IIETA International Information and Engineering Technology Association
Advancing the World of Information and Engineering

Instrumentation Mesure Métrologie
Vol. 18, No. 4, August, 2019, pp. 389-396
Journal homepage: http://iieta.org/journals/i2m

The Isothermal Sorption Measurement and the Isosteric Heats Determinations for the South Algerian Date Varieties

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https://doi.org/10.18280/i2m.180408

Received: 18 April 2019
Accepted: 10 July 2019

Keywords:
sorption isotherm, solar drying, gravimetric method, isosteric heats, date varieties, GAB and SMITH models

ABSTRACT

The equilibrium water content of a product to be dried is essential during a forced convection solar drying and storage operation. These values are often taken from the so-called isothermal sorption curves. The isotherms determination is an essential step to know the distribution and the intensity of the water connections in the products.

They make it possible to determine the products final water content to be reached in order to optimize the drying conditions and give valuable information on the hygroscopic equilibrium. As a result, this experimental study aims to determine the physicochemical components' and the desorption isotherms curves for deferent date type L’hmira, Bamakhlouf and Tegazza from the Touat region, in South-West Algeria, using the saturated salt solution method. The experiment is carried out for three temperature values 40 ° C, 50 ° C and 60 ° C, to study the influence of the temperature on the equilibrium curves of the product and finally to describe the isothermal moisture desorption for deferent date types for three temperatures using known models of GAB, SMITH, LANGMUIR and OSWIN.

1. INTRODUCTION

Many agricultural products, consumed in large quantities are not always available during the seasons. Several solutions are proposed to overcome this handicap for example: greenhouse cultivation, freezing and the preservation by drying. The solar drying use is a simple, safe and adequate solution for a large number of agricultural products. The Southern Algerian regions are beginning to experience more or less rapid development. A large part of the agricultural production of these regions: tomato, dates, henna, mint. etc., lends itself perfectly to conservation by drying to ensure continuity in their availability and allow their transport as well as to reducing the produces cost. The dates are the historical food of great and high energy and commercial values. According to FAO statistical data in 2012, Algeria is the world's sixth largest date producer, with an estimated average annual production of 789357 tones, for more than 12 million date palms, particularly in TOUAT areas.

Certain dates varieties have excess production, such as: Aghammou or L’L’hmira, which is destined for export to the African countries: Mali, Niger, and Chad. As well as, some local applications such as livestock or for industrialization (manufacture of soup, vinegar). Date varieties are generally drayed after harvest using traditional solar dryer. Agriculture use solar energy; free, non-polluting and excellently clean. Those methods are subjects of several researches.

Sorption isotherms are of great importance in the food industry, especially in solar drying, storage and food preservation. Their knowledge tells us about the hygroscopic or hydric balance of the product under consideration, the isosteric heats, and the lifetime's duration. We do not know the product stability types. This equilibrium is characterized by these curves whose experimental determination requires a large number of measurements. They are also useful during a solar drying process. Indeed, it is from these sorption curves that we can determine the final product water content, so as to ensure the physicochemical and microbiological stability during the storage, in addition the product shelf life can be predicted [1-6].

studies on sorption isotherms are carried out by mathematical modeling, eight mathematical models (in the literature) empirical and semi-empirical based on more or less physical bases [7-10], describing the correlation of water content with balance with the water activity of the products. among these models: GAB [11], modified BET [12], Henderson-Thompson [13], modified Halsey [14], modified Oswin [15], Peleg [16]. GAB is the most widely used model for various foods [17-20].

The present Works focus firstly the experimental determination of the sorption curves forL’L’hmira, Bamakhlouf and Tegazza varieties, secondly we are interested in defining among the existing mathematical models, the most appropriate model for the studied product and finally, we are interested in modelling the isosteric heats for deferent dates.

