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Research Article

Mathematical Modeling of Moisture Sorption Isotherms and Determination of Isosteric Heats of Sorption of *Ziziphus* Leaves

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Desorption and adsorption equilibrium moisture isotherms of *Ziziphus spina-christi* leaves were determined using the gravimetric-static method at 30, 40, and 50°C for water activity (a_w) ranging from 0.057 to 0.898. At a given a_w, the results show that the moisture content decreases with increasing temperature. A hysteresis effect was observed. The experimental data of sorption were fitted by eight models (GAB, BET, Henderson-Thompson, modified-Chung Pfost, Halsey, Oswin, Peleg, and Adam and Shove). After evaluating the models according to several criteria, the Peleg and Oswin models were found to be the most suitable for describing the sorption curves. The net isosteric heats of desorption and adsorption of *Ziziphus spina-christi* leaves were calculated by applying the Clausius-Clapeyron equation to the sorption isotherms and an expression for predicting these thermodynamic properties was given.

1. Introduction

Ziziphus spina-christi (L.) Desf. is a multipurpose tree species belonging to the botanical family Rhamnaceae [1]; it grows wild in Asia and tropical Africa. The plant is originally of the Middle East and spread to Saharan Oases across Africa into the Sahel [2]. The tree and its various parts have been an important source for pharmaceuticals since antiquity [3]. *Ziziphus spina-christi* has been used in folk medicine as a demulcent, depurative, anodyne, emollient, stomachic, and astringent and as a mouth wash [4, 5]; the powdered leaves of the plant are used as a shampoo and are believed to leave the hair clean and lustrous [6–11].

Some useful phytochemicals that include flavonoids, lipids, terpenes, alkaloids, steroids, saponins, and carbohydrates have been isolated from the plant [12–20].

Moreover, aqueous extract of various parts of the plant have showed analgesic, sedative, antinociceptive, anti-inflammatory, and antimicrobial effects [21–25].

People from Algerian Sahara use different parts from *Ziziphus spina-christi* to treat several ailments, such as abdominal pains, diarrhea, lips herpes, fever, diabetes, sores, and burns [26–28].

Since vegetables are highly perishable products, the quality is affected by postharvest handling, transportation, storage, and marketing. The improper handling, storage, and transportation may result in decay and production of microorganisms [29].

Water activity has long been considered as one of the most important quality factors especially for long-term storage;it affects the shelf-life, safety, texture, flavor, and smell of foods. Water activity is defined as the ratio of the partial pressure of water over the wet solid system to the equilibrium vapor pressure of water at the same temperature [30].

The knowledge and understanding of sorption isotherms for foods are of particular importance especially in the determination of a drying end point which ensures economic viability and microbiological safety.

Several empirical and semiempirical equations have been proposed for the correlation of the equilibrium moisture content with the water activity of food products. Among them the GAB equation has been applied successfully to various foods [31].

The object of this research is to obtain experimental equilibrium moisture isotherms for *Ziziphus spina-christi* leaves at 30, 40, and 50°C, determine the most suitable model describing the isotherms, determine hysteresis phenomena, and calculate the isosteric heat as a function of moisture content.

Table 1: Selected salts used for preparing saturated salt solutions and their corresponding water activities.

Salt	Water activity		
	30°C	40°C	50°C
KOH	0.0738	0.0626	0.0572
$MgCl_2$	0.3238	0.3159	0.3054
K_2CO_3	0.4317	0.423	0.4091
$NaNO_3$	0.7275	0.71	0.6904
KCl	0.8362	0.8232	0.812
$BaCl_2$	0.898	0.891	0.8823

2. Materials and Methods

2.1. Description of Experimental Procedure. Ziziphus spina-christi leaves for experiments were obtained from Bechar region of South-Western Algeria. Harvest was done between April and June 2011. Discolored and dried out samples were discarded.

2.2. Determination of Sorption Isotherms. The equilibrium moisture content of *Ziziphus spina-christi* leaves at 30, 40, and 50°C was determined by a gravimetric technique, which is based on the use of saturated salt solutions to obtain constant relative humidity of surrounding air.

