

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Prediction of Optimum Humidity Condition for Storage of Some Egyptian Dry Fruits and its Effect on Physical Properties of Fruits

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Abstract

This work aimed to establish sorption isotherms curves for the dry fruits of apricots, raisins and Saidi dates and their relationship to the expected shelf life, color and hardness of the tested fruits. Results showed that the obtained sorption curves are of sigmoidal shape and that content and type of sugars in the fruits is crucial for the level of equilibrium moisture content (Me) at water activity level of 0.11 to 0.85 at 25±5℃, whereby sucrose rich fruits (apricots) recorded Me – values lower than those of high levels in reducing sugars (raisins and Saidi dates). GAB and Halsey isotherms equations were suitable for presenting sorption curves of raisins and dates (Relative deviation percent" P" <10%), while Henderson – equation gave a high R^2 – value of 0.93 for apricot, s sorption data but with patterned residual (P%). Monolayer moisture content Mm was in the range of 0.0547 (apricots) and 0.119 to 0.147 g $_{H2O}/$ g_{DM} for raisins and dates. Expected shelf life of the tested dry fruits was estimated from the slope of multilayer portion of the sorption curves to be 7.16 to 7.40 months at 25℃ and 70% relative humidity in the surrounding atmosphere outside a polyethylene package containing the dried fruits and it could be extended to 10-11 months if surrounding relative humidity maintained at 50% level. At water activity levels 0.11 and 0.22, the fruits showed high hardness values of 1738 to 1412 N/m² for dry apricots and raisins, while dates showed values of 198 to 84 N/m². Increasing aw – level to 0.85 drastically reduced hardness values. L, b, chroma and Hue∘- values of the dried fruits were decreased by increasing a_w – level from 0.11 to 0.85, while redness (a-values) were increased. Browning reached a maximum at 0.7 to 0.75 a_w – level and decreased at lower a_w – values.

Keywords: dried fruits, sorption isotherms, apricots, raisins, Saidi dates, shelf life, color, hardness

1. Introduction

Dry fruits of apricots (*prunus armeniaca L*.), raisins (*Vitis vinifera L*.) and dates *(phoenix dactylifera*.) are consumed in large amounts in Egypt, especially during the fasting seasons. They are very nutritious and could be eaten in dry form or after rehydration. On other side, Egypt as the leading country in date palm cultivation, produces 17% of the annually 8 million tons of date fruits produced worldwide. Saidi date variety is a semi dry date fruit representing13% of Egypt's dates production, considered one of the best varieties suitable for packing, processing and export. [1, 2]. Apricots and raisins were produced from fresh fruits after sulfuring treatment and drying, while dates were harvested at the end of semi-dry phase and were marketed at a moisture level of 18-20%. However, this low moisture content can keep the product safe for long time provided that the storage conditions (temperature and relative humidity) are in the suitable range to prevent microbial and biochemical deterioration and to avoid weight loss, structure collapse and brittleness, [3, 4,5,6] . According to [7], water activity gives more information than moisture content because it is the basic controlling factor in the preservation of dried fruits (especially rate of biochemical reactions). Understanding interaction of H₂O with dry fruits is useful for better storage conditions and more suitable packaging. Therefore, the moisture sorption isotherms experiments would allow correctly finding the suitable relative humidity, water activity (a_w) and equilibrium moisture content specifying safe storage, packing, handling and shelf-life prediction of the product and understanding incident changes.

The equilibrium state will be attained when the water vapor adsorption or release between the active sites of the food and the acting vapor pressure in the surrounding atmosphere ceased and the food approached the corresponding equilibrium moisture content (Me), and a sorption isotherms curve $(a_w$ versus Me) could be established for the specific tested food product. Mathematical modelling used to describe sorption isotherms curves of food are important for drying and storage processes as well as for shelf-life prediction of the food [8]**.** They give important information about suitable end point of drying as well as safe storage and stability of the dry product [9]. Depending on the parameters included in the model for describing sorption curves, the models are of one, two, three or more parameters [10]**.** There are several researches have been carried out to establish sorption isotherms curves for apricots, [5, 8, 11, 12] for raisins, [13,16] and for date fruits, [6, 17,27]**.** Most of these researches studied fruits grown and stored in other countries using fruit varieties other than those grown or marketed in Egypt. Therefore, the aim of this work was to establish sorption isotherms curves for Saidi dates,

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apricots and raisins consumed in Egypt, mathematical modelling of the sorption curves to obtain useful data for optimum fruit storage and Studying effect of water activity levels on color parameters and hardness of the tested fruits.

2. Material and Method

2.1. Materials

Dry and sulfured apricots and Thompson seedless raisins, were of Turkish import, while Saidi dates (of average 18% moisture content) were purchased from a central Nuts and dry fruits packaging and distribution company in Cairo. Good quality fruits with variety specific appearance and a normal size were used. Samples were thoroughly mixed to ensure uniformity. In order to preserve the initial characteristics and avoid any alterations that may lead to change in properties, the samples were stored at +4°C until analysis. All used chemicals and reagents were of analytical grade and purchased from Al Goumhoria Company for Medicines and Medical Supplies, Cairo -Egypt.

2.2. Methods

2.2.1. Proximate composition and some physical properties of the fruits:

Moisture content; total, reducing and non-reducing sugars, crude fibers, crude Protein (N x 6.25), crude fates and ash were determined according to AOAC [28]. Water activity of the raw dry fruit samples was measured by hygrometric method throw analyzing the relative humidity of air surrounding tested samples using a water activity device (Aqualab, Novasina AG., Switzerland) at 25°C as described by [12]. The device was calibrated by saturated solution of lithium chloride ($a_w = 0.112$). Hardness of the fruits, was measured by an automatic penetrometer (AP 4/3, Feinmess, Dresden, Germany) by using a cone of 37◦, total load of 203.5 g or 109.3g, according to the expected hardness of the fruit, and a total penetration time of 20 seconds. The penetration depth (in cm) was changed to hardness value $(N/m²)$ according to following formula,[29].

$$
Hardness = \frac{P\cos(\epsilon)}{\pi \cdot d^2 \cdot (\tan \epsilon) * 10} \qquad \qquad \dots \dots \dots \dots \dots \dots \dots (1)
$$

Where: P = total weight of penetration device (g), ϵ = slope angle of the penetrating cone and (d) = penetration depth (cm) .

