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Sorption Isotherm Properties of Date (*Phoenix Dactylifera* L., Mejhoul Variety)

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Abstract

The objective of this study is to model the water adsorption isotherms of the dates of the Moroccan variety "Mejhoul" at three temperatures (25 °C, 30 °C and 40 °C) using the static gravimetric method. Experiments have measured the change in water content of samples according to water activity, with maximum values of 32% at 25 °C, 46.4% at 30 °C and 51.2% at 40 °C for water activities close to 95%. The experimental results were adjusted to GAB (Guggenheim, Anderson and Boer), BET (Brunauer, Emmet and Teller) and Langmuir models in order to determine characteristic parameters such as water content of the mono-molecular layer (Xm) and sorption heat isosteric. The GAB model was the most accurate, with a correlation coefficient of 99.5%. The net sorption isosteric heat varies between 37 and 44 kj/mol, decreasing gradually with increasing water content, indicating a reduction in affinity between water and fruit matrix as humidity increases. These results allow a better understanding of the hygroscopic behaviour of Mejhoul dates and provide useful indications for improving drying, conditioning and preservation processes.

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Introduction

In Morocco, dates are spread over an area of 50,000 hectares which represents about 5 million palm trees. The palm groves of Drâa Tafilalet contribute about 90% of the Moroccan date production, The date production in Morocco estimated at nearly one hundred thousand tons per year contributes to the generation of income 40% to 60% for nearly 2 million of the oasian population. However date production is subject to constraints related to good harvesting practices, post-harvest treatments, conservation and storage.[1]

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The variety Mejhoul considered among the noble dates in Morocco, it is known by its appearance and taste appreciated by the Moroccan consumer, this date variety is characterized by a high weight of an average value of 22.43 g also with a weight of pulp of 20.87 g and nuclei of 1.56 g, the length of this variable equal to 4.64 cm and with a width of 2.47 cm, the thickness equal to 0.70 cm.[2]

Water is the most abundant component of food. It is a key determinant of food storage and plays many roles in its processing.[3] During the food quality assessment, the moisture content of the food may be used as a relevant criterion that provides information on the degradation of the food quality). Water in food is generally measured in terms of water activity (aw), which can be measured as the ratio of the water vapor pressure at the product surface to the pure water vapor pressure (T) at the product temperature.[4]

The water content in a product and its temperature influences its water activity. An adsorption isotherm curve is called when the curve represents for a given temperature, the water content X of a product according to the value of the activity of the water aw or the relative humidity of the air in equilibrium HR, and we can distinguish between:

- Adsorption isotherm if experimentally determined from a dry product
- Desorption isotherm if experimentally determined from a water-saturated product.[4]

The interest in adsorption isotherms is significant, especially in food storage and preservation operations.[5,6]

In a given temperature, Sorption isotherms are curves of the X_{eq} equilibrium water content as a function of the value of water activity a_w [6,7] In fact In the operation of preservation or storage of food. The adsorption isotherm has the objective of determining the behavior of a product towards the conditions of the storage medium precisely the value of relative humidity influence on the stability of the product. The adsorption isotherm allows to estimate the amount of water that would be adsorbed according to the storage time and to predict the shelf life of the product under specified conditions.[8] In macroscopic chemistry the impact of water on reactions and food quality is more important than any other chemical constituent.[3] The functional importance of water in biological products goes well beyond its quantitative aspect. Water contributes to the texture and appearance of vegetables and plays a key role in the development of biochemical reactions and microbial growth.[3] Controlling the moisture content of food during processing is an old conservation technique, still widely practiced today.[9]

Recently, several studies have been carried out on food sorption isotherms, as well as on the influence of temperature on isotherms and the study of mathematical models describing sorption isotherms. For the mathematical modelling of sorption isotherms, several models have been proposed based on different theories. Some 80 correlations one based on theory, and the other models are purely empirical.[10,11]

Control of the hygroscopic properties of dates is essential to ensure their stability and preserve their quality throughout storage and marketing. Among the most popular varieties, the Mejhoul date occupies a special place due to its organoleptic characteristics and high commercial value. However, few studies have been interested in modelling the adsorption isotherms of this variety especially under different temperature conditions.

In this context, the main objective of this work is to experimentally determine the adsorption isotherms of Mejhoul dates at temperatures of 25 °C, 30 °C and 40 °C, using the static gravimetric method. The study also aims to model these isotherms by applying three theoretical reference models (GAB, BET and Langmuir) and to determine key parameters such as water content of the mono-molecular layer and net sorption heat. These results will improve the understanding of the hygroscopic behaviour of Mejhoul dates and optimize their drying, storage and conservation conditions.