Six saturated salt solutions (KOH, $MgCl_2$, K_2CO_3, $NaNO_3$, KCl, and $BaCl_2$) were prepared by dissolving an appropriate quantity of salt in distilled water at a higher temperature than equilibration to insure that they remain saturated when cooled.

The experimental apparatus utilized consists of six glass jars of 1 L each with insulated lid. Every glass jar is filled to one-quarter depth with a prepared saturated salt solution. A layer of solid salts was maintained during the whole period of equilibration to confirm that the solutions always remain saturated. A tripod was also put in each jar to place *Ziziphus spina-christi* leave sample. The jars are then put in a controlled temperature oven for 24 h to be stabilized at experiment temperature. As indicated in Table 1, the saturated salt solutions allow obtaining a water activity ranging from 0.07 to 0.89 [32]. Duplicated samples, each of 0.02 g (±0,0001 g) for desorption and 0.01 g (±0,0001 g) for adsorption, were weighted using an analytical balance (±0,0001 g) and placed into the glass jars. The glass jars, containing saturated salt solutions and *Ziziphus spina-christi* leaves samples, were then tightly closed and then put in an oven at a fixed temperature (30, 40, or 50 ± 1°C) for equilibration.

The *Ziziphus spina-christi* leaves samples were weighted every 2 days, until there is no change in mass. Fresh *Ziziphus spina-christi* leaves were used for desorption experiments. Samples used for adsorption isotherms were dried 24 h in an oven at 105°C until reaching maximum dehydration. The moisture content of each sample was determined in a drying oven at 105°C (±0,1°C) for 24 h [33]. The hygroscopic equilibrium of *Ziziphus spina-christi* leaves was reached in 08 days for desorption and 06 days for adsorption. The difference in mass before (m_w) and after (m_d) drying in the oven gives the moisture content X_e of the product at hygroscopic equilibrium:

$$X_e = \frac{m_w - m_d}{m_d}. \quad (1)$$

2.3. Data Analysis

2.3.1. Modeling of Sorption Isotherms. Eight mathematical equations were used for describing desorption and adsorption isotherms of *Ziziphus spina-christi* leaves in the range of temperature varying from 30 to 50°C. The expressions and the parameters of the eight models used to fit the data are presented in Table 2.

Nonlinear regression analysis was used to estimate the constants of the models from the experimental results of sorption isotherms for *Ziziphus spina-christi*. The goodness of fit was determined by using three statistical parameters: the standard error (S), the correlation coefficient (r), and the percent average relative deviation (P). These statistical parameters were defined as follows:

$$S = \sqrt{\frac{\sum_{i=1}^{n_{\exp\cdot\text{data}}} \left(X_{ei} - X_{e_{\text{cali}}}\right)^2}{n_{\exp\cdot\text{data}} - n_{\text{param}}}},$$

$$r = \sqrt{1 - \frac{\sum_{i=1}^{n_{\exp\cdot\text{data}}} \left(X_{ei} - X_{e_{\text{cali}}}\right)^2}{\sum_{i=1}^{n_{\exp\cdot\text{data}}} \left(\overline{X_e} - X_{ei}\right)^2}}, \quad (2)$$

$$P\,(\%) = \frac{100}{n} \sum_{i=1}^{n_{\exp\cdot\text{data}}} \left(\frac{X_{e_{\text{cali}}} - X_{ei}}{X_{ei}}\right),$$

where $X_{e_{\text{cali}}}$ is the calculated value of equilibrium moisture content by using the tested model, X_{ei} is the experimental value of equilibrium moisture content, n_{param} is the number of parameters of the particular model, and $n_{\exp\cdot\text{data}}$ is the number of experimental points. The arithmetic average value of the experimental equilibrium moisture content ($\overline{X_e}$) is calculated as follows:

$$\overline{X_e} = \frac{1}{n_{\exp\cdot\text{data}}} \sum_{i=1}^{n_{\exp\cdot\text{data}}} X_{ei}. \quad (3)$$

2.3.2. Determination of the Net Isosteric Heat of Sorption. The net isosteric heat of sorption can be determined from

Table 2: Mathematical models used to describe desorption and adsorption isotherms of *Ziziphus spina-christi* leaves.