2. OPERATING MODE

The obtaining isotherm point's principle is as follows: a sample of the product is placed in an enclosure maintained at temperature T and at relative humidity Hr of the constant air: this is the gravimetric method. The sample is weighed at regular intervals until its mass no longer varies, it is then in equilibrium with the air at (T, Hr). Knowing its wet mass, it is then sufficient to determine its dry mass to deduce its water content X, the couple (Hr, X) provides a point of the isotherm of sorption or desorption. The product is suspended in a sealed container inside which a solution maintains a constant relative humidity. The solutions used are saline solutions saturated with salt. The temperature is kept constant by placing the containers in a thermostatically controlled enclosure. We use as many salts as we want to obtain points on the isotherm [19-21].

3. SATURATED SALINE SOLUTIONS PREPARATION

The saturated saline solutions used are: KOH, KF, MgC_{l2}, K_2CO_3, NaCl, KCl, BaC_{l2}, K_2CO_4. These solutions make it possible to obtain relative moistures ranging from 5.5 to 95.8 % (Table 1).

Figure 1. Saturated saline solutions preparation

Table 1. Standard values of relative humidity given by different salts as a function of temperature [22]

	T1 = 40 °C	T2 = 55 °C
Salts	**HR (%)**	**HR (%)**
KOH	5,7	5,5
KF	20,8	20,8
$MgCL_2$	30,5	30,5
K_2CO_3	45,6	45
NaCL	74,4	74,5
KCL	81,2	81,2
$BaCL_2$	88,2	88,2
K_2SO_4	95,8	95,8

But, before beginning the preparation of the eight salt solutions, we consulted the different values of the solubility of the salts used and that to have a prior idea on their masses (Table 2).

Each saturated solution is obtained by dissolving, in a bottle containing 120 ml of distilled water, a mass weighed in a 0.001g precision digital display scale; during the preparation, stirring of the solutions was necessary. The masses of salts added to obtain these saturated salt solutions are given in Table 3.

Table 2. Used salts solubility [16]

Solution	KOH	KF	$MgCl_2$	K_2CO_3	NaCl	KCl	$BaCl_2$	K_2SO_4
Solubility (g /l)	1100	449	542	1120	359	340	360	120

Table 3. Used saturated salts masses

Solutions	KOH	KF	$MgCl_2$	K_2CO_3	NaC_l	KCl	$BaCl_2$	K_2SO_4
Masses mi (g)	132	35.9	43.3	134.4	28.7	27.2	28.8	9.6

4. SAMPLE PREPARATION OF L'HMIRA, BAMAKHLOUF AND TEGHAZZA DATES:

We brought samples of Takarboucht / Tegazza dates from the Adrar (Bouda) area, to the URER / MS laboratory, in a fresh and healthy state for the purpose of the study. The fresh samples destined for the Takarboucht / Tegazza date desorption process are small pieces of about the same mass (1 g) weighed using a precision 10^{-3}g digital scale. Each sample is then placed in the jar (on a perforated stainless steel grid prepared in advance), above the acid solution, and thus remains in an environment stabilized in temperature and hygrometry.

Figure 2. L'hmira, Bamakhlouf and Tegazzadates preparation

5. EXPERIMENTAL METHOD

The VACUCELL oven (VU) is a vacuum chamber, electrically heated, in which it is possible to heat the objects placed there after the evacuation. The evacuated objects are placed on the shelves in the room. The device operates up to a temperature of 200 ± 1 °C. The desorption experiments carried out were carried out at two different temperatures: 40 °C., 55 °C. and eight relative humidity values. For each working temperature, an oven was used. So, in each oven, there are eight hermetic jars (Figure 2).

Figure 3. The product arrangement in the oven

Before placing the product to be studied in the jars, they are introduced into the controlled oven at a fixed set temperature. The jars are left in the oven for 24 hours so that the saline solutions can adapt to the working temperature. Then the samples are introduced into the jars and using an electronic balance 0.001g precision digital display. They are weighed every two days until the mass variation between two successive measurements becomes less than 2 %. The thermodynamic equilibrium is reached after a few days.

As soon as the equilibrium wet masses are determined, the samples are introduced into the oven at 105 °C. for 24 hours in order to determine their dry masses.