Color parameters of the fruits were measured according to the tristimulus X (red), Y (Green) and Z (blue) coordinates of CIE – color space using a Spectrophotometer (MOM, 100 D, Hungary). Values of X, Y and Z were converted to Hunter L, a and b- parameters applying formulas of the producing company. At least five replicates were taken for each sample and averaged. Chroma, Hue angle and Browning index (BI-Values) were calculated from basic L, a and b – values as described by [30].

2.2.2. Determination of equilibrium moisture contents:

The standard gravimetric method was used to obtain equilibrium moisture content (M) of the fruit samples as described [31]. In this method, saturated solutions of lithium chloride, potassium acetate, Magnesium chloride, Barium carbonate, Barium nitrate, sodium nitrate, sodium chloride and potassium chloride were placed separately in 8 glass desiccators to achieve an atmosphere of 0.11, 0.22, 0.33, 0.45, 0.55, 0.65, 0.75 and 0.85 water activity (a_w) , respectively at 25±5°C. According to [21,32] ,differences in equilibrium moisture content at 20 to 40°C are negligible. Duplicate samples of whole fruit (raisins) and halved fruits (apricots and dates) of about 10g each were spread inside petri dishes and placed on the perforated porcelain stage above the corresponding saturated solutions. 5 ml of 1% sodium azide were added to salt solutions of 0.75 and 0.85 a_w to avoid mold growth. The samples were weighed using analytical balance with accuracy of 0.001g on intervals of two days until constant weight has been achieved and the corresponding final moisture content was assigned as the equilibrium moisture content (M_e) and a sorption isotherms curve $(M_e$ versus a_w) was established.

2.2.3. Fitting sorption data to isotherms equations

According to [33] as well as., [34] sorption isotherms curves of sugar-rich foods could be represented by several isotherms equations as follows:

Henderson:

$$
\log(-\ln(l - a_w)) = B \cdot \log(M_e) + \log(AT) \dots (2)
$$

Halsey: Ln M_e = $\frac{1}{B}$ ln A - $\frac{1}{B}$ [ln (ln (- $\frac{1}{a_w}$)].... (3)

Iglesias and chirife: Ln [M^e + & + .' (] = β. aw + A ……………………………………………. (4)

GAB :
$$
\frac{a_w}{M_e} = α
$$
 . $a_w^2 + β$. $a_w + γ$ (5)
BET : $\frac{a_w}{M_m(1-a_w)} = \frac{1}{M_m} + \frac{C-1}{M_m} \cdot a_w$ (6)

Where: a_w = water activity (-), M_e = Equilibrium moisture content (g_{H2O} / g_{DM}), M_m = Monolayer moisture content (g_{H2O}/g_{DM}), T= Absolute temperature (k), M 0.5 = Moisture content at $a_w = 0.5$ (g_{H2O}/g_{DM}), C = Constant related to sorption heat of monolayer water molecules adsorbed on the active sites, A and B are constants and T is temperature (K). GAB is (Guggenheim – Anderson - Deboer) and BET is (Brunnauer, Emett and Teller) [35, 36]. The constants of GAB equation (α, β, γ) contain parameters related to binding energy (C and K) for first and condensed water molecules on adsorption sites of fruits as well as indicator for the monolayer water molecules (M_m) as follows:

α = 2.*, -/ ………………………………………. (7) β = (*, / − 1 ……………………………….(8)

 $\gamma = \frac{1}{\gamma}$ *, ./.2 ………………………………………(9)

The validity of the applied moisture isotherms equations for presenting the sorption behavior of the fruits is judged by three statistical parameters and by comparison between experimental(M_e) and calculated(M_{ca}) as follows: [37-39] Correlation Coefficient (R^2)

Sum squares of error (SSE) =
$$
\sum_{i=1}^{N} (M_e - M_{ca})^2
$$
-(10)

Root mean square of error, (RMSE):

RMSE = $\left(\frac{SSE}{df}\right)$;< (------------………………………..(11) Mean relative percent of deviation (P%): $P\% = \frac{100}{N} \sum_{i=1}^{N} \frac{M_e - M_{ca}}{M_e}$ \boldsymbol{n} 78 ------------------………….(12)

The BET equation is valid for only M_e range= 0.11 to 0.55 and provides value of M_m .

Calculation of excess energy required for water evaporation during drying (q_{st}) could be estimated from sorption data according to following equation [40 ,41,34]., as follows:

Ln aw = A B.C ………………………………………(13)

where: $R = Gas constant (8.31 J/mole H₂O vapor)$

2.2.4. Shelf-life prediction (τ^s):

(**τs**) estimation could be obtained from sorption isotherms curves according to following equation [26]:

 $\tau_s = \frac{W_s}{4}$ $\frac{N_S}{A} \cdot \frac{X}{P_e}$ $\frac{X}{P_e} \cdot \frac{b}{P_e}$ $\frac{b}{P_0}$. $Ln \frac{M_0 - M_i}{M_0 - M_C}$ ………………………….(14)

where: W_s = dry weight of fruits inside a flexible bag (g_{DM}), A = area of package (m²), X = thickness of packaging film (μ m), P_e = water vapor permeability of packaging film (for polyethylene: 100 g_{H2O}. μ m / m².d.KP_a), b = slope of sorption isotherms curve in multilayer water region between $a_w = 0.2$ and $a_w = 0.55$ (in g_{H2O}/g_{DM} unit a_w), P_0 is the saturation pressure of water vapor in surrounding atmosphere at 25°C (2.1 K_{Pa} for 70% relative humidity), M_m , M_i and M_c are maximum allowed safe, initial and critical moisture content of dry fruit (g_{H2O}/g_{DM}) .

