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PRESERVATION OF CASHEW NUTS (*Anacardium occidentale* L.): WATER DESORPTION ISOTHERMS AND ISOSTERIC HEAT OF SORPTION

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Abstract: *Knowledge of a food product's sorption isotherms is of vital importance in determining its stability zone during storage. Thus, this study focuses on determining the desorption isotherms of cashew kernels at 40, 50 and 60°C. This was done experimentally, using solutions of increasing sulfuric acid concentration from 20 to 90%. Modelling was then carried out on the basis of eight (8) models described in the literature, using the non-linear GRG algorithm of the Excel 2021 solver. This was used to determine the net isosteric heat of desorption of cashew nuts. The experimental isotherms obtained have a type III sigmoidal shape. The BET model was used to calculate monolayer water contents of 3.07, 2.67 and 2.07% at 40, 50 and 60 °C respectively. Calculation of the correlation coefficient shows that Henderson's model has the highest coefficients (1 for 50 and 60 °C), with the exception of 40 °C, where Smith's model has the highest one (1 vs. 0.994 for Henderson). The net isosteric heat of desorption tends towards zero for high water contents (0.58 kJ/mol at 23.05% water). To guarantee good preservation of cashew nuts at 7% moisture content, the isosteric heat of the monolayer must be maintained at 0.79 kJ/mol.*

Keywords: *Cashew nuts, preservation, desorption isotherms, modelling, monolayer water content, isosteric heat.*

1. Introduction

Since 2016, Côte d'Ivoire has been in first place in terms of cashew nut production, with 968,676 tons in 2021, i.e. over 40% of world supply [1]. Dried and roasted almonds have a number of uses in the food industry (confectionery, pastries, cashew butter). It is roasted, salted and eaten on its own or mixed with other nuts or dried fruits [2]. Its shell also yields cashew balsam, another high value-added product, which is highly acidic and corrosive. It is used in the manufacture of friction elements [3]. In view of the many economic advantages of marketing cashew nuts, Côte d'Ivoire has focused its economic and social policy in recent years on increasing production, processing and export. These challenges take into account the problems of quality of the end product for export, in order to maintain its leading position. According to the Conseil National Coton-Anacarde, measures to guarantee the quality of marketed nuts include controlling the commercial moisture content, which is set at 7%, and controlling hygiene quality. To predict the behaviour of a product during storage, it is essential to know how the water content changes as a function of water activity in the product at a given temperature [4]; [5]; hence the importance of determining desorption isotherms for drying and preserving foodstuffs. Knowledge of water activity is of considerable interest in food technology. The thermodynamic activity of water in a plant product determines the

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speed and intensity of chemical reactions (oxidation, Maillard reaction), enzymatic reactions, the development of micro-organisms and modifies most rheological, mechanical and organoleptic properties [6]; [7]. The fact that Côte d'Ivoire is a hot and humid country, the phenomenon of moisture pick-up can be detrimental to cashew nuts. Thus, the general objective of this work was to contribute to cashew nut conservation by determining and modelling its desorption isotherms.

2. Materials and methods

2.1. Biological materials

The biological material consisted of 1 kg of cashew nuts freshly harvested from a cashew plantation located 5 km from the National Polytechnic Houphouët-Boigny Institute (INP-HB) on the Yamoussoukro-Didiéviroad. The nuts were collected from under the cashew trees and separated from their apples before being packed in plastic bags and transported to the laboratory.

2.2. Description of the laboratory experimental set-up

The static gravimetric method has been used to determine cashew nut desorption isotherms [8]; [9]. Two hundred (200) mL of sulfuric acid solution at increasing concentrations (20, 30, 40, 50, 60, 70, 80 and 90% volume/volume) were introduced into eight jars (Table 1). Samples weighing 5 g in glass cups were suspended in each jar above the sulfuric acid solution. The eight samples were stabilized in terms of temperature and humidity in an oven at 40, 50 and 60 °C (Figure 1). These temperatures correspond to cashew nut drying temperatures. The samples were weighed at regular 4-day intervals until they reached a stable mass. They were then assumed to be in equilibrium with the ambient air at temperature and relative humidity. Equilibrium is considered to have been reached when the variation in mass between two successive measurements is less than or equal to 0.001 g [10]. Knowing the wet masses, the dry ones were obtained by placing the samples in an oven at 105 °C±2 °C for 24 hours.