Models names	Models equations	References
GAB	$X_e = \frac{CABa_w}{(1 - Ba_w)(1 - Ba_w + ABa_w)}$	Iglesias and Chirife (1995) [34]
BET	$X_e = \frac{ABa_w}{(1 - a_w)(1 + (A - 1)a_w)}$	BET (1938) [35]
Henderson-Thompson	$X_e = \left(\frac{\ln(1 - a_w)}{-A(T + B)}\right)^{1/C}$	Henderson (1952) [36]
Modified Chung and Pfost	$X_e = A - B\ln(a_w)\ln(-T + B)$	Chung and Pfost (1967) [37]
Halsey	$X_e = A\left(-\frac{B}{\ln(a_w)}\right)^{1/C}$	Halsey (1948) [38]
Oswin	$X_e = A\left(\frac{a_w}{(1 - a_w)}\right)^B$	Chen (1990) [39]
Peleg	$X_e = Aa_w^{\ B} + Ca_w^{\ D}$	Peleg (1993) [40]
Adam and Shove	$X_e = A + Ba_w + Ca_w^{\ 2} + Da_w^{\ 2}$	Chirife and Iglesias (1978) [41]

Where A, B, C, and D are parameters of the equations, T is temperature (°C), X_e is equilibrium moisture content (kg/kg d.b.), and a_w is the water activity.

moisture sorption data by using the following equation, which is derived from the Clausius-Clapeyron equation [42, 43]:

$$\ln(a_w) = -\left(\frac{q_{st}}{R}\right)\left(\frac{1}{T_k}\right) + K, \tag{4}$$

where q_{st} is the net isosteric heat of sorption (kJ/mol), (a_w) the water activity (dimensionless), T_k the absolute temperature (K), R the universal gas constant (kJ/mol K), and K a constant. The sorption isotherms are plotted as $\ln(a_w)$ versus $1/T$ for fixed values of equilibrium moisture contents (the isosteres curves). The net isosteric heats of sorption could be calculated at each value of equilibrium moisture content from the slope of the isosteres curves which is equal to $-(q_{st}/R)$.

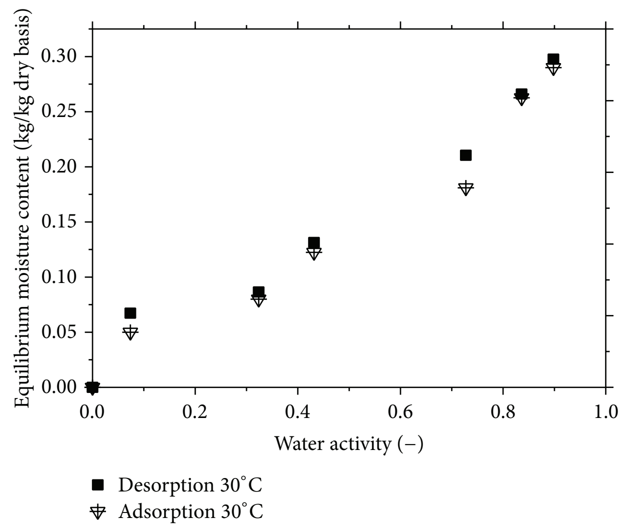

Figure 1: Desorption and adsorption isotherms of *Ziziphus spina-christi* leaves obtained at 30°C. Des: desorption; Ads: adsorption.