2.2.5. Statistical Analysis

All experiments were carried out in triplicates. Means, standard deviation, SSR, RMSE and R^2 were computed by applying linear and non – linear regression programs of SAS - statistical package SAS, [42] at a confidence level of 95% .

3. Results and Discussion:

3.1. Proximate composition and some physical characteristics

Composition of the dry and semi – dry fruits is essential for the interpretation of their sorption behavior during storage.

Table (1) Shows the proximate composition of the tested dry fruits. The moisture content of the fruits was in the range of 16.92% to 17.52%, which agree with the Egyptian standard ES, [43] for dry and semi-dry fruits. The Saidi date fruits showed the highest total sugar content (70.71%) followed by raisins (69.42%) and apricot (54.40 %). Date fruits showed the highest content a reducing sugar (68.60 %), followed by raisins (65.38 %), while apricots showed the lowest content in reducing sugar (20.46 %). The major sugar in apricots was of the non- reducing type (34.26%), while dates and raisins showed very low content of non- reducing sugar (3.47 and 3.91 %, respectively). The highest crude fiber content was found in apricots (15.96 %), followed by raisins (5.25 %) and dates (3.92 %). Similarly, apricots contained the highest protein and ash content (5.12 and 3.95 % respectively), while their content in raisins and date was 2.71, 2.57, 2.47 and 2.20 %, respectively for protein and ash content. Saidi dates showed the highest fat content (1.49 %) compared with raisins (0.23%) and apricots (0.48 %). Saidi date fruits were very poor in acids (0.152 %), compared with raisins (1.37 %) and apricots (1.54 %). Based on the aforementioned data, the sugar /acid ratio for the tested dry fruits was 473.87 for Saidi dates, while raisins and apricots showed moderate sweetness ratio of 50.53 and 35.32, respectively. The measured water activity (a_w -values) for the tested dry fruits were 0.66, 0.67 and 0.71, respectively for Saidi dates, raisins and apricots. Hardness results showed that the dry apricots possess the highest hardness value (53.07 N/m^2) followed by raisins 36.16 $N/m²$, while Saidi- dates was relatively soft with a hardness value of only 8.80 $N/m²$, which agree with the crude fiber content of the tested fruits (15.96, 5.25 and 3.92 %, respectively for apricots, raisins and Saidi dates). Also, the percent of reduced sugars influences the moisture binding and consequently the softness degree of the fruits. Apricots showed the lowest content a reducing sugar (20.46 %), while Saidi dates contained the highest ratio of reducing sugar (68.60 %). The color parameters of the tested fruits are also given in Table (1). Dry apricots and raisins showed, respectively, Hue°- values of 90.31°, and 93.43°, which speaks for an almost pure yellow color type, while Saidi dates showed yellow-orange color type with a Hue -value of 71.08°. Correspondingly, apricots and raisins showed higher chroma and browning index values than those of Saidi dates.

The obtained results agree with those of [11,44,45,46] .They reported moisture content in the average range of 19, 17.45 and 19.67 %, respectively for dry apricots, raisins and dates. The corresponding average dry matter was in the range of 81-82.55 % of the tested dry fruits. Reported total sugars of dry apricots were in the range of 55±4.6 g/100 g dry fruits consisting of sucrose (61.84 %) and reducing sugars (38.16 %). On contrary, raisins and dry dates showed total sugar content of 69.42 to 70.71%, being 130 % higher than those of apricots with reducing sugars making 92 to 95 % of the total

sugars. Also, the obtained fibers, protein, fat and ash contents (Table 1) agree with those reported by the aforementioned researches. Measured values of water activity agree to a great extent with the a_w – values reported by [47] for apricots (0.62-0.69), [44,48] for raisins (0.425 to 0.6) as well as [49] and[11] for dry dates (0.66 to 0.693). In Table (1), measured initial Hue◦ color parameter for apricots and raisins are slightly higher than those reported by [50] due to differences in variety and applied pre sulfurization method. However, the results agree with those reported by [5,46,51]. The measured initial hardness values of the tested dry fruits agree with those reported by [11,52] where they reported hardness values of 2.2 N to 40 N according to the moisture content and type of fruit.

3.2. Equilibrium moisture content of the dried fruits: -

Figure (1) shows the course of moisture change during incubation time at different aw - levels (0.11 to 0.85) at room temperature (25°C) until final equilibrium has been attained for dry apricots, raisins and Saidi dates. The equilibrium moisture content of each tested dry fruit is defined as the final moisture content, when the curve of moisture change has been flattened versus humidity a_w - level. The initial moisture content (on dry weight basis) was 0.211, 0.203 and 0.207 g_{H2O}/g_{DM} for apricots, raisins and Saidi dates, respectively. As seen in Figure (1), it took 30 to 40 days to reach the final equilibrium moisture content. For apricots, the equilibrium moisture content has been achieved by moisture loss until a_w value of 0.65, while for raisins and Saidi dates, moisture losses have been maintained until aw-values of 0.45.