Table 1.

Standard Aw values for sulfuric acid solutions [7]

Sulfuric acid (%v/v) **Aw**	**20**	**30**	**40**	**50**	**60**	**70**	**80**	**90**
Aw (40 °C)	87.85	75.37	57.48	36.69	17.80	4.97	0.69	0.05
Aw (50 °C)	88.86	77.07	58.26	38.05	18.92	5.59	0.83	0.06
Aw (60 °C)	89.03	77.65	58.44	38.83	19.81	6.11	0.98	0.08

The (Aw, Xeq) pairs provide the points of the desorption isotherm {Eq. (1 and 2)} [7].

$$Xi = \frac{Mi - MS}{MS} \times 100 \qquad (1)$$

$$Xeq = \frac{M}{Mi}(Xi + 100) - 100 \qquad (2)$$

With **Mi**: Mass of the product at the initial time (g),

M: Mass of the product at time t (g),

Ms: Dry mass of the product (g),

Xi: Water content of the product at the initial time (% g m.s)

Xeq: Equilibrium water content of the product (% m.s).

Fig. 1. Experimental set-up for determining cashew nut desorption isotherms

2.3. Modelling and determining the parameters of the equations

In the present study, eight (8) models most commonly used in the food industry for fitting and modelling sorption isotherms were used to interpret the experimental curves obtained (Table 2) [11]. The parameters X0, A, B and C, of the different models are the coefficients of the equations of the desorption curves which are determined by identification with the experimental curves, by minimizing the sum of the Mean Squared Deviations (MSE) according to the following formula:

$$MSE = \frac{1}{N}\sum_{1}^{N}\left|X_{eqi,exp} - X_{eqi,pre}\right|^2 \quad (3)$$

With:

Xeqi, **exp**: i^{ième} experimental equilibrium water content (% d.b), **Xeqi**, **pre**: i^{ème} predicted equilibrium moisture content (% d.b), **N**: number of experimental points.

2.4. Calculating isosteric heat

The isosteric heat was calculated from a relationship derived from the Clausius-Clapeyron equation [12]:

$$\frac{\partial \mathrm{Ln(aw)}}{\Delta(\frac{1}{T})} = -\frac{Qst-Lv}{R} = \frac{qst}{R} \quad (4)$$

Qst: isosteric heat of desorption (kJ/mol);
qst: net isosteric heat of desorption at equilibrium water content (kJ/mol);
Xeq: Equilibrium water content (kg/mol);
T: Absolute temperature (K);
R: Perfect gas constant (8.314J/mol.K);
Lv: Latent heat of vaporization of pure water (43.53 kJ/mol) at 35 °C.

The net isosteric heat represents the additional heat to the heat of vaporization of pure water that would have to be supplied to the product in order to dehydrate it [12]; [13]: The net isosteric heat of desorption is calculated from the relationship -Ln(Aw) as a function of 1/T at a constant water content with an accuracy of R [14]. This heat is also the slope of the isosteric curve, which is deduced for each water content Xeq by plotting the equation qst= f (Xeq). Isosteric heat can be calculated using the equation:

$$Qst = qst + Lv \quad (5)$$

2.5. Enthalpy-entropy compensation theory

The heat and differential entropy of sorption are linked by equation Eq. (06) [14].

