Moisture sorption characteristics of *wadi*, a legume-based traditional condiment

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Abstract Moisture sorption characteristics of *wadi*, a condiment in the Indian Subcontinent, were investigated at temperature of 15–45 °C and water activities (a_w) of 0.11–0.97. The sorption isotherms were sigmoidal. Nine different mathematical models were found to effectively describe the moisture sorption data on the basis of regression analyses and goodness of fit. Each model was statistically evaluated by means of percent root mean square and coefficient of determination. The GAB model gave the best fit in the entire range of a_w . Temperature dependence of the GAB constants and good fit were determined. Using the Caurie's model, the properties of sorbed water, such as monolayer moisture content, number of adsorbed monolayers, density of sorbed water, bound water content and surface area of adsorption, which decreased with an increase in temperature, were calculated. The monolayer moisture content ranged from 102.7 to 128.7 g/kg solids in *wadi*. The optimum storage temperature and a_w for *wadi* were 15–25 °C and <0.7, respectively.

Keywords *Wadi* · Legume · Moisture sorption · Modelling · Adsorption isotherm

Wadi is a legume-based ready-to-fry product, popularly consumed in India, Pakistan and Bangladesh. To prepare *wadi*, blackgram (*Phaseolus mungo* L.) dal (dehusked split beans) is washed and soaked for about 10–12 h at room temperature (20–25 °C), soaked water is drained off, and

the dal is ground to a smooth thick batter. The batter is left for natural fermentation at room temperature for 18–24 h, and then hand beaten for proper aeration. Finally, the batter is shaped into balls or cones and sun-dried for 4–8 days over bamboo or palm mats, smeared with oil. The product is marketed as hollow, brittle cones, which are fried briefly in a small amount of oil and used as an adjunct to cooked vegetables.

Water is one of the most important components determining the physical, chemical and sensory properties of foods (Fennema 1996). Water activity (a_w), which is defined as the ratio of the vapour pressure of water in equilibrium with a material to the vapour pressure of pure water at the same temperature, is used to express the amount of water available in a food system for microbial growth and biochemical reactions (Fennema 1996). An important aspect of a_w concept is the moisture sorption isotherm which is a graphical representation of the relationship between moisture content of a product and a_w at a particular temperature. Moisture sorption isotherms give vital information about sorption behaviour and interaction of various components of a food with water (Kaymak-Ertekin and Gedik 2004). It is also an important property of dried products, like *wadi*, as it helps to determine the degree of drying required to obtain a stable product (Lerici et al. 1985). Temperature has an important influence on the moisture sorption characteristic of a product (Manuel and Sereno 1993). Hence, information obtained from moisture sorption data of a food is important for the prediction of its quality and shelf-life during its packaging and storage.

Wadi is shelf-stable to a certain extent; so its cottage-scale production is restricted during the winter seasons only when the ambient temperature is 10–25 °C. However, its storage becomes problematic during the monsoons when relative humidity of the atmosphere is high; it absorbs

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moisture from the air and becomes mouldy, thus making it unfit for consumption. Besides, improper packaging and storage conditions bring about undesirable changes in the sensory qualities of *wadi*. So, the moisture sorption study of *wadi* is important in order to understand its sorption mechanics and exploit this information to improve its quality and shelf-life by applying hurdle technology.

The objectives of this research were to generate data on the sorption characteristics of *wadi* at four different temperatures (15–45 °C) to determine the water-binding mechanisms and suggest suitable mathematical models to describe the isotherms.

Materials and methods

Sampling Unpackaged locally marketed samples of black-gram *wadi* were collected in sterile sampling bags and analysed within a week of collection.

