Vol. 21, No. 3(2022) Alim2813
Revista Mexicana de Ingeniería Química

Investigation of drying kinetics and drying conditions on biochemical, sensory, and microstructural parameters of "Sefri" pomegranate arils (*Punica granatum* L. a Moroccan variety)

Investigación de la cinética de secado y las condiciones de secado sobre parámetros bioquímicos, sensoriales y microestructurales de arilos de granada "Sefri" (*Punica granatum* L. una variedad marroquí)

S. El Broudi[1], N. Zehhar[2], N. Abdenouri[3], A. Boussaid[4], A. Hafidi[5], H. Bouamama[1]*, F. Benkhalti[1]

[1]*Laboratory of Sustainable Development and Health Research (LRDDS), Faculty of Sciences and Techniques, Cadi Ayyad University, PO Box: 549, Marrakech, 40000, Morocco.*

[2]*Laboratory of Biotechnology and Molecular Bioengineering (LBMB), Cadi Ayyad University, PO Box: 549, Marrakech, 40000, Morocco.*

[3]*Laboratory of Electrical Systems and telecommunications (LSET), Faculty of Sciences and Techniques, Cadi Ayyad University, PO Box: 549, Marrakech, 40000, Morocco.*

[4]*Laboratory of Bioprocess Engineering, Faculty of Sciences and Techniques, Cadi Ayyad University, PO Box: 549, Marrakech, 40000, Morocco.*

[5]*Laboratory of Biotechnology and Molecular Bioengineering, Faculty of Sciences Semlalia, Cadi Ayyad University, PO Box: 2390, Marrakech, 40000, Morocco.*

Received: April 23, 2022; Accepted: August 18, 2022

Abstract

The study evaluated the application of four drying temperatures inside a newly developed hybrid indirect solar dryer in forced convection (at 40, 50, 60, and 70°C) compared to a direct solar dryer (Greenhouse) for the processing of the Moroccan Sefri pomegranate arils. The thermodynamical analysis allowed characterizing the physical properties of the arils using some parameters such as optimal water activity, net isosteric heat of sorption, and effective moisture diffusivity. The biochemical analysis studied included total proteins, sugars, vitamin C, and total anthocyanins, hygrometric properties, microstructure modification, and sensory evaluation. Generally, drying led to a reduction in all parameters. However, lower drying temperature processes (indirect convective solar drying at 40°C and 50°C) give the best results for pomegranate arils. Greenhouse dried samples had almost the lower quality in all parameters. The color is more preserved at indirect convective solar drying at 40°C. However, indirect convective solar drying at 50°C was most appreciated by consumers with higher scores on the sensory evaluation test. As the first study of the thermodynamic and biochemical investigation of dried pomegranate arils in Morocco, this work intended to be the first step in developing controlled new high-quality products for the Moroccan market.

Keywords: Pomegranate arils "Sefri variety", drying methods, drying kinetics, biochemical parameters, sensory analysis.

Resumen

El estudio evaluó la aplicación de cuatro temperaturas de secado dentro de un secador solar indirecto híbrido recientemente desarrollado en convección forzada (a 40, 50, 60 y 70°C) en comparado con un secador solar directo (Invernadero) para el procesamiento de los arilos de la granada Sefri marroquí. El análisis termodinámico permite caracterizar las propiedades físicas de los arilos mediante algunos parámetros como la actividad óptima del agua, el calor isostérico neto de sorción y la difusividad efectiva de la humedad. El análisis bioquímico estudiado incluye proteínas totales, azúcares, vitamina C y antocianinas totales, propiedades higrométricas, modificación de la microestructura y evaluación sensorial. Los procesos de menor temperatura de secado (secado solar convectivo indirecto a 40°C y 50°C) dan los mejores resultados. Las muestras secadas en invernadero presentaron casi los valores más bajos. El color se conserva mejor a 40°C. Sin embargo, el secado solar convectivo indirecto a 50°C fue el más apreciado por los consumidores en la prueba de evaluación sensorial. Como primer estudio de la investigación termodinámica y bioquímica de los arilos de granada secos en Marruecos, este trabajo pretende ser el primer paso en el desarrollo de nuevos productos controlados de alta calidad para el mercado marroquí.

Palabras clave: Arilos de granada "variedad Sefri", métodos de secado, cinética de secado, parámetros bioquímicos, análisis sensorial.

* *Corresponding author. E-mail*: h.bouamama@uca.ac.ma, bouamamahafida@gmail.com
https://doi.org/10.24275/rmiq/Alim2813
ISSN:1665-2738, *issn-e*: 2395-8472

1 Introduction

Pomegranate (*Punica granatum* L.), belonging to the *Punicaceae* family, is one of the oldest known and widely consumed edible fruits in various cultures. The pomegranate is native to the region that extends from Iran to northern India (Stover & Mercure, 2007). Nowadays, pomegranate is cultivated in most areas of the world. As a producer country, Morocco cultivates a broad area of 5000ha and ensures an average harvest of 58,000 tons of fruit per year (Hmid *et al*., 2018).

Many local cultivars have been known in Morocco, such as Grenade Jaune, Grenade rouge, Ounk Hmam, Gjebali, Djeibi, Chelfi, Bzou, and Sefri. This last is one of Morocco's very popular pomegranate cultivars because of its shiny pink-red color; it has the lowest acidity, the highest juice percentage, and a very sweet taste due to the higher sugar content (Hmid *et al*., 2017). Also, it is very rich in phenolic compounds with a high antioxidant potential (Benchagra *et al*., 2021). However, it is very sensitive to water stress and does not tolerate drought conditions the world faces in the actual climate challenge (Adiba, Hssaini, *et al*., 2021; Adiba, Razouk, *et al*., 2021). For all of this, preserving the quality of fresh pomegranate arils for more extended periods is becoming a necessity.

The edible parts, called "Arils," are consumed mostly as fresh seeds and juices, jellies, jams, coloring, and flavoring agents in drinks and as a garnish in desserts and salads. Pomegranates are an immune booster and contain many antioxidants that delay aging impact. Grenades arils are full of vitamin C, B, and iron and can relieve certain diseases like anemia and Alzheimer's. Also, rich in phytochemical compounds with high levels of flavonoids and polyphenols. These antioxidants have a known effect to protect against heart disease and cancer.

The commercial interest in pomegranate is growing over time, non just as fresh but also the processed products have gained attention like drinks, syrups, and candies, etc. Among all conservation processes, drying has often been used to add value and preserve this product over the season because of the short shelf life of less than 12 to 14 days in an ambient environment (Dak *et al*., 2014). Drying is often considered simple and can be handled near the harvest area without transporting the product to industrial areas for processing.

The drying operation requires advanced technology to control the process to obtain a good quality product in the end. Convective solar drying is considered to be a potential alternative method in developing countries because of its simplicity, uses renewable energy, reduces transportation costs, and solves many problems in traditional solar drying. The product is exposed to dust, moisture, and insects. Besides, uncontrollable fluctuations in temperature lead in many cases to undesired changes in the final product. Food products containing high sugar content may suffer from significant damage to the dried product's chemical composition, flavor, structure, and color (Calín-Sánchez *et al*., 2012).

Thermodynamical parameters are special tools that describe the product's stability and hygroscopic behavior (Zhao *et al*., 2015). In addition, water activity and optimum moisture content can be inferred by the sorption curves at a steady-state, determining the hygrometric conditions for better preservation of the dried product. Very minimal studies investigate only some drying kinetics of pomegranate. To this moment, our work presents the first study on drying kinetics, biochemical variations, and sensory analysis of dried Moroccan *Punica granatum* L. arils.

This study aimed to develop a new dried high-quality product for the Moroccan market, and investigate the effect of drying conditions (forced convective indirect solar drying and greenhouse drying) on physicochemical properties of Moroccan pomegranate arils, such as drying kinetics, hygroscopic stability, biochemical composition (Total protein, sugar, vitamin C, and anthocyanins contents), rehydration capacity, sensory evaluation, microstructure, and color change.

2 Material and methods

2.1 Pomegranate arils preparation

Pomegranate fruits were bought from the wholesale market of fruits and vegetables of Marrakech one day after harvesting. The studied variety was "Sefri" which was harvested at the region of Loudaya (27 km from Marrakech). Twenty-five fruits were randomly selected and weighed from different harvested batches in order to obtain a representative sample of the harvested area. After separating the arils from the peel, each part was weighed to determine their ratio in the sample. The arils manually separated, washed, and prepared for drying.

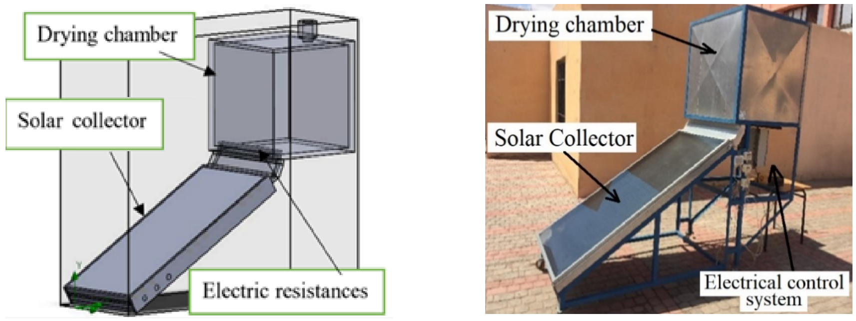

Fig. 1 Synoptic and the designed hybrid solar-electric dryer used for the convective drying of pomegranate arils.

2.2 Drying methods

2.2.1 Convective solar drying

The solar dryer used in this work (Fig. 1) is the standard one equipped with electrical resistance inside the drying chamber, designed and developed at the physique department of the Caddi Ayyad unUniversity (El Ferouali *et al*., 2016). The main parts of this solar dryer are a solar collector, a drying cell, a fan installed on the top of the drying chamber, and the electrical resistance placed between the outlet collector and the chamber inlet (max 4 kW).

- The drying chamber can support four trays which represent about $4m^2$.
- The hybrid solar-gas-electric dryer is equipped with PID temperature regulation control.
- Outlet airflow was controlled by an Arduino Uno, which acts on a fan of 10 cm diameter (Orion 12HBVXC model) in the chimney.
- PT100 temperature sensors (±0.5K) (ref. TM110, KIMO) were placed inside the drying chamber.
- The solar irradiation on a 30° inclined surface was recorded using a Kipp and Zonen SMP10 pyranometer with a sensitivity of 14.69×10^{-3} mV.
- Weather station Vantage Pro2 (ref. 6162CFR) for measuring ambient temperature and wind velocity.
- We performed the drying experiments at 40, 50, 60, and 70°C (± 0.5°C), under an airflow rate of 0.025 kg/s. The respective drying time was 75, 10, 6, and 4 hours.

