Adsorption of moisture on dried juniper berries (*Juniperus communis* L.) at various temperatures and properties of sorbed water

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Summary

Moisture adsorption characteristics of juniper berries were studied using Brunauer-Emmet-Teller (BET), Guggenheim-Anderson-de Boer (GAB) and Peleg sorption equations. A reasonable goodness of fit to the experimental data over the entire range of water activity was obtained with the GAB equation. A negative temperature effect was observed, particularly at the curves measured at 10 °C. The monolayer moisture contents decreased with an increase in temperature of adsorption using the GAB model. Caurie´s equation was used to obtain data on non-freezing water, surface area of sorption, density of the sorbed water and the number of adsorbed monolayers. Values of these parameters decreased with an increase in the temperature. The linear form of Henderson isotherms intersected at moisture contents ranged from 158.5 $g.kg^{-1}$ to 114.8 $g.kg^{-1}$ (on dry weight basis) representing the transition from multilayer sorbed water to mobile water. Dehydrated juniper berries kept at ambient temperature would require less hydration to be enzymatically active (i.e. above 0.40 a_w). Isosteric heat of adsorption obtained by applying Clausius-Clapeyron equation decreased exponentially with the increasing moisture contents. Results showed that the refrigerated storage of dry juniper berries may be beneficial to preserve their nutritional value.

Keywords

juniper berries; moisture isotherm; sorbed water; isosteric heat

Knowledge on the moisture sorption contents and water activity relationships of most fruits plays a key role in their preservation. Such a relationship can be formulated by determining the moisture sorption isotherm, which can be used to define storage conditions and to determine the shelf life [1]. In this concern, another key property of food is the isosteric heat of sorption of the bound water, which can be used to estimate the energy requirements of drying and provides important information on the state of water in food products. The moisture contents (MC) of a material, at which the net isosteric heat of sorption reaches the heat of vaporization of pure water, is considered an indication of "bound water" existing in the foods [2, 3].

Common juniper, *Junipers communis* L. (*Cupressaceae*), is an aromatic shrub, whose berries are used in medicine or as a food ingredient. The physiological action of berries including diuretic activity [4], hypoglycemic activity [5], anti-inflammatory activity [6] and vasorelaxing action [7] has been documented. Juniper essential oils also exhibited antimicrobial activity against fungi, yeasts, bacteria [8, 9], viruses [10] and activity against insects [11]. The essential oils also showed significant antioxidant activities [12, 13]. The chemical substances probably responsible for antioxidant and antimicrobial properties of common juniper were identified in recent studies as α-pinene, β-myrcene and other terpenoids and others [5, 14].

In folk medicine, berries are mainly used as a drug with diuretic activity [15] or for treating respiratory illness [16]. The "pseudo-fruits" of *Juniperus* sp. are widely used as a food ingredient for gin aromatization [14] and, in some regions of Eastern Europe, for preparation of meat products (sausages) [17].

A variety of sorption models have been published in the literature [2]. For practical purposes, a sorption model should be as simple as possible and should utilize parameters with physical meanings [18]. Among them, Brunauer-Emmet-Teller (BET) [19] and Guggenheim-Anderson-de Boer (GAB) [20] are the equations most applied to various food products including fresh or processed

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fruits [3, 21–26]. The BET equation usually fits well the experimental data in the lower range of water activity (from 0.1 to 0.5) whereas the GAB equation is valid in the entire range of a_w. The Peleg's model is applicable in the wide range of water activity and was proposed as a standard model for cataloguing the sorption data of a large group of food and other biological materials. The BET and GAB models assume the existence of a certain MC level that corresponds to a monolayer being determined from the shape of the moisture sorption isotherm. Dehydrated foods are considered stable during storage when their MC is close to that of a monolayer [27]. Other properties of sorbed water such as non-freezing water, surface area of sorption or density of sorbed water on the active site of adsorbent can be useful to fully understand the moisture behaviour in the particular food under different storage conditions. Dry products may adsorb significant amounts of water when stored at a low temperature or at a high relative humidity.