Figure 4. The products in the oven at 105 °C

After obtaining the wet and dry mass of all test shots. Equilibrium water contents are been calculated using the relationship:

The dry extract of the sample is determined: the water content of the sample is then known for a given hygrometry. A desorption curve is obtained: that is, the sample loses water to reach equilibrium because the sample placed in the chamber was wet.

6. EXPERIMENTAL AND SIMULATION RESULTS

After obtaining the wet and dry masses of all test samples and calculating equilibrium water contents, summarize the equilibrium moisture content values of all date samples are plotted for 40 °C, 50 °C and 60 °C, in Tables 4, 5 and 6.

Table 4. Obtained water equilibrium for three temperatures of L'hmira dates

	Xeq(g H_2O/gMS)		
Aw	**θ=40°C**	**θ=50°C**	**θ=60°C**
90	0.75492958	0.99679487	0.75787728
87.56	0.48064085	0.63623596	0.61764706
81.66	0.30213904	0.33895921	0.33811802
74.93	0.22765363	0.21938089	0.24613003
66.51	0.16395664	0.16504854	0.16845329
56.83	0.15529753	0.08860759	0.14772727
45.88	0.14306569	0.12046444	0.1011236
35.57	0.15316642	0.1194690	0.09210526
25.89	0.07959479	0.1166180	0.09656301

Table 5. Obtained water equilibrium for three temperatures of Tegazza dates

	Xeq(g H_2O/gMS)		
aw	**θ=40°C**	**θ=50°C**	**θ=60°C**
99	0.87727273	/	1.003125
83	0.5290611	0.53277545	0.55102041
75.3	0.38026125	0.36592179	0.39665653
68	0.24404762	0.22759602	0.20574887
48.8	0.18333333	0.17804154	0.20623145
33.1	0.1496063	0.15159944	0.14157973
24	0.15981013	0.15142429	0.10576923
15.5	0.18456884	0.13149847	0.38660578
9	0.13855422	0.14637681	0.09177215
6.8	0.18597561	0.15428571	0.14710744

Table 6. Obtained water equilibrium for three temperatures of Bamakhlouf dates

	Xeq(g H_2O/gMS)		
aw	**θ=40°C**	**θ=50°C**	**θ=60°C**
99	1.13492036	1.12714286	0.88166667
83	0.9	0.62243286	0.7
75.3	0.39365079	0.35060976	0.35225376
68	0.43174603	0.28219971	0.29259897
48.8	0.26349206	0.2944	0.21086262
33.1	0.18888889	0.15494978	0.22182469
24	0.24920635	0.13817664	0.122571
15.5	0.14126984	0.16148148	0.1443299
9	0.18412698	0.14092954	0.15913556
6.8	0.12857143	0.13969571	0.11908932

6.1 Dates isotherm desorption

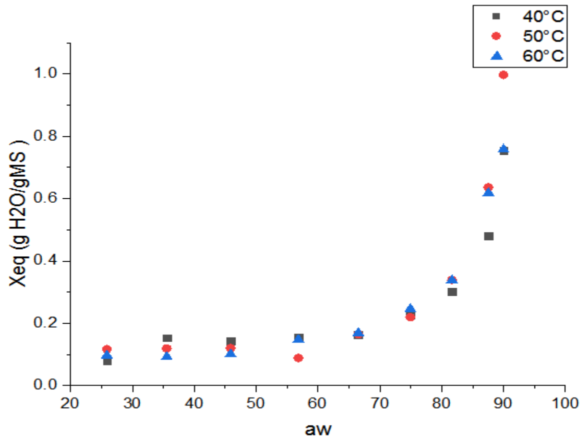

Figure 5. L’hmira date desorption isotherm: Experimental points at three working temperatures

