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EXPERIMENTAL STUDY OF PRODUCTION AND CHARACTERIZATION OF DATE FRUIT POWDERS AND SYRUP

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	ABSTRACT In the present work, date pulp and pomace powders were produced under freeze drying conditions and syrup was extracted from Garn Ghzel date variety. Date products were characterized in terms of physicochemical properties (moisture, water activity, ash, soluble solids content, titrable acidity, pH and color) and functional properties (water holding capacity, wettability index, dispersibility and density). Freeze drying kinetic of date pulp and pomace was modeled using five empirical models (Newton, Page, Henderson and Papis, Logarithmic and Wang and Singh). Results showed that there is a slight difference between powders properties. Page and Logarithmic models best fitted the freeze drying kinetic of date pulp and pomace with the highest determination coefficient, R^2 (0.9635) and R^2 (0.9987) and the lowest chi-square χ^2 (0.000021) and χ^2 (0.000052) values respectively. Fick's law was used to determine the effective moisture diffusivity. Its values were 9.74×10^{-11} and 5.15×10^{-11} m^2/s for date pulp and pomace respectively. These results contribute added value to date technology.

1.Introduction

Date palm *(Phoenix dactylifera L.)* is extensively cultivated for its edible fruit belonging to the *Palmae (Arecaceae)* family (Yahaya *et al.*, 2015). The fruit of date palm is used due to its remarkable nutritional, health and economic value, in addition to its aesthetic and environmental benefits. Date composition is rich in carbohydrates, minerals, dietary fiber, vitamins, fatty acids, amino acids and protein diet (Al-Shahib and Marshall, 2003).

Algeria is one of the largest date producers in the world with a wide production diversity. Garn Ghzel variety is not widely consumed despite its high total and reducing sugar contents (Mrabet *et al.*, 2008). Date processing industries produce various date products such as paste, syrup, jam and vinegar. Date syrup is probably the most common derived date product (Ganbi,

2012). Production of date powder could improve handling, storage and blending ability of several products such as baked foods (Manickavasagan *et al.*, 2015).

In recent years, drying operations have made possible the production of various added value food products (Omolola *et al.*, 2015). Freeze drying is a drying method used to manufacture pharmaceutical and food thermolabile products (Nireesha *et al.*, 2013). It is considered a low processing drying method.

To our knowledge, no work has been reported on the production of powders from date pomace as well as the processing of Garn Ghzel variety.

Hence, the objective of this work is to produce pulp, pomace powders and syrup from Garn Ghzel date variety, to determine their

physicochemical and functional properties and to model their freeze drying kinetic.

2. Materials and methods
2.1. Vegetable materials

Date fruit (Garn Ghzel variety) was harvested at full maturity (Tamr) and stored at 4°C in a refrigerator, (SUMSUNG, South Korea). It was purchased from the region of Sidi Okba (Biskra, Algeria). The fruit was washed using water and divided into 2 parts; the first part used to characterize the pulp, the second part to produce date syrup and pomace.

Date syrup was prepared according to Al-farsi (2003) method with a slight modification: A chopper was used to cut dates into pieces of 1 cm, then dates were mixed with an equal amount of distilled water. The mixture was stirred for 20 min at 60°C in a water bath, (DK-420, China).

Finally, the juice was extracted through a muslin cloth. Filtration was performed in two steps; coarse (50 μ filter paper) and fine (3 μ filter paper).

Concentration was performed on a hot plate, (IKA-COMBIMAG RCT, Germany) (90°C) (Daas Amiour *et al.*, 2014), until syrup reached 35°Brix.