Determination of equilibrium moisture content The sorption apparatus used by Wolf et al. (1985) was slightly modified to study the sorption characteristics of *wadi* using static gravimetric method. Wide-mouthed glass bottles (1 l) with air-tight lids were used as sorbostats. Saturated solutions of LiCl, MgCl₂, K₂CO₃, Mg(NO₃)₂, NaNO₂, NaCl, (NH₄)₂SO₄, KCl, BaCl₂ and K₂SO₄, providing a_w 0.11–0.97, were prepared. Each salt solution, poured into a separate sorbostat, acted as the source of sorbate. The salts were dissolved in distilled water a week ahead, and occasionally mixed daily in order to bring stability. Approximately 2 g finely powdered *wadi* was weighed into tared weighing bottles (10 ml) in triplicate and placed over glass supports inside the sorbostats. Before putting the samples, the weighing bottles were allowed to come to equilibrium with the surrounding environment inside the sorbostat for 24 h. Approximately 5 mg potassium sorbate (HiMedia RM1311) was added to each sample to prevent microbial growth. The sorbostats were tightly closed and placed inside incubators at 15, 25, 35 and 45 °C. The samples were weighed periodically until (20–30 days) the difference between the two successive weighings was ≤1 mg. The samples were exposed for less than 10 s while weighing them to avoid absorption of moisture from the surrounding which can lead to erroneous results. The moisture content in the samples was determined gravimetrically (Helrich 1990). All the chemicals used were of analytical grade.

Sorption models There are several mathematical models to describe the moisture sorption isotherms of foods. In this study, nine moisture sorption models were selected to fit the experimental moisture sorption data of *wadi*

(Table 1). The GAB (Guggenheim-Anderson-de Boer) and modified Mizrahi models have three parameters, while the rest have two parameters each. The most commonly used 2-parameter model is BET (Brunauer-Emmett-Teller), but its applicability is limited only to the lower (0.1–0.5) a_w . On the other hand, the GAB model is the most successful 3-parameter model applicable even at a higher (0.1–0.9) a_w , and has been recommended by the European Project Group COST '90 on physical properties of foods (Wolf et al. 1985). There are ample reports on the successful application of the GAB model in moisture sorption studies for many food materials (Menkov and Durakova 2005). All the equations were converted into linear form, and the moisture sorption data were analysed and fitted to different equations either in the whole range of isotherm or part of it. The goodness of fit for each equation was evaluated in terms of coefficient of determination (R^2), obtained by plotting the experimental (M_{exp}) and calculated (M_{cal}) sorption moisture and by percent root mean square (%RMS):

$$\%RMS = \sqrt{\frac{1}{n} \sum_{i=1}^n \left(\frac{M_{exp} - M_{cal}}{M_{exp}} \right)^2} \times 100 \quad (1)$$

where, n denotes the number of observations.

The appropriate constants of the equations were determined by regression analysis using MS Excel software.

Properties of sorbed water The different properties of sorbed water that were determined are:

- Number of adsorbed monolayers (N), which was calculated using the formula (Caurie 1981):

$$N = \frac{M_c}{C_c} \quad (2)$$

where, M_c was the monolayer moisture content (g/kg solids), calculated from Caurie's model, and C_c was the Caurie's constant.

- Bound water content was expressed as $M_c \times N$. (3)
- Assuming Caurie's constant (C_c) to be equivalent to the density of adsorbed water in the monolayer, the surface area of adsorption (A) was calculated using the formula (Sahu and Jha 2008):

$$A = \frac{M_c}{C_c \times d \times 10^8} \quad (4)$$

where, d was the diameter of a water molecule (3.673×10^{-10} m).