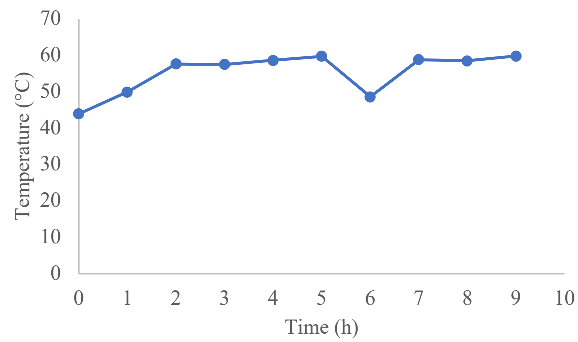

Fig. 2 Variation of temperature inside the greenhouse chamber during drying.

2.2.2 Greenhouse drying

Samples were placed in a 1 × 0.8 × 0.5 m greenhouse with natural ventilation. The internal temperature was about 55 ± 3°C. Fig. 2 showed the variation of temperature inside the greenhouse, and the relative drying time was 9 hours at discontinued mode. The drying was occurred during the hottest periode of the day (12 am to 4 pm in July 2019 (35-43°C)) to maintain the inner temperature of the greenhouse. The samples were preserved in hermetic bags until the next day to finish the drying.

All the drying experiments were conducted in triplicates. The initial moisture content of fresh *P. granatum* (78.0 ± 0.11%) was determined by drying the samples in an oven at 105°C for 24 hours.

The moisture content based on a dry weight (M_t):

$$X_t = \frac{M_{th} - M_{td}}{M_{td}} \tag{1}$$

The experimental moisture content accodring to Koukouch *et al*., (2015) by the following Equation (2):

$$X^* = \frac{X_t - X_{eq}}{X_0 - X_{eq}} \tag{2}$$

2.3 Isothermes of sorption

The isotherms of sorption were determined by the static gravimetric technique. The product was placed in the presence of a specific saturated saline solution. Six saturated salt solutions covering a relative humidity range of 5-90% were considered (KOH, $MgCl_2$ $6H_2O$, K_2CO_3, $NaNO_3$, KCl, and $BaCl_2$ $2H_2O$) at three temperatures of 30, 40, and 50°C (Table 1). The samples were weighed for both adsorption and desorption every two days until they reached 1% of the difference between two successive weighings; the thermodynamic equilibrium was then reached. Once the equilibrium wet masses are determined, the samples are placed in a 105°C oven for 24 hours to calculate their dry masses (Greenspan, 1977).

2.4 Mathematical fitting of sorption isotherms

The relationship between the equilibrium moisture content (Xeq) and water activity (a_w) was simulated by four best-known mathematical models (GAB, Modified Henderson, Peleg, and the Enderby model) shown in Table. 2.

The best model, describing the sorption isotherm of the product, has the highest value of the correlation coefficient r and the smallest values of Standard Error of Moisture (SEM). These statistical parameters are defined respectively by equations (3), (4) (Senadeera *et al*., 2020):

$$r = \sqrt{\frac{\sum_{i=1}^{N}(Xeq_{i,pre} - \overline{Xeq}_{i,exp})^2}{\sum_{i=1}^{N}(Xeq_{i,exp} - \overline{Xeq}_{i,exp})^2}} \tag{3}$$

The experimental average moisture content is defined by:

$$SEM = \sqrt{\frac{\sum_{i=1}^{N}(Xeq_{i,pre} - \overline{Xeq}_{i,exp})^2}{d_f}} \tag{4}$$

$Xeq_{i,exp}$ is the experimental moisture content, $Xeq_{i,pre}$ is the predicted moisture content, and df is the freedom degree of the regression model.

2.5 The optimal water activity for drying and storage

Sorption isotherm provides accurate information about the optimal conditions for conserving and storing dried products. In this case, the optimal water activity for the conservation of pomegranate arils.

All experimental data points of the sorption isotherm for desorption and adsorption at 30, 40, and 50°C were gathered in the same graph. The data points may particularly be described by a 3rd-degree polynomial. The value of water activity of which the second derivative equals to zero f''(x)=0 (inflection point) presents the optimal water activity for storage a_{wopt} (Moussaoui *et al*., 2019).

2.6 Isosteric heat of sorption

The net isosteric heat of sorption (Δh_d) or differential enthalpy stands for the binding energy of the water to the substrate. This energy must be added to the heat vaporization energy (Δh_{vap}) for dehydration (Equation (5)). The net isosteric heat of sorption was calculated from the experimental data using the Clausius Clapeyron equation given by Equation (6) (Beristain *et al*., 1996):

$$\Delta H_d = \delta h_d + \Delta h_{vap} \tag{5}$$

$$\left[\frac{d(\ln a_w)}{d(1/T)}\right]_{vap} = \frac{-\Delta h_d}{R} \tag{6}$$

where
Δh_d : net isosteric heat of sorption (J/mol),
a_w : the water activity,
T: the absolute temperature (K) ,
R: the universal gas constant (J/(mol.K)).

This equation involves determining the isotherms at different temperatures to calculate the logarithmic variation of the water activity ($\ln(a_w)$) as a function of inverse temperature ($1/T$) for fixed equilibrium moisture content. It is assumed that isosteric heat does not vary with the temperature. Hence, Δh_d is determined from the slope -Δh_d/R (Equation (7)).

$$\ln(a_w) = \frac{-\Delta h_d}{RT} + \frac{\Delta s_d}{R} \tag{7}$$

2.7 Effective moisture diffusivity and activation energy determination

2.7.1 Effective moisture diffusivity

The drying process of food products is characterized by various thermodynamic phenomena. The migration of water from inside to the surface of the product is the simplest definition of this process. The effective moisture diffusivity D_{eff} is the parameter controlling this migration of moisture in the function of time and temperature. This parameter could be modeled based on Fick's second low of diffusivity Equation (8) (Crank, 1975) :

$$\frac{\partial X^*}{\partial t} = D_{eff}\nabla^2 X^* \tag{8}$$

The solution of equation (8) was given by Crank for slab geometry (Crank, 1975):

$$X^* = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left[(-2n+1)^2\frac{\pi^2 D_{eff}t}{4L^2}\right] \tag{9}$$

Where:
D_{eff} : the effective diffusivity (m^2/s),
L: the half-thickness of the sample (0.001 m).

This equation can be simplified for long drying time and reduced to the following Equation (10):

$$X^* = \frac{8}{\pi^2}\exp\left(-\frac{\pi^2 D_{eff}t}{4L^2}\right) \tag{10}$$

Plotting $\ln(X^*)$ against the drying time gaves a straight line allowing the determination of D_{eff} using the slope (S):

$$S = -\frac{\pi^2 D_{eff}}{4L^2} \tag{11}$$

Activation energy: The activation energy (E_a) presents the minimum amount of energy needed for the drying process to be achievable (Tagnamas *et al*., 2020). The law of Arrhenius was used to determine the Ea as the same procedure as effective diffusivity D_{eff} using equation (12):

$$D_{eff} = D_0 e^{E_a/RT} \tag{12}$$

Where:
D_0: Arrhenius equation's factor;
E_a: Activation Energy (kJ/mol).

2.8 Compensation theory

The compensation theory or the enthalpy-entropy compensation theory is also known as the isokinetic theory. According to this theory, Δh_d and Δs have a linear relationship based on the Gibbs-Helmholtz equation, and the relation between these two parameters is given by the following Equation (13) (Hssaini *et al*., 2020).

$$\Delta h_d = T_\beta \Delta S_d + \Delta G_d \tag{13}$$

T_β is the isokinetic temperature at which all the reactions in the series occur at the same rate, and ΔG_d is Gibbs free energy used to indicate the spontaneity of the sorption process. It is considered to be spontaneous when ΔG_d is negative and non-spontaneous when the free energy is positive. The compensation theory is validated by comparing the isokinetic temperature T_β with the harmonic mean temperature T_{hm} calculated mathematically as follows:

$$T_h m = \frac{n}{\sum_1^n 1/T} \tag{14}$$

n is the number of isotherms.

The compensation theory is applied only when the isokinetic temperature T_β is different than the harmonic mean temperature T_{hm}. The sorption process is enthalpy driven when $T_\beta > T_{hm}$. On the other hand, the process is entropy-driven.

2.9 Rehydration capacity determination

Rehydration experiments were based on the methodology detailed by Horuz & Maskan, (2013). 5g of dried aril samples were soaked into hot tap water at 50°C. At 10 min intervals, the samples were removed from the water, placed into the absorbent paper to eliminate the surface water, and then weighed.

Rehydration capacity was calculated using the equation described by Bhardwaj *et al*., (2019):

$$\text{Rehydration capacity} = \frac{\text{Wg. of rehydrated sample}}{\text{Wg.of dried sample}} \tag{15}$$

2.10 Titratable acidity and total ascorbic acid content

Titratable acidity was measured (TA) by titration to pH 8.1 with 0.1 M NaOH solution and expressed the results as g of citric acid per L of juice (Ismail *et al*., 2014). The total ascorbic acid content was determined using iodometric titration (Chowdhury *et al*., 2016). The I2 solution was titrated first with sodium thiosulfate to verify its concentration.

2.11 Total Protein and sugar content

Total protein content was measured using the following protocol: 50 mg of pomegranate arils were ground in 2 ml of phosphate buffer (pH 6.8), then centrifuged for 20 minutes at 12500×g. A volume of 0.1 ml of supernatant was added to 0.1 ml of distilled water and 2 ml of Bradford reagent (Bradford, 1976). After 5 minutes of incubation at 27°C, the absorbance was read at 595nm. We have used the bovine serum albumin (BSA) as standard.

Total sugar content was analyzed as described: 100 mg of pomegranate arils were ground in 2 ml of ethanol (80%) then centrifuged for 10 minutes at 5000×g. 0.1 ml of the supernatant was added to 1 ml of 5% phenol solution and 5 ml of concentrated sulfuric acid The reaction was held for 20 min at 100 °C then cooled in the dark. The absorbance was read at 495 nm. Different concentrations of glucose (50-500 μg/ml) were used for the standard curve (Dubois *et al*., 1956).

2.12 Total anthocyanins content

Total anthocyanins content was determined according to the pH differential method described by Ghasemnezhad *et al*., (2015). Absorbance (A) was measured in a UV/Vis spectrophotometer at 510 and 700nm in buffers at pH 1.0 and 4.5 using the following equation:

$$A = [(A_{510} - A_{700})_{pH(1.0)} - (A_{510} - A_{700})_{pH(4.5)}] \quad (16)$$

The molar extinction coefficient of cyanidin-3-glucoside is 26,900 for pomegranate fruit juice. We expressed the results as milligrams of cyanidin-3-glucoside per 100 g of dried pomegranate arils.

2.13 Color measurement

CIE L^*, a^*, b^* color parameters are used as a base for color analysis. Those parameters were deduced from an imaging process of samples. Where:

- L^* represents lightness, ranging from 0 (black) to 100 (white),
- a^* ranges between -128 (green) and 127 (red),
- b^* ranges between -128 (blue) and 127 (yellow).

The L^*, a^*, and b^* color parameters were delivered using a Konika Minolta Chromameter CR400. Values of different parameters were expressed as the mean ± standard deviation. Fig. 3 pressent the pomegranate arils before and after drying by different processes.