$$\mathbf{Ln}\ (A_w) = -\frac{qst}{RT} + \frac{\Delta S}{R} \quad (6)$$

The differential sorption entropy is simply the y-intercept of the various isostere lines obtained previously. From the y-intercept and for each water content Xeq, we can deduce the corresponding differential sorption entropy and plot the curve ΔS= f(Xeq). According to compensation theory, the linear relationship between enthalpy and entropy for a specific reaction is given by:

$$qst = T\beta . \Delta S + \Delta G\beta \qquad (7)$$

$T\beta$, is the isokinetic temperature for which all reactions in the series proceed at the same rate; $\Delta G\beta$, is the free energy at temperature, $T\beta$, which provides a criterion for assessing whether the water sorption process is spontaneous (-ΔG) or not (+ΔG). $T\beta$ and $\Delta G\beta$ are obtained by plotting the curve qst= f (ΔS).

3. Statistical analysis

The modelling of adsorption isotherms requires the statistical methods of regression and correlation analysis. The regression analysis was carried out using the non-linear GRG algorithm of the solver in Excel 2021. The regression coefficients (R^2) of the lines were then determined. The correlation coefficient (r) was the first criterion for selecting the best equation to describe the desorption curves [15]. In addition to r, calculations of the values of RME (Relative Mean Error), SE (Standard Error of Water Content) and ei (Residual Error) were used to justify the choice of model [16].

The e_i is used to determine whether the residuals between the values predicted by the model and those obtained from experience are minimal.

The best model is the one with the highest value of r and the lowest values of RME, SE and e_i [17].

$$\mathbf{r}= \frac{\sum(Xeqi,exp - \bar{X}eqi,exp)(Xeqi,pre - \bar{X}(eqi,pre)}{\sqrt{\sum(Xeqi,exp - \bar{X}(Xeqi,exp)2(Xeqi,pre - \bar{X}(eqi,pre)2}} \qquad (8)$$

$$\mathbf{RME}= \frac{100}{N}\sum_{1}^{N}\frac{Xeqi,exp - Xeqi,pre}{Xeqi,pre} \qquad (9)$$

$$\mathbf{SE}= \sqrt{\frac{(Xeqi,exp - Xeqi,pre)2}{df}} \qquad (10)$$

$$e_i = Xeqi,pre - Xeqi,exp \qquad (11)$$

With: Xeqi, exp: $i^{ième}$ experimental equilibrium water content (% ms),

$$\bar{X}eqi,exp = \frac{1}{N}\sum_{1}^{N} Xeqi,exp \qquad (12)$$

Average experimental water content.

$$\bar{X}eqi,pre = \frac{1}{N}\sum_{1}^{N} Xeqi,pre \qquad (13)$$

predicted mean water content, N: Number of experimental points, df: degree of freedom of the model regression; df = N-n, n: Number of variables in each model.

4. Results and discussion

4.1. Graphical representation of cashew nut desorption isotherms

After 44 days in the oven, hygroscopic equilibrium of the cashew nuts was reached at the last point. Figure 2 shows the desorption isotherm curves for cashew nuts at 40 °C, 50 °C and 60 °C. They have a sigmoidal shape, similar to those commonly found for plant products [12; 18]. The desorption isotherms at higher temperatures are lower than those obtained at lower temperatures. In other words, for the same water activity, the equilibrium water content of the product decreases with increasing temperature. Indeed, during drying, the increase in temperature progressively excites the water molecules involved in the bonds (hydrogen and covalent) with the product [19]. This weakens the bonds

Table 2.