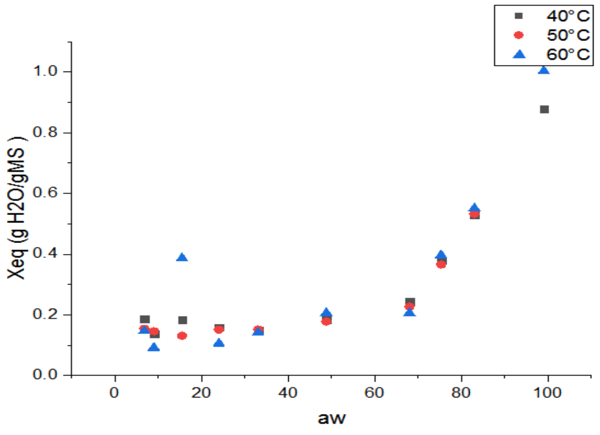

Figure 6. Tegazza date desorption isotherm: experimental points at three working temperatures

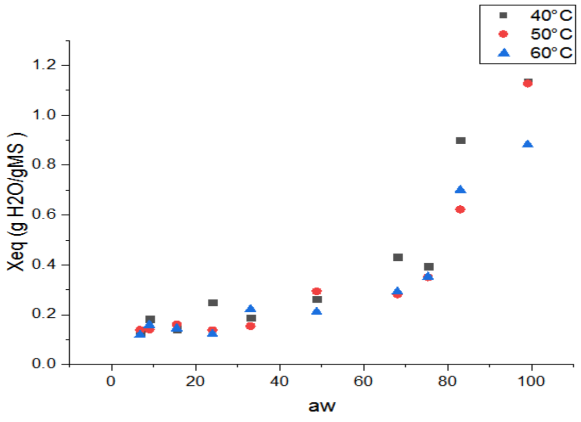

Figure 7. Bamakhlouf date desorption isotherm: experimental points at three working temperatures

The hygroscopic equilibrium for dates desorption is realized. Figures 5, 6 and 7 show that the desorption isotherm has a sigmoidal appearance. This is consistent with the behavior of other agri-food products [23-30].

6.2 Curve modeling

Using Curve Expert software, we test the equations of the above models on water content (Xeq) curves as a function of water activity (aw) for different samples at temperatures of 40, 50 and 60 ° C [31-39].

In the results pane, software gives different models compatible with the curves in the order of standard error values (S) and Coefficient of determination (r^2). The following Tables 7, 8 and 9 summarize the three most compatible dates desorption isotherm models for each type of date at different temperatures.

Note that the most suitable model for date desorption isotherm curves is the GAB model, since it has the lowest values for the standard errors (S) and the largest values for the coefficient of determination (r^2) at experimental temperatures.

Table 7. Model parameters of L’hmira date desorption isotherms at three temperatures

models	**parameters**	**Temperatures**		
		40 °C	**50 °C**	**60 °C**
GAB	A	-5.1928179E-02	-4.7702490E-02	-5.777748E-02
	B	***6.8131185***E+01	1.2786111E+02	7.6595657E+01
	C	***9.9984864***E-01	9.9991728E-01	9.9986577E-01
	S	4.6094014 E-02	3.2860925E-02	2.2042156E-02
	r^2	9.6579273E-01	9.9155055E-01	9.9389568E-01
CAURIE	A	-5.3542922E+00	-8.963677E+00	-5.8860118E+00
	b	5.4353015E-02	9.8643388E-02	6.1617524E-02
	S	9.3439722E-02	9.5634732E-02	6.3774799E-02
	r^2	8.3600174E-01	9.1650760E-01	9.4038246E-01
PELEG	A	-2.2790668E-05	7.4502921E-02	-1.2596080E-07
	k	2.1485148E+00	3.8039716E+00	3.7199177E+00
	B	5.1676668E-06	-7.4503108E-02	7.6501601E-08
	q	2.6870899E+00	3.8039709E+00	3.8874526E+00
	S	1.35130297E-01	1.4905582E-01	9.4802419E-02
	r^2	7.5500705E-01	8.5512766E-01	9.0590064E-01

Table 8. Model parameters of Tegazza date desorption isotherms at three temperatures