Materials and Methods

Experimental Procedure

The dates of Mejhoul variety origin from the Er-Rachidia province (Morocco) used in this work, the sample studied is collected in the companion of the year 2021.

The equilibrium water content (Xm) is derived from the following formula:

Xm=(humid mass-dry mass)/(dry mass)

The dates were cuted into thin slices, 2 cm long and 1 to 2 mm thick. The samples weight was about 30 g placed in a stainless-steel capsule weighed beforehand, and the latter is placed in a glass jar containing sulphuric acid with different concentrations ranging from 10% to 85% to maintain the desired water activity in the medium. The glass pot is closed and placed in the oven (figure 1).

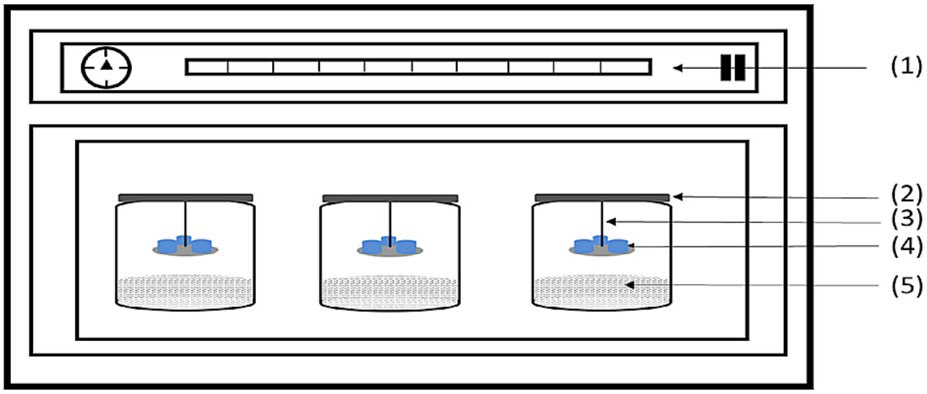

Fig.1: Experimental apparatus for sorption isotherm: (1) thermostated bath; (2) sample jars (3) sample holders; (4) date sample; (5) sulphuric acid solutions

The study is carried out on six samples in three temperatures 25 °C, 30 °C and 40 °C. Water activity up to 0.94,data showed in table 1.5

Samples are weighed every 24 hours until equilibrium is reached. Equilibrium is reached in 15 days.

To avoid the influence of the medium conditions on the sample, the weighing time is about 30 s. To determine the water content, the samples are placed in the oven under a temperature of 105 °C.

The weight of the sample is followed over time, and the balance is reached when three successive measurements almost do not change (the difference between three weighings is less than 0.001).[5]

Table 1: Values of water activity aw sulphuric acid solutions as a function of concentration (% vol./vol.) and temperature

Concentration (%vol/vol)	Temperature of the air solution (°C)		
	25 °C	30 °C	40 °C
10	0.9429	0.9427	0.9562
25	0.8756	0.8736	0.8785
40	0.5683	0.5656	0.5748
55	0.2598	0.2605	0.2675
70	0.0434	0.0453	0.0497
85	0.0016	0.0018	0.0023

Mathematical Treatment

To model the adsorption isotherms obtained by experiment method, there are many mathematical models in the literature for adjusting these isotherms. In the above table 2 the three models used in this work have been grouped:

GAB

To estimate the parameters of the models used in this work based on experimental results of sorption isotherms. Among the most widely used models is the GAB model, one of the best food models. It is widely used and accepted in food technology by all European researchers. It is used to describe food behaviour with water activities between 0.05 and 0.95. This model determines the monomolecular layer's water content, the monomolecular layer's sorption heat and multilayer. The GAB equation, is as follows:[11]

X= (Xm*aw*C*K)/((1-aw*K)(1+aw*C*K-aw*k))

Including:

X: Moisture content of the product; Xm: Water content corresponding to the occupation of all the sorption sites of the monomolecular layer of water in the product (expressed in % relative to the dry matter); aw: Water activity; C: GAB constant; K: Correction factor for multilayer properties.

GAB parameters depend on temperature and product, determined from the experimental results.