Component	Saidi date	Raisina	Dry apricot		
Moisture	17.52 ± 0.26 ^A	16.92 ± 0.25 ^A	$17.19 \pm 0.12^{\rm A}$		
Total solid	82.52 ± 0.29 ^A	83.04 ± 0.58 ^A	82.87 ± 0.57 ^A		
Total sugars:	70.71 ± 0.67 ^A	69.42 ± 0.29 ^A	$54.40 \pm 0.23^{\rm B}$		
Reducing sugars	68.60 ± 0.13 ^A	$65.38 \pm 0.09^{\rm B}$	$20.46 \pm 0.14^{\circ}$		
Non-reducing sugars	$3.47 \pm 0.27^{\rm B}$	$3.91 \pm 0.06^{\rm B}$	34.26 ± 0.09 ^A		
Crude Fiber	$3.92 \pm 0.01^{\circ}$	$5.25 \pm 0.03^{\rm B}$	15.96 ± 0.34 ^A		
Crude protein	$2.47 \pm 0.04^{\circ}$	$2.71 \pm 0.06^{\rm B}$	$5.12 \pm 0.06^{\rm A}$		
Crude Fats	1.49 ± 0.06 ^A	$0.23 \pm 0.01^{\circ}$	$0.48 \pm 0.05^{\circ}$		
Ash	$2.20 \pm 0.01^{\circ}$	$2.57 \pm 0.01^{\rm B}$	3.95 ± 0.03 ^A		
Total acids	$0.152 \pm 0.00^{\rm B}$ (as citric)	$.37 \pm 0.01^{\circ}$ (as tartaric)	$1.54 \pm 0.01^{\rm A}$ (as malic)		
Sugar/Acid Ratio	$437.87 \pm 1.76^{\rm B}$	50.53 ± 0.29 ^A	$.35.32 \pm 0.18$ ^C		
Water activity (a_w)	$0.66 \pm 0.01^{\rm B}$	$0.67 \pm 0.00^{\rm B}$	$0.71 \pm 0.00^{\rm A}$		
Hardness (N/m2)	$8.80 \pm 0.01^{\circ}$	$36.16 \pm 0.01^{\rm B}$	$53.07 \pm 0.04^{\text{A}}$		
Color parameter:					
	$23.78 \pm 0.05^{\rm B}$	$40.27 \pm 0.04^{\rm A}$	40.40 ± 0.06 ^A		
A	$5.23 \pm 0.02^{\rm B}$	$4.26 \pm 0.03^{\circ}$	$6.25 \pm 0.03^{\rm A}$		
B	15.64 ± 0.12 ^C	41.42 ± 0.29 ^A	$40.75 \pm 0.09^{\rm B}$		
Chroma	16.53 ± 0.02^C	41.37 ± 0.04 ^A	$41.23 \pm 0.02^{\rm B}$		
Hue ^o	$71.08 \pm 0.05^{\circ}$	$93.43 \pm 0.06^{\rm A}$	$90.31 \pm 0.06^{\rm B}$		
BI	$59.28 \pm 0.06^{\circ}$	$86.73 \pm 0.02^{\rm B}$	89.53 ± 0.08 ^A		

Table (1): Proximate composition (% wet bases) and some physical characteristics of Saidi date, raisins and dry apricots

A, B & C: There is no significant difference (P>0.05) between any two means, within the same row have the same superscript letter.

At aw-values of 0.65 or higher, the equilibrium moisture content has been attained by gain through adsorption of water vapor molecules on the surface of polymers present in the fruits (Polysaccharides and proteins) and by dissolving the di-and mono- saccharides at a_w-level 0.75 and higher. The equilibrium moisture data of Saidi dates were higher than those of raisins and apricots (Table 2). At water activity level of 0.85, the equilibrium moisture content of apricots was relatively low $(0.38 \text{ g H2O/g DM})$, while those of raisins and Saidi-dates were high in the range of 0.59 to 0.63 g $_{H2O}$ /g $_{DM}$. The difference in M_e-values for the tested fruits could be referred to their sugar content and type. Apricots contains 54.41% sugars mainly from disaccharides, while raisins and Saidi dates contains higher sugar contents (69.28 and 72.04) mainly of monosaccharides (especially fructose), which are more hygroscopic than sucrose. The equilibrium moisture curves obtained in the present work (Figure 1) agree with those published by [53] .

Moisture equilibrium curves could be analyzed according to [54] using following equation:

M = Meⁱ ± KƮ -----------------------……………..(15)

Where: M_i = initial moisture content (dry basis), M = moisture content at any time (T) during the period of sorption experiment until equilibrium, $T=$ Time of contact (days) and $K=$ The rate of moisture desorption or adsorption (g_{H2O} / g_{DM} d).

Table (2) includes the K-values of the tested dry fruits. The negative sign means that the equilibrium has been achieved by moisture loss (Desorption), while positive sign means that it had been achieved by adsorption (moisture gain). The equilibrium has been achieved by moisture loss until a_w - values of 0.65 for apricots, while for raisin and dates it turns positive at a_w - values higher than 0.33, reflect the hygroscopicity of dry raisins and Saidi – dates. However, rate of moisture change for apricots did not exceed \pm 0.015 g_{H2O} / g_{DM}. d, while those of raisins and Saidi – dates were higher by double or more (+ 0.0329 and +0.0389 g_{H2O} / g_{DM} . d).

The high hygroscopicity of mono-saccharides has been confirmed by [65], who reported that plant sugars are amorphous and can adsorb high amounts of moisture.

3.3. Sorption isotherms characteristics:

Fig (2) shows the sorption isotherms curves of dried apricots, raisins and Saidi date variety at $(25\pm5\degree C)$. The sorption curves represent the values of equilibrium maximum moisture content (M_e) of the fruits, when stored at constant temperature in an atmosphere with definite constant relative humidity (water activity) for enough time to allow the moisture content of the fruit to reach an equilibrium state with the vapor pressure of the storage atmosphere. As seen, all isotherms sorption curves took the Sigmoidal shape, typical for type two isotherms described by [55] of sugar rich fruits.

 This type of isotherms curves is characterized (as seen in Fig. 2) with very low equilibrium moisture content at water activity values between 0.11 and 0.33 applied water activity level (aw).

Figure (1): Change in moisture content of dried fruits during storage at different a_w levels.

