

## Moisture Sorption Characteristics of Freeze Dried Blueberries

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### ABSTRACT

Moisture sorption isotherms of freeze dried lowbush blueberries at 4, 16, 25, 35, and 45°C were determined using indirect gravimetric method. Moisture sorption kinetics at these temperatures and 75% RH were also investigated. The isotherms followed a type III shape with a temperature inversion effect at 0.9 water activity. They were best described by the GAB equation. Net isosteric heat of moisture sorption in freeze dried blueberries, determined using the Clausius Clapeyron equation, varied from 5.5 to 0.25 kJ/mol as moisture content changed from 8 to 36% (dry basis). Moisture adsorption kinetics in freeze dried blueberries were well described by the first order kinetics equation. The rate constant followed the Arrhenius relationship with an activation energy of 38.6 kJ/mol.

Key Words: blueberries, freeze dried, moisture sorption, isotherms

### INTRODUCTION

LOWBUSH BLUEBERRIES, *Vaccinium augustifolium* Ait., are a native crop of North America. The production of this crop has been increasing steadily and reached over 120 million pounds in 1990 (Statistic Canada, 1994). Fresh blueberries are highly perishable. After harvest, they can be stored without spoilage for only 2 wk under refrigerated conditions. Blueberries may be kept frozen for a long time. However, frozen products entail increased storage and transportation costs. Prolonged frozen storage results in fractures at the cellular level. Leaching of cellular content and soft texture when thawed limit applications for frozen blueberries. Alternative preservation methods including dehydration were investigated to prolong shelf life of blueberry products. Kim and Toledo (1987) reported that high temperature fluidized-bed drying reduced dehydration time as compared to conventional air drying, and the dried product had lower bulk density, larger diameter and higher rehydration ratio. Sullivan et al. (1982) used a continuous explosion puffing process to dry rabbiteye blueberries. They found the berries were very susceptible to skin rupture which caused severe 'bleeding'. Yang and Atallah (1985) compared the effects of freeze drying, forced air, vacuum oven and micro-convection methods on the quality of intermediate moisture (16–25%) lowbush blueberries. Higher vitamin C and soluble solids retention were observed in freeze dried products. These products also had the highest rehydration ratio and lowest bulk density.

Water activity influences microbial growth, enzymatic reactions and lipid oxidation in food products (Labuza, 1968). In order to better control the quality of freeze dried blueberries during production and storage, data are needed on physical properties including equilibrium moisture content (EMC)/equilibrium relative humidity (ERH) relationships, which may be expressed in moisture sorption isotherms. The heat of sorption data are also useful when assessing energy requirements for drying. Understanding moisture sorption kinetics may help in selecting appropriate stor-

age conditions and evaluating moisture sorption during storage. Our objectives were to determine moisture sorption isotherms at 4, 16, 25, 35, and 45°C, develop a suitable model to describe them, and determine the heat of moisture sorption of freeze dried blueberries, and kinetics involved at various temperatures.

### MATERIALS & METHODS

LOWBUSH BLUEBERRIES which were fluidized frozen at -40°C were obtained from a local food processor (Cobi Foods Inc., Berwick, NS). Frozen berries were sorted and cut into halves manually using a sharp blade. Samples were placed on a stainless steel tray and stored at -30°C for 24 hr prior to freeze drying.

#### Freeze drying

Blueberry samples were freeze dried in a Modulyo 4K freeze dryer (Edwards High Vacuum International, Crawley, England) for about 48 hr. The pressure in the drying chamber was set at 20 Pa and the temperature in the condensing chamber was controlled at -40°C. Sublimation heat was provided by a heater mat set at 30°C underneath the drying tray. The freeze dried samples at 2–3% moisture content (dry basis) were separated into two lots. One lot was powdered with a food blender, while the other was untreated. Samples were stored in air tight containers in a desiccator prior to moisture sorption experiments.

#### Sorption data

Saturated salt solutions were used to provide constant relative humidity (RH) environments ranging from 24 to 86% at 4, 16, 25, 35, and 45°C (Table 1). RH data of these saturated salt solutions were obtained from published data (Labuza, 1984; Hutchinson and Otten, 1984; Pappas and Rao, 1987) and confirmed by a CX-1 Water Activity System (Decagon Devices, Pullman, WA).