3. Results and Discussion

3.1. Sorption Isotherm of Ziziphus spina-christi. The initial moisture content of *Ziziphus spina-christi* leaves was 1.06 kg of H_2O per kg dry matter. The experimental desorption and adsorption isotherms obtained at 30, 40, and 50°C are shown in Figures 1, 2, and 3. The sorption isotherms have the sigmoidal-shaped profile according to the BET classification. These curves are typical of plant products as reported by Ait-Mohammed et al. [44], Kouhila et al. [45], and Idlimam et al. [46]. As seen in the figures, the equilibrium moisture content increased with water activity at constant temperature. Two bending regions are noted, one around 0.1 to 0.3 and another at 0.6 to 0.7. The isotherm is therefore divided into three zones. According to Aguilera and Stanley [47], in zone I (water activity between 0.05 and 0.2), minimal water is contained in the product, and the water molecules present are tightly bound to active sites (e.g., polar groups in molecules) mainly by hydrogen bonding. In zone II (water activity between 0.2 and 0.6) the water is more loosely bound, initially as multilayer above the monolayer; later, as moisture content increases, this water successively fills micropores and macropores in the system. In this region, chemical and biochemical reactions requiring solvent water start to take place because of the increased mobility of solutes. In zone III (water activity between 0.6 and 0.9), excess water is present in macrocapillaries, exhibiting nearly all the properties of bulk water. Microbial growth becomes a major deteriorative reaction in this region.

The figures also show the effect of hysteresis between adsorption and desorption over almost the entire range of water activity at the three temperatures, in which water content on the desorption isotherm is higher than that on the adsorption side at the same water activity. One of the reasons for differences in moisture content between the two closure points is that, during drying (desorption), some solutes may supersaturate below their crystallization water activity and thus hold more water as water activity is lowered especially for products with high sugar content (Table 4) [47, 48].

There is also a decrease in the equilibrium moisture content with increasing temperature, at a constant water

Table 3: Results of fitting of the desorption isotherms of *Ziziphus spina-christi* leaves.

Models names	T (°C)	Parameters				r	S	P (%)
		A	B	C	D			
GAB	30	0.0125	0.684	3.171	—	0.98	0.028	14.74
	40	0.0125	0.487	4.324	—	0.97	0.030	18.40
	50	−0.707	−0.438	0.678	—	0.96	0.037	22.76
BET	30	0.0184	15.422	—	—	0.98	0.027	14.44
	40	0.0192	13.474	—	—	0.97	0.028	18.19
	50	0.0963	17.708	—	—	0.99	0.017	17.30
Henderson	30	11.054	17.086	−29.39	—	0.99	0.019	10.97
	40	34.723	1.838	−38.65	—	0.99	0.017	8.90
	50	10.513	1.195	−49.05	—	0.98	0.020	17.99
Chung and pfost	30	7.683	−29.09	15.607	—	0.97	0.107	57.06
	40	8.256	−39.074	17.287	—	0.97	0.024	11.34
	50	8.180	−48.768	18.330	—	0.93	0.040	28.41
Halsey	30	−6.516	0.063	2.092	—	0.98	0.028	9.08
	40	−6.646	0.058	2.140	—	0.98	0.019	6.98
	50	−9.262	0.102	1.558	—	0.99	0.009	6.99
Oswin	30	0.142	0.356	—	—	0.99	0.017	9.17
	40	0.132	0.341	—	—	0.99	0.010	4.91
	50	0.095	0.500	—	—	0.99	0.009	8.38
Peleg	30	0.108	0.216	0.251	2.544	0.99	0.010	5.51
	40	0.200	0.544	0.262	9.730	0.99	0.012	6.15
	50	0.343	6.529	0.120	0.474	0.99	0.004	3.16
Adam and Shove	30	0.023	0.178	−1.128	1.261	0.98	0.018	19.80
	40	0.019	0.209	−1.163	1.226	0.98	0.013	15.34
	50	0.0165	0.044	−1.076	1.3137	0.97	0.013	21.09