Table 1 Sorption isotherm models for fitting experimental data

Model	Equation	Constants	Reference
Smith	$M = b - aa_w$	a, b	Chirife and Iglesias (1978)
Bradley	$\ln\left(\frac{1}{a_w}\right) = ab^M$	a, b	Chirife and Iglesias (1978)
Iglesias and Chirife	$\ln\left(M + \sqrt{M^2 + M_{0.5}^2}\right) = a + ba_w$	a, b	Chirife and Iglesias (1978)
BET	$M = \frac{M_b C_b}{(1-a_w)(1+(C_b-1)a_w)}$	M_b, C_b	Caurie et al. (1976)
Freundlich	$M = b(a_w)^{\frac{1}{a}}$	a, b	Ertugay et al. (2000)
Oswin	$M = a\left(\frac{a_w}{1-a_w}\right)^b$	a, b	Chirife and Iglesias (1978)
Caurie	$\frac{1}{M} = \frac{1}{C_c M_c} \left(\frac{1-a_w}{a_w}\right)^{\frac{2C_c}{M_c}}$	M_c, C_c	Caurie (1981)
Modified Mizrahi	$M = \frac{a+a_w(ca_w+b)}{a_w-1}$	a, b, c	Rao et al. (2006)
GAB	$\frac{M}{M_g} = \frac{C_g ka_w}{(1-ka_w)(1-ka_w+C_g ka_w)}$	M_g, C_g, k	Bizot (1983)

Results and discussion

Sorption isotherms The moisture isotherms of *wadi* were sigmoid and described as type II (Fig. 1). Such isotherms are typical of foods rich in carbohydrates (Bolin 1980). Marketed *wadi* had moisture content of 147 g/kg. *Wadi* contained (per kg dry weight): 746 g carbohydrate, 210 g protein, 19 g crude fat, 34 g total nitrogen, 23 g soluble nitrogen, 10 g titratable acid (as lactic acid), 25 g free fatty acid (as linoleic acid) and 25 g ash. The coefficient of variation was approximately 3% among the triplicate sets.

Fig. 1 Moisture sorption isotherms of *wadi* at different temperatures (lines drawn are best eyefit). Each observation is a mean of three replicate experiments

Since *wadi* contained a high level of carbohydrate, it exhibited a typical S-shaped isotherm as expected and resembled the isotherms of some food products, like papaya and ginger powder (Alakali and Satimehin 2009; Vega-Gálvez et al. 2008). Moisture sorption isotherms of several sugar-rich foods such as dried figs, prunes, date pastes, cake mixes have also been described by other workers (Leiras and Iglesias 1991).

The adsorption isotherms showed three regions as reported for other food products, such as *dudh churpi*, milk *burfi*, *sandesh* and ginger powder (Alakali and Satimehin 2009; Caurie 1981; Hossain et al. 2002; Vega-Gálvez et al. 2008). Region I corresponding to a_w 0–0.11 represents the monolayer moisture content which is strongly bound to the polar groups of the food components. Region II, also called the multimolecular phase, corresponds to a_w 0.11–0.65 for 25, 35 and 45 °C. However, for 15 °C, this region corresponds to a_w 0.11–0.82. In these regions, the slope of the isotherm is gentle as the multimolecular water is converting into free water and becomes available for chemical reactions. This is the region of adsorption of additional layers over the already existing monolayer. Above a_w 0.82 there is a steep rise in the isotherm, especially for 45 °C. This is Region III in which water is in the free state and condensed into the pores of the material, gaps and capillaries (Kinsella and Fox 1987). The steep rise in the isotherms suggests rapid changes in the product quality, because the soluble materials of the product begin to dissolve in the condensed water of this region (Chetana et al. 2005). A series of chemical reactions which begins in region II ultimately leads to the deterioration of food quality, and the beginning of steep rise in moisture indicates the onset of physicochemical deterioration (Labuza and Contreras-Medellin 1981).