On the arrangement for the moisture sorption experiments (Fig. 1), the saturated salt solutions, together with undissolved salts, were introduced into 500 mL air-tight plastic containers. Duplicate samples (1.0g) of powdered and untreated freeze dried blueberries were weighed in trays and placed on supports above the solutions. Containers were covered with lids and allowed to equilibrate in incubators set at selected temperatures. The samples were removed and weighed every 3 days until differences between two consecutive measurements were <0.5% of sample weights. For kinetics measurements, samples were placed in containers with saturated NaCl solutions at 4, 14, 22, 34 and 44°C. Relative humidities of saturated NaCl at these temperatures were 75.6, 75.6, 75.4, 74.9, and 74.6%, respectively. Sample weights were measured at 2 hr intervals during the first 24 hr, and at 1/2 day intervals towards the end of the experiment (about 20 days). The equilibrium moisture contents were determined by drying samples in a vacuum oven at 70 ± 1°C for 6 h under pressure < 100 mmHg (AOAC, 1990).

Table 1—Relative humidities provided by saturated salt solutions at 4, 16, 25, 35, and 45°C

Salt	Temperature				
	4° C	16° C	25° C	35° C	45° C
CH <sub>3</sub> COOK		0.236			
MgCl <sub>2</sub>	0.336	0.338	0.330	0.330	0.330
Mg(NO <sub>3</sub> ) <sub>2</sub>	0.592	0.563			
NaCl		0.755	0.750	0.750	0.750
NaBr			0.570	0.550	0.520
NaNO <sub>2</sub>			0.645	0.625	0.620
KCl				0.860	0.850
K <sub>2</sub> CO <sub>3</sub>	0.431				
KOH	0.143				
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0.824	0.814	0.790		

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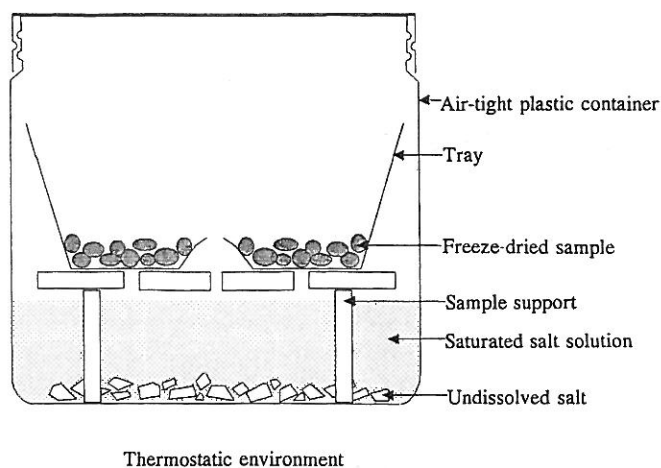


Fig. 1—Arrangement for moisture sorption tests.

### Isotherm models

The following five equations frequently used to describe EMC/ERH relations in foods were selected from published studies:

**GAB equation** (Berg, 1985; Weisser, 1985; Kiranoudis et al., 1993):

$$\frac{M}{M_m} = \frac{ACa_w}{(1 - Aa_w)(1 - Aa_w + ACa_w)} \quad (1)$$

where,

$$C = C_0 \exp\left(\frac{\Delta H_c}{RT}\right), \quad \Delta H_c = H_m - H_n \quad (2)$$

$$A = A_0 \exp\left(\frac{\Delta H_A}{RT}\right), \quad \Delta H_A = H_l - H_n \quad (3)$$

**Modified Handerson equation** (Handerson, 1952; Thompson et al., 1968; Sokhansanj et al., 1986):

$$M = \left( -\frac{1}{A(T+C)} \ln(1 - a_w) \right)^{1/B} \quad (4)$$

**Modified Chung Pfost equation** (Chung and Pfost, 1967; Chen and Morey, 1989a):

$$M = \frac{1}{B} \left( \ln\left(\frac{A}{T+C}\right) - \ln(-\ln(a_w)) \right) \quad (5)$$