Figure (2): Sorption isotherms curves of tested dry fruits in the aw-range of 0.11 to 0.85

In this region of water activities, sugar molecules of the fruits are in hard crystalline form , adsorb negligible amount of water vapor from the surrounding atmosphere, but the high molecular weight component of the fruit tissues (such as polysaccharides and proteins) starts to adsorb low amount of water vapor and bind it on the surface chain of these macromolecule to cover their active OH \cdot , H⁺ and N+ sites on the backbone of the molecules to protect it from oxidative reactions (damage). In the second region of the sorption isotherms curves (between $a_w = 0.33$ and $a_w = 0.65$), the sugar molecules present in the fruits starts also to adsorb water vapors from the surrounding atmosphere, which lead to swelling and surrounding the sugar molecules with layers of H_2O units and change their state from hard crystalline to the soft amorphous for with increase in the equilibrium moisture content. In the third phase of the sorption curves (at $a_w > 0.65$), the amorphous sugar molecules start to dissolve and adsorb, therefore, rapid increasing water vapor amount from the surrounding atmosphere to meet the hygroscopic and hydrophilic nature of these sugars. In the same time, the surface area as well as internal capillary system of the macromolecules starts to increase and adsorb large amounts of water vapor to fill the capillaries and to cover the bounded water molecules with more layers of condensed and free water. Hence, the equilibrium moisture content increases sharply in this region with increasing a_w -level of the storage atmosphere. Significant differences were found in the sorption isotherms curves of the tested fruits (apricot, raisins and Saidi dates). The equilibrium moisture contents of apricot fruits were lower than those of raisins and dates at all tested a_w - levels. The M_e -value of apricots at $a_w = 0.85$ was 0.382 g_{H2O} / g_{DM} , while the corresponding values of raisins and dates were 0.587 and 0.631 g_{H2O}/g_{DM} , respectively. These values agree to a great extent with the values published by [13,6,31,12] as well as [55]. The reason for the lower M_e values of apricots could be referred to the type of sugar present in fruits. As seen in Table (1) and according to [5], sucrose was found to be the dominant sugar present in apricot fruits, while fructose and glucose are the major sugar components of raisins and Saidi dates. As mentioned by [56], fructose is more hygroscopic than glucose and sucrose with M_e at 0.85=0.9, 0.7 and 0.3 g_{H2O} / g_{DM} for fructose, glucose and sucrose, respectively. Therefore, apricots showed lower M_e -values than those of raisins and Saidi date fruits, because sucrose and glucose absorb very little water

vapor until $a_w = 0.7$, then it begins to dissolve at higher a_w -values than those of fructose. This phenomenon is important for estimating the shelf life of such fruits by determining the critical a_w -value, at which the equilibrium moisture content (M^e) starts to increases sharply and accelerating the deterioration reactions.

3.4. Fitting Sorption isotherms curves to isotherms models:

The moisture sorption data of dry apricots, raisins and dates (presented in Fig. 2) were subjected to statistical analysis applying the selected mathematical equations (GAB, BET, Henderson, Halsey as well as Iglasis and Chirife) and the results are given in Table (3). As seen, Halsey equation gave R^2 values of 0.977 for raisins, 0.963 for dates and 0.877for apricots, being, in general higher than those of Henderson, Iglasis and Chirife as well as GAB -equations.

Table (2): Final experimental equilibrium moisture content and rate of change (g_{H20}/g_{DM}**.** d) during equilibrium period **of the tested dry fruits.**

Applied water	Final equilibrium moisture content (g_{H2O}/g_{DM})			% change compared with initial moisture content of the fruits			Rate of change (K-value) g_{H2O}/g DM. d		
activity level a_w	Apricots	Raisins	Dates	Apricots	Raisins	Dates	Apricots	Raisins	Dates
0.11	0.0074 ^{gC}	0.1080^{hB}	0.1395^{gA}	-95.69	-46.89	-34.01	-0.1897	-0.0151	-0.0157
0.22	0.0086 ^{gC}	0.1279^{gB}	$0.1420^{\overline{gA}}$	-94.99	-37.11	-32.82	-0.1293	-0.0125	-0.0017
0.33	0.0535 ^{fC}	0.1712^{fB}	0.2190^{fA}	-74.20	-15.82	$+3.602$	-0.0878	-0.0044	$+0.002$
0.44	0.1036^{eC}	0.2200^{eB}	0.2650^{eA}	-50.04	$+8.17$	$+25.36$	-0.0402	$+0.0007$	$+0.012$
0.54	0.1084 ^{dC}	0.2261 ^{dB}	0.2752^{dA}	-47.73	$+11.18$	$+30.18$	-0.0292	$+0.0008$	$+0.0145$
0.65	$0.1185^{\overline{cC}}$	0.2546^{cB}	$0.3030^{c\overline{A}}$	-42.86	$+25.19$	$+43.34$	-0.0171	$+0.0106$	$+0.0157$
0.75	0.2304^{bC}	0.3629^{b}	0.4090 ^{6A}	$+11.09$	78.44	$+93.48$	$+0.0028$	$+0.0164$	$+0.0229$
0.85	$0.3816^{\rm aC}$	0.5873^{aB}	0.6310^{aA}	$+83.99$	188.78	$+198.51$	$+0.015$	$+0.0329$	$+0.0389$

Means within a column with the same letters a, b, c ... and means within a row with the same capital letters A, B, C, ... are not different at 95% significancy level

Although, GAB -equation has been recognized as the mathematical model suitable to represent sorption data over a wide range of water activity values of foods from 0 to 0.9 [3,8,55] there are some critics that GAB does not satisfactory describes the sorption data of all foods due to the following reasons : GAB- model is not always suitable for interpreting sorption data obtained by gravimetric method [57] no single equation has been found to accurately depict all range of sorption isotherms [55]. GAB model was better in fitting desorption data, while other models were better in representing adsorption data [34,58].Also, the suitability of GAB- equation decreases, if the energy parameter (C) is lower than 2 [59]. In the present work, C- parameter for GAB – equation of dry apricots was 2.8 (Table 3-B) with a low R^2 – value of 0.59 compared to the satisfactory \mathbb{R}^2 -values for raisins and dry dates (0.873 and 0.841, respectively).

The model showed also a relative deviation between experimental and predicted M_{e} - values (P% - ratio) of 32.82%, which by far exceeds the level of accepted good and satisfactory P% ratio (<15%). As seen in Table (3), the only model (equation) representing sorption data of dry apricots is that of Iglasias and Chirife with P- value of 25.90%. Other applied equations (Henderson, Halsey and GAB showed higher P%- values. The reason could be referred to the type of common sugar in apricots (sucrose), which differ in hygroscopicity from fructose [56]. The most relevant models (equation) representing sorption data of raisins and apricots are Halsey and GAB with a very good P% -values in the range of 6.78 to 8.35% (Table 3), which are within the limit of good ratio (0- 10 %) [60,8].