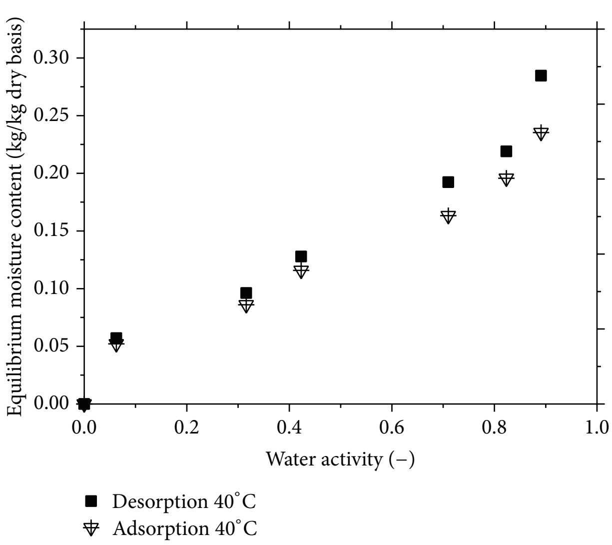

Figure 2: Desorption and adsorption isotherms of *Ziziphus spina-christi* leaves obtained at 40°C. Des: desorption; Ads: adsorption.

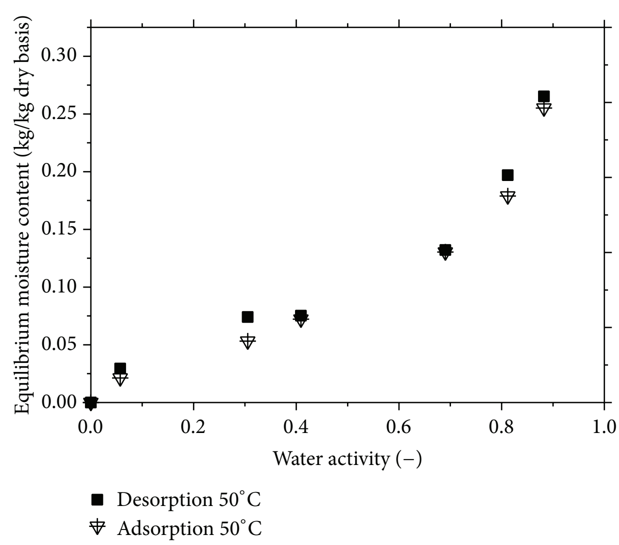

Figure 3: Desorption and adsorption isotherms of *Ziziphus spina-christi* leaves obtained at 50°C. Des: desorption; Ads: adsorption.

activity; this can be explained by the change in the excess enthalpy of water binding, dissociation of water, or increase in solubility of solute in water as temperature increases [49]. Similar trends for many medicinal plants have been reported [50, 51].

Table 4: Results of fitting of the adsorption isotherms of *Ziziphus spina-christi* leaves.

Models names	T (°C)	Parameters A	B	C	D	r	S	P (%)
GAB	30	1.084	0.318	0.633	—	0.98	0.026	16.59
	40	0.091	0.323	8.037	—	0.97	0.025	18.20
	50	0.047	1.061	3.329	—	0.99	0.006	9.51
BET	30	0.03	7.88	—	—	0.98	0.023	15.47
	40	0.007	31.976	—	—	0.97	0.025	18.41
	50	0.1143	1.3399	—	—	0.99	0.009	13.32
Henderson	30	11.129	1.517	−29.24	—	0.99	0.019	12.15
	40	17.986	1.989	−39.59	—	0.99	0.011	7.82
	50	10.988	1.086	−48.90	—	0.99	0.014	16.17
Chung and pfost	30	7.529	−29.00	15.97	—	0.96	0.035	20.63
	40	8.487	−39.11	20.47	—	0.98	0.015	8.98
	50	8.081	−48.66	19.019	—	0.92	0.041	36.65
Halsey	30	−6.44	0.065	1.928	—	0.98	0.027	10.74
	40	−6.91	0.049	2.259	—	0.98	0.017	6.90
	50	−6.23	0.072	1.46	—	0.99	0.009	10.07
Oswin	30	0.127	0.395	—	—	0.99	0.017	7.86
	40	0.1178	0.317	—	—	0.99	0.006	3.32
	50	0.084	0.542	—	—	0.99	0.005	4.23
Peleg	30	0.143	0.420	24.601	4.147	0.99	0.014	7.02
	40	0.157	0.446	0.157	5.892	0.99	0.007	4.25
	50	0.155	0.832	0.374	9.498	0.99	0.006	8.32
Adam and Shove	30	0.018	0.142	−1.111	1.278	0.98	0.021	8.74
	40	0.017	0.205	−1.184	1.205	0.98	0.020	7.40
	50	0.011	0.026	−1.069	1.3203	0.98	0.013	10.59