**Modified Halsey equation** (Halsey, 1948; Iglesias and Chirife, 1976):

$$M = \left( \frac{-\exp(C+BT)}{\ln(a_w)} \right)^{1/A} \quad (6)$$

**Modified Oswin equation** (Chen, 1988):

$$M = (A+BT) \left( \frac{a_w}{1-a_w} \right)^C \quad (7)$$

In the equations,  $a_w$  is water activity (or ERH) and  $M$  is equilibrium moisture content (EMC).  $M_m$  is the monolayer moisture content;  $A$ ,  $B$  and  $C$  are constants;  $R$  is the universal gas constant;  $T$  is the absolute temperature;  $H_m$  sorption heat of monolayer,  $H_n$  multilayer moisture, and  $H_l$  condensation heat of water. The GAB (Guggenheim Anderson de Boer) equation was selected for its theoretical significance. The others were chosen because temperature is included as one of the variables, and EMC can be explicitly expressed as a function of  $a_w$ . In practice, a temperature dependent equation is more convenient as only one set of parameters is needed in describing isotherms at different temperatures. When using the GAB model, Eq. (2) and (3) were combined with Eq. (1). The resulting equation contains five parameters ( $M_m$ ,  $\Delta H_c$ ,  $\Delta H_A$ ,  $C_0$  and  $A_0$ ). This method presents an advantage over indirect regression. When successive regressions are involved, the first estimation may introduce notable uncertainties into subsequent regressions (Tsami et al., 1990a; Kiranoudis et al., 1993).

Ajibola (1986) found that the standard error of estimated equilibrium moisture content (EMC) was lower when water activity ( $a_w$ ) was the

independent variable than when it was the dependent variable. Therefore, all five models were arranged so that EMC was expressed as a function of  $a_w$ . Sorption data were fitted to the EMC/ERH equations using the nonlinear regression procedure PROC NLIN in SAS (SAS Institute Inc., 1988). Three criteria were used to evaluate fit of models:

**Mean sum of squares of residuals (MSE):**

$$MSE = \frac{\sum (Y - Y')^2}{df} \quad (8)$$

$Y$  and  $Y'$  are the measured and predicted moisture contents, respectively;  $df$  is the degrees of freedom of the residuals.

**Mean relative percentage deviation  $P$**  (Boquet et al., 1978, 1979; Chen and Morey, 1989a, b):

$$P = \frac{100}{N} \sum \frac{|Y - Y'|}{Y} \quad (9)$$

where  $N$  is the number of data points.  $P$  value indicates the relative error of the predictions.

**The plots of the residuals**, where the residuals are defined as:

$$e_i = Y - Y' \quad (10)$$

The residual plot for an ideal model should be randomly distributed around zero without definite pattern.

### Isosteric heat of sorption

The net isosteric heat of sorption  $\Delta H_{st}$  was determined from the following expression derived from Clausius Clapeyron equation (Weisser, 1985; Ayrançi et al., 1990; Kiranoudis et al., 1993):

$$\Delta H_{st} = -R \left( \frac{\partial(\ln a_w)}{\partial(1/T)} \right)_M \quad (11)$$

where  $\Delta H_{st}$  (J/mol) is the total molar enthalpy change during the phase transition minus, at a fixed level of moisture content  $M$ , the heat of vaporization of pure water.  $R$  is the universal gas constant (8.314 J/mol K). If  $\Delta H_{st}$  is independent of temperature, it can be estimated from the slope of the linear relation between  $\ln(a_w)$  and  $1/T$ . Alternatively, Eq. (11) can be integrated at fixed equilibrium moisture content to give:

$$\ln\left(\frac{a_{w1}}{a_{w2}}\right) = -\frac{\Delta H_{st}}{R} \left( \frac{1}{T_1} - \frac{1}{T_2} \right) \quad (12)$$

where  $a_{w1}$  and  $a_{w2}$  are water activities at temperatures  $T_1$  and  $T_2$ , respectively.

### Kinetic of moisture sorption

The changes in moisture content of freeze dried blueberries during moisture sorption were characterized by high moisture sorption rates at initial stages and gradual decreasing rates as moisture contents approached equilibrium values. Due to the porous structure of freeze dried blueberries, moisture sorption was expected to be primarily rate controlled rather than diffusion controlled. The following first order kinetics equation was used to predict moisture adsorption in the freeze dried blueberries (Singh et al., 1981):

$$\frac{M_e - M}{M_e - M_0} = Ae^{-kt} \quad (13)$$

where  $M_0$  and  $M_e$  are the initial and equilibrium moisture contents, respectively.  $M$  is the transient moisture content,  $k$  is the rate constant for hydration and  $A$  is a constant. Parameters in Eq. (13) were estimated using the nonlinear regression procedure PROC NLIN in SAS (SAS Institute Inc., 1988).