Mathematical models and their areas of use

Model name	Model		Area of validity
BET (1938)	$X_{eq} = X_0 C A_w / [(1 - A_w)(1 + (C - 1)A_w)]$	**(14)**	0.05 <Aw < 0.35
Oswin (1946)	$X_{eq} = A[\frac{A_w}{1-A_w}]^B$	**(15)**	0.05 < Aw< 0.90
Hasley (Rahman, 1995)	$X = X_0(-\frac{A}{RT\ln A_w})^{1/B}$	**(16)**	0.05 < Aw< 0.80
Chung et Pfost (1967)	$X_{eq} = \frac{1}{B}[Ln(A - Ln(-Ln(A_w))]$	**(17)**	0.20 < Aw< 0.90
GAB (1966)	$X_{eq} = \frac{X_0 C K A_w}{(1-KA_w)(1-KA_w+CKA_w)}$ $C = c_0 exp\left[\frac{H_m - H_n}{RT}\right]$ $K = k_0 exp\left[\frac{H_1 - H_n}{RT}\right]$	**(18)**	0.05 < Aw< 0.95
Smith (1947)	$X_{eq} = A + B\, log(1 - A_w)$	**(19)**	0.50 < Aw< 0.95
Henderson (1952)	$X_{eq} = \left[\frac{\ln(1-A_w)}{-A}\right]^{1/B}$	**(20)**	0.50 < Aw< 0.95
Harkins & Jura (1944)	$X_{eq} = \left[\frac{-B}{\ln(A_w)-A}\right]^{1/2}$	**(21)**	0.50 <Aw

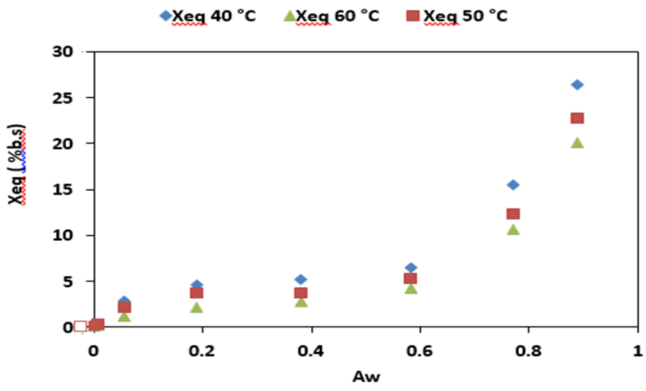

Fig. 2. Experimental desorption isotherms for cashew nuts at temperatures of 40, 50 and 60 °C

between the water molecules and between the water molecules and the product, causing them to detach, resulting in water loss [20].

4.2. Modelling desorption isotherms

The parameters of the mathematical models studied and their statistical selection criteria are given in Table 3. All the correlation coefficients r and the coefficients of determination R^2 are high (over 0.85) for the different temperatures. The correlation coefficient r varies from 0.93 to 1 for 40 and 50 °C, then from 0.81 to 1 for 60 °C. Then the coefficient of determination R^2 varies from 0.83 to 1 for 40 and 50 °C, then from 0.86 to 1 for 60 °C. The lowest correlation coefficients r and determination coefficients R^2 are observed with the Chung and Pfost model, while the highest values appear with the Henderson and Smith models on the one hand, and with the Harkins and Jura model on the other. With regard to the experimental values of the two coefficients R^2 and r, it emerges that, in general, the models all predict the regression between the theoretical and experimental parameters well, with a percentage of over 80%. Better still, the Henderson, Smith and Harkins' and Jura models predict this correlation. The same is true for the Mean Square Errors (MSE), which generally decrease for all models with increasing temperature, except for the BET and Oswin models. At 60 °C, the MSE is higher than at 40 and 50 °C with the BET model, and at 50 °C, the MSE is higher with Oswin. The lowest values are recorded with the Henderson and Smith models. The highest values are found with the Chung and Pfost model on the one hand and the Harkins and Jura model on the other. Given that the aim of the modelling is to minimise MSE errors, this shows that the best models for predicting the hygroscopic behaviour of cashew nuts are the Henderson and Smith models. The models of Chung and Pfost and that of Harkins and Jura, which give the highest values, show that they are not suitable for this study. The Relative Mean Errors (RME) vary very little at the different temperatures of 40, 50 and 60 °C. The lowest values are observed in the Smith and Henderson models, as in the case of the Mean Square Errors. The highest values are obtained with the BET models for all temperatures. The Henderson and Smith models still show a better fit to the description of the hygroscopic behaviour of cashew nuts. The high values express the poor adaptation of the model to the reality of studying the equilibrium moisture content of the product at these temperatures. For the parameters A and B, the values of A increase while those of B decrease with increasing temperature in the Henderson and Smith models. These values are negative with Smith and tends towards zero in the Henderson model. The parameter A is high for Hasley and close to zero for the Chung and Pfost models, and the Harkins and Jura ones. The opposite is true of the B parameter for the same models. Statistical analysis of the eight models used shows that the two-parameter Henderson model has the highest correlation coefficients (1.000) for 50 and 60 °C with the exception of 40 °C where the Smith model has the highest coefficient. However, for 40 °C, the Smith model has the lowest MSE, RME and SE estimation errors, while these values fall with the Henderson model at 50 °C and 60 °C. Only two models provide information on the water content of the monolayer (the BET and GAB models). These monolayer water contents are temperature-dependent, with relatively close values (respectively 3.07