The objective of the current study was to establish the equilibrium moisture contents and water activity data for common juniper berries at different temperatures, to analyse them with different models and to generate moisture sorption parameters, to determine other properties of the sorbed water as well as to evaluate the isosteric heat of sorption.

MATERIALS AND METHODS

Sample preparation

Pre-dried berries of *Juniperus communis* L. were purchased in local supply. The initial moisture contents of approximately 46.5 g.kg^{-1} (on dry weight basis) were determined after drying the samples at a temperature of 102 °C to give a constant weight using moisture analyzer MLB50-3 (Kern & Sohn, Balingen, Germany). The total amount of ash (88.0 g.kg^{-1}) and crude protein (34.2 g.kg^{-1}) were determined according to AOAC procedures [28, 29]. The amount of reducing sugars (248.4 g.kg^{-1}) was also determined [30] and the results were expressed on the dry weight basis. Prior to moisture adsorption study, the sample was dried to crispness over silica gel in a desiccator at room temperature. Thereafter, berries were manually grinded in a mortar to give a particle size of < 1.0 mm. The milled sample was subsequently dried over silica gel to weight constancy.

Determination of moisture adsorption isotherms

Moisture adsorption isotherms were determined gravimetrically by exposing berry samples at 10, 25 or 40 °C to different salt slurries (in the approximate range of 10% to 85%). Approximately 500 mg amounts of the dried sample were spread on glass vials and put into desiccators containing the salt slurries. The desiccators were kept in temperature-controlled chambers. The mass of each sample was measured periodically using an analytical balance (sensitivity, ± 0.001 g) until less than 1% weight change was found after two consecutive measurements. A glass vial containing toluene was placed into desiccators to prevent mould growth, which might take place in particular at high a_w. The adsorption isotherms were obtained by plotting MC on the dry basis versus a_w. Each isotherm was constructed using data of three replicates.

Mathematical modeling of adsorption data

The experimental adsorption data of samples at three different temperatures were fitted to three sorption equations with two parameters (BET), three parameters (GAB) and four parameters (Peleg) [31]. The BET and GAB equations were used in the following forms:

$$M = \frac{M_0 C a_w}{\left[(1-a_w)(1-a_w+Ca_w)\right]} \quad (1)$$

$$M = \frac{M_0 C K a_w}{\left[(1-Ka_w)(1-Ka+CKa_w)\right]} \quad (2)$$

where a_w is the water activity, M is the experimental moisture contents, M_0 is the monolayer moisture contents on dry basis (g.kg^{-1}), C is the constant related to the heat of sorption in BET equation or to monolayer properties of water in GAB equation, and K is the constant related to multilayer properties of water in GAB equation. In addition, the influence of temperature on GAB constants may be calculated using following equations:

$$M = M_0^{'} \exp\left(\frac{\Delta H}{RT}\right) \quad (3)$$

$$C = C_0 \exp\left(\frac{\Delta H_c}{RT}\right) \quad (4)$$

$$K = K_0 \exp\left(\frac{\Delta H_k}{RT}\right) \quad (5)$$

where C_0, K_0 and M'_0 are the pre-exponential factors, ΔH, ΔH_c and ΔH_k (kJ.mol^{-1}) are the molar sorption enthalpies of the monolayer, multilayer and bulk liquid, respectively. T is the absolute temperature (K) and R is the universal gas constant (kJ.mol^{-1}.K^{-1}).

The form of Peleg's model is:

$$M = k_1 a_w^{n_1} + k_2 a_w^{n_2} \quad (6)$$

where a_w is the water activity, k_1, k_2, n_1 and n_2 are constants ($n_1 < 1$ and $n_2 > 1$).

The parameters of the BET, GAB and Peleg's adsorption models were estimated from the experimental results using non-linear regression analysis (QCExpert v. 3.0, Trilobyte, Pardubice, Czech Republic). GAB and BET models were used to calculate the monolayer moisture contents.