BET

In this work, we also used the BET model known by the following expression:

X= (Xm*aw*C) / (1-aw) (1-aw+aw*C)

Including:

C: BET constant; X: Moisture content of the product (g/100 g dry matter); aw: Water activity Xm: Water content corresponding to the monomolecular layer (g/100g dry matter).[12]

Langmuir

the Langmuir model is known by the following expression:[13]

X = Xm (C·aw) / (1 + C·aw)

Including:

Xm: Water content corresponding to the monomolecular layer (g/100g dry matter); C: Langmuir constant

For water activities not exceeding 0.90, the GAB model can correctly write the adsorption isotherms of agri-food products. on the other hand, the use of the BET model is limited.[5,14]

Isosteric Sorption Heat

The isosteric sorption heat is an important parameter. The main objective of this measurement is to allow the calculation of the integral sorption heat to estimate the amount of heat needed to dry the product. The most common method used to calculate sorption heat is the Clausius-Clapeyronl equation: [5]

$$\ln a_w 1/a_w 2 = \Delta Hb/R(1/T1-1/T2)$$

ΔHb is sorption heat (kJ/mole). $a_w 1$ and $a_w 2$ are water activities at T1 and T2 temperatures respectively, R is a gas constant. The isosteric heat (Qst) is then calculated using the equation:

Qst = ΔHb + ΔH0

Including:

ΔH0 is the heat of vaporization of pure water.

Data Analysis

A non-linear regression was performed using the CurveExpert Professional 2.7.1 software to estimate the parameters of the mathematical models GAB (Guggenheim, Anderson and Boer), BET (Brunauer, Emmet and Teller) and Langmuir. These models have been more widely used in the literature for several years for food and feed, but there is a difference between the two models. Now there is a favor of the GAB model to describe the water sorption isotherms of food products, compared to the full range (0.1 to 0.9) of water activity. The BET model is now less cited but still used in some occasions.[12] The quality of the fit of the models to the experimental data was assessed by the adjusted coefficient of determination (R^2).

Results

Adsorption Isotherms

The results of adsorption isotherms at three temperatures (25 °C, 30 °C, and 40 °C,) are presented in the table 3.

The table below the evolution of water activity (aw) and water content at three different temperatures: 25°C, 30°C and 40°C. At 25°C, water activity varies from 0.9429 to 0.0016, with a water content increasing from 32% to 3.1%. At 30°C, the water activity fluctuates between 0.9427 and 0.0018, and the water content varies from 46.4% to 3.4%. At 40°C, the water activity reaches 0.9562, corresponding to a water content of 51.2%, and decreases with decreasing water activity.

Adsorption of water is essential when the activity of water is maximum. In shallow water activities, the adsorption is also low, which is true for all three temperatures (25°C, 30°C and 40°C)

Table 2: Mathematical models of adsorption isotherms

Auteurs	Models	Parameter
Guggenheim Anderson Boer (GAB)	X= (Xm*aw*C*K) / (1-aw*K) (1+aw*C*K-aw*k)	Xm C K
Brunauer Emmet Teller (BET)	X= (Xm*aw*C) / ((1-aw) (1-aw+aw*C)	Xm C
Langmuir	X = Xm (C·aw) / (1 + C·aw)	C Xm

Xm: Water content corresponding to the monomolecular layer
C: GAB constant or BET constant or Langmuir constant
K: GAB constant
a_w: Water activity

Table 3: Adsorption isotherms of date Mejhoul variety (25 °C,30 °C and 40 °C)

Temperature	water activity	Water content (%)
25 °C	0.9429	32
	0.8756	14.3
	0.5683	5.9
	0.2598	3.5
	0.0434	3.3
	0.0016	3.1
30°C	0.9427	46.4
	0.8736	14.7
	0.5656	5.7
	0.2605	3.5
	0.0453	3.4
	0.0018	3.4
40°C	0.9562	51.2
	0.8785	15.1
	0.5748	6.0
	0.2675	2.3
	0.0497	2.1
	0.0023	2.2

Mathematical Modelling

The experimental values are adjusted using Cuve expert basic software 2.2.3 the model parameters results are presented in the following table:

The table below, which presents the adsorption isotherms of dates from the Majhoul variety, indicates that the experimental and calculated data curves require further clarification. The models were assessed by comparing their correlation coefficients R2. In general, the R2 values for all models at the temperature set are greater than 0.9 except for the Langmuir model. This means that the models agree with the experimental results. The GAB model provides a better fit, with high correlation coefficients of 99.5, 99.6, and 99.9 for temperatures of 25°C, 30°C, and 40°C, respectively, compared to 98.6, 99.8, and 99.7 for the BET model.