Table 3.

Estimated parameter values and statistical selection criteria for the mathematical models used

MODELS	PARAMETERS	TEMPERATURES			MODELS	PARAMETERS	TEMPERATURES		
		40	50	60			40	50	60
	X0	**3.700**	**2.671**	**2.074**		X0	**3.000**	**3.000**	**13.077**
	C	**40.115**	**38.335**	**7.048**		C	**2.002**	**1.454**	**0.423**
	MSE	**0.162**	0.164	1.573		K	0.817	0.891	0.990
BET	RME	**50.042**	45.148	131.945	**GAB**	MSE	3.374	2.024	0.396
	SE	0.520	0.524	1.619		RME	**26.280**	**34.293**	**23.480**
	r	**0.981**	0.965	0.813		SE	2.904	2.012	0.890
	R²	**0.963**	0.931	0.660		r	0.986	0.992	0.997
	A	8.977	19.690	4.465		R²	0.972	0.984	0.993
	B	0.571	0.118	0.734		A	-6.929	-6.976	-9.077
Oswin	MSE	2.518	5.292	0.438		B	-36.449	-30.877	-30.371
	RME	**22.222**	**39.923**	**26.062**		MSE	0.027	0.139	0.008
	SE	2.048	2.818	0.811	**Smith**	RME	1.301	3.696	1.370
	r	0.986	0.964	0.997		SE	0.285	0.647	0.155
	R²	0.971	0.929	0.993		r	1.000	0.999	1.000
	X0	39.108	35.823	33.894		R²	1.000	0.997	1.000
	A	17.004	15.575	14.736		A	0.219	0.300	0.370
	B	0.081	0.088	0.092		B	0.690	0.637	0.595
Hasley	MSE	3.690	1.478	0.341		MSE	0.390	0.006	0.004
	RME	**21.776**	**24.378**	**13.973**	**Henderson**	RME	5.655	0.910	0.830
	SE	3.037	1.719	0.826		SE	1.081	0.139	0.103
	r	0.990	0.996	0.997		r	0.997	1.000	1.000
	R²	0.981	0.992	0.995		R²	0.994	1.000	1.000
	A	1.137	1.055	0.996		A	0.000	0.000	0.000
	B	0.050	0.059	0.067		B	72.249	47.147	35.409
Chung	MSE	10.309	8.148	6.851		MSE	**10.712**	**8.509**	**8.049**
& Pfost	RME	**36.184**	**39.764**	**45.325**	**Harkins**	RME	**29.965**	**33.158**	**39.521**
	SE	4.541	4.037	3.702	**& Jura**	SE	5.669	5.052	4.914
	r	0.926	0.925	0.924		r	0.996	1.000	1.000
	R²	0.857	0.856	0.855		R²	0.992	1.000	1.000