The goodness of fit of the models was evaluated with the mean relative percentage deviation (E). The E values were calculated from the experimental (M_e) and predicted (M_p) moisture contents in a modified formula

$$E = \frac{100}{n}\sum\left|\frac{M_e - M_p}{M_e}\right| \tag{7}$$

A model is considered acceptable if the E values are below 10% [32].

Caurie's equation was applied in its linear form previously published by CAURIE [33]:

$$\ln\left(\frac{1}{M}\right) = -\ln(DM_0) + \frac{2D}{M_0}\ln\frac{1-a_w}{a_w} \tag{8}$$

where a_w is the water activity, M and M_0 are the experimental and monolayer moisture contents (g.kg^{-1}) on dry basis, respectively. D is the constant related to the density of sorbed water (g.ml^{-1}).

Monolayer values (M_0) were evaluated using Caurie's plot of ln $(1 - a_w)/a_w$ versus ln $(1/M)$ in the a_w range from 0.1 to 0.9. The surface area (A, m^2.g^{-1}) and the number of adsorbed monolayers (N) was calculated according to CAURIE [33] using the formulae:

$$A = \frac{54.45}{S} \tag{9}$$

$$N = \frac{2}{S} \tag{10}$$

where S is the slope of Caurie's plot.

The bound or non-freezing water was calculated from monolayer moisture contents and the number of adsorbed monolayers was derived from Caurie's plot. Similarly, the isotherms were analysed using the Henderson equation in the linear form [34]:

$$\log\left[-\ln(1-a_w)\right] = n\log M + \log k \tag{11}$$

where a_w is the water activity, M is the moisture contents (g.kg^{-1}) on dry basis, n and k are the constants depending on the material.

Using a_w values of up to 0.9, the constants of Caurie and Henderson equations were computed by the least squares method.

Net isosteric heat of sorption

The net isosteric heats of adsorption (q_{st}) were calculated from the following form of Clausius-Clapeyron equation.

$$q_{st} = -R\frac{\partial \ln a_w}{\partial(1/T)} \tag{12}$$

where a_w is the water activity, q_{st} is the net isosteric heat of sorption (kJ.mol^{-1}), R is the universal gas constant (kJ.mol^{-1}.K^{-1}) and T is the absolute temperature (K). The net isosteric heat of sorption was calculated from Eq. (12) by plotting the sorption isostere as ln(a_w) against $1/T$ for a specific moisture contents of the material and determining the slope which is equal $-q_{st}/R$. This procedure was repeated for different moisture contents and subsequently the dependence of q_{st} on the moisture contents was determined [3].

Statistical differences were evaluated by one-way ANOVA test on the probability level $p = 0.05$.

RESULTS AND DISCUSSION

Moisture adsorption isotherms

The results of the experimental measurements of the equilibrium MC of juniper berries at different temperatures are given in Fig. 1. The shape of the isotherms at all temperatures is characteristic for high-sugar-containing foods, which sorb relatively small amounts of water at low water activities and large amounts of water at high water activities, particularly above 0.70 a_w. Fig. 1 also shows the effect of temperature on the adsorption isotherms of juniper berries. It can be seen that equilibrium moisture contents were generally

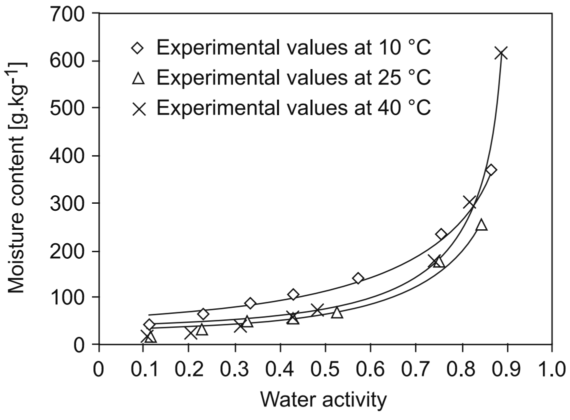

Fig. 1. Experimental equilibrium moisture contents for juniper berries at different temperatures, and moisture adsorption isotherms predicted by the GAB equation (curves).