3.2. Modeling of Sorption Isotherms. Tables 2 and 3 show, respectively, the results of the nonlinear regression analysis of desorption and adsorption isotherms of *Ziziphus spina-christi* leaves obtained at 30, 40, and 50°C. The values of constants of the eight models, that is, GAB, BET, Handerson-Thompson, modified Chung and Pfost, Halsey, Oswin, Peleg, and Adam and Shove, fitted to the desorption and adsorption data along with their standard error (S), the correlation coefficient (r), and the percent average relative deviation (P) for the studied temperatures are given.

These results indicate that all the models are acceptable for predicting the equilibrium moisture content. However, the Peleg and Oswin models gave the best fitting of adsorption and desorption isotherms for the three temperatures, with lowest standard error and the highest coefficient of correlation.

Peleg and Oswin equations were found to be satisfactory for many other plant species [52–54].

Comparisons were done between experimental and calculated (Peleg and Oswin models) data of desorption and adsorption isotherms obtained for *Ziziphus spina-christi* leaves at the three temperatures and are shown, respectively, in Figures 4 and 5.

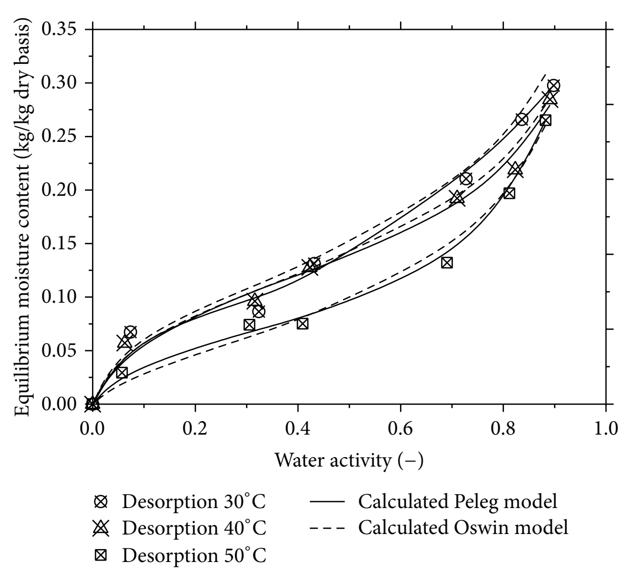

Figure 4: Comparison between experimental and calculated (Peleg and Oswin models) data of desorption isotherms of *Ziziphus spina-christi* leaves obtained at 30, 40, and 50°C. Cal.: calculated.

3.3. Isosteric Heats of Sorption. The net isosteric heats of sorption of *Ziziphus spina-christi* leaves obtained for different moisture contents were determined by using both Peleg and Oswin models in combination with (4).

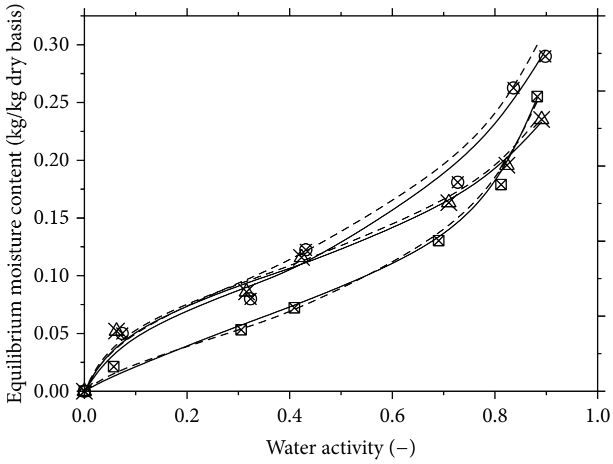

FIGURE 5: Comparison between experimental and calculated (Peleg and Oswin models) data of adsorption isotherms of *Ziziphus spina-christi* leaves obtained at 30, 40, and 50°C. Cal.: calculated.