The color parameters were calculated using the following equations (García-Valladares *et al*., 2022):

$$\text{Chroma:} \quad C^* = \sqrt{a^{*2} + b^{*2}} \quad (17)$$

$$\text{Hue:} \quad h^* = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (18)$$

$$\text{Browning Index:} \quad BI = \frac{100 \times (A - 0.31)}{0.172} \quad (19)$$

$$\text{Where:} \quad A = \frac{a^* + (1.75 \times L^*)}{(5.645 \times L^*) + a^* - (0.3012 \times b^*)} \quad (20)$$

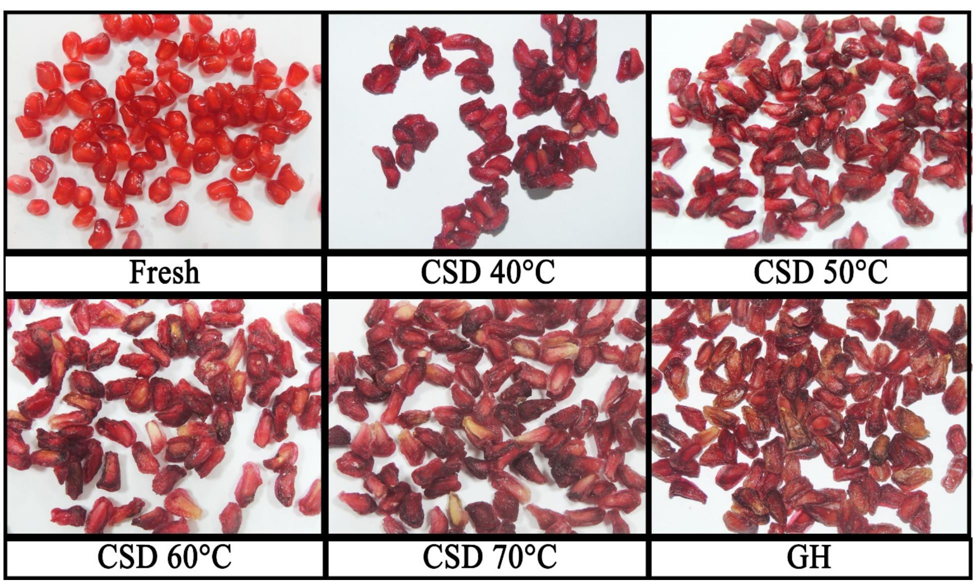

Fig. 3 Fresh and dried pomegranate arils at different process. A: CSD40°C; B: convective solar drying at 50°C; C: convective solar drying at 60°C; D: convective solar drying at 70°C; E: greenhouse drying.

2.14 Experimental design

The experimental design of this work was divided on two parts. The first, consisted of a unifactoral desing of two levels (Drying technique: convective drying and greenhouse drying). The second part was adopting a bifactoral design, factor 1: Drying technique (convective drying) and factor 2: drying temperature (40,50,60, and 70°C) as independing variables, while the response variables are : titrable acidity, ascorbic acid, protein content, sugar content,anthocyanins content, L^*, a^*, b^*, h^*, C*, rehydration capacity, taste, and appearance.

2.15 Sensory evaluation

Seventy untrained panels were used to evaluate the sensory attributes of dried pomegranate arils (42 female and 28 male) in three age subgroups (under 20 years old; between 20 and 40 years old, and above 40 years old) based on a 9-point hedonic scale (Kar *et al*., 2013), where 9 denoted highly desirable and 1 designates highly undesirable. We divided the experiment into two tests (taste and color) and the samples were coded differently for each test.

2.16 Effect of drying on the microstructure of pomegranate arils

2.16.1 Microscopic analysis

Punica granatum dried arils surface texture was observed in an optical microscope (VWR® Professional Plus VisiScope® BL254T1) at 100× and a stereomicroscope (Optika SZM-45B2) at 20×. Thin slices (0.5 mm) of the surface for different samples were observed under the optical microscope. The stereo microscope observation was carried out for a whole pomegranate aril of different drying processes.

2.16.2 Scanning electron microscopy (SEM) analyses

The microstructural observation of the surface of dried arils was realized using scanning electron microscopy (SEM). The samples were firstly coated with carbon using a carbon coaster (Cressington 108 carbon/A) to reduce the effect of superficial charging. The SEM images were obtained using a VEGA3 TESCAN SEM at an acceleration voltage of 20 kV, and the photos were taken at a magnification scale of 2000×.

2.17 Statistical analysis

The statistical analysis was conducted using the SPSS 21.0 Software and a data variance analysis (ANOVA) follow-up by post hoc Student-Newman-Keuls (SNK). This analysis was carried out to perform multiple stepwise comparisons to identify sample means significantly different from each other. All experimental measurements were carried out in triplicate, and the means ± standard deviations were reported. The PCA analysis was performed using the software R version 3.6.2, and the significance level was set to $p<0.05$.

3 Results and discussion

3.1 Drying kinetics and hygroscopic stability of Punica granatum arils

3.1.1 Isotherms of sorption and mathematical fitting

The effect of temperature on sorption isotherms of *Punica granatum* arils is presented in Fig. 4a for adsorption and Fig. 4b for desorption. *Xeq* increases by decreasing temperature at constant water activity. This result may be explained by the higher excitation state of water molecules at higher temperatures, decreasing the attractive forces between them. All sorption isotherms of pomegranate arils have a sigmoidal shape characterizing of the food materials recognized as type II of the IUPAC classification. Mundada and Hathan, (2012) found the same isotherms shape for pomegranate arils. This is consistent with the behavior of other food materials as fruits and vegetables (Mujumdar, 2006) like figs (Hssaini *et al*., 2020) and mashroom (Pascual-Pineda *et al*., 2020). At a constant water activity, the *Xeq* desorption isotherms are higher than *Xeq* of adsorption forming a gap in between them that characterize the phenomenon of hysteresis (Fig. 5). This gap reach its higher expantion at the water activity range of 0.4 and 0.8. as the food material is a complex in matter of the chemical constituent, the hysteresis phenomenon could not have a single explaination. However, it is explained generally by the thermodynamically irreversible processes during desorption or adsorption or both (Barbosa-Cánovas *et al*., 2007).

In the temperature range of 30-50°C, the Peleg model shows a better fitting to the experimental data with a correlation coefficient *r* and the standard error of humidity SEM of 0.9916; 0.9846, and 0.1073;

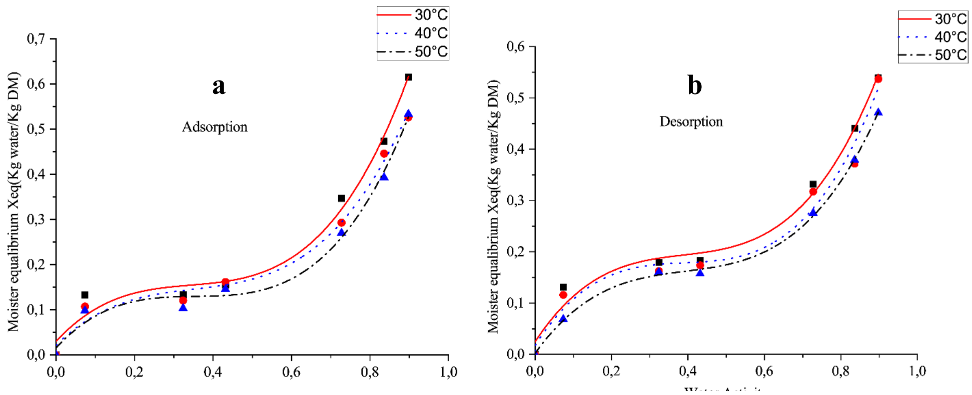

Fig. 4 Adsorption (a) and desorption (b) isotherms of *Punica granatum* arils at 30. 40 and 50 °C.

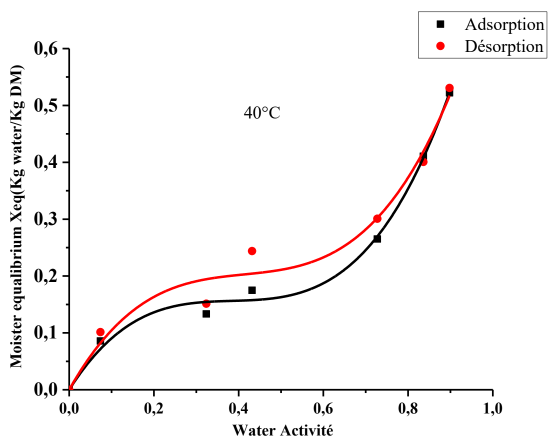

Fig. 5 Hysteresis of desorption and adsorption isotherms of *Punica granatum* arils at 40°C.

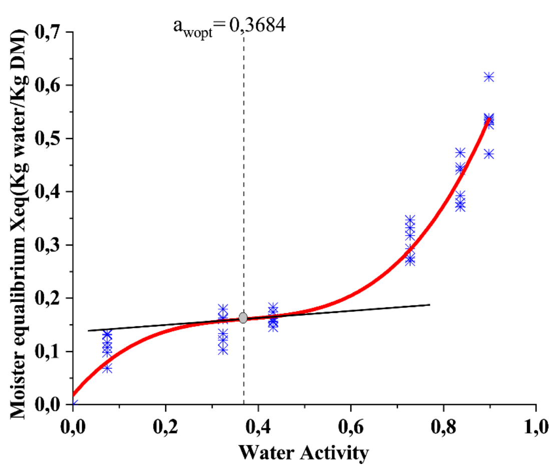

Fig. 6 Determination of the optimal water activity for storage of *Punica granatum* arils.

0.1028 respectively (Table 3). The net isosteric heat of sorption (Δh_d) or differential enthalpy stands for the binding energy of the water to the substrate. This energy must be added to the heat vaporization energy (Δh_{vap}) for dehydration (Equation 5). The net isosteric heat of sorption was calculated from the experimental data using the Clausius Clapeyron equation given by Equation (6) (Beristain *et al*., 1996).

3.1.2 Optimal water activity for drying and storage

The physical characteristics of pomegranate arils are provided in Table 5. *Punica granatum* arils' optimum water activity for drying and storage is included in the known general stability range for biological products, between 0.2 and 0.4 (Le Meste *et al*., 2001) (Fig. 6).

The optimal water for drying and conservation of pomegranate arils is a_w = 0.3684± 0.0309 calculated by the following polynomial equation:

$$X_{eq} = 2.29348a_w^3 - 2.53503a_w^2 + 1.00555a_w + 0.02764 \quad (21)$$

3.1.3 Net isosteric heat of sorption

The net isosteric heat of sorption of *Punica granatum* arils at temperatures ranging between 30°C and 50°C is presented in Fig. 7. For the small moisture content values (Xeq=0.07kg water/kg db), a higher net isosteric heat of sorption (Δh_d) is observed (154.21±1.41 kJ.mol^{-1}), indicating the highest binding energy for water removal and decreases with the increase of Xeq. The same trend of results was found by Wojdyło *et al*., (2019) for jujube fruit and Hamza *et al*., (2020) for anchovy. High moisture content requires lower levels of isosteric heat than samples with lower moister content (Prasantha, 2018).