higher at 10 °C for constant values of a_w in comparison with those measured at 25 °C and 40 °C ($p < 0.05$). The adsorption isotherm obtained at 40 °C visually crossed the isotherm representing at 25 °C at water activity about 0.60, however, the difference in MC was not significant ($p > 0.05$). This means that the temperature effect was negligible at a_w of up to 0.75. The negative temperature effect on equilibrium MC has been observed in many foods of high sugar contents [3, 24, 25]. This phenomenon can be related to the fact that sugars sorb relatively small amount of water compared to polymers (protein, starch) at low water activities, whereas high water uptake of sugars or other soluble components was found at high a_w values. Therefore, the negative temperature effect is eliminated at higher water activities, as documented for dried apricot [20], dried plantain slices [23] and dried mango and pineapple [25], with the crossings of the isotherms in the a_w range of 0.70–0.80. Different results were obtained by Alhamad and Hassan [26], who determined the crossing of the isotherm of date pastes at a_w of 0.45–0.55. The crossing of the isotherms was at 0.80 a_w in our study; however, the adsorption at 10 °C was still higher than at 25 °C ($p < 0.05$).

Moisture adsorption models

Three isotherm equations (GAB, BET and Peleg) were used for establishing the degree of fit to experimental data using non-linear regression analysis. Estimated parameters for these models are presented in Tab. 1. GAB and BET equations exhibited the similar $\%E$ values, the residual sum of squares being better for the BET model. It was previously published that equilibrium MC for low water activities showed low deviations (using both BET and GAB models), whereas higher deviations were obtained for high values of water activities using the GAB model [25]. Although the GAB model gave a goodness of fit similar to that of the BET model; it was applicable in the entire range of water activities. The GAB equation can be recommended for predicting the moisture adsorption of juniper berries in the temperature range from 10 °C to 40 °C. Peleg's equation described well only the adsorption data at 25 °C ($E = 9.5$).

Properties of sorbed water

The physical state of water in foods determines its spoilage. It is therefore important to generate information related to various aspects of the sorbed water. Among them, a monolayer moisture concept is widely used by food technologists because these values correlate with stability of foods during storage [27]. The monolayers MC of juniper berries at each temperature were derived from GAB and BET equations and are presented in Tab. 1. The monolayer moistures were similar for both GAB and BET models. The values of parameters obtained from the GAB model suggest that the monolayer moisture contents decreased with an increase in temperature of adsorption, as has been observed in other food systems [35–37], and were similar to those obtained for various dried fruits [18, 21, 25]. The monolayer MC derived from the BET isotherm did not show any temperature dependence. The estimated ΔH_c value had a large negative value (Tab. 2), similarly to other dried fruits such as grape, apricot and apple [3]. The higher heats of sorption (ΔH_c) indicate a stronger interaction between water vapour and

Tab. 1. Estimated parameters for selected models of adsorption isotherm equations for juniper berries at different temperatures.

Sorption model	Temperature [°C]	Parameters							
		M_0	C	K	k_1	n_1	k_2	n_2	E
BET	10	64.1	11.1	–	–	–	–	–	5.7
	25	38.8	5.4	–	–	–	–	–	10.8
	40	57.6	1.8	–	–	–	–	–	10.3
Peleg	10	–	–	–	187.5	0.7	520.9	6.9	14.3
	25	–	–	–	9.2	0.8	39.2	4.9	9.5
	40	–	–	–	20.9	1.5	223.6	14.1	16.8
GAB	10	70.2	9.4	0.9	–	–	–	–	4.6
	25	61.5	1.9	0.9	–	–	–	–	10.8
	40	43.8	3.0	1.1	–	–	–	–	11.3

M_0 – monolayer moisture contents [g.kg^{-1}] on dry weight basis, C, K, k_1, k_2, n_1, n_2 – constants of sorption models, E – the mean relative percentage deviation.