The variations of the heats of desorption and adsorption of *Ziziphus spina-christi* leaves with moisture content are shown in Figure 6.

For the most part of the curves, the heat of desorption has been observed to present a higher magnitude than the corresponding heat of adsorption. Iglesias and Chirife [42] considered this to be due to structural modifications which takes place during desorption; this modifies the overall energy of binding of the sorbate through cooperative binding or entrapment effects. This phenomena not only explains the difference between the adsorption and desorption heats, but also is capable of explaining the difference between the moisture content of the adsorption and the desorption branch of the isotherm for a given water activity [55].

The figure shows also that the net isosteric heat of sorption decreased with an increase in moisture content. As shown in the curves, a steep slope of the curves is observed; this is indicative of intermolecular attraction forces between sorptive sites and water vapour.

At low moisture contents, the isosteric heat of sorption is high and then decreased at high moisture contents. According to Tsami et al. [56] and Iglesias and Chirife [42], the higher heat of sorption at lower moisture content might be due to the fact that the water is tightly bound to the material, corresponding with high interaction energy. At increasing moisture content, the most active sites become occupied and sorption occurs on the less active sites giving lower heats of sorption.

An exponential function was used to describe the relationship between the isosteric heat of sorption and the equilibrium moisture content:

$$Q_{st} = 252.73 \exp\left(-15.13X_e\right) + 49.42 \quad (5)$$

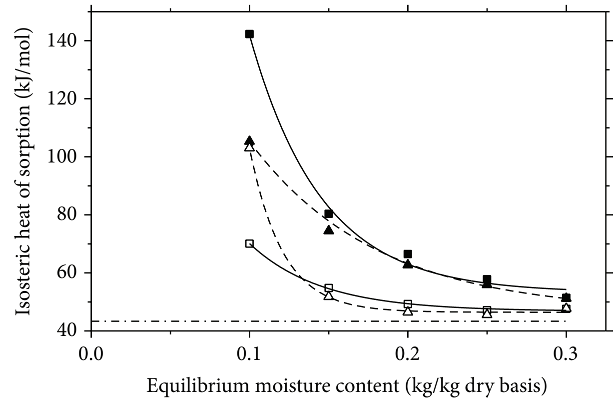

FIGURE 6: Variations of calculated and experimental values of desorption and adsorption isosteric heats of *Ziziphus spina-christi* leaves versus equilibrium moisture content.

for desorption where $r = 0.99$ and $S = 1.83$. Consider

$$Q_{st} = 6629.33 \exp\left(-47.64X_e\right) + 46.43 \quad (6)$$

for adsorption where $r = 0.99$ and $S = 1.37$.

The best statistical parameters show that the exponential function can be used to calculate the heat of sorption of *Ziziphus spina-christi* leaves for different moisture contents.

4. Conclusion

The moisture desorption and adsorption isotherms of *Ziziphus spina-christi* leaves have been determined at 30, 40, and 50°C by gravimetric method. The sorption isotherms had a sigmoid shape. The equilibrium moisture contents were found to decrease with increasing temperature at constant water activity. They were also found to increase with increasing water activity at constant temperature.

Both Peleg and modified Oswin equations were the best models for prediction of desorption and adsorption phenomena among eight commonly used models investigated. The desorption and adsorption isotherms show the occurrence of moisture sorption hysteresis.

The net isosteric heat of desorption and adsorption were calculated using the Clausius-Clapeyron equation.

The net isosteric heats of sorption of *Ziziphus spina-christi* leaves were found to increase with decrease in moisture content and it approached the value of heat of vaporization of free water at higher moisture content.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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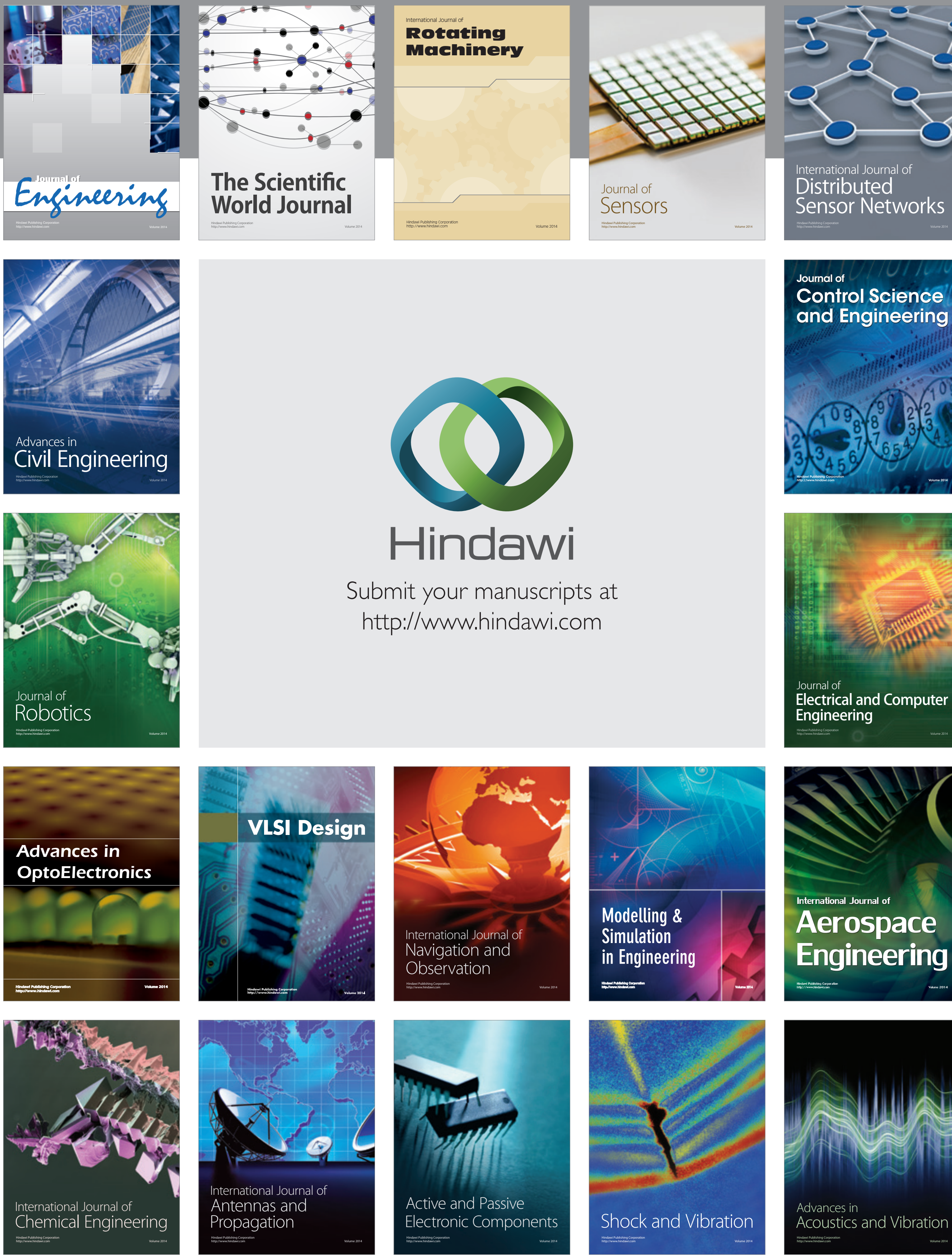
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