On other side, Henderson and Iglesias- Chirife equations gave satisfactory P%-values (12.73 to 15.98 %) for raisins and dates, but Halsey and GAB – equations gave better fit to the sorption data of these two fruits. The statistical parameters (RMSE) were lower for Halsey as well as Iglasias and Chirife equation than those of Henderson model (Table 3) and those for raisins and dates were lower than those obtained for apricots. These differences could be referred to the type of common sugar in the tested fruits (sucrose in apricots and fructose in raisins and dates), which affect the crystallinity and ability of fruits to adsorb water vapor molecules, especially at lower level of water activity which influence the amount of bound water at this low range $(a_w \le 0.3)$ [56].

The constants A and B for Henderson, Halsey as well as Iglasis and Chirife equations are within the values for apricots, raisins and dates given by other researchers [61,31,8,24]. Table (3) includes also the values of relative deviation P%. Only GAB – equation gave random distribution of the calculated M_{e} -values of the tested fruits, also for apricots in spite its high P% – value (32.82 %). Halsey equation gave also random distribution for raisins and dates moisture data, while equation of Iglasis and Chirife gave random distribution for dates -M_e only. The most important parameters of GAB and BET equation (Table 3) are the values of M_m , C and K.

The expression M_m gives the amount of the first water molecules tightly bound to the adsorption sites of the dry fruits, (i e. the first water molecule layer bounded to the H^+ and OH groups of the sugars and polymer chains of the fruit. This layer is very important for keeping quality of the dry fruit and prevent their oxidation by O_2

It is neither freezable nor be able to remove during drying. As seen, the M_m - value of raisins and dates (0.1186 and 0.147 g_{H2O}/g_{DM} , respectively) was higher than those of apricots (0.054 g_{H2O}/g_{DM}) due to the high hygroscopicity of the reduced sugar present in these two fruits. BET-equation gave M_m -values lower than those of GAB- equation (Table 3), because the later takes in consideration also the second, water molecule condensed on the first layer and also is strong bounded to the backbone of the adsorbing sites. bounded to the backbone of the adsorbing sites.

Statistical parameters	Applied equation								
	Henderson			Halsey			Iglesias and Chirife		
	Apricots	Raisins	Dates	Apricots	Raisins	Dates	Apricots	Raisins	Dates
A	0.507	0.822	0.775	-3.346	-1.753	-1.564	-1.341	-0.702	-0.562
	± 0.052	±0.147	± 0.145	± 0.222	± 0.035	± 0.042	± 0.105	± 0.090	± 0.078
B	0.625	1.615	-1.739	-1.538	-0.622	-0.572	1.181	0.936	0.897
	± 0.070	±0.214	± 0.237	±0.242	± 0.039	±0.045	± 0193	±0.165	± 0.143
SSE	0.080	0.109	0.1146	1.87	0.0485	0.0661	0.106	0.0776	0.0580
RMSE	0.116	0.135	0.138	0.558	0.089	0.105	0.133	0.1138	0.0983
R^2	0.930	0.904	0.900	0.870	0.977	0.963	0.861	0.842	0.867
$P\%$	34.27	12.73	12.79	41.33	7.02	8.35	25.90	15.98	13.03
	Patterned	Patterned	Patterned	Patterned	Random	Random	Random	Random	Random

Table (3): Estimated parameters of the applied models for sorption data of dry apricots, raisins and dates: (3-A): Henderson, Halsey as well as Iglasias and Chirife equations

Table (3-B): GAB and BET Equations:

The term (C) expresses the binding every of the mono- layer water to the adsorbing sites and it was found that it in raisins is relatively higher, (61.8), than that of dates (40.02), while that of apricots is low (2.8). Similar trend could be observed also by the C- values obtained from BET- equation (Table 3).

The term K expresses the strength of binding energy for the condensed $H_2O -$ layers above the first mono – layer. Kvalues for raisins and apricots were lower than unity $(0.734$ and 0.868 , respectively) which indicate that these H₂O- layer are free and could be easily removed during drying and are freezable.

K- values of apricots (1.59) is > 1 and expresses the difficulty of apricots drying to an acceptable low moisture content. The obtained results agree with [6,26,4,34].

3.5. Comparison between experimental and calculated moisture data:

To validate the suitability of tested isotherms models to simulate the sorption behavior of tested fruits (dry apricots, raisins and dates), the differences between the calculated and experimental moisture data (residuals) as well as the comparison between the experimental and predicted sorption isotherms curves were plotted and established. Figure (3) shows the plot of experimental and calculated sorption isotherms curves for the tested fruits using 4 sorption isotherms equations. As seen, Henderson, Iglesias and Chirife equations failed to represent the sorption data of the tested fruits, due to the clear differences in isotherms curves between experimental and calculated curves, especially at medium and higher water activity level for all tested fruits. On other side, Halsey and GAB – equations showed a very close course of experimental and predicted sorption isotherms curves of raisins and dates (high reducing sugars fruits), while their curves for apricots were of less quality, where GAB- equation was better than Halsey equation in representing sorption data, of apricots (high sucrose containing fruit), especially at high water activity levels $(a_w > 0.75)$. So, it could be concluded that GAB- equation gave the best results for residuals and isotherms course of the tested fruits (dry apricots, raisins and dates) followed by Halsey – equation for raisins and dates. These results agree with those reported by [63,23,3,62,41,34].

They mentioned that most 2- constants and 3 – constants isotherms model cannot fit sorption data of sugar rich foods towards high water activity level ($> a_w(0.8)$) due to solubility and exudation of sugars affecting the surface area of the absorbing sites of the fruit.

3.6. Shelf-life prediction for dry apricots, raisins and dates:

Determination of safe moisture content for storage and the corresponding suitable relative humidity are important issues for sorption isotherms experiments of dry foods.

The major risk in storage of sugar rich dry foods is mold infection, which could be avoided if the a_w -value of the dry foods remains below 0.65. The suitable relative humidity is that, where the dry food neither gain nor loose moisture with the surrounding atmosphere.