Models	parameters	Temperatures		
		40°C	50°C	60°C
GAB	A	- 1.3538144E-01	-1.0631120E-01	-1.2812661E-01
	B	2.2469293E+01	1.1509816E+00	2.9950693E+01
	C	9.9961891E-01	9.9168426E-01	9.9970525E-01
	S	4.2051312E-02	2.0605969E-02	5.1158684E-02
	r^2	9.8166800E-01	9.8560635E-01	9.8124378E-01
Peleg	A	9.6031872E-01	3.6439506E-09	4.9769361E-09
	k	1.7766861E+00	4.1812376E+00	4.1275782E+00
	B	-9.6009453E-01	1.7125941E-01	1.0723777E-01
	q	1.7766861E+00	-6.4888186E-02	4.9980316E-02
	S	1.3579015E-01	1.6384339E-02	2.8781669E-02
	r^2	8.4707540E-01	9.9271998E-01	9.9525071E-01
BET	A	2.0398098E-01	1.4425471E+02	1.3298921E-01
	C	1.8043961E+00	1.2419379E-04	1.4677305E+00
	n	-1.6527645E-01	7.8424171E-01	-2.3463105E-01
	S	3.4129795E-02	9.0425647E-02	1.9655943E-02
	r^2	9.8792414E-01	7.2281607E-01	9.9723118E-01

Table 9. Model parameters of Bamakhlouf date desorption isotherms at three temperatures

Models	parameters	Temperatures		
		40°C	50°C	60°C
GAB	A	-1.1187980E-01	-1.1317039E-01	-1.1908188E-01
	B	1.4747643E+01	6.9670627E+00	6.0415004E+00
	C	9.9936626E-01	9.9869601E-01	9.9855391E-01
	S	3.9256465E-02	1.8564819E-02	1.8789033E-02
	r^2	9.9473497E-01	9.9785484E-01	9.96125917E-01
Peleg	A	4.3363487E-01	3.8895813E-09	-5.5001654E-01
	k	-3.4262062E-01	4.1914788E+00	2.4991706E+00
	B	7.796049E-11	5.2414657E-02	5.5002524E-01
	q	5.1308094E+00	2.2404252E-01	2.4991706E+00
	S	7.5851909E-02	8.0255226E-02	1.4094793E-01
	r^2	9.8427461E-01	9.6792881E-01	8.2559136E-01
BET	A	1.7965963E-01	6.9558197E+00	1.6357597E-01
	C	2.1273255E+00	7.1301578E-10	1.7340500E+00
	n	-1.3360631E-01	3.8934301E+00	-1.7686765E-01
	S	4.7175214E-02	1.4448316E-01	2.7244222E-02
	r^2	9.9239663E-01	8.7006898E-01	9.9185466E-01

6.3 Isosteric sorption heats determination

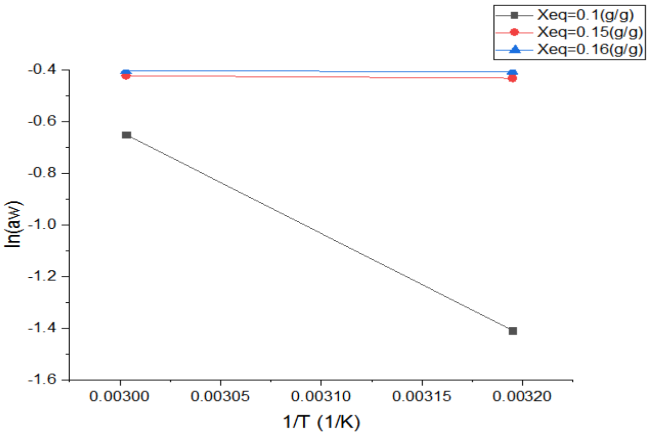

Figure 8. Desorption isosters for L'hmira dates

To calculate the isothermal heat of sorption, we use the data accompanying the GAB model in the results pane, set the water content value and record the water activity values for each experimental temperature, then plot the curves

$$\ln a_w = f\left(\frac{1}{T}\right) \qquad (1)$$

after that, we draw the curve

$$Q_{st} = f(X_{eq}) \qquad (2)$$

Desorption isosters for L'hmira, Tegazza and Bamakhlouf dates are illustrate in Figure 8, 10 and 12, respectively.