Date pulp and pomace were freeze dried in a laboratory freeze dryer model (LD 2-8 CHRIST BETA PLUS, Germany) for 24 hours at a pressure and temperature of 0.12 mbar and -40°C respectively. The thickness of date pulp and pomace was measured using digital caliper, LCD screen 0.01mm-150mm (Electronic Vernier, UK) (Goula Athanasia and Adamopoulos Konstantinos, 2004). Samples were ground and sieved in a sieve of 1mm diameter (Figure 1).
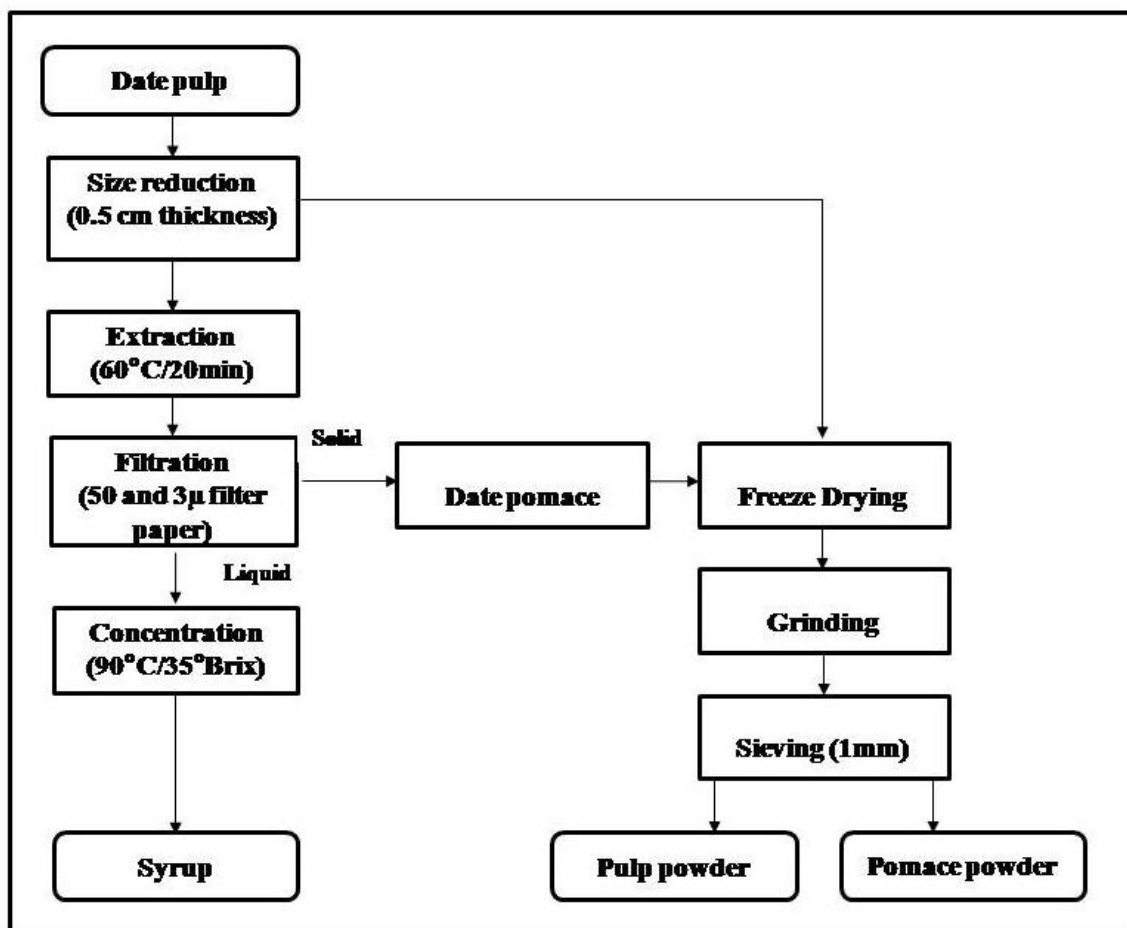

Figure 1. Simplified flowchart of date powders and syrup processing

2.2. Physicochemical and functional analysis of powders and syrup

2.2.1. Physicochemical analysis

For powders, moisture content was determined by drying at 70°C to obtain constant weight in a moisture analyzer, (RADWAG MA 110.R.NS, Poland) and moisture loss expressed as a percentage of (100 kg water / kg wet material) (Goula Athanasia and Adamopoulos Konstantinos, 2004). For syrup, moisture content was obtained by drying at 105°C in a forced convection laboratory oven, (ESCO isotherm, USA) until constant weight (Gurak *et al.*, 2014).

Water activity of date pulp and pomace powders was measured using water activity meter model (rotronic HYGROSCOP BT-

RS1, Gemini BV), set at 24.70 ± 1°C (Caparino *et al.*, 2012). For date syrup, water activity was measured according to Fennir *et al.* (2003) with a slight modification, where samples have the same concentration and prepared without dilution.

For date powders and syrup, ash was determined by combustion of the sample in a muffle furnace, (Nabertherm 30-3000°C, Germany) at 550°C for 8 h (Abbès *et al.*, 2011).

Soluble solids content of date powders and syrup was determined using a digital refractometer, (ATAGO, HSR500, Japan) (Kulkarni *et al.*, 2008).

Titrable acidity was determined by titrating a known quantity of sample with 0.1 N NaOH and expressed as citric acid (Kulkarni *et al.*, 2008).

pH was determined according to standard ISO 10390 (1994); the method consists of preparing a suspension component in five times its volume of distilled water, leave for 5 minutes then rest for at least two hours but no more than 24 hours. pH is measured using a pH meter, (HACH, France) (Kulkarni *et al.*, 2008).