According to [21,64,26,12], the critical moisture content (M_c) of the dried fruits could be obtained from the intersection point between the slowly and fast increasing segments of the sorption isotherms curve, which represent the end point of H₂O multi -layer region (between $a_w = 0.2 - 0.6$) and that of condensed water region $(a_w > 0.65)$.

Figure (3): Comparison between experimental () and calculated () mo) moisture content of dried fruits using different sorption isotherms equations. (Y axis : $g_{\text{ H2O}}/g_{\text{ Dm}}$ **and X axis : Water activity Value)**

The isotherms curve could be considered as a straight line (in this region) and the slope(b) in $(g_{H2O}/g_{DM}$. unit water activity) could be used to estimate shelf life of the dry fruits. The values for critical moisture content M_c , maximum safe moisture content (M_0) , critical water activity level (a_{wc}) and slop (b) of multi-layer sorption region of the tested fruits have been obtained from sorption curves (Fig 2) and are given in (Table 4).

The critical moisture content (M_c) for dry apricots was 0.28 g_{H2O}/g_{DM} at a critical water activity of 0.73, while M_c for raisins and dates were respectively 0.26 and 0.28 g $_{H2O}/g_{DM}$ at corresponding critical water activity of 0.65. $a_{\rm wc}$ – values of dry apricots were relatively higher than those of raisins and dates due to the nature of dominant sugar in apricots (sucrose) and the presence of absorbed SO_2 , which reduces the amount of adsorbed water molecules.

Hence a_w – values higher than 0.65 could be tolerated without incidence of mold growth Sharma et al. [65] Maximum (end) water activity (a_{w0}) of apricots was chosen to be 0.78 (Maximum water activity level without incidence of mold growth, [65] and 0.7 for both raisins and dates (Begins of water vapor condensation region). The corresponding maximum (end) moisture contents for safe storage were 0.31 ,0.305 and 0.33

 g_{H2O}/g_{DM} , respectively for apricots, raisins and dates. The slope (b), rate of moisture increase, in the slowly rising section of sorption curve (Multilayer H₂O region), was 0.19, 0.225 and 0.25 $_{\text{BHO}}$ / $_{\text{BDM}}$ unit a_w for apricots, raisins and dates, respectively. With references to [50], if 500 g dry fruits packed in 600 cm² polyethylene bag of 30 μm thickness were taken as basis for normal storage of the dry fruits, expected shelf life according to equation (14), could be predicted and the results are given in Table (4). As seen, the expected shelf life of the tested fruits was 223, 214.9 and 219 days for apricots, raisins and dates, respectively. The calculation showed that the shelf life of the fruits could be extended to 7.16 - 7.4 months at 25℃ and 70% RH without mold growth and remarkable loss or gain in moisture. Each reduction in relative humidity by 10% will increase the expected shelf life by 12%.

Also, increasing the thickness of polyethylene bag by 10 μm will increase the shelf life by 34%. For one year shelf life, the thickness should be at least 50 μm. Available standard for storage of dry fruits ES, [43] gives 26, 31 and 30 % allowed moisture contents – on wet basis – for apricots, raisins and dates, respectively. As seen in Fig (2), the corresponding M_e on dry weight basis will be 0.351, 0.449 and 0.428 g $_{H2O}$ /g $_{DM}$, respectively, which lies in the region of condensed water for these fruits and subjected them to mold growth and other deterioration reactions. In light of the obtained results in the present work, it could be recommended to revise these allowed moisture limits.

3.7. Heat of water molecules adsorption:

The nominal (base) energy required to evaporate one mole of free water is 43 KJ/ mole. This value is valid also for free water molecules condensed on the adsorbing sites of the fruits. By increasing the binding energy of condensed water molecules or the binding energy on the adsorbing sites, the energy required to evaporate this water will exceeds the nominal evaporation energy. This excess energy value is termed as excess evaporation energy (q_{st}) and its magnitude increases with changing the state of water layer towards " bound water".

The calculations of the q_{st} – values are essential for proper designing of dehydration process of the fruits as well as storage and handling operations. According to [40], heat of sorption (q_{st}) can be determined from sorption data. According to equation (13).

For this purpose, the water activity values for definite equilibrium moisture contents from 0.63 to 0.007 g_{H2O}/g_{DM} were obtained from Figure (2) and were used to calculate the corresponding excess (q_{st}) evaporation energy for apricots, raisins and dates, and the results are illustrated in Fig (4). As seen, the excess evaporation heat remains almost constant at <1 Kj/mole in the high moisture region from 0.6 to 0.4g $_{H2O}/g$ _{DM}, after which the values of q_{st} increases slowly between 0.4 to 0.2 g_{H2O}/g_{DM} . In the low moisture region < 0.2 g_{H2O}/g_{DM} , the values of q_{st} increases sharply and reaches level of 6 Kj/mole.

This value agrees with those reported by $[41,21,16,26,18]$. They reported that q_{st} value depends on physio -chemical and structural composition of the fruit. As seen in Fig (4), the qst curves of dates and raisins differ than those of apricots, where apricots showed lower qst – value in a_w – region <0.2. Similar behavior has been reported for date varieties [18] as well as for Mango and jackfruit [16] .

According to [6] crystallized sugar has a low hygroscopicity since sucrose (dominant sugar of apricot) has higher glass transitions temperature (50 - 60℃) than that of reducing sugars (15 -17℃), [66] then it is expected that apricots show lower qst -values than those of dates and raisins.

3.8. Effect of water activity level on color and hardness of the tested fruits:

Water activity level induces changes in color and hardness of the stored dry fruits because water availability is the most factor influences these reactions and structural changes.

Table (5) gives the change in color parameters (L, a, b, C, H and BI) for apricots, raisins and dates. As seen, lightness (L), yellowness (b) and Chroma (C) values for the tested three fruits were decreased by changing the storage atmosphere from $a_w = 0.11$ to 0.85. On other side, redness (a- value was increased by increasing a_w value due to darkening of the dry fruits.