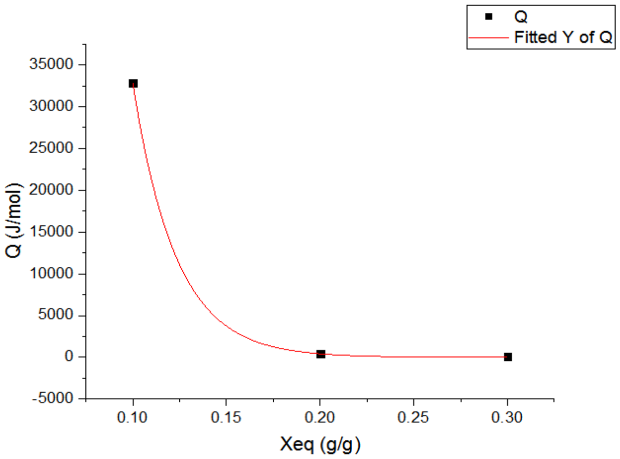

Figure 9. Net Isosteric heat desorption of L'hmira dates according to equilibrium water content

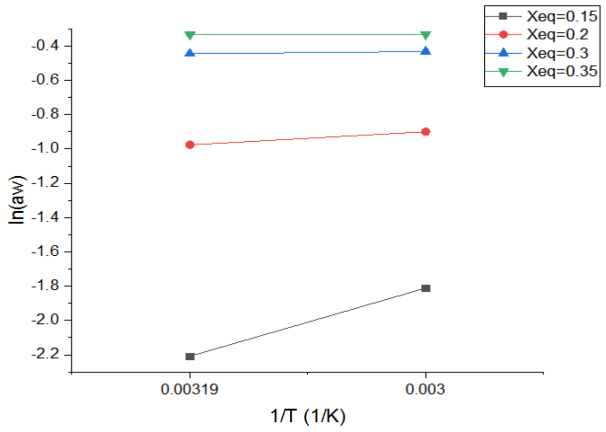

Figure 10. Desorption isosters for Tegazza dates

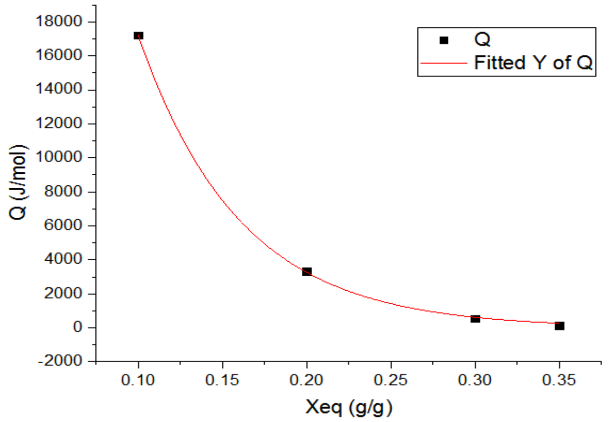

Figure 11. Net Isosteric heat desorption of Tegazza dates according to equilibrium water content

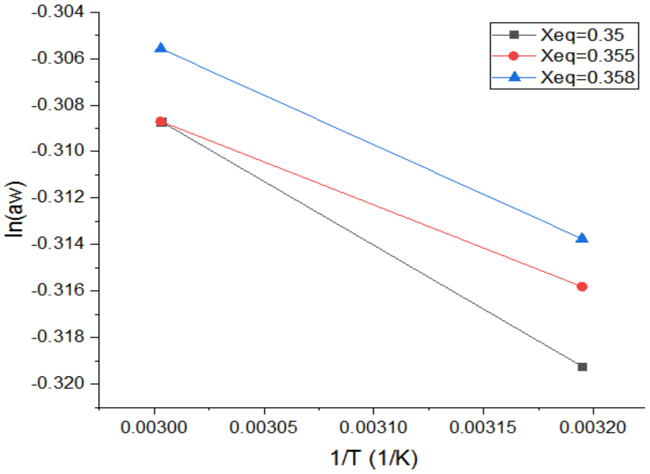

Figure 12. Desorption isostersfor Bamakhloufdates

In Figure 9, 11 and 13, there is also an increase in net desorption heat as the degree of dehydration increases. This is due to the existence of highly active polar sites in the product that are been covered by water molecule forming the molecular layer [20].