and 3.00% at 40 °C, and 2.67 and 3.00% at 50 °C for BET and GAB), with the exception of the values of 2.07 and 13.07% observed at 60 °C, where the difference is significant. The type of isotherm is determined by the values of the parameter C in the GAB and BET equations. When C ≤10, the isotherm is type III, whereas for C ≥10, the isotherm is said to be type II [21]. In this study, two out of six C values for the two models mentioned above are greater than 10 [22]. The isotherms obtained could therefore be termed type III sigmoidal shape. This implies the formation of a monolayer, then a multilayer [12]. This type of isotherm is characteristic of a non-porous or macro porous medium and little interaction between the adsorbed gases during dehydration. These isotherm results are consistent with the behaviour of plant products [23]. When it comes to preserving food products and plants, the water content of the monolayer is of vital importance, especially when the product is stored for a long time. A low value for the parameter X0 (water content of the monolayer) is a better indicator of good product preservation. Considering the acceptable market moisture content of cashew nuts of 7%, the water content of the monolayers in the BET and GAB models could guarantee good product preservation (recording the lowest X0 values). In fact, the loss of quality due to chemical reactions, for most dehydrated products, is negligible below the value of the water content of the monolayer.

4.3. Net isosteric heat and differential entropy between 40 and 60 °C

The net isosteric heat of desorption (qst) for different water contents in the temperature range between 40 and 60 °C is shown in Figure 3. It therefore falls as the water content of the material increases. In fact, its value drops sharply from 8.98 to 1.20 kJ/mol between 0 and 5% moisture content to reach 0.6 kJ/mol above 10% moisture content. The same observation is made with the differential entropy of desorption (ΔS) in Figure 4. This explains that the high value of the heat of desorption at low water content, is due to the existence of highly active polar locations on the product surface [7]. The water molecules form a mono-molecular layer.

The net isosteric heat and differential entropy of desorption of cashew can be calculated from the smoothing of desorption isosteres which are expressed as an exponential function of water content. The experimental data of net isosteric heat (qst) and differential entropy (ΔS) were satisfactorily correlated according to the following relationships:

qst = 29.6654×EXP (Xeq/1.7123) (kJ/mol).
with r = 0.9756 and MSE=28.78% **(22)**

ΔS =1312.501 × EXP(-Xeq/1.6166) (J/mol) with r = 0.9762 and MSE= 43.4% **(23)**

According to equation 23, if we are looking for a marketable water content in the cashew nut set at 7% by the Cotton and Cashew Council from Côte d'Ivoire, the isosteric heat to be applied to one mole of water in the cashew nut is 0.79 kJ.

Thus, to guarantee good preservation of the cashew nut and ensure better commercial quality, the producer should dry the nuts at a moisture content of 7%, maintaining the isosteric heat of the monolayer at 0.79 kJ/mol (Figure 4).

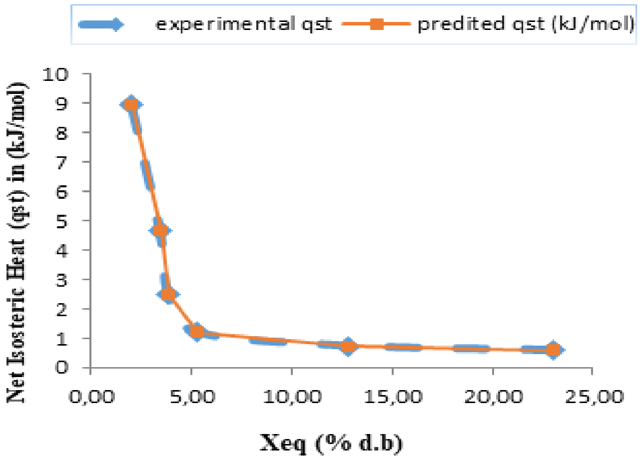

Fig. 3. Evolution of the isosteric heat of desorption of cashew nuts as a function of water content between 40 and 60 °C