Tab. 2. Temperature dependencies of GAB constants for juniper berries.

$M_0(T)$			C(T)			K(T)		
M'_0	ΔH	R^2	C_0	ΔH_c	R^2	K_0	ΔH_k	R^2
0.54	11.5	0.926	4.5×10^{-5}	-28.6	0.512	2.8	-2.7	0.695

M'_0, C_0, K_0 – pre-exponential factors, ΔH, ΔH_c, ΔH_k – heats of sorption [kJ.mol^{-1}], R^2 – coefficient of determination, T – absolute temperature [K].

Tab. 3. Properties of the sorbed water of juniper berries at different temperatures.

Temperature [°C]	D [g.ml^{-1}]	N	A [m^2.g^{-1}]	Bound or non-freezable water [%]	Henderson plot	
					Breakpoint	Moisture [g.kg^{-1}]
10	1.3	3.6	97.7	16.9	1.2	158.5
25	1.2	2.7	72.8	8.3	1.1	114.8
40	1.3	2.2	60.7	6.6	1.1	125.9

D – density of sorbed water, N – number of adsorbed monolayers, A – surface area of sorption.

primary sorption sites of juniper berry samples. The estimated ΔH_k value, which was found to be negative, corresponded to the heat of sorption of the multilayer slightly greater than the heat of condensation of water.

Caurie's equation provided information on various aspects of bound water in juniper berries such as its density, its relation to the surface area of the adsorbent and the number of adsorbed layers. The results are summarized in Tab. 3. The bound or non-freezing water is adsorbed onto the exposed surface of the adsorbent (berries) in different layers yielding particular surface area. It is evident that non-freezing water, surface area of sorption and the number of adsorbed monolayers of juniper berry samples decreased with the increase in temperature, as was expected. The decrease was pronounced for temperatures ranging from 10 °C to 25 °C, whereas further increase in temperature lead to slightly decreased values of these parameters. In addition, density of the bound water decreased from 1.3 g.ml^{-1} at 10 °C and to 1.2 g.ml^{-1} at 25 °C, followed by an increase to 1.3 g.ml^{-1} at 40 °C for juniper berries. It has been well established that the amount of adsorbed water depends on the number of hydrophilic group capable of binding water through hydrogen bond formation (carbonyl, amino and hydroxyl groups). Our preliminary analysis showed that juniper berries were relatively rich in reducing sugars and Hyaouni et al.[13] found high contents of polyphenolic compounds in juniper "pseudo-fruits". It could, therefore, be implied that the number of active polar groups in juniper berries decreased as a result of crosslinking and interactions of proteins, polyphenols or sugars at higher temperatures. The increase in the density of the sorbed water in the temperature range from 25 °C to 40 °C could be explained by the fact that density of water is also determined by surface forces acting between the molecules. Closer intermolecular distances and hence greater forces acting between them result in a higher density [33]. The density of the sorbed water better corresponded to the BET monolayer values.

As was expected, more than one straight line could be fitted applying Henderson's equation to the experimental data. According to the hypothesis of Rockland [38], the Henderson isotherm should be, theoretically, divided into three local isotherms corresponding to monolayer, multilayer and free water physical state, respectively. Thus the breaks in the curves indicate changes in the type of water binding. In our study, only one intersection point has been observed (Fig. 2) representing the transition from the multilayer state of the sorbed water to mobile water, as was similarly reported for other food items [34, 39, 40]. For juniper berries, the

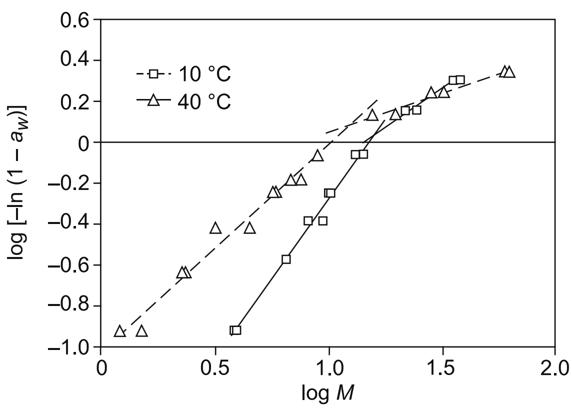

Fig. 2. The predicted Henderson plots for juniper berries at 10 °C and 40 °C.