## RESULTS & DISCUSSION

### Moisture sorption isotherms

During tests, no yeast or mold contamination was observed, except for one sample exposed to 86% RH at 35°C. The data for that sample were discarded. The difference between EMC of powdered and untreated samples was insignificant ( $p > 0.05$ ). Similar observations were reported by Lima and Vidal (1983) with freeze dried bananas. Thus, mean moisture contents were

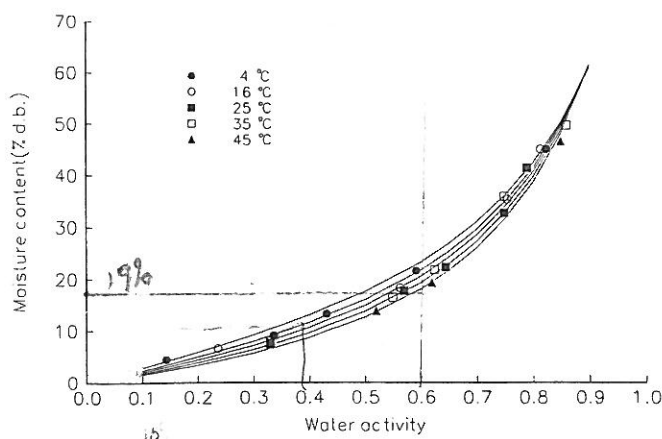


Fig. 2—Experimental data for freeze dried blueberries and predicted sorption isotherms using GAB equation.

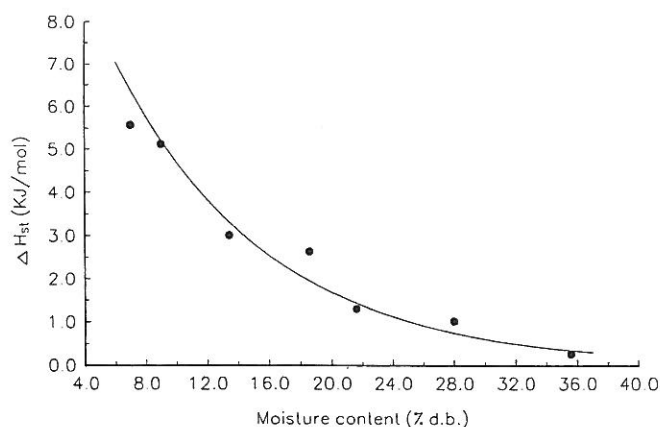


Fig. 3—Net isosteric heat data for freeze dried blueberries; the best fit curve is based on Eq. (14).

Table 2—Estimated parameters for five equilibrium moisture sorption models

Models	Parameter					Statistic	
	A	B	C			MSE	P
Modified Chung-Pfost	301.50	0.04970	163.79			7.9857	17.348
Modified Halsey	1.1749	-0.0035	2.9080			5.1181	11.557
Modified Oswin	16.230	-0.0443	0.7009			2.7967	6.2018
Modified Handerson	0.0002	0.8477	326.69			1.9362	6.2876
GAB equation	$M_m$ (% d.b.)	$C_0$	$A_0$	$\Delta H_c$ (J/mol)	$\Delta H_A$ (J/mol)		
	17.387	0.00481	1.115	13816.75	-653.33	1.909	5.434

obtained by averaging moisture contents of powdered and untreated berry samples for each set of storage conditions.

The experimental moisture sorption isotherms (Fig. 2) agreed well with dried orinithian raisins, sultana raisins, figs, prunes, strawberries, pears, guavas, mangos and pineapples (Iglesias and Chirife, 1978; Bolin, 1980; Ayranci et al., 1990; Tsami et al., 1990a; Pääkkönen and Mattila, 1991). They showed type III isotherm patterns (J shape) commonly observed in high sugar content food products (Labuza et al., 1985). Sugar content in raisins, figs, and apricots ranged from 51.5 to 82.3% (dry basis) (Tsami et al., 1990a), while blueberries contain  $\approx 50\%$  sugar (Yang and Atallah, 1985). Similar to isotherms of those high sugar fruits (Ayranci et al., 1990; Tsami et al., 1990a), isotherms of blueberries intersected, but at a higher water activity level (about 0.9). According to Weisser (1985), the intersection point depends on the type of sugar present, sugar size distribution, and composition of the foods.

#### EMC/ERH model

Among the five equations, the GAB equation predicted the isotherms with smallest MSE and P value (Table 2). Residuals from the GAB model were also randomly scattered. Therefore, this model appeared to best describe the sorption data. With the Modified Oswin equation, the residuals spread as water activity increased. A U shape residual plot was observed when using the Modified Handerson equation. The estimated parameters of the GAB equation compared well with those for other fruits determined by Tsami et al. (Table 3). The isotherms predicted by the GAB equation were illustrated as solid curves (Fig. 2).

In the GAB equation,  $\Delta H_c$  is the difference between enthalpy of monolayer and multilayer sorption (Eq. 2). Its value was estimated to be 13.82 kJ/mol for freeze dried blueberries. This suggests that stronger bonds exist between solids and monolayer water molecules than between the monolayer and subsequent multilayers of water molecules. A negative value ( $-0.65$  kJ/mol) was obtained for  $\Delta H_A$ , defined in Eq. (3) as the difference between heat of sorption of water and heat of sorption of

Table 3—Comparison of estimated GAB parameters for freeze dried blueberries with those for raisins, currants, figs, prunes, apricots obtained by Tsami et al. (1990a)

Dried fruit	$M_m$ (% d.b.)	$C_0$	$\Delta H_c$ (kJ/mol)	$A_0$	$\Delta H_A$ (kJ/mol)
Raisins	14.0	0.01557	10.5	1.30	-0.793
Currants	17.3	0.05725	5.7	1.58	-1.341
Figs	9.7	0.00023	22.5	1.22	-0.531
Prunes	12.6	0.00064	18.5	1.42	-0.994
Apricots	11.7	0.00186	14.6	1.44	-0.980
Blueberries	17.4	0.00481	13.8	1.12	-0.653

multilayers. This suggests that more energy was needed to overcome the sorption energy of the multilayer water in the hygroscopic blueberry system than that for pure water.

#### Heat of sorption

The net sorption heat was considerably more at lower moisture contents, and it decreased to approach the latent heat of vaporization of pure water ( $\Delta H_{st} = 0$ ) as moisture content increased (Fig. 3). Thus, the heat evolved during sorption of the first few layers of water molecules was substantially more than the heat of condensation of free water. This was probably due to strong interactions between these water molecules and the hydrophilic groups of the food solid.

Tsami et al. (1990b) proposed an empirical equation to describe the relationship between  $\Delta H_{st}$  and the equilibrium moisture content:

$$\Delta H_{st} = \Delta H_0 \exp\left(-\frac{M}{M_0}\right) \quad (14)$$

where  $M$  is the equilibrium moisture content,  $\Delta H_0$  is the isosteric heat of sorption of the first molecule of water,  $M_0$  is a characteristic moisture content of the food material. Eq. (14) was transformed into a linear form by using the natural logarithm on both sides. The transformed equation was fitted to  $\Delta H_{st}$  data as a

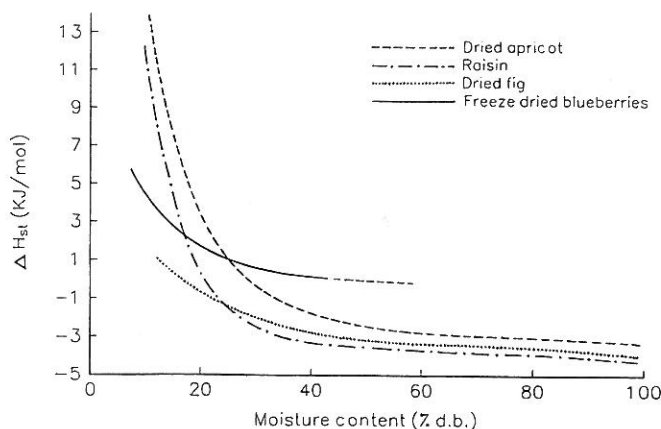


Fig. 4—Comparison of the net isosteric heat of sorption for freeze dried blueberries with dried apricot, raisin and dried fig. The data for the latter three fruits are adapted from Ayranci et al. (1993).

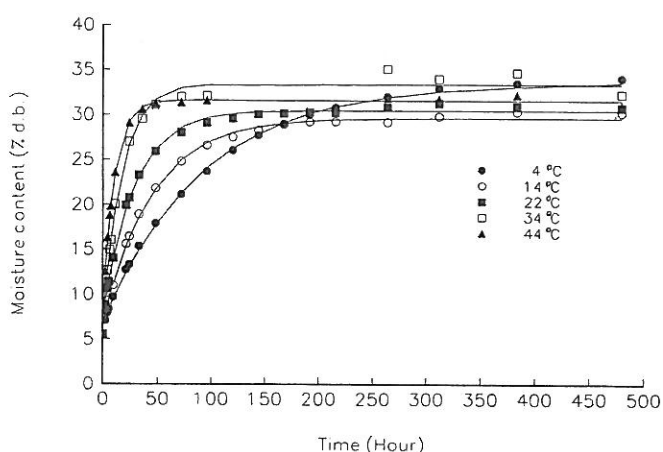


Fig. 5—Moisture sorption kinetics of freeze dried blueberries at 4, 14, 22, 34, and 44°C. The solid curves were predicted by Eq. (13).

function of moisture content by linear regression. The estimated  $\Delta H_0$  was 13.0 kJ/mol and  $M_0$  9.8% dry basis, with  $r^2 = 0.96$ .

The sorption heat curve was compared with other fruits reported by Ayranci et al. (1990). No negative  $\Delta H_{st}$  was observed in our system (Fig. 4) up to a moisture content of 35% d.b. When extrapolated above the tested moisture range, we could obtain negative  $\Delta H_{st}$ . As shown in Eq. (12), negative  $\Delta H_{st}$  results from the effects of cross over in the moisture sorption isotherms. This cross over may be a result of endothermic dissolution of sugar, which is more prominent at high moisture content than the exothermic sorption process in high sugar systems.

### Sorption kinetics

The sorption data for the freeze dried blueberries (Fig. 5) showed rates of moisture sorption were highest during initial stages and gradually decreased as moisture contents approached equilibrium. Initial sorption rates also increased with increasing temperature. The predictive model Eq. (13) was used to describe experimental data, and good agreement was observed. The minor deviation during initial moisture adsorption (Fig. 6) may be attributed to the limited moisture diffusion rates as compared to the rate of adsorption.

The rate constant,  $k$ , in Eq. (13) was temperature dependant and may be described by the Arrhenius equation:

$$k = k_0 e^{-\frac{E_a}{RT}} \quad (15)$$

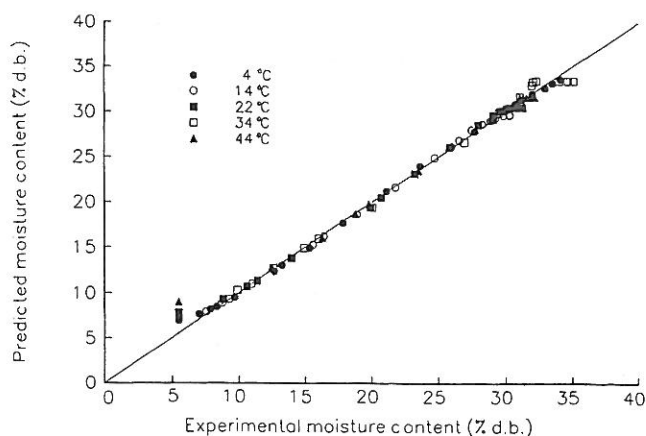


Fig. 6—Correlation between experimental and predicted moisture contents using Eq. (13).

where  $E_a$  is the activation energy,  $R$  the gas constant,  $T$  the absolute temperature and  $K_0$  a constant.  $\ln(k)$  was linearly related to  $1/T$  with  $r^2 = 0.994$ . The activation energy was estimated to be 38.6 kJ/mol. This value was comparable with the  $E_a$  published for hydration reactions of polished barley 39.9 kJ/mol (Mok et al., 1983) and vapor sorption of wheat flour 25.0–50.2 kJ/mol (Singh et al., 1981).

### CONCLUSION

SORPTION DATA indicated that the moisture sorption isotherms of freeze-dried blueberries followed the type III isotherm pattern common for high sugar foods. An inversion effect of temperature took place at  $a_w$  0.90, resulting in a cross-over in isotherms. The equilibrium moisture sorption isotherms were best described by the GAB model. The estimated isosteric heats indicated that affiliation between adsorbed water molecules and the adsorbent increased with decreasing moisture. Heat of vaporization of freeze dried blueberries ranged from 5.5 to 0.25 kJ/mol in excess of the value for free water, when moisture content increased from 8 to 36% moisture. The first order kinetic equation described well the moisture sorption kinetics in freeze-dried blueberries. The rate constant was temperature dependent as described by the Arrhenius equation, and the  $E_a$  was 38.6 kJ/mol.

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