Nonlinear regression was used to evaluate the parameters of the empirical Eq. (2) with the Levenberg-Marquardt method (Table 10). These results are in perfect agreement with the results obtained by Tsami et al. [21] for other food products.

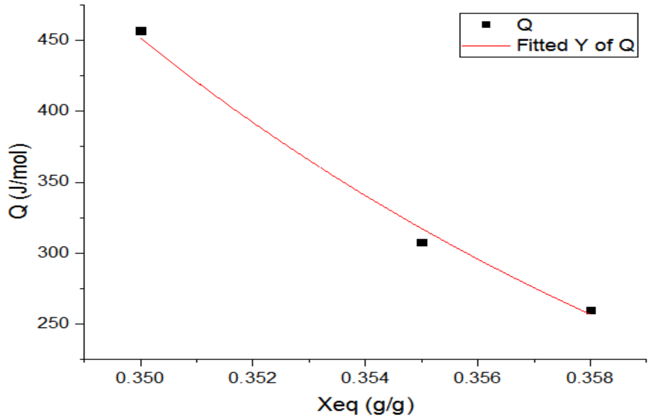

Figure 13. Net Isosteric heat desorption of Bamakhlouf dates according to equilibrium water content

Table 10. Ompirical parametres estimation

	Q_0(kJ/mol)	X_0(g H_2O/g MS)	r^2
L'hmira date	2515.87787723	0.023	0.99997
Tegazza date	90.78397681	0.060	0.99977
Bamakhloouf date	23.0067.E09	0.0142	0.99394

7. CONCLUSION

The objective, after having the experimental curves of the desorption isotherms curves of the three varieties of dates (L'hmira, Tegazza, Bamakhlouf) is to determine by following the most well-known models, by describing this phenomenon and of proposing a suitable model, thus calculating the isosteric heat and predicting the storage life.

Our study allowed us to conclude by noting, through previous theoretical and experimental work, that the determination of these curves is indispensable in the drying field and that the understanding of the equilibrium between the drying air and the wet product to be dried is imperative. Our determination that the BET model is suitable to describe the desorption isotherm of the L'hmira date, as well as the Peleg model is suitable to describe the isotherms of the desortions for both Tegazza and Bamakhlouf dates. At the same time the proposed model also has acceptable results, where the coefficient of determination (r^2) has exceeded 0.99 in most cases. On the other hand, we calculated the isosteric heat. And we left the door open to those who want to complete this study for determining the adsorption curves and the estimated lifetime.

The desorption curves of the three types of dates were in good agreement with those found in many previous scientific works. Indeed, the water content at equilibrium is inversely proportional to the temperature, this was very clear on the isotherms, when the activity of the water exceeds 0.4.

Finally, the smoothing of the experimental points allowed us to find experimental equations characterizing the desorption of the date at 40 °C, 50 °C and 60 °C.

ACKNOWLEDGMENT

The present work was ben effectuated in the solar drying

laboratory at the Thermal and Thermodynamics Conversion Division, within the Research Unit Energies in Renewable Energies in the Sahara Medium. Development Centre of Renewable Energies, Adrar, Algeria

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NOMENCLATURE

aw	water activity (dimensionless)
r^2	correlation coefficient [-]
T	temperature [°C]
Xeq	equilibrium moisture content [kg/kg]
Hr	relative humidity %
A,C,k,B, q,S	model coefficients
Q_0	net isosteric heat of desorption [J/mol]