Table 4: Parameters result of the three models for the temperatures 25 °C, 30 °C and 40 °C

Model	Parameter	Temperature		
		25 °C	30 °C	40 °C
GAB	Xm	2.065709056046638	1.732844370594615	1.790881794032999
	Standard Error	1.436646968808225	1.910605308709850	1.113754914153893
	Correlation Coefficient	0.9952499142	0.996206527	0.99900045
BET	X_m	3.112477346173817× 10	4.427560955342391	$2.372330733966039\times 10^2$
	Standard Error	2.448801775505522	1.343883792574919	1.674321052020111
	Correlation Coefficient	0.986135731	0.9981250003	0.997739658
Langm -uir	X_m	$1.401876328283412\times 10^4$	$2.295657312546490\times 10^4$	$2.695080338394310\times 10^4$
	Standard Error	7.294811118197206	1.246904066203142× 10	1.406720521427568× 10
	Correlation Coefficient	0.821090256	0.75495933	0.75828695

Table 5: The sorption heat values for the temperatures 25 °C

a_w(25°C)	Water content (%)	a_w (40°C)	Water content (%)	∆Hb (j/mol)	Qst (j/mol)
0.9429	32	0.9427	51.2	10.9778	44055.69
0.8756	14.3	0.8736	15.1	118.3387	44163.33
0.5683	5.9	0.5656	6	246.4483	44291.16

Net Isosteric Heat of Sorption

The sorption heat values are calculated is presented in the following table 5

The table below presents water activity aw values at 25°C and 40°C, along with the corresponding water content. As aw decreases, the water content generally declines. The differential enthalpy (∆Hb) shows positive values at higher aw and negative values at lower aw. The isosteric heat of sorption (Qst) remains relatively stable within the range of approximately 38,000 to 44,500 J/mol for medium aw values but decreases at very low aw levels.

Discussion

The adsorption isotherm in the three temperatures (25 °C, 30 °C and 40 °C) (table 3) of the Moroccan dates variety Mejhoul have a sigmoidal form of type II,[15] same result found by Zhang *et al*,[16] A type II sigmoid isotherm according to the Brunauer classification, characteristic of biological products such as oil-rich tubers, has been observed, where the water content at equilibrium of tiger nuts slowly increases with low water activities.[16] This type of curve is known by the fruits rich in carbohydrates. 5,9Most food sorption isotherms are divided into three regions and have a sigmoid form, according to Lewicki.[9] The results obtained are similar to several results of the sugar-rich products, dried apricots (*Prunus armeniaca* L.),[17] fig (*Ficus carica* L.),[9] Banana (*Musa spp*.), Mango (*Mangifera indica* L.), and Pineapple (*Ananas comosus* L.).[18]

The water activity value between a_w =0 and a_w =0.4 is called the monolayer, in which the water molecules are firmly bound. Van der Waals' strength in this area is significant. The product water molecules in this interval are stable due to the action of the Van der Waals force between these molecules and hydrophilic groupings. Thus, water molecules with high sorption heat cannot participate in any reactions. The increase in water activity values influences the state of carbohydrates, as there is a transition from a crystalline to an amorphous state, along with the dissolution of carbohydrates. The increase in the amount of water absorbed causes an increase in adsorption sites. Thus, the crystalline or amorphous state of carbohydrates influences water retention.[5,12,17]

Note that for water activity below 0.9, the water content does not change with large values. For water activities above 0.9, the water content is essential. It is also noted that for the same water activity, the water content at temperature 25 °C is higher than the water content at temperature 40°C. This is noticed when the water activity is less than 0.9. The results found are similar to that found by Benseddik *et al*.[19] Most carbohydrate-rich foods have the same behaviour, which can be explained by the increase in sorption heat (high water binding energy).[5] For water activity which is less than 0.9, we notice the opposite. The water content at a temperature of 40 °C is higher than at a temperature of 25°C. This observation can be explained by the change of the sorption property of the dates to the temperature and the activity of high water. The state of carbohydrates influences adsorbed water. At low water activity, the crystalline state absorbs smaller amounts of water than the amorphous state, which absorbs more water,[17] according to Shweta Yadav The equilibrium moisture of the sample increases with water activity, which could be explained by the increase in porosity and water content of the powder at higher levels of water activity. This increase in product moisture promotes a high hygroscopicity. Furthermore, the potential for water fixation by the powder decreases as the temperature increases.[20] In another study Adil *et al* showed that the equilibrium moisture content decreases with increasing temperature, while the physical state of the material and water activity influence water retention. The material gradually reaches its hygroscopic equilibrium during storage.[21]