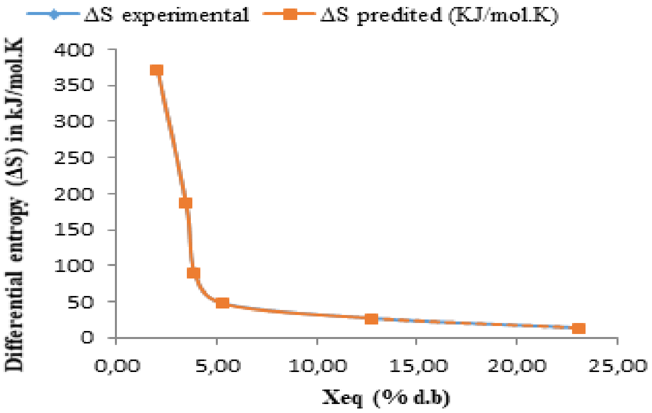

Fig. 4. Evolution of the differential entropy of desorption of cashew nuts as a function of water content between 40 and 60 °C

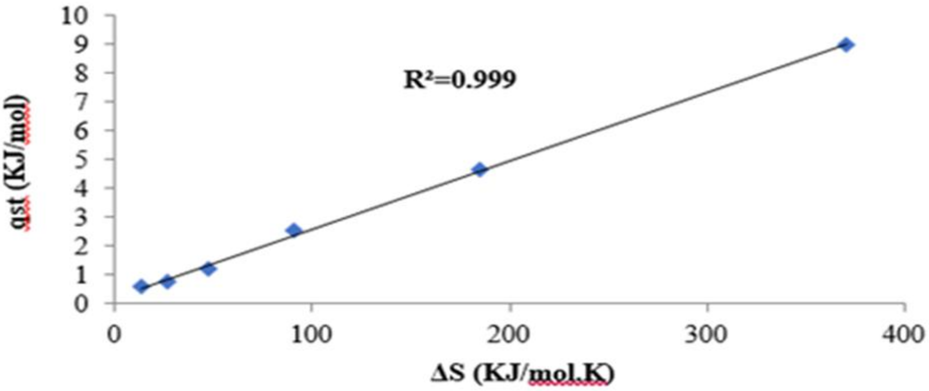

Fig. 5. The entropy/enthalpy compensation theory for fresh cashew nuts

4.4. Cashew nut entropy/enthalpycompensation theory

Figure 5 shows the entropy/enthalpy compensation theory for fresh cashew nuts. This curve shows linearity between enthalpy (net isosteric heat) and differential entropy of desorption at 99%. According to equation (24), the net isosteric heat is:

$$qst = T\beta . \Delta S + \Delta G\beta \qquad (24)$$

hence,

qst = **0.0238 x ΔS + 0.196**

This equation allows us to determine, the isokinetic temperature $T\beta$ which is 0.0238 K and the free energy $\Delta G\beta$ which is 0.1964 kJ/mol. There is an equilibrium between entropy and free enthalpy during the desorption of cashew nuts. The enthalpy-entropy compensation process thermodynamically manifests the structuring - destructuring of water. The positive free energy of the calculated enthalpy-entropy equation explains why the desorption phenomenon during the change of state of water is progressive [15].

5. Conclusion

The aim of this work was to contribute to cashew nut conservation by determining and modeling its desorption isotherms. The experimental study showed that cashew nut desorption isotherms are type III sigmoidal shape. The data on the parameters of the different models clearly show that the empirical Henderson model fits the experimental results well. This model is therefore better able to predict the hygroscopic behaviour of cashew nuts for a water activity of between 0.5 and 0.95. It therefore remains limited compared with the GAB model, whose validity range is between 0.05 and 0.95. However, the BET model proved effective in determining the water content of the monolayer, as is the case for most food products. To preserve cashew nuts properly and ensure better commercial quality, the producer should dry and maintain the nuts at a moisture content of 7% Hr, with an isosteric heat of the corresponding monolayer of 0.79 kJ/mol and a relative humidity less than 70%.

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