	0.001	0.667
50	D'Arcy –Watt	4.520	0.005	0.000	0.997
50	Henderson	55.172	0.062	0.005	0.218
50	Halsey	94.883	0.077	0.008	*
60	GAB	6.516	0.005	0.000	0.989
60	BET	13.413	0.019	0.000	0.734
60	D'Arcy –Watt	6.553	0.005	0.000	0.984
60	Henderson	58.215	0.068	0.006	0.181
60	Halsey	98.834	0.083	0.009	*

*: Shows negative R^2

Table 5. Fitting constants for the fits of the models to the sorption data for black grape seeds

| GAB	m	K_1	K_2		
20	0.134	0.758	10.099		
30	0.108	0.704	7.242		
40	0.075	0.840	36.952		
50	0.065	0.834	3.054		
60	0.054	0.877	4.960		
BET	m	k			
20	0.049	18.525			
30	0.030	27.861			
40	0.036	18.284			
50	0.028	10.015			
60	0.032	9.767			
Darcy-watt	k_1	k_2	k_5	k_3	k_4
20	-3.253	-0.002	0.423	1.828	1E-07
30	8.969	0.085	-0.135	0.522	0.362
40	40.221	0.128	-0.548	0.526	0.753
50	0.456	0.902	-0.394	0.654	0.227
60	0.702	1.204	-0.895	0.608	0.478
Henderson	k_1	k_2			
20	0.504	1.607			
30	0.667	1.727			
40	0.417	1.601			
50	0.223	1.084			
60	0.187	1.089			
Halsey	k_1	k_2			
20	0.015	2.228			
30	0.005	2.477			
40	0.008	2.189			
50	0.009	1.630			
60	0.015	1.465			
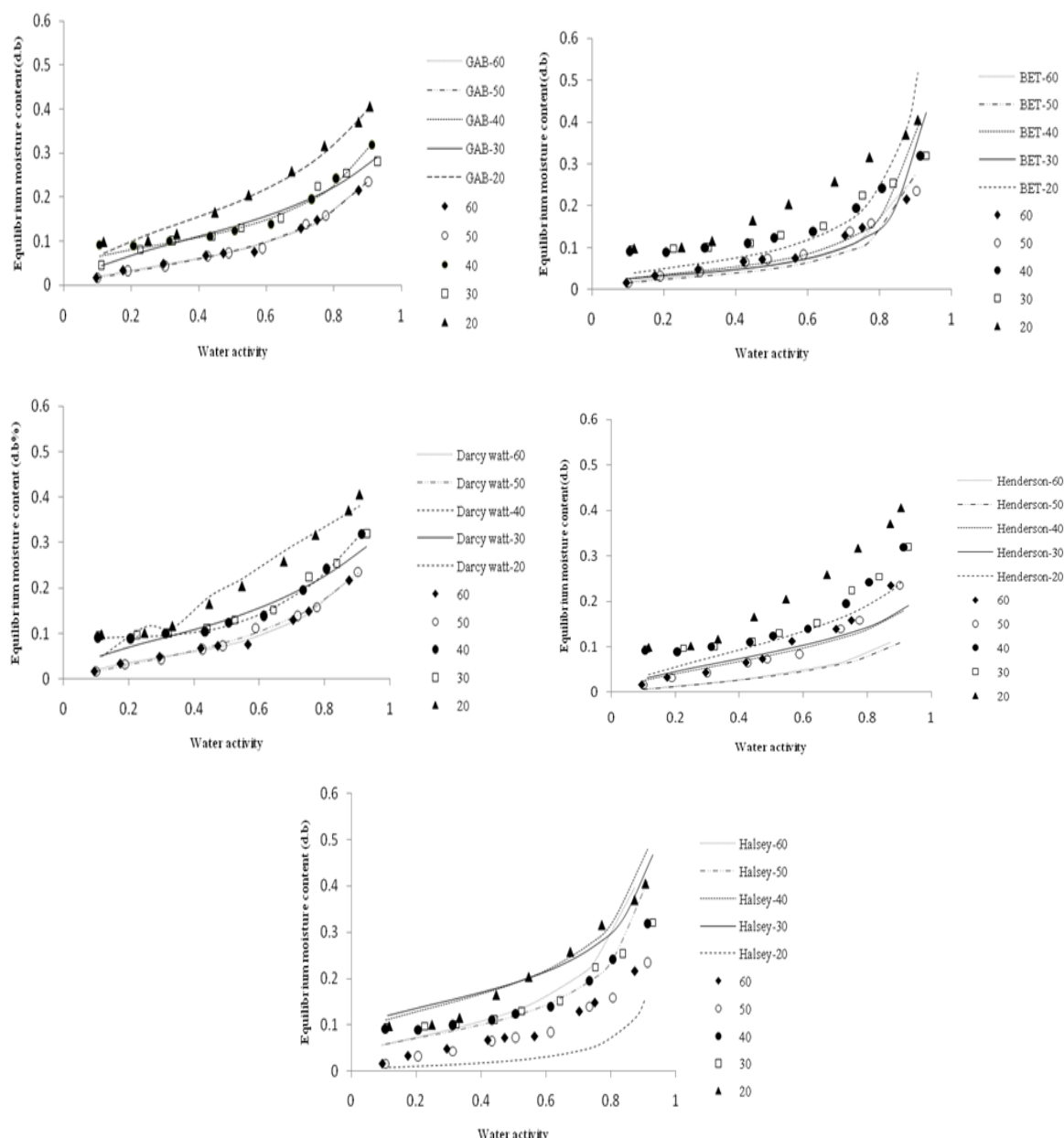
Figure 2. Moisture sorption isotherms of black grape seeds at various temperatures. The symbols are experimental data and the lines are from the equations achieved by fitting the experimental data to Guggenheim, Anderson and De Boer, Brunauer-Emmett-Teller, D'Arcy-Watt, Henderson and Halsey equations
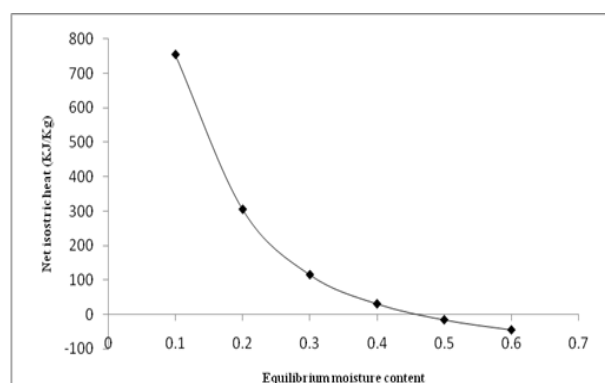

Figure 3. The net isosteric heat of sorption for the grape seeds as a function of moisture contents

Thermodynamic properties

The isosteric heats of sorption are plotted versus moisture content in Fig. 3. The heat of sorption (differential enthalpy) was calculated and increased as moisture content decreased.

Discussion

Sorption isotherms

The EMC of samples increased with increases in a_w at specific temperatures. Similar results were reported for tomato (21), lime (30), cucumber (19), teff (26), Brazil nut (31) and sunflower (32) seeds. At constant a_w, increases in temperature led to decreases in EMC. Analogous trends

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were seen in prickly pear (17), tomato (21), cucumber (19) and lime (30) seeds.

Model use

To keep foods and food products for the longest time, they should be held at low moisture contents, the optimum of which is the monolayer. The monolayer water content (m_m) corresponds to moisture levels; at which, rates of oxidation and chemical reactions in foods are really low since water molecules are bound strongly to the surface of this layer (33, 34). However, above the monolayer, decay rate accelerates (35). For grape seeds, the monolayer moisture content can be used to estimate shelf stability and efficient use of energy in drying processes. This critical moisture content can be calculated using the GAB model (36, 37). In a temperature range of 20–60 °C, monolayer moisture content of the black grape seeds varied 0.13–0.05 (d.b%), (Table 5). It was seen that m_m decreased with increasing temperatures, which can be attributed to the reduction of hydrogen bonding degree of food polymers induced by increasing temperatures and consequently decreasing availability of active sites for water binding and monolayer moisture contents (38-41). Similar results have been reported for the moisture sorption of African star apples, mangos (42) and figs (43). A parameter in BET model, namely the sorption energy constant (k), can be used for the classification of sorption isotherms based on the Brunauer classification. Since k values for the grape seeds were higher than 2 at 20–60 °C (Table 5), their sorption isotherms were classified as Type II. Achieved isotherms in this study were included in this type ($k = 9.8$–27.8) and presented the characteristic of the S-shaped curve (Type II), typical of sorption isotherms of many plants and food materials. This behavior has been shown in other fruits such as apples (42), grapes (44), spray-dried tomato pulps (45) and grape seeds (24).

Thermodynamic properties

The net isosteric heat of sorption (the latent heat of vaporization) was assessed using the Clausius-Clapeyron equation. Due to the proper description of the phenomenon by the GAB model, the model was used to calculate the heat change of sorption. Figure 3 demonstrates effects of moisture contents on isosteric heat of sorption for the black grape seeds. The curve shows that by decreasing EMC from 0.6 to 0.3 (% d.b), the heat of sorption increased slowly (from -45 to 115.1 kJ/kg) and later increased quickly (from 305.18 to 754.30 kJ/kg) by decreasing the moisture contents from 0.2 to 0.1 (% d.b). It could occur due to the greater resistance to moisture migration from the surface to the inside (46), revealing the highest energy of binding for removing the water of seeds. This means that polar sites exist on the surface of the seeds, which are extremely active and should be covered with the monomolecular layers of water molecules (17, 29, 47), as seen in several other food systems (24, 32, 43, 48, 49).

Conclusion

Of the highlighted equations for the prediction of sorption behaviors of black grape seeds, GAB model presented the best fitting with experimental data at all water activities and temperatures. It was verified that the thermodynamic properties of seeds were affected by the water contents. Isosteric desorption heat (-45–754.30 kJ/kg) increased with decreasing moisture contents (0.6–0.1% d.b), showing increases of necessary energy to remove water from the products during desorption. Monolayer water contents, as criteria for increasing shelf stability, varied 0.13–0.05 (d.b %) at a temperature range of 20–60 °C. To keep seeds for the longest time, moisture content should be reduced to less than the moisture content of monolayer. Furthermore, it has been suggested that spoilage of black grape seeds could be retarded by keeping seeds at a relative humidity less than 0.3 at 20 °C and less than 0.6 at other temperatures.

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