The equilibrium moisture content increased with the increase in a_w at a particular temperature. This may be due to an increase in the vapour pressure of water within the foods with an increase in the surrounding vapour pressure (Shivhare et al. 2004). The equilibrium moisture content at a particular a_w decreased with an increase in temperature at $a_w < 0.70$, as was observed in the case of oolong tea (Chen and Weng 2008). For instance at a_w 0.32, the equilibrium moisture content of *wadi* decreased from 144.5 g/kg solids at 15 °C to 133.3, 127.0 and 124.4 g/kg solids at 25, 35 and 45 °C, respectively. Increase in temperature activates the water molecules causing them to break away from their binding sites leading to a decrease in the water sorption with an increase in temperature (McMinn and Magee 2003). This phenomenon can also be explained from the aspect of kinetic molecular theory which says that samples adsorb more water at low temperatures than at higher temperatures as water molecules at lower temperatures have a lower kinetic energy which is not enough to overcome the corresponding sorption energy. However, in the present study, at $a_w > 0.70$, the equilibrium moisture content increased with an increase in temperature, possibly due to the solubilization of sugars at higher temperatures, as reported in the case of date pastes where the equilibrium moisture content increased with an increase in temperature for a_w 0.45–0.55 (Alhamdan and Hassan 1999).

The sorption isotherms at $a_w > 0.68$ intersect due to an increase in the moisture content for these water activities, mainly due to rise in the sugar solubility of *wadi* as observed in some foods, like papaya (Vega-Gálvez et al. 2008). This phenomenon is found to occur in foods rich in sugar (Kaymak-Ertekin and Gedik 2004) and *wadi*, being rich in carbohydrates, shows this phenomenon. Another reason for the intersection of the isotherms may be due to increased enzymatic activity in the multimolecular phase and intermixing of water molecules, carbohydrates and proteins (Erbaş et al. 2005). As the temperature increased, the isotherms tended to shift towards the right hand (Fig. 1). For a given moisture content, a_w increased with an increase in temperature. However, this effect diminished above a_w 0.68.

Evaluation of sorption models The moisture sorption data obtained for *wadi* at 15, 25, 35 and 45 °C were fitted to nine different mathematical models in the a_w range of 0.11–0.97 (Table 2). Among these, Oswin and modified Mizrahi models exhibited a good fit over the entire a_w range at all the four test temperatures, as these models were found with lower %RMS values and higher R^2 values. Smith, Bradley, Iglesias and Chirife, Caurie, and GAB models showed good fit in a_w 0.11–0.91. Among these five models, the GAB model yielded the lowest %

RMS values and the corresponding R^2 values were also high. Hence, the GAB model provided the best fit to the experimental data. This is in agreement with other findings (Chetana et al. 2005; Menkov and Durakova 2005), where the GAB model, among others, gave a very good fit to the experimental data for milk *burfi* and bean flour. From the GAB model, the sorption constants, C_g and k were evaluated (Table 2). Both the constants were temperature dependent.

The moisture adsorbed in the monolayers or the monolayer moisture content (M_g) was also evaluated from the GAB equation and it ranged from 93.1 to 139.0 g/kg solids. The M_g values decreased with an increase in temperature, while C_g and k increased with an increase in temperature.

The temperature dependence of M_g and both the sorption constants (C_g and k) were evaluated, and the following expressions with high 'r' values indicating excellent fit were obtained

$$M_g(T) = 0.2131e^{\frac{9933.411}{RT}}; \quad r = 0.9825 \quad (5)$$

$$C_g(T) = 2207.0527e^{\frac{-8299.352}{RT}}; \quad r = 0.9785 \quad (6)$$

$$k(T) = 213.6732e^{\frac{-14783.974}{RT}}; \quad r = 0.9792 \quad (7)$$

After the GAB equation, the next best fit for the experimental sorption data was obtained from the Caurie's equation. This equation was used to determine the various properties of sorbed water such as the number of adsorbed monolayers, monolayer moisture content, density of sorbed water, bound water content and surface area of adsorption (Table 3). A relationship of the Caurie's constants with temperature of sorption was established as:

$$M_c(T) = 1.0577e^{\frac{5994.555}{RT}}; \quad r = 0.9901 \quad (8)$$

$$C_c(T) = 5.8734e^{\frac{-3640.216}{RT}}; \quad r = 0.9343 \quad (9)$$

The BET and Freundlich models were applicable in the a_w range of 0.11–0.55. The BET equation has two parameters, M_b and C_b , with meanings similar to the GAB parameters. The BET monolayer values were less than those of the GAB monolayers. However, the monolayer moisture content obtained from the Caurie's model was the highest.