Color: Powders and date syrup were poured into Petri dishes, formed a layer of 10 mm thick and covered with a transparent film. The colorimeter, (KONICA MINOLTA SENSING, INC CR-10, Japan) was calibrated with a

standard white ceramic plate before playback (L = 95.97, a= − 0.13, b = − 0.30). L, a, b parameters were measured for all samples. The average values L, a and b were obtained from six readings for each sample (Abonyi *et al.*, 2002).

2.2.2. Functional analysis

Density of syrup was calculated using the following equation:

$$Density = \frac{weight\ of\ syrup\ volume}{weight\ of\ the\ same\ volume\ of\ distilled\ water\ at\ 4°C} \quad (1)$$

Weighing is conducted using a precision balance model (RADWAG, AS 220.R2, Poland) with reading accuracy of 0.0001g and maximum capacity of 220g (Mimouni, 2015).

Water holding capacity (WHC) is determined by the method described by AACC (1995); 2 g of powders put in 200 ml of distilled water, were stirred for 30 seconds with a glass rod. The mixture rested 10 minutes, the stirring operation was repeated five times. Tubes were centrifuged for 20 min at 4000 rev/min in a centrifuge, (SIGMA 6-16 KS, Germany). The supernatant was carefully decanted, then the content of the tube was dried at 45°C for 10 minutes in an oven, (ESCO isotherm, USA) before being weighed, using a standard scale, (RADWAG, AS 220.R2, Poland). The water holding capacity was expressed as percentage increase in the sample's weight (Adepeju *et al.*, 2014).

The index of wettability is measured from the results obtained during the rehydration of powder without stirring. It is expressed as the time in seconds required for a given amount of powder to enter the water through its free surface at rest (Schuck *et al.*, 2012).

Dispersibility: 10 ml of distilled water at 25°C were poured into a beaker of 50 ml. 1 g of powder was added in the beaker. The stopwatch started and the sample stirred vigorously with a spoon for 15 seconds by 25 full movements back and forth across the diameter of the container. Reconstituted honey was poured through a sieve (112 microns). 1 ml of sieved honey was transferred into an aluminum pan weighed and dried for 2 hours in

a vacuum oven 70 ± 1°C model (BINDER, Germany).

Dispersibility of the powder was calculated as follows:

$$D\ (\%) = \frac{(10+m)\ x\ TS}{m\ x\ ((100-MC)/100))} \qquad (2)$$

m: amount of powder (g) being used, MC: Moisture content in the powder (db) and TS%: dry matter in of reconstituted honey after it has been passed through the sieve (Koç and Kaymak-Ertekin, 2014).

True density: determined as the ratio of the mass of dry solids (m) to total volume (Vs) of the sample excluding the air ports (Calín-Sánchez *et al.*, 2014):

$$\rho_t = \frac{m}{Vs} \qquad (3)$$

Samples were weighted with an analytical balance, (RADWAG, AS 220.R2, Poland), while Vs was measured with a pycnometer model (ISO LAB In 20°C, 10ml BORO 3.3 A, Germany).

Bulk density: Determined as the ratio of solid mass to the bulk volume (Vb). Samples were weighted with the analytical balance, (RADWAG, AS 220.R2, Poland) with reading accuracy of 0.0001g and the bulk volume was measured with an 80 mL graduated container (Calín-Sánchez *et al.*, 2014):

$$\rho b = \frac{m}{Vb} \qquad (4)$$

2.2.3. Mathematical modeling of freeze drying kinetics

Moisture ratio (MR) of date (pulp and pomace) during freeze drying was calculated using the following equation:

$$MR = \frac{Mt-Me}{M0-Me} \qquad (5)$$

Where: Mt, M0 and Me are moisture content at any time, the initial moisture and the equilibrium moisture (g water / g dry matter), respectively (Erbay and Icier, 2009).

The effective moisture diffusivity can be defined from Fick's second law of diffusion, which describes the movement of moisture within the solid.

$$\frac{\delta M}{\delta t} = D\frac{\delta^2 M}{\delta Z^2} \qquad (6)$$

Analytical solution of this law in the case of drying an infinite slab of thin layer, assuming moisture migration being by diffusion, one-dimensional moisture movement, uniform initial moisture distribution, negligible shrinkage, constant moisture diffusivity, and negligible external resistance to heat and mass transfer can be developed in the form of the following equation (Crank, 1975).

$$\frac{M-M_e}{M0-M_e} = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)}\exp\left[-(2n-1)^2\frac{\pi^2}{4}\frac{Dt}{L^2}\right] \quad (7)$$

Where L is the thickness (m); t is the time (min); and D is the effective diffusivity (m²/s). Simplifying the previous equation by taking the first term of the series solution and assuming Me equal to 0:

$$MR = \frac{M}{M0} = \frac{8}{\pi^2}\ .\exp\left[-\frac{\pi^2}{4}\frac{Dt}{L^2}\right] \qquad (8)$$

Where L the thickness of the sample and D the effective diffusion coefficient.