Table. 1 standard water activities of saturated salts solutions used at 30, 40, and 50°C.

	KOH	$MgCl_2, 6H_2O$	K_2CO_3	$NaNO_3$	KCl	$BaCl_2, 2H_2O$
30°C	0.738	0.3238	0.4317	0.7275	0.8362	0.898
40°C	0.626	0.3159	0.423	0.71	0.8232	0.891
50°C	0.572	0.3054	0.4091	0.6904	0.812	0.8823

Table. 2 Mathematical models used for the fitting for experimental data of sorption isotherms.

	Equation	Reference
GAB	$X_{eq} = \frac{ABCa_w}{[1-Ba_w][1-Ba_w+BCa_w]}$ $B = B_0 e^{h_1/RT}, C = C_0 e^{h_2/RT}$	(van den Berg and Bruin, 1981)
Modified Henderson	$1 - a_w = e^{[-A(\theta+B)X_{eq}^C]}$	(Thompson *et al.*, 1986)
Peleg	$X_{eq} = A(a_w)^C + B(a_w)^D$	(Peleg, 1992)
Enderby	$X_{eq} = \left[\frac{A}{1-Ba_w} + \frac{C}{1-Da_w}\right] a_w$	(Popovski and Mitrevski, 2004)

Table. 3 coefficients of models describing the sorption isotherms of pomegranate arils.

	Parameters	**Adsorption**			**Desorption**		
		30°C	**40°C**	**50°C**	**30°C**	**40°C**	**50°C**
GAB	*r*	0.98273	0.99424	0.99332	0.98187	0.9688	0.97815
	SEM	0.11253	0.10785	0.10863	0.10891	0.10761	0.10873
Henderson modified	*r*	0.8199	0.86772	0.9112	0.83001	0.86588	0.90175
	SEM	0.15346	0.14116	0.13268	0.13974	0.13365	0.13008
Peleg	*r*	0.98423	0.99583	0.9948	0.99607	0.97779	0.97992
	SEM	0.11021	0.10543	0.10635	0.10199	0.10251	0.10407
Enderby	*r*	0.97779	0.99347	0.99682	0.99536	0.97486	0.97717
	SEM	0.11129	0.10627	0.1774	0.10299	0.1019	0.10526

Table. 4 Effective moisture diffusivities obtained for dried Moroccan pomegranate arils.

Drying technique	**T(°C)**	D_{eff} **($m^2 \cdot s^{-1}$)**	*r*
Convective drying	40	$2.27\ 10^{-7}$	0.9947
	50	$6.73664\ 10^{-7}$	0.9978
	60	$1.27\ 10^{-6}$	0.9543
	70	$5.62414\ 10^{-6}$	0.9847
Greenhouse drying	≈ 55	$1.14914\ 10^{-6}$	0.9675

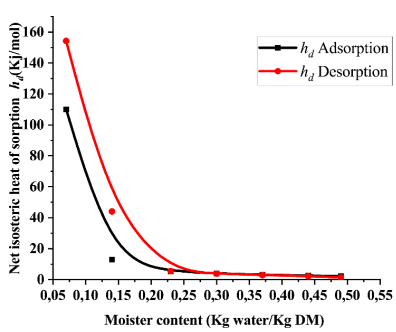

Fig. 7 Net isosteric heat of sorption of dried pomegranate arils.

3.1.4 Effective moisture diffusivity and activation energy

The effective moisture diffusivities of dried pomegranate arils under different conditions are shown in Table 4. In convective solar drying, an increase in drying air temperature induces a proportional increase in effective moisture diffusivity, this positive correlation between drying temperature and effective moisture diffusivity was in accordance with many studies on drying kinetics of biological and food products (Hidar *et al.*, 2020; Tagnamas *et al.*, 2020). The effective moisture diffusivity value for greenhouse drying is higher than convective dried

Table. 5 Physical characteristics of *Punica granatum* arils.

	Values ± SD
Arils ratio (%)	60.08 ± 6.04
Peel ratio (%)	39.92 ± 6.03
Water content (%)	78.0 ± 0.11%
Optimal water activity	0.37 ± 0.03
Net isosteric heat of sorption (kJ.mol^{-1})	154.21 ± 1.41

Values are expressed as means±standard deviation of replicated three times.

Table. 6 Biochemical parameters of *Punica granatum* arils as affected by different drying techniques.

	Fresh	SD 40°C	SD 50°C	SD 60°C	SD 70°C	GH
Titratable acidity (mg citric acid/L)*	4.22±0.41[a]	3.20±0.13[b]	2.36±0.38[c]	1.86±0.15[cd]	1.43±0.14[d]	2.62±0.39[bc]
Ascorbic acid content (mg/g DM)*	3.93±0.34[a]	1.53±0.05[b]	0.96±0.11[c]	0.73±0.05[cd]	0.53±0.05[d]	0.73±0.05[cd]
Total Protein (mg/g DM)*	163.4±7.02[a]	86.82±5.86[b]	77.10±7.20[bc]	68.56±3.36[cd]	60.80±2.54[d]	70.73±4.76[cd]
Total sugars (mg/g DM)*	785.80±54.50[a]	396.89±26.30[d]	491.63±15.01[c]	554.49±31.20[bc]	616.50±16.88[b]	244.65±5.71[e]
Anthocyanins (mg Cyanidine-3-glucoside/100g DM)*	80.8±0.23[a]	59.0±0.15[bc]	62.8±0.09[b]	58.0±0.20[c]	49.8±0.11[d]	29.0±0.08[e]

Values are expressed as means±standard deviation of replicated three times.

SD: indirect solar drying at 40, 50, 60, and 70°C; GH: greenhouse drying.

Values with different letters in the same ligne are significantly different (p<0.05).

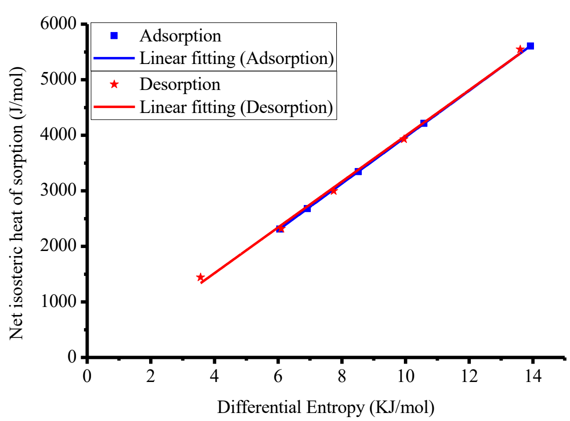

Fig. 8 Determination of the optimal water activity for storage of *Punica granatum* arils.

samples at 50ºC even if the temperature inside is about ≈ 55°C and approaches 60°C in convective drying. It could be attributed to the concentrated solar irradiations and lower air velocity inside the chamber. Increased moisture diffusivity at higher temperatures is explained by increasing the water molecules' activity as a consequence of increasing the heating energy (Doymaz & Altiner, 2012).

The activation energy presents the level of energy needed by the process of diffusion to start. For the pomegranate arils, the activation energy Ea is 91.84 KJ.mol^{-1}. Similar results were found for other food materials, such as Moroccan truffle, for which the activation energy value was 76.37 kJ.mol^{-1} (Tagnamas *et al*., 2020).

Compensation theory: The correlation plot of net isosteric heat of sorption (Δh_d) against differential entropy (Δs_d) is presented in Fig. 8 showing a high linear correlation coefficient r (0.996 for adsorption and 0,999 for desorption), assuming the existence of the compensation theory. This regression allowed us to determine the isokinetic temperatures (T_β) and the free energy (ΔG_d), which are presented in the following equations:

$$\Delta h_{adsorption} = 364.68\Delta s_d + 297.52 \qquad (22)$$

$$\Delta h_{desorption} = 337.96\Delta s_d + 597.06 \qquad (23)$$

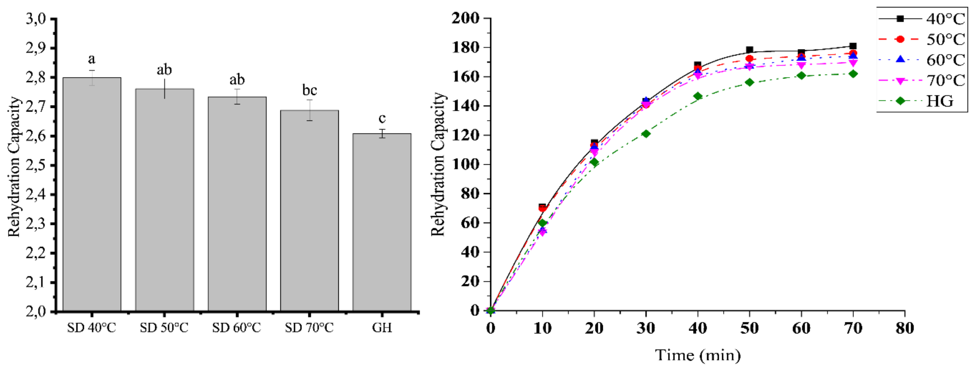

Fig. 9 Rehydration capacity and weight gain percentage of dried samples of *Punica granatum* arils.

The compensation theory is validated by comparing the isokinetic temperature to the harmonic mean temperature (T_{hm} = 313.25K). The two temperature parameters were very different ($T_\beta \neq T_{hm}$), confirming the compensation theory for pomegranate arils. For both adsorption and desorption, $T_\beta > T_{hm}$, and as previously explained, the sorption process is enthalpy driven. Similar results were found for sweet cherry powder (Ouaabou *et al*., 2021) and dried figs (Hssaini *et al*., 2020), concluding that the sorption process of dried figs was enthalpy driven and its microstructure is stable without suffering from any changes during the process.

The free energy values (ΔG_d) for both adsorption and desorption were positive, which suggests that the sorption process is non-spontaneous, as also found for dried figs (Hssaini *et al*., 2020).

3.2 Rehydration capacity

The rehydration capacity analysis is one of the most discussed parameters used to investigate the quality attributes of dried products. Rehydration reveals the physical and structural changes and damages induced by different drying processes.

The rehydration capacity of dried pomegranate arils by different drying techniques is presented in Fig. 9. The highest value was obtained by convective solar-dried samples at 40°C (2.79 ± 0.025), followed in order by 50°C, 60°C, and 70°C. In contrast, the lowest value was found at the greenhouse dried samples (2.76 ± 0.034; 2.73 ± 0.025; 2.68 ± 0.035 and 2.60 ± 0.014, respectively). The results showed that drying techniques and drying temperature significantly affected the rehydration capacity. The increase in drying temperature was associated with a decrease in the rehydration ratio. The highest temperature of 70°C showed the lowest rehydration ratio for the convective solar-dried samples, and the highest ratio was obtained at 40°C. This attitude has already been brought up in the work of Horuz & Maskan, (2013) on the pomegranate arils. Higher temperatures could cause damage in form and structure by reducing the capillaries responsible for conducting water into the cells and then decreasing the rehydration capacity (Meda & Ratti, 2005). The greenhouse dried samples had a lower rehydration ratio than convective solar drying, which could be explained by the direct contact of the product with solar radiations (Sidhu, 2016).