isotherms intersected at moisture contents ranging from 158.4 g.kg-1 to 114.8 g.kg-1 (on dry weight basis) at 10 °C and 40 °C, respectively. These moisture contents represent the highest or critical values at which the water molecules are bound to other chemical groups. At higher values of MC, water is considered as unbound or free, found in interstitial pores of the food sample [38]. Extrapolated from Fig. 1, the corresponding water activities were in the range from 0.63 to 0.67 at temperatures ranging from 10 °C to 40 °C. At higher water activities or higher relative humidity of the surrounding air, juniper berries may be, theoretically, vulnerable to fungal spoilage. Despite of the antimicrobial properties of essential oils or other components, a high incidence of toxigenic fungi including species from *Aspergillus* genus was determined in fresh and dry medicinal plants [41]. Enzymatic action has also influence on the stability of the dry plant materials. For instance, the study of Raina et al. [42] indicated that prolonged storage of saffron affected the pigments and flavour concentration to a great extent and suggested that the minimal MC to reduce deterioration should be 50.0 g.kg-1. Generally, enzymatic reactions leading to the lost of beneficial properties during storage may be active $a_w > 0.40$ [27]. Setting the water activity of 0.40, safe MC for enzymatic stability can be read from Fig. 1: 94.9 g.kg-1 at 10 °C, 52.6 g.kg-1 at 25 °C and 50.6 g.kg-1 (on dry weight basis) at 40 °C. Since initial MC of dried juniper berries used in this study was 46.5 g.kg-1 (on dry basis), which is close to monolayer values, dehydrated pseudo-fruits kept at ambient temperature would require less hydration to be enzymatically active. This result shows the benefit of refrigerated storage of dehydrated berries as was also documented for rosehip fruits [18].

Net isosteric heat of sorption

Values of the net isosteric heat of sorption were calculated from the equilibrium data at different temperatures using Eq. (12) and the dependence of q_{st} on the moisture contents was plotted (Fig. 3). The curve shows that the heat of adsorption decreased with an increase in MC, initially rapidly up to 150.0 g.kg-1 (on the dry basis) and later slowly, as observed in many other food systems [1, 23, 43, 44]. The high q_{st} value indicates a strong link between the adsorbed water and the adsorbent (juniper berries). Starting from MC > 150.0 g.kg-1 on dry weight basis, q_{st} decreased gradually approaching zero, which means that the isosteric heat was equal to the heat of condensation of water. The corresponding moisture contents can be considered as the limit of "bound" water [45]. The

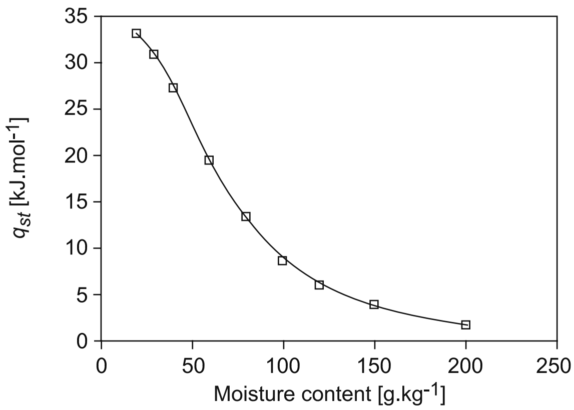

Fig. 3. Net isosteric heat of adsorption of juniper berries at temperatures ranging from 10 °C to 40 °C.

equilibrium MC of juniper berries, at which isosteric heat of adsorption was close to zero (i.e. 150.0 g.kg-1 on dry weight basis) corresponded to the safe moisture contents derived from Henderson's equation.

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