The results found are similar to that found by Belarbi,[12] who worked on eleven dates variety, the curve shape is sigmoidal type II according to Brunaue.[12] The behavior of dates sorption isotherms studied by Belarbi [12] is similar to that of our studied variety (Mejhoul), the difference is in the value of the water activity in which we have a high water content 0.9 for the variety Mejhoul and 0.5 for the variety studied by Belarbi[12] and 0.8 for the variety Deglet Nour.[5]

After adjusting the experimental values to the mathematical sorption models (table 4), we can conclude that the GAB model correctly describes the adsorption isotherms of the Mejhoul variety dates, the GAB model is suitable for most carbohydrate-rich foods, the isotherm of dry apricot adsorption[17] and date varieties.[5] However,According to the study conducted by Belarbi[12] the BET model correctly describes the adsorption isotherms of the [11] varieties of dates, which can be explained by their composition and behaviour under storage conditions.

The table 4 presents the water content values of the monomolecular layer (Xm) determined using the GAB, BET, and Langmuir equations. The Xm values obtained from the BET equation are slightly higher than those determined by the GAB equation, a finding consistent with the observations of Belarbi.[12]

Date adsorption isotherms are influenced by temperature and water content, which changes the affinity of water for the substrate. At ambient temperature and low water contents, the adsorption of water is usually stronger (table 5) due to the existence of many sorption sites available. As the water content increases, the interaction between water and product components becomes more complex as sorption sites fill up, changing the sorption heat. This can be observed by the variation of the values of ΔHb and Qst.[16,22]

The calculated ΔHb values change inversely to the water content values. These results are similar to those found by Ferradji.[5] The results shows that the sorption heat decreases when the increase in water content can be attributed to the strong interaction between the adsorbent components of the date and water molecules. Additionally, the reduction in available sorption sites results in a decrease in the ΔHb value.[17]

At low moisture content (< 0.06), the binding energy (Δ Hb) is negative, indicating an exothermic interaction, where water is strongly bound to the substrate, and its adsorption releases energy. This is typical of processes where a monolayer of water molecules is formed, requiring a significant amount of energy to be removed, as observed by Lahsasni *et al*.[23] As the moisture content increases, the binding energy decreases and approaches zero, indicating that the isosteric heat of sorption becomes equal to the heat of water condensation (Qst = Δ h0), meaning a weaker interaction between water and the substrate.

At higher moisture contents, the heat (Δ Hb) becomes positive, suggesting an endothermic interaction where energy is required to bind the water, typically due to the saturation of available sorption sites. This explains the reduction in sorption heat as the moisture content increases.

These results are similar to those reported by Samapundo *et al*.[24] who observed a similar trend for various food products. In the case of dates, as moisture increases, the interactions between the components and water decrease, reducing the energy required for adsorption.[22]

Conclusion

This study determined experimentally the sorption isotherms of dates of the Mejhoul variety at 25°C, 30°C and 40°C. Analysis of the results showed that the equilibrium water content increases with activity in water at constant temperature and decreases with temperature increase for a given water activity. Three models (GAB, BET and Langmuir) were applied to adjust the experimental data. Among them, the GAB model presented the best fit, making it particularly suitable for describing the hygroscopic behavior of Mejhoul dates. In addition, the sorption isosteric heat, estimated at 37 to 44 kj/mol, showed that the binding energy between water and solid increases as the water content decreases.

These results provide useful data for the optimization of storage conditions of Mejhoul dates, in particular the control of humidity and temperature to limit the risk of degradation.

For future research, it would be interesting to study the sorption behavior after different drying or storage methods, as well as to assess the impact of these conditions on the microbiological and nutritional quality of dates during storage.

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This research did not involve human participants, animal subjects, or any material that requires ethical approval.

Informed Consent Statement

This study did not involve human participants, and therefore, informed consent was not required.

Clinical Trial Registration

This research does not involve any clinical trials.

Permission to Reproduce Material from Other Sources

Not Applicable.

Author Contributions

- **Oubouali Morad:** Wrote the document, revised it, and edited it to ensure clarity and coherence.
- **Zine-eddine Yassine and Ajbli Nouhaila:** Edited the text and reviewed it for structure and logic.
- **Ellaite Mohammed, Kzaiber Fouzia, and Oussama Abdelkhalek:** Reviewed the content, checked the data, and edited to improve overall clarity.
- **Boutoial Khalid:** Supervised the entire process, ensuring scientific quality and guiding all contributors.

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