3.3 Titratable acidity and total ascorbic acid content

TA of fresh and dried pomegranate arils by different techniques is given in Table 6 The fresh samples revealed a higher TA (4.22 ± 0.41 mg citric acid/L); which is higher than what was found in the work of Loukhmas *et al*., (2021) while comparing different Moroccan varieties of pomegranate, and revealed that different varieties had a TA ranging from 1.83 ± 0.11 to 2.09 ± 0.01 mg citric acid/L. In our study, all dried samples showed lower values compering to fresh ones which rangs between 3.20 ± 0.14 and 1.43 ± 0.15 mg citric acid/L. The drying caused a decrease in acidity due to the evaporation of some sensible heat acids as found for hot air dehydrated figs (Piga *et al*., 2004). The highest TA of the fresh product was spotted for dried ones at 40°C and the lowest at 70°C. The AAC results follow the same variation for all the tested methods and temperatures, except its higher sensitivity for heat (60% loss) compared to acidity (25%) from 40 to 70°C. The fresh samples and the samples dried at convective solar drier at 40°C had the highest content (3.93 ± 0.34 and 1.53 ± 0.06 mg/g DM, respectively), and the lowest was obtained for the samples dried by convective solar drier at 70°C (0.53 ± 0.058 mg/g DM). The AAC was significantly affected by the drying method and temperature. The degradation of vitamin C could explain those variations at high temperatures and for a long drying period or both by the oxidation of L-ascorbic acid into dehydroascorbic acid through enzymatic degradations (Montoya-Ballesteros *et al*., 2017; Wang *et al*., 2017) . The same conclusions were cited by Ruvini *et al*., (2017). However, Turkiewicz *et al*. (2019) suggested that the increase in temperatures in convective drying could generate new compounds from L-ascorbic acid that might explain the massive loss in its content. For the two parameters, greenhouse dried samples had higher contents than samples dried at SD70°C due to the low temperature inside the greenhouse (55 ± 3°C).

3.4 Total protein and sugar content

The content of total sugars and protein of pomegranate arils at the different drying techniques are presented in Table 6 and expressed as mg/g of dry matter. Both parameters were significantly affected by the drying technique and temperature. The highest protein content was obtained from convective solar-dried samples at 40°C (86.82 ± 5.86 mg/g DM) followed by 50°C (77.10 ± 7.20 mg/g DM). The lowest content was observed in convective solar-dried samples at 70°C (60.80 ± 2.54 mg/g DM). In convective solar drying, when the drying temperature goes from 40 to 70°C, protein content drops by 30%.

Tian *et al*., (2016) had the same conclusions for mushrooms. Drying caused protein denaturation and cell embrittlement, leading to protein loss (Chen *et al*., 2016). no significant differences existed between greenhouse and convective dried samples at 60°C (70.73 ± 4.76 and 68.56 ± 3.36 mg/g DM, respectively). The evaporation rate could explain these results in both methods related to the variable temperature in the greenhouse drier (Guiné *et al*., 2015).

The sugar content shifted inversely with drying compared to protein content. It increases with drying temperature. This variation could be explained by reducing moister during drying allows for increased sugar concentrations (Krešić *et al*., 2004). Also, the increase of drying temperature leads to decreased drying time which means less exposition time to heat and consequently more preservation of sugars. The highest sugar content following the fresh samples (785.80 ± 54.50 mg/g DM) was observed for convective solar-dried samples at 70°C (616.50 ± 16.88 mg/g DM) followed by convective dried samples at 60°C (554.49 ± 31.20 mg/g DM). The increase of the drying temperature from 40 to 70°C causes an increase in the sugar content by 35%. The lowest sugar content was found in greenhouse dried samples, which could be explained by the direct contact with solar irradiations causing caramelization and sugar degradations by Maillard reactions (Guiné *et al*., 2015; Slatnar *et al*., 2011).

3.5 Total anthocyanins content

As antioxidants, secondary metabolites (polyphenols, flavonoids, and anthocyanins) are essential compounds for human health as they contribute to minimizing the risks of many diseases (Ruvini *et al*., 2017). The effect of the drying techniques on total anthocyanins of *Punica granatum*'s arils is presented in Table 6. The results are presented as mg of Cyanidine-3-glucoside/100 g DM.

Drying pomegranate arils caused a decrease in anthocyanins contents compared to fresh samples. Results were significantly affected by the drying technique and temperature. The Anthocyanins content of fresh samples was 80.8 ± 0.23 mg Cyanidine-3-glucoside/100 g DM. The same value was found by Benchagra *et al*., (2021) on the same variety, concluding that the Moroccan sefri pomegranate had higher anthocyanins levels compared to Italian, Iranian, Turkish, Indian, and Tunisian varieties. However, Loukhmas *et al*., (2020) light on more anthocyanins content in different varieties of Moroccan pomegranate aril juices. The highest value was found for convective dried samples at 50°C (62.8 ± 0.09 mg Cyanidine-3-glucoside/100 g DM), and then the samples dried at 40°C (59;0 ± 0.15 mg Cyanidine-3-glucoside/100 g DM). The lowest value was given by greenhouse dried samples (29.0 ± 0.08 mg Cyanidine-3-glucoside/100 g DM). The anthocyanins content decreased as follows:

$$\text{Fresh} > \text{Convective drying } 50°\text{C} > \text{Convective drying } 40°\text{C} > \text{Convective drying } 60°\text{C} > \text{Convective drying } 70°\text{C} > \text{Greenhouse.} \quad (24)$$

Anthocyanins belong to the polyphenol family. This could explain its low value when drying at 40°C for a long exposure (long drying time). Thus, resulting in a delayed deactivation of oxidative enzymes (polyphenols oxidase and peroxidase) (Ruvini *et al*., 2017). Direct sun-dried samples have the lowest polyphenols content compared to indirect solar-dried samples. Also, changes in chemical structures, such as binding to proteins, could cause lower values (Sharma & Thakur, 2016).

3.6 Color measurement

The color measurements are used as a quality indicator to visually evaluate the deterioration level caused by the drying process (Doymaz & Altiner, 2012). L^*, a^*, and b^* are mainly applicable to calculate different parameters (chroma, hue, and browning index) which describes the color changes during the drying process of fruits and vegetables. Changes are related to more factors, such as the degradation of vitamins, pigments, phenolic compounds, enzyme activities, and chemical reactions between the product's components (Turkiewicz *et al*., 2019).

The color parameter changes in fresh and dried pomegranate arils by the different techniques are presented in Table 7. For L^*, the highest value was found for fresh samples (33.31 ± 2.45), followed by greenhouse dried samples (23.73 ± 1.06), then convective solar-dried samples at different temperatures of 40°C, 50°C, 60°C, and 70°C (20.13 ± 0.44; 19.72 ± 0.62; 18.01 ± 0.82 and 16.32 ± 0.58 respectively). An increase in drying temperature has caused a decrease in lightness values which means that drying darkened the samples, as Turkiewicz *et al*., (2019) on dried Japanese quince fruit. Convective dried samples at different temperatures have the highest values of a^* 26.63 ± 0.56; 24.92 ± 0.24; 22.92 ± 0.84, and 21.61 ± 1.08, respectively), followed by fresh and greenhouse dried samples (22.84 ± 0.99 and 13.67 ± 0.59 respectively). Horuz & Maskan, (2013) found the same trend on convective dried pomegranate arils, explained that the changes result from the anthocyanin compound destruction occurring in Maillard reactions that lead to a change of natural color from red to darker red. The analysis of variance showed that the color parameters were significantly affected by the drying technique and temperature. The increase in drying temperature causes a decrease in parameters L^* and a^* and a slight increase in parameter b^*. Indeed, the highest value of b^* was obtained by the convective solar at 70°C and greenhouse dried samples (10.07 ± 0.12 and 9.06 ± 0.98, respectively), and the lowest value was found for fresh samples (6.08 ± 0.48) (Guiné *et al*., 2015). Color difference ΔE* points out the variations in color between the fresh and dried samples as the literature mentioned that a ΔE* higher than 3 is considered very distinct. All dried samples had shown higher values. Samples dried at higher temperatures had the most important color differences. Convective dried samples at 70°C and 60°C had the most noticeable differences significantly based on the analysis of variance with values of 83.31±4.81 and 79.19±4.47, respectively. In contrast, no significant differences were noticed for the other samples.

The values of chroma (C*) revealed a higher intensity or saturation in convective dried samples at 40°C and 50°C (27.52±0.68 and 26.19±0.35 respectively) when compared to the other samples, including the fresh ones; this is due to an elevation of concentrations of the pigments after water evaporation after the drying. In addition, the hue (h^*) values are inversely correlated to chroma values indicating that the lowest the hue angle, the highest the red color intensity(García-Valladares *et al*., 2022). Both parameters C* and h^* conclude that drying at 40°C and 50°C preserve and concentrate the red color of pomegranate arils, while greenhouse drying (16.41±0.88 and 0.58±0.05 respectively for C* and h^*) deteriorate the color properties.

Table. 7 Color parameters of dried *Punica granatum* arils as affected by drying techniques.

	Fresh	SD 40°C	SD 50°C	SD 60°C	SD 70°C	GH
L^*	33.31±2.45a	20.13±0.44c	19.72±0.62c	18.01±0.82cd	16.32±0.58d	23.73±1.06b
a^*	22.84±0.99bc	26.63±0.56a	24.92±0.24ab	22.92±0.84bc	21.61±1.08c	13.67±0.59d
b^*	6.08±0.48d	6.93±0.55cd	8.04±0.43bc	8.41±1.02abc	10.07±0.12a	9.06±0.98ab
ΔE*	-	13,59±1,53a	13,74±2,06a	13,88±1,98ab	15,48±1,73b	17,50±1,90a
C*	23.64±1.07b	27.52±0.68a	26.19±0.35a	24.44±0.54b	23.85±1.01b	16.41±0.88c
h^*	0.26±0.01d	0.25±0.014d	0.31±0.01cd	0.35±0.05c	0.44±0.02b	0.58±0.05a
BI	45.51±2.58b	80.07±3.07a	77.99±1.27a	79.19±4.47a	83.31±4.81a	41.31±2.95b

Values are expressed as means±standard deviation of replicated three times.

SD: indirect solar drying at 40, 50, 60, and 70°C; GH: greenhouse drying.

Values with different letters in the same ligne are significantly different ($p<0.05$).

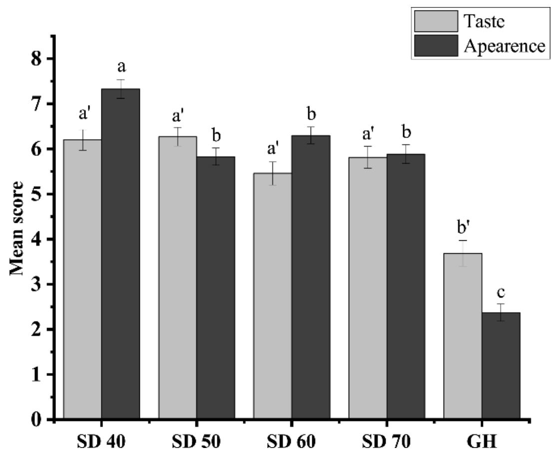

Fig. 10 Sensory evaluation of *Punica granatum* arils dried samples by different drying techniques.?

The browning (BI) indicates the degree of browning reactions occurring during the drying process. The highest values were given to the convective dried samples, showing that the browning increased with increased drying temperature(García-Valladares *et al*., 2022). The analysis of variance showed that the drying method significantly affects the browning index. However, for convective drying, the temperature does not affect the browning index significantly, even though the values vary according to drying temperatures. The greenhouse showed the lowest value of the browning index due to the higher value of b^* and the lowest value of a^*, indicating an increase in yellow color against the red color (García-Valladares *et al*., 2022).

3.7 Sensory analysis

The results of sensory analysis of dried pomegranate arils by different drying techniques are presented in Fig. 10. There were no significant differences between all drying temperatures of convective solar-dried samples for taste, but there is a slight advantage between 50 and 40°C (6.27 ± 0.204 and 6.20 ± 0.229). At the same time, the lowest score was found for greenhouse dried samples (3.69 ± 0.284). For the appearance, the samples were divided based on the panel's responses into three groups; the first group comprised convective solar-dried samples at 40°C, which recorded the highest score (7.33 ± 0.210). The second group contains the convective dried samples at 50, 60, and 70°C with no significant differences in-between (5.83 ± 0.189; 6.30 ± 0.184 and 5.89 ± 0.208, respectively).

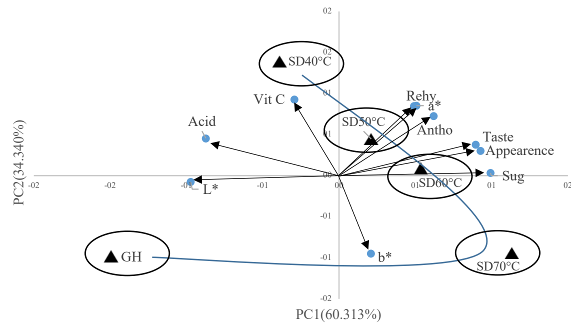

Fig. 11 PCA biplot of biochemical and sensory analysis parameters Acid: Acidity; Vit C: Vitamin C content; Rehy: rehydration capacity; Sug: total sugars content; L^*. a^*. and b^*: color parameters; SD: indirect solar drying at 40. 50. 60. and 70°C; GH: greenhouse drying.

However, the last group contains the greenhouse dried samples (2.37 ± 0.190). These results showed that the drying technique and temperature affect the taste and appearance parameters. The more the drying temperature increased, the less the panel appreciated the product's taste and appearance. For appearance, the results are associated with the degradation of pigments color known by anthocyanins responsible for the red color of pomegranate arils (Yalin *et al*., 2010).

Anthocyanins are chemically unstable and quickly degraded when exposed to higher temperatures (Beaudry *et al*., 2004). For the taste, all panel members have confirmed and agreed that the greenhouse dried samples with the lowest sugar content are not sweet compared to convective dried samples. This effect results from the sugars degradation due to the Maillard reaction at higher temperatures, causing color browning and sugar burning, resulting in an undesired taste (Dipersio *et al*., 2006).

3.8 Principal component analysis (PCA)

The sensory analysis scores and different biochemical and color parameters of dried pomegranate arils were subjected to a principal component analysis (PCA) to evaluate the relationships between those parameters under different drying conditions (Fig. 11).

The PCA analysis presents a gradient of drying intensity from indirect solar drying at 40°C (less intense drying) to indirect solar drying at 70°C and greenhouse drying (intense drying). The analysis reduces the factors into two principal components (PC1 and PC2), representing 94.653% of the information. The variables: appearance, taste, L^*, sugars, and acidity are correlated to PC1 (60.313%) plotted on the x-axis. While, variables: b^*, anthocyanins, a^*, and vitamin C are positively correlated to PC2 (34.340%) plotted on the y-axis. PC1 is explained by the panel's appreciation (taste and appearance) correlated positively to sugar content and negatively to acidity. However, PC2 is explained primarily by vitamin C and the red color of highly correlated samples to anthocyanins.

Greenhouse drying is characterized by direct solar irradiation and lack of ventilation, which form a distinct group from solar drying (SD) treatment but have some similarities with SD40°C and SD70°C. The SD70°C and GH dried samples are correlated inversely to PC1; the first sample was more appreciated, had more sugar content, and had the lowest acidity. While the second is the less liked product by the panel in both tests (taste and appearance), the lowest content explains this in anthocyanins and a^* value. That could be due to the Maillard reactions, resulting in changes in color (browning) and undesired taste (Dipersio *et al.*, 2006). In addition, GH and SD40°C samples are a little similar according to PC1 in acidity and lightness L^*.

3.9 *Effect of drying on the microstructure of pomegranate arils*

3.9.1 *Microscopic analyses*

Microscopic analyses revealed the effect of different drying temperatures and processes on the surface texture of pomegranate arils (Fig. 12). Convective drying at 40°C was characterized by minor structural damage in cells, followed by samples dried at 50°C, regardless of the degree of shrinkage caused by the force of water evaporation and longer drying time at lower temperatures (Turkmen *et al.*, 2020). In addition, the degradation cell's structure was increased proportionally with the drying temperature, which is observed in samples dried at 60°C and 70°C. Greenhouse dried samples appeared to be the most damaged at different stages of observation. The surface texture is sharper and more destructed, shown at 100× magnification. These changes could have resulted from the direct contact with solar irradiation and the longer drying time the samples undergo in these conditions.

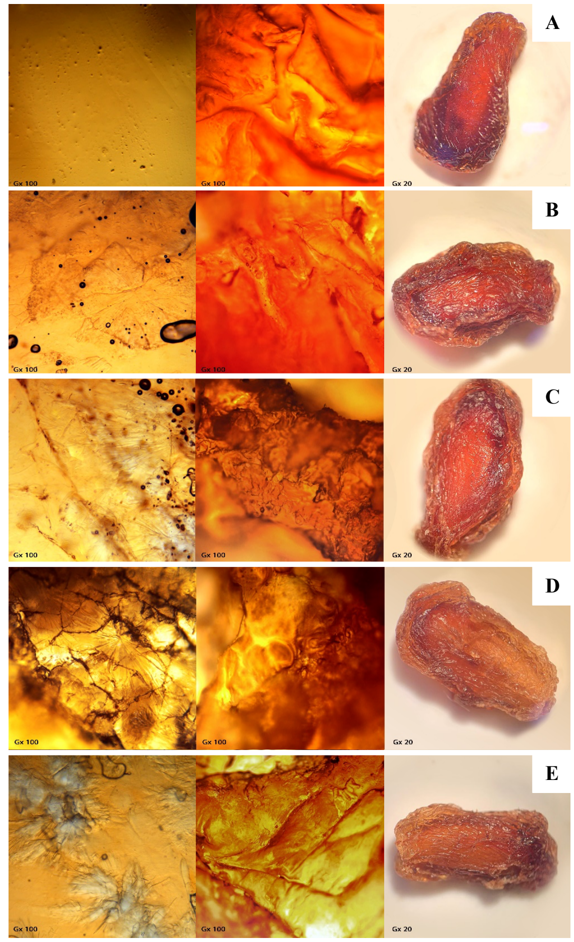

Fig. 12 Microscopic images of dried pomegranate arils. A: CSD40°C; B: convective solar drying at 50°C; C: convective solar drying at 60°C; D: convective solar drying at 70°C; E: greenhouse drying.

3.9.2 *Scanning electron microscopy (SEM) analyses*

SEM images obtained for dried pomegranate arils using convective solar drying at 40, 50, 60, and 70°C, and arils dried in the greenhouse are resumed in Fig. 13. The microstructural analysis highlighted the effect of drying temperature on the structure surface of dried arils. Increasing drying temperature caused structural damage as the cells and tissues collapsed and shrinkage. These damages are more pronounced at higher temperatures. Samples dried at 70°C had a more shrunken texture with a reduction of smother areas than samples dried at 40°C, presenting a more homogeneous surface, concluding that increasing drying temperature the surface of dried arils regularity reduces as also found by Santacruz-Vázquez *et al.* (2008).

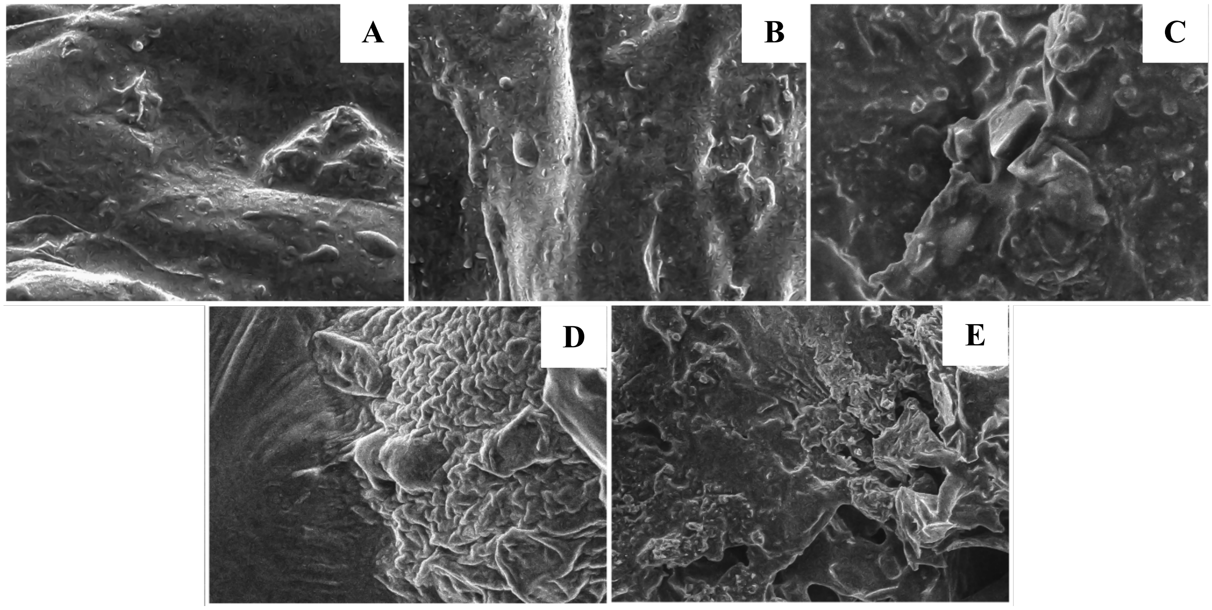

Fig. 13 Scanning electron microscopy (SEM) images at 2000× of dried pomegranate arils. convective solar drying at 40°C (A); 50°C (B); 60°C (C); and 70°C (D); and samples dried at greenhouse (E).

However, Horuz & Maskan, (2013) found that higher drying temperatures resulted in a lower shrinkage ratio from 78.57% at 50°C to 75.62% at 70°C for pomegranate arils, but the differences are non-significant. The increase in damage at higher temperatures could be because of the higher moisture diffusion and evaporation rates from the interior to the surface of the dried product (Morales-Delgado *et al*., 2014; Turkmen *et al*., 2020). The greenhouse dried samples showed the highest damage in all dried samples presented in shrinkage and cracked texture, which could be caused by higher temperature and direct contact with solar irradiations inside the chamber because of the greenhouse effect. These conditions encourage the increase in water diffusion and the residual stress on the product's surface, turning it into a glassy form, which takes cracking as the shape of damage (Bonazzi & Dumoulin, 2014).

Conclusions

The optimal water activity for drying and storing the arils is 0.3684±0.0309, and the isosteric heat of sorption is 154.21±1.41 kJ.mol^{-1}. The effective moisture diffusivity ranges from 1.27× 10^{-6} to 2.27× 10^{-7} KJ.mol^{-1}, with an average activation energy of 91.84 KJ.mol^{-1}. Convective drying at 40 and 50°C gives the product the highest quality at different parameters under study compared to different drying processes used. More precisely, Convective dried samples at 50°C provide the best combination of the nutraceutical and organoleptic (appearance, crispiness, and sweetness) parameters. The greenhouse dried samples had the lowest contents in sugar and anthocyanins (244.65±5.71; 29.0±0.08, respectively) and the most substantial structural damage and color deterioration. Higher drying temperatures also give lower quality samples for different parameters, especially for forced convective drying at 70°C. Dried pomegranate arils could be stored for long periods and consumed as healthy food in many ways: in salads, as snacks, sweets, and desserts. More studies are required to adjust the drying conditions to enhance the quality and shelf life of the dried pomegranate arils, which widen the range of applications and enhance the income of farmers and workers in this industry in Morocco.

Acknowledgments

The research institute for solar energy and new energies (IRESEN) supported this work as part of the project SSH. Japan International Cooperation Agency (JICA)-Japan Science and Technology Agency (JST)'s Science and Technology Research Partnership for Sustainable Development (SATREPS) project, and the

Ministry of Higher Education, Scientific Research and Executive Training (MHESRET) of the Kingdom of Morocco.

Nomenclature

Xeq	Equilibrium moisture content
a_w	Water activity
SEM	Standard Error of Moisture
r	Correlation coefficient
$Xeq_{i,exp}$	Experimental moisture content
$Xeq_{i,pre}$	Predicted moisture content
d_f	Freedom degree of the regression model
Δh_d	Net isosteric heat of sorption
Δh_{vap}	Heat vaporization energy
ΔG_d	Gibbs free energy
D_{eff}	Effective moisture diffusivity
E_a	Activation energy
Δs	Entropy
T_β	Isokinetic temperature
T_{hm}	Harmonic mean temperature
TA	Titratable Acidity
BSA	Bovine Serum Albumin
CIE	International Commission on Illumination
SEM	Scanning Electron Microscopy
PCA	Principal Component Analysis
ANOVA	Analysis of variance

References

Adiba, A., Hssaini, L., Haddioui, A., Hamdani, A., Charafi, J., El Iraqui, S., and Razouk, R. (2021). Pomegranate plasticity to water stress: attempt to understand interactions between cultivar, year, and stress level. *Heliyon 7(6)*, 1-9. https://doi.org/10.1016/j.heliyon.2021.e07403

Adiba, A., Razouk, R., Charafi, J., Haddioui, A., and Hamdani, A. (2021). Assessment of water stress tolerance in eleven pomegranate cultivars based on agronomic traits. *Agricultural Water Management 243*, 1-7. https://doi.org/10.1016/j.agwat.2020.106419

Barbosa-Cánovas, V., G., J. Fontana Jr., A., Schmidt, S. J., and Labuza, P., T. (2007). *Water Activity in Foods Fundamentals and Applications*.

Beaudry, C., Raghavan, G. S. V., Ratti, C., and Rennie, T. J. (2004). Effect of four drying methods on the quality of osmotically dehydrated cranberries. *Drying Technology 22(3)*, 521-539. https://doi.org/10.1081/DRT-120029999

Benchagra, L., Berrougui, H., Islam, M. O., Ramchoun, M., Boulbaroud, S., Hajjaji, A., Fulop, T., Ferritti, G., & Khalil, A. (2021). Antioxidant effect of Moroccan pomegranate (*Punica granatum* L. Sefri Variety) extracts rich in punicalagin against the oxidative stress process. *Foods 10*, 2219. https://doi.org/https://doi.org/10.3390/foods10092219.

Beristain, C. I., Garcia, H. S., and Azuara, E. (1996). Enthalpy-entropy compensation in food vapor adsorption. *Journal of Food Engineering 30*, 405-415.

Bhardwaj, A. K., Kumar, R., and Chauhan, R. (2019). Experimental investigation of the performance of a novel solar dryer for drying medicinal plants in the Western Himalayan region. *Solar Energy 177*, 395-407. https://doi.org/10.1016/j.solener.2018.11.007

Bonazzi, C., and Dumoulin, E. (2014). Quality changes in food materials as influenced by drying processes. In *Modern Drying Technology*, 1-20. https://doi.org/10.1002/9783527631728.ch14

Bradford, M. M. (1976). A rapid and sensitive method for the quantitation of microgram quantities of protein utilizing the principle of protein-dye binding. *Analytical Biochemistry 72(1-2)*, 248-254.

Calín-Sánchez, Á., Lech, K., Szumny, A., Figiel, A., and Carbonell-Barrachina, Á. A. (2012). Volatile composition of sweet basil essential oil (*Ocimum basilicum* L.) as affected by drying method. *Food Research International 48(1)*, 217-225. https://doi.org/10.1016/j.foodres.2012.03.015

Chen, X., Li, X., Mao, X., Huang, H., Miao, J., and Gao, W. (2016). Study on the effects of different drying methods on physicochemical properties, structure, and *in vitro* digestibility of *Fritillaria thunbergia* Miq. (*Zhebeimu*) flours. *Food and Bioproducts Processing 98*, 266-274. https://doi.org/10.1016/j.fbp.2016.01.008

Chowdhury, M. S., Ahmed, A., Hoque, M. R., Rahman, A., Saied, S. U. H., and Ealahe, H. M. H. K. (2016). Determination of amount of vitamin C (Ascorbic Acid) from the supplied drug by using iodometric titration. *Department of Pharmacy International Islamic University Chittagong*.

Crank, J. (1975). *The Mathematics of Diffusion* (Second Edition). Clarendon Press, Oxford.

Dak, M., Sagar, V. R., and Jha, S. K. (2014). Shelf life and kinetics of quality changes in dried pomegranate arils in flexible packaging. *Food Packaging and Shelf Life 2-7*. https://doi.org/10.1016/j.fpsl.2014.04.005

Dipersio, P. A., Kendall, P. A., and Sofos, J. N. (2006). Sensory evaluation of home-dried fruit prepared using treatments that enhance the destruction of pathogenic bacteria. *Journal of Food Quality 29(1)*, 47-64. https://doi.org/10.1111/j.1745-4557.2006.00055.x

Doymaz, I., and Altiner, P. (2012). Effect of pretreatment solution on drying and color characteristics of seedless grapes. *Food Science and Biotechnology 21(1)*, 43-49. https://doi.org/10.1007/s10068-012-0006-4

Dubois, M., Gilles, K. A., Hamilton, J. K., Rebers, P. A., and Smith, F. (1956). Colorimetric method for determination of sugars and related substances. *Analytical Chemistry 28(3)*, 350-356. https://doi.org/10.1021/ac60111a017

El Ferouali, H., Doubabi, S., El Kilali, T., and Abdenouri, N. (2016). CFD study of a designed forced convection solar dryer. application to the drying of *Punica granatum legrelliae*'s flowers. Gifu, Japan: *The 20th International Drying Symposium (IDS 2016)*.

García-Valladares, O., Cesar-Munguia, A. L., López-Vidaña, E. C., Castillo-Téllez, B., Ortiz-Sánchez, C. A., Lizama-Tzec, F. I., & Domínguez-Niño, A. (2022). Effect by using a modified solar dryer on physicochemical properties of carambola fruit (*Averrhoa carambola* L.). *Revista Mexicana de Ingeniería Química 21(1)*, 1-14. http://www.redalyc.org/articulo.oa?id=62029966013

Ghasemnezhad, M., Zareh, S., Shiri, M. A., and Javdani, Z. (2015). The arils characterization of five different pomegranates (*Punica granatum*) genotypes stored after minimal processing technology. *Journal of Food Science and Technology 52(4)*, 2023-2032. https://doi.org/10.1007/s13197-013-1213-6

Greenspan, L. (1977). Humidity fixed points of binary saturated aqueous solutions. *Journal of Resources of the National Bureau of Standards - Section A. Physics and Chemistry 81(1)*, 89-96.

Guiné, R. P. F., Almeida, I. C., Correia, A. C., and Gonçalves, F. J. (2015). Evaluation of the physical, chemical, and sensory properties of raisins produced from grapes of the cultivar Crimson. *Journal of Food Measurement and Characterization 9(3)*, 337-346.

Hamza, L., Mounir, K., Younes, B., Zakaria, T., Haytem, M., Hind, M., Abdelkader, L., and Ali, I. (2020). Physicochemical study of the conservation of Moroccan anchovies by convective solar drying. *Renewable Energy 152*, 44-54. https://doi.org/10.1016/j.renene.2020.01.039

Hidar, N., Ouhammou, M., Mghazli, S., Idlimam, A., Hajjaj, A., Bouchdoug, M., Jaouad, A., and Mahrouz, M. (2020). The impact of solar convective drying on kinetics, bioactive compounds, and microstructure of stevia leaves. *Renewable Energy 161*, 1176-1183. https://doi.org/10.1016/j.renene.2020.07.124

Hmid, I., EloT_{hm}ani, D., Hanine, H., Oukabli, A., and Mehinagic, E. (2017). Comparative study of phenolic compounds and their antioxidant attributes of eighteen pomegranate (*Punica granatum* L.) cultivars grown in Morocco. *Arabian Journal of Chemistry 10*, S2675-S2684. https://doi.org/10.1016/j.arabjc.2013.10.011

Hmid, I., Hanine, H., EloT_{hm}ani, D., and Oukabli, A. (2018). The Physico-chemical characteristics of Morrocan pomegranate and evaluation of the antioxidant activity for their juices I. *Journal of the Saudi Society of Agricultural Sciences 17(3)*, 302-309. https://doi.org/10.1016/j.jssas.2016.06.002

Horuz, E., and Maskan, M. (2013). Hot air and microwave drying of pomegranate (*Punica* *granatum* L.) arils. *Journal of Food Science and Technology 52(1)*, 285-293. https://doi.org/10.1007/s13197-013-1032-9

Hssaini, L., Ouaabou, R., Charafi, J., Idlimam, A., Lamharrar, A., Razouk, R., and Hanine, H. (2020). Hygroscopic proprieties of fig (*Ficus carica* L.): Mathematical modeling of moisture sorption isotherms and isosteric heat kinetics. *South African Journal of Botany*. https://doi.org/10.1016/j.sajb.2020.11.026

Ismail, F. A., Abdelatif, S. H., El-Mohsen, N. R. A., and Zaki, S. A. (2014). The physicochemical properties of pomegranate juice (*Punica granatum* L.) extracted from two Egyptian varieties. *World Journal of Dairy and Food Sciences 9(1)*, 29-35. https://doi.org/10.5829/idosi.wjdfs.2014.9.1.1131

Kar, C. El, Mtimet, N., Ferchichi, A., and Bouajila, J. (2013). Relationships between fruit acceptability and health-case of seven pomegranates (*Punica granatum* L.) juices. *Food and Nutrition Sciences 04(08)*, 119-130. https://doi.org/10.4236/fns.2013.48a015

Koukouch, A., Idlimam, A., Asbik, M., Sarh, B., Izrar, B., Bah, A., and Ansari, O. (2015). Thermophysical characterization and mathematical modeling of convective solar drying of raw olive pomace. *Energy Conversion and Management 99*, 221-230. https://doi.org/10.1016/j.enconman.2015.04.044

Krešić, G., Lelas, V., and Šimundić, B. (2004). Effects of processing on nutritional composition and quality evaluation of *candied celeriac. Sadhana - Academy Proceedings in Engineering Sciences 29(1)*, 1-12. https://doi.org/10.1007/BF02706997

Le Meste, M., Roudaut, G., Chiotelli, E., Simatos, D., and Colas, B. (2001). Propriétés fonctionnelles de l'eau dans les aliments: Dossier eau. *Industries Alimentaires et Agricoles 118(5)*, 21-28.

Morales-Delgado, D. Y., Téllez-Medina, D. I., Rivero-Ramírez, N. L., Arellano-Cárdenas, S., López-Cortez, S., Hernández-Sánchez, H., Gutiérrez-López, G., and Cornejo-Mazón, M. (2014). Effect of convective drying on total anthocyanin content, antioxidant activity and cell morphometric parameters of strawberry parenchymal tissue (*fragaria x ananassa Dutch*). *Revista Mexicana de Ingeniera Quimica 13(1)*, 179-187.

Mundada, M., and Bahadur Singh, H. (2012). Studies on moisture sorption isotherms for osmotically pretreated and air-dried pomegranate arils. *Journal of Food Processing and Preservation 36(4)*, 329-38.

Loukhmas, S., Kerak, E., Outaki, M., Ettalibi, F., Kharbouch, H. A., and Harrak, H. (2021). Physicochemical criteria, bioactive compounds, antioxidant activity, and sensory attributes of ten Moroccan pomegranate cultivars. *European Journal of Horticultural Science 86(4)*, 339-353. https://doi.org/10.17660/eJHS.2021/86.4.1

Loukhmas, S., Kerak, E., Outaki, M., and Belaqziz, M. (2020). Assessment of minerals, bioactive compounds, and antioxidant activity of ten Moroccan pomegranate cultivars. *Journal of Food Quality 2020*, 10. https://doi.org/10.1155/2020/8844538.

Meda, L., and Ratti, C. (2005). Rehydration of freeze-dried strawberries at varying temperatures. *Journal of Food Process Engineering 28*, 233-246.

Montoya-Ballesteros, L. C., Gonzales-Leon, A., Martinez-Nunez, Y. J., Robles-Burgueno, M. R., Garcia-Alvarado, M. A., Rodriguez-Jimenes, G. C. (2017). Impact of open sun drying and hot air drying on capcaisin, capsanthin, and ascorbic acid content in chiltepin (*Capsicum Annum* L. var. *Glabriusculum*). *Revista Mexicana de Ingeniería Química 16(3)*, 813-25. http://www.redalyc.org/articulo.oa?id=62029966013.

Moussaoui, H., Bahammou, Y., Idlimam, A., Lamharrar, A., and Abdenouri, N. (2019). Investigation of hygroscopic equilibrium and modeling sorption isotherms of the argan products: A comparative study of leaves, pulps, and fruits. *Food and Bioproducts Processing 114*, 12-22. https://doi.org/10.1016/J.FBP.2018.11.002

Mujumdar, A. S. (2006). *Handbook of Industrial Drying* (3rd ed.). Ed. Marcel Dekker Inc.

Ouaabou, R., Ennahli, S., Lorenzo, C. Di, Hanine, H., Bajoub, A., Lahlali, R., Idlimam, A., Oubahou, A. A., and Mesnaoui, M. (2021). Hygroscopic properties of sweet cherry powder: thermodynamic properties and microstructural changes. *Journal of Food Quality*.

Pascual-Pineda, L. A., Hernández-Marañon, A., Castillo-Morales, M., Uzárraga-Salazar, R., Rascón-Díaz, M. P., and Flores-Andrade, E. (2020). Effect of water activity on the stability of freeze- dried oyster mushroom (*Pleurotus ostreatus*) powder. *Drying Technology*, 1-14. https://doi.org/10.1080/07373937.2020.1739064.

Peleg, M. (1992). Assessment of a semi-empirical four-parameter general model for sigmoid moisture sorption isotherms. *Journal of Food Process Engineering 16(1)*, 21-37.

Piga, A., Pinna, I., Özer, K. B., Agabbio, M., and Aksoy, U. (2004). Hot air dehydration of figs (*Ficus carica* L.): drying kinetics and quality loss. *International Journal of Food Science and Technology 39(7)*, 793-799. https://doi.org/10.1111/j.1365-2621.2004.00845.x

Popovski, D., and Mitrevski, V. (2004). Some new four-parameter models for moisture sorption isotherms. *Electronic Journal of Environmental, Agricultural and Food Chemistry 3(3)*, 698-701.

Prasantha, B. D. R. (2018). Prediction of moisture adsorption characteristics of dehydrated fruits using the GAB isotherm model. *Annals of Agricultural Crop Science 3(1)*, 1-4. www.austinpublishinggroup.com

Ruvini, L., Dissanayaka, W., Chathuni, J., Rizliya, V., Swarna, W., and Barana, C. J. (2017). Effect of different drying methods on antioxidant activity of star fruits (*Averrhoa Carambola* L.). *Journal of Nutrition and Diet Supplements 1(1)*, 1-6.

Santacruz-Vázquez, V., Santacruz-Vázquez, C., Welti-Chanes, J., Farrera-Rebollo, R. R., Alamilla-Beltrán, L., Chanona-Pérez, J., Gutiérrez-López, G. F. (2008). Effects of air-drying on the shrinkage, surface temperatures and structural features of apples slabs by means of fractal analysis. *Revista Mexicana de Ingeniería Química 7(1)*, 55-63.

Senadeera, W., Adiletta, G., Önal, B., Matteo, M., Di Russo, P. (2020). Influence of different hot air drying temperatures on drying kinetics, shrinkage, and colour of persimmon slices. *Foods 9(101)*, 1-12.

Sharma, A., and Thakur, N. S. (2016). Influence of active packaging on quality attributes of dried wild pomegranate (*Punica granatum* L.) arils during storage. *Journal of Applied and Natural Science 8(1)*, 398-404. https://doi.org/10.31018/jans.v8i1.806

Sidhu, S. J. (2016). Thesis: Optimization of greenhouse drying process for white button mushroom (*Agaricus bisporus*). Punjab Agricultural University, India.

Slatnar, A., Klancar, U., Stampar, F., and Veberic, R. (2011). Effect of drying of figs (*Ficus carica* L.) on the contents of sugars, organic acids, and phenolic compounds. *Journal of Agricultural and Food Chemistry 59(21)*, 11696-11702.

Stover, E., and Mercure, E. W. (2007). The pomegranate: A new look at the fruit of paradise. *HortScience 42(5)*, 1088-1092. https://doi.org/10.21273/HORTSCI.42.5.1088

Tagnamas, Z., Bahammou, Y., Kouhila, M., and Hilali, S. (2020). Conservation of Moroccan truffle (*Terfezia boudieri*) using solar drying method. *Renewable Energy 146*, 16-24. https://doi.org/10.1016/j.renene.2019.06.107

Thompson, T. L., Peart, R. M., and Foster, G. H. (1986). Mathematical simulation of corn drying, a new model. *Transactions of the ASAE 11(4)*, 582-586.

Tian, Y., Zhao, Y., Huang, J., Zeng, H., and Zheng, B. (2016). Effects of different drying methods on the product quality and volatile compounds of whole shiitake mushrooms. *Food Chemistry 197*, 714-722. https://doi.org/10.1016/j.foodchem.2015.11.029

Turkiewicz, I. P., Wojdyło, A., Lech, K., Tkacz, K., and Nowicka, P. (2019). Influence of different drying methods on the quality of Japanese quince fruit. *LWT - Food Science and TechnologyFood 114*, 108416. https://doi.org/10.1016/j.lwt.2019.108416

Turkmen, F., Karasu, S., and Karadag, A. (2020). Effects of different drying methods and temperature on the drying behavior and quality attributes of cherry laurel fruit. *Processes 8(7)*. https://doi.org/10.3390/PR8070761

Van den Berg, C., and Bruin, S. (1981). Water activity and its estimation in food systems: Theoretical aspects. In *Water Activity: Influences on Food Quality* (pp. 1-61). Elsevier. https://doi.org/10.1016/B978-0-12-591350-8.50007-3

Wang, J., Yang, X. H., Mujumdar, A. S., Wang, D., Zhao, J. H., Fang, X. M., Zhang, Q., Xie, L., Gao, Z. J., and Xiao, H. W. (2017). Effects of various blanching methods on weight loss, enzymes inactivation, phytochemical contents, antioxidant capacity, ultrastructure, and drying kinetics of red bell pepper (*Capsicum annuum* L.). *LWT - Food Science and Technology 77*, 337-347. https://doi.org/10.1016/j.lwt.2016.11.070

Wojdyło, A., Lech, K., Nowicka, P., Hernandez, F., Figiel, A., and Carbonell-Barrachina, A. A. (2019). Influence of different drying techniques on phenolic compounds, antioxidant capacity, and color of *Ziziphus jujube* mill. Fruits. *Molecules 24(13)*. https://doi.org/10.3390/molecules24132361

Yalin, M. D., Glazer, I., Bar-ILan, I., Kerem, Z., Holland, D., and Amir, R. (2010). Color, sugars, and organic acids composition in aril juices and peel homogenates prepared from different pomegranate accessions. *Journal of Agricultural Food Chemistry 58*, 4342-4352. https://doi.org/10.1021/jf904337t

Zhao, P., Zhong, L., Zhao, Y., and Luo, Z. (2015). Comparative studies on the effect of mineral matter on physicochemical properties, inherent moisture, and drying kinetics of Chinese lignite. and Management. *Energy Conversion 93*, 197-204.