The adsorbent capacity and the intensity of adsorption can be measured from the values of the intercept ($\ln a$) and the slope ($1/b$) in the Freundlich isotherm (Lowell and

Table 2 Sorption ranges, constants, coefficient of determination (R^2) and %RMS values of different sorption models at different temperatures for *wadi*

Isotherm model	Temp. (°C)	a_w	Constants of linear fitting			R^2	%RMS
			M_b	C_b			
BET	15	0.11–0.55	8.5339	–25.6353		0.9912	11.7271
	25		7.6293	–32.1044		0.9752	14.2602
	35		7.6182	–38.7596		0.9777	10.0345
	45		7.4478	–48.6007		0.9899	7.4121
Freundlich	15	0.11–0.55	a	b		0.9757	2.0980
	25		19.1140	4.2968		0.9862	2.3969
	35		19.9802	3.0466		0.9870	2.3765
	45		19.3647	2.9081		0.9949	1.3618
Bradley	15	0.11–0.91	a	b		0.8809	10.0421
	25		10.9400	0.8464		0.9927	2.9590
	35		16.9447	0.8138		0.9622	5.9072
	45		9.2089	0.8452		0.9603	10.5986
Iglesias and Chirife	15	0.11–0.91	a	b		0.8953	8.4029
	25		5.2693	0.8773		0.9459	7.4594
	35		2.3256	0.9920		0.9578	7.6839
	45		2.1563	1.1834		0.9658	7.1206
Smith	15	0.11–0.91	a	b		0.9011	7.5665
	25		2.0483	1.3726		0.9782	7.1873
	35		1.9860	1.4851		0.9450	8.5671
	45		11.0758	7.3255		0.9850	4.8658
GAB	15	0.11–0.91	M_g	C_g	k	0.9904	2.9880
	25		13.9046	67.1061	0.4238	0.9795	3.8975
	35		11.3389	79.7294	0.5818	0.9676	4.6069
	45		10.2190	87.6049	0.6699	0.9866	2.6705
Caurie	15	0.11–0.91	M_c	C_c		0.9175	7.4843
	25		9.3113	93.5776	0.7732	0.9895	3.3250
	35		12.8746	1.3110		0.9701	6.3072
	45		12.3914	1.2546		0.9727	6.3175
Modified Mizrahi	15	0.11–0.97	a	b	c	0.9883	6.8861
	25		–10.4289	–0.8225	10.2716	0.9954	4.1670
	35		–8.3433	–6.3640	14.4705	0.9895	11.6313
	45		–7.6031	–7.4073	14.4178	0.9833	4.6184
Oswin	15	0.11–0.97	a	b		0.9048	9.9293
	25		–7.5867	–5.2023	11.1122	0.9899	2.9674
	35		16.8788	0.2037		0.9812	5.1246
	45		15.5443	0.2123		0.9727	8.6655
	15		15.4026	0.2628			
	25		15.4280	0.3004			
	35						
	45						

Shields 1991). In the present study, it was found that the sorption capacity of the adsorbent decreased with an increase in temperature (Table 3), which accounts for the

reduced monolayer moisture values at higher temperatures. This finding is consistent with the results obtained from BET, GAB and Caurie's models.

Table 3 Properties of sorbed water in *wadi*

Temp (°C)	Number of adsorbed monolayers	Monolayer moisture content (g/kg solids)	Density of sorbed water (g/mL)	Surface area of adsorption (m ² /g)	Bound water (%)
15	9.8204	128.746	1.3110	267.3684	126.4337
25	9.2781	121.024	1.3044	252.6037	112.2873
35	7.6108	108.271	1.4226	207.2089	82.4029
45	6.8636	102.652	1.4956	186.8663	70.4562

Properties of sorbed water Properties of the water sorbed by *wadi* were calculated according to the Caurie's model (Table 3). The monolayer moisture content (M_c), number of adsorbed monolayers, bound water content and surface area of adsorption decreased with an increase in temperature.