Taking logarithm on both sides of previous equation gives the following equation (Nag and Dash, 2016):

$$Ln\ MR = \left(Ln\frac{8}{\pi^2}\right) - \frac{\pi^2 Dt}{4L^2} \qquad (9)$$

The diffusion coefficient (D) is calculated using the method of slopes.

$$Slope = -\left(\frac{\pi^2 D}{4L^2}\right) \qquad (10)$$

Drying kinetics of date pulp and pomace were fitted to five models (Newton, Page, Henderson and Papis, Logarithmic, Wang and Singh). The quality of the adjustment was determined using the coefficient of

determination (R^2), the reduced χ -square (χ^2) (Ergun *et al.,* 2014) and root mean square error (RMSE) as follows:

$$\chi^2 = \frac{\sum_{i=1}^{N} \left(MR_{exp(i)} - MR_{pre(i)}\right)^2}{N-n} \qquad (11)$$

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N} \left(MR_{pre(i)} - MR_{exp(i)}\right)^2}{N}} \qquad (12)$$

Where MR_{expi} is the *i*th experimental moisture ratio, MR_{prei} is the *i*th predicted moisture ratio, N is the number of observations, n is the number of constants in drying model (Pardeshi *et al.*, 2009).

2.2.4. Statistical analysis

Analyses were performed with three replications, while color with six and results expressed as the mean ± standard deviation (SD). Results were submitted to the analysis of variance (ANOVA) and means were compared by Tukey's test using Origin Pro 2016 32 Bit software. Differences were considered to be significant at $P < 0.05$.

3.Results and discussions
3.1. Production of powders

100 g of pulp and pomace were freeze dried at a temperature of −40°C and a pressure of 0.12 mbar (Fig 1). After 24h, samples weight became 96.70 g and 63.50 g respectively.

Pulp and pomace samples have an initial water content of 0.11 and 0.45 (kg H_2O/ kg product) respectively. The relative water content of pomace decreases and reaches a level of 0.55 after 12 h. However, pulp reaches a value of 0.94 after 12 h, these results agree with Sahari *et al.* (2008) who studied the effect of initial moisture and date thickness on the drying process.

3.2. Physicochemical properties of powders

Physicochemical properties of date powders are shown in Table 1.

Table 1. Physicochemical properties of pulp and pomace powders

Parameters	Pulp powder	Pomace powder
Moisture (%)	2.50±0.05	7.64±0.22
Water activity	0.17±0.22	0.32±0.02
Ash (g/100g)	0.84±0.04	1.00±0.01
Soluble solids content (°Brix)	60±0.00	59.90±0.17
Titrable acidity (% citric acid)	3.03±0.20	2.57±0.20
pH	6.28±0.05	6.36±0.02
Color (L*)	78.57±0.23	68.9±0.36
Color (a*)	4.41±0.12	7.10±0.09
Color (b*)	22.43±0.29	25.00±0.17

Results are expressed as mean values of three determinations ± SD;
analysis of variance (ANOVA) p < 0.05 with Tukey's tests.
Color results are expressed as mean values of six determinations ± SD

Moisture content of pulp and pomace powders were 2.50±0.05 and 7.64±0.22% respectively. These results are similar to moisture content values of freeze dried papaya and pineapple, 2 and 7% (wb) respectively (Marques *et al.*, 2011).

However, they were higher than freeze dried marionberries and strawberries cultivars (0.10-0.20%) and corn cultivars (0.70-1.10%) (Asami *et al.*, 2003).

Titratable acidity of pulp and pomace powders were 3.03±0.20, 2.57±0.20 (% citric acid) respectively. They were higher than

freeze-dried papaya pulp powders with a value of 1.38±0.07% citric acid (Canuto *et al.*, 2014).

pH of date powders was almost similar with values of 6.28±0.05 and 6.36±0.02 of pulp and pomace powders respectively. These results were closer to those reported by Mahendran (2010) for freeze dried guava.