Reduction ratio in lightness $(L - value)$ reached 24.38, 44.79 and 54.87% for apricots, raisins and dates, respectively. The low loss in lightness (L-value) of apricots could by referred to the SO_2 – treatment, which protect fruit color. On other side, the corresponding loss in yellowness (b-value) was 44.02, 44.79 and 97.02, respectively. For apricots, the loss in b-value could be referred mainly to the loss in carotenoid pigments [53].

In all tested fruits, redness (a-value) was substantially increased by increasing a_w -level and reached 171.86 % for apricots and 390.96 % for raisins compared with 547.75 % for dates. The main reasons for the increase in (a-value) are the pigments formed through the Maillard reaction products. Correspondingly, the Hue- value of the fruits was changing from yellow and yellow- green color type $(82.87, 97.35 \text{ and } 99.75^{\circ})$ at $a_w = 0.11$ to the orange and dark reddish color with Hue- values of 85.78, 55.59 and 15.64 \degree , respectively at a_w 0.85 for apricots, raisins and dates. Browning index (BI), reached its maximum level at $a_w = 0.75$ (68.98, 58.51 and 72.89 for apricots, raisins and dates, respectively), while the minimum values of BI were obtained at $a_w - val$

ues \leq 0.33, (63.36, 45.89 and 21.56, respectively). The slow rate of browning reaction at $a_w \leq 0.3$ could be explained by the low mobility of the reagents, while at $a_w > 0.75$, excess adsorbed water causes inhibition of the reaction due to dilution of the reactants, especially because Maillard reaction itself produce water molecules contributes also to diluting the medium. The slow decrease in BI- value in apricots at low a_w levels, in comparison to those of raisins and dates, may confirm the assumption that carotenoid pigments are involved in surface discoloration of the dry fruits. According to [67] sucrose in dry fruits changed to the crystalline state at

 $a_w \leq 0.33$. In this state, oxygen diffusion through the fruit tissues increased and carotenoids degraded quickly, which keeps the level of BI at high values. The obtained color parameters of dry apricots in this work agree with [51,5]. They reported L-values in the range of 35 to 44 and Hue- values of 58 - 67° for dry apricot at a_w range between 0.6 and 0.8.

Table (4): Shelf-life prediction of dry apricots, raisins, dates.

Figure (4) : Relationship between qst and fruits moisture content

Also, [68] reported L- values in the range of 33.58 to 40.14 for Thompson raisins, with a, b and Hue- values similar to those of 0.55 to 0.75 water activity levels given in the present work for dry dates. The color parameters (L, a, b and Huevalues) given by [69,70] agree to a great extent with the values given in Table (5). The effect of water activity level on color parameters has been discussed by Rhim and Hong, [71,72,22]. In all these works, L and b – values of the dry products were decreased, while a- value was increased by increasing a_w - level.

Hardness of the dry fruits is an important factor for their palatability, processing and handling as well as for rehydration. The effect of water activity level on hardness of the tested fruits is given in Table (6). As seen, the hardness value of the dry fruits at the medium range of water activity level $(a_w=0.55$ to 0.75 %) was 78.93 to 29.75, 139.09 to 8.77 and 14.62 to 6.60 N/m² for apricots, raisins and Saidi dates, respectively. This agrees with hardness rang of 5 N to 46.95 N published for apricots [73,11,52]. The hardness values of raisins were in the range of 60- 150 N [68]. The lowest values of hardness were recorded for dates with a range of 3.73 to 57.02 N. [45,69,70].

As seen in Table (6), dried fruits recorded very high hardness values from 1738.28 N to 84.55 N at very low water activity level (0.11and 0.22), with apricots showing the highest hardness values followed by raisins, while dates was relatively the softest fruit at a_w level of 0.11 and 0.22. The high level of hardness could be referred to crystallization of sugar at this very low moisture content of the fruits, creating a very solid texture. [16,74] related this behavior to moisture content and crystalline state, since water molecules plasticizes upon transition of the fruits to the glassy state, where structure changes, collapses and become dense. At higher a_w - levels, moisture content increases forming more liquid bridges between particles and the structure changes to a sticky form. According to [66], the glass transition temperature of sucrose (apricots) is 50ºC, while that of fructose (raisins and dates) is 15ºC. Therefore, it could be expected that apricots will be very hard at room temperature when the moisture content becomes very low at $a_w < 0.33$.

Also, results of [75] showed hardness of 1000 and 38 N/m² for apricots at 8% and 30% moisture content, respectively. As seen in Table (6), tested fruits becomes very soft at a_w level of 0.85 and recorded low hardness level of 4.02 to 8.21 $N/m²$.

Table (6): Hardness (N/m²) of tested fruits in relation to water activity values

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Means with same subscripts in the same column are not different at significant level (p<0.05)

4. Conclusion

Storage condition are essential for ensure safety of the dry fruits. The aim of the present work was, therefore, to establish sorption isotherms curves for dry apricots, raisins and Saidi dates. The course of the obtained isotherms differed between the tested dry fruits, mainly because the differences in their drying pretreatments and the differences in sugar type dominating in the fruits, sulfurization as pretreatment and sucrose as the major sugar type in apricots led to lower equilibrium moisture content at all tested a_w – values (0.11 to 0.85) compared with those of raisins and dates, where fructose and glucose are the dominating sugars. However, all tested fruits showed isotherms curves of sigmoidal shapes and could be satisfactory represented by GAB and Halsey equations. Some essential parameters such as minimum moisture content (M_m) , energy constant (C), excess evaporation heat (q_{st}) and expected shelf life (T_s) for the tested fruits could be obtained from their sorption isotherms curves. Maintaining relative humidities of 50% in the surrounding atmosphere could extent the expected safe shelf life of the fruits to 10-11 months. Storage of the dried fruits at relative humidity values lower than 30% for long time, drastically increases their hardness to an implantable level, while increasing a_w – values to level higher than 0.7 encourages the browning reaction and make the fruits very soft and susceptible for microbial and chemical deterioration reactions. Further researches on this subject are essential for optimization of safe shelf life of dry foods.

5. References

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