The M_c signifies the amount of water strongly binding to the specific sorption sites. It is at these values the food is most stable. The M_c values for *wadi* decreased from 128.7 g/kg solids at 15 °C to 121.0, 108.3 and 102.7 g/kg solids at 25, 35 and 45 °C, respectively. This may be due to the loss of some moisture sorption sites as a result of some physical and chemical changes on temperature rise (Mazza 1982). In the region of lower a_w , the proteins and carbohydrates in *wadi* are the preferred sorption sites at lower temperatures than at higher temperatures, because these food components have polar hydrophilic groups with high water-binding capacity in them. Water also forms hydrogen bonds with some specific groups leading to hydrophobic hydration (Kinsella and Fox 1987), but with an increase in temperature there is a decrease in the number of hydrophilic groups and the hydrophobic hydration of the biopolymers also begins to break at higher temperatures. This explains reduced monolayers at higher temperatures.

Dried foods, like *wadi*, have a greater shelf-life if they are kept at low moisture contents, because under such conditions the rate of chemical reactions within the food is greatly reduced (Bolin 1980). They feature a critical moisture content called the monolayer value at which they show maximum physical and chemical stability (Iglesias and Chirife 1976). When moisture content in the food crosses this monolayer value, it leads to food spoilage (Labuza 1968). In the present study, the safe storage moisture content values (M_c) predicted by the Caurie's model was between 102.7 and 128.7 g/kg solids. The initial moisture level of *wadi* was 153.9 g/kg solids which is higher than the calculated monolayer values. This explains why *wadi* is shelf-stable only to a certain extent.

Surface area of the globular proteins also shows a direct relation with the monolayer moisture content. A larger surface area means greater number of polar groups that are exposed, resulting in an increase in water sorption. With an increase in temperature the surface area decreases (Zettlemoyer 1968) as

reported in some products, like peeled ginger powder (Alakali and Satimehin 2009) and *dudh churpi* (Hossain et al. 2002), where this value decreased from 203.8 to 151.1 m²/g in the former and from 239.7 to 214.1 m²/g in the latter as the temperature increased from 15 to 45 °C. Bound water content of *wadi*, which is directly related to monolayer moisture content, was found to decrease from 126.4 to 70.4 g/kg solids with the rise in temperature from 15 °C to 45 °C.

Density of sorbed water increased from 1.30 g/ml at 25 °C to 1.42 and 1.49 g/ml at 35 °C and 45 °C, respectively. Possibly at higher temperatures the rate of chemical reactions at the multimolar phase and capillary water condensation in region III also increased which may have led to the increased solubilization of sugars present in considerable amounts in *wadi*. This intermingling of the sugars with the water molecules may have caused an increase in the density of the sorbed water as adding sugar or salt to water increases its density.

Conclusion

The present study was conducted with an intention to understand the sorption characteristics of *wadi* at 15–45 °C and describe the various properties of sorbed water such as monolayer moisture content, number of adsorbed monolayers, density of sorbed water, bound water content and surface area of adsorption. We conclude that the optimum storage temperature for *wadi* lies at 15–25 °C and $a_w < 0.7$. This study can serve as the possible explanation for the onset of various microbiological events in *wadi* that may lead to its spoilage. For example, the present study reveals that the amount of bound water was more at lower temperatures and it decreased with an increase in temperature. This water is not available for microorganisms; so the shelf-life of *wadi* at lower temperatures will be more than at higher temperatures. Besides, monolayer moisture content is an indication of the stability of a food, and from this study we obtained the optimum moisture levels at the four test temperatures.

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