Microbiological, chemical and enzymatic stability of food products is related to water activity content (a_w), values of water activity ranging between 0.20 and 0.40 ensure stability of the product (Nur Dirim and Çalişkan, 2012), the a_w of pulp and pomace powders were 0.17±0.22 and 0.32±0.02 respectively. These values were similar to water activity value of freeze dried pumpking puree powder found as 0.20 (Nur Dirim and Çalişkan, 2012). Ashes and total soluble solids have values of 0.84±0.04, 1.00±0.01 (g/100g) and 60±0.00, 59.90±0.17°Brix for pulp and pomace powders respectively. These values were similar to those

reported in literature for samples of Mech Degla date powder 1.44±0.20 (g/100g) and 60°Brix.

Food color is a major determinant of product quality and affects consumer preferences. Color may be used as an indicator to predict chemical and quality change due to thermal processing (Valdenegro *et al.*, 2013). Color parameters (L*, a*, b) of pulp and pomace powders were shown in Table 1. Pulp and pomace powders were slightly orange (a*4.41±0.12) and (a*7.10±0.09), slightly yellow (b*22.43±0.29) and (b*25.00±0.17) respectively, the difference in color parameters is due to product composition. Similar L*, a*, and b* values were reported by Sablani *et al.* (2008) for date powder (Mech degla variety).

3.3. Functional properties of powders

Table 2 shows functional properties of pulp and pomace powders. Water holding capacity (WHC), wettability index and dispersibility show the ability of powders to rehydrate.

Table 2. Functional properties of pulp and pomace powders

Parameters	Pulp powder	Pomace powder
Water holding capacity (g water/g dry matter)	0.63±0.23	0.70±0.02
Wettability index (s)	930±0.50	1425±0.33
Dispersibility (%)	91.76±0.21	68.51±0.40
True density (Kg/m^3)	884.96±0.00	454.55±0.00
Bulk density (Kg/m^3)	909.00±0.00	505.05±0.00

Results are expressed as mean values of three determinations ± SD; analysis of variance (ANOVA) $p < 0.05$ with Tukey's tests.

According to Gurak *et al.* (2014), WHC is defined as the amount of water retained by the sample without being subjected to any stress. Pulp powder has a WHC value less than pomace with values of 0.63±0.23 and 0.70±0.02 (g of water / g dry matter) respectively. These results were lower than those obtained by Nguyen (2014) with values of 0.90 to 1.00 (g eau/ g powder) for soy powders produced by atomization and between 3.97 and 6.20 g/g dry fiber for date fiber concentrates varieties (Borchani *et al.,* 2010). The difference is due to composition and varieties. The wettability or the ability of powder to absorb water is one of the

most important physical properties related to reconstituting powders (Sarabandi *et al.*, 2014). The obtained results were 930±0.50 (s) for pulp which is lower than 1425±0.33 (s) for pomace. According to Manickavasagan *et al.* (2015), date powder obtained by spray drying has a wettability time ranged between 145.70 and 162.70 (s), dispersibility values of pulp and pomace powders were 91.76±0.21 and 68.51±0.40% respectively. These values are higher than results obtained by Koç and Kaymak-Ertekin (2014), ranging between 51.30 and 100% for spray dried honey powder.

Bulk density of powders is determined by particle density, which is determined by solid density and particle internal porosity, and also by spatial arrangement of particles in the container (Micha, 2005). True and bulk density (Table 2) of powder samples were 884.96±0.00-909.00±0.00 Kg/m^3 and 454.55±0.00 - 505.05±0.00 Kg/m^3 respectively. Pulp powder density was higher than pomace powder and bulk density was also higher than the true density. The difference is justified since powder heap reduces the space between particles, so volume will decrease. The obtained results were similar to Calín-Sánchez *et al.* (2014) results regarding freeze dried chokeberry fruits.

3.4. Physicochemical properties of syrup

Date syrup is an important date by-product and a natural sweetener considered as a suitable replacement of sugar in food products formulation, in order to reduce the harmful effect of sugar, and improving the nutrient properties (Raiesi Ardali *et al.*, 2014). Physicochemical properties of syrup were shown in Table 3.

Table 3. Physicochemical properties of date syrup

Parameters	Date syrup
Moisture (%)	61.81±0.08
Water activity	0.92±0.01
Ash (g/100g)	1.02±0.99
Soluble solids content (°Brix)	35±0.00
Titrable acidity (% citric acid)	0.58±0.20
pH	6.22±0.06
Color (L*)	33.06±0.23
Color (a*)	0.60±0.00
Color (b*)	6.93±0.14
Density (g/ml)	1.20±0.02

Results are expressed as mean values of three determinations ± SD; analysis of variance (ANOVA) $p < 0.05$ with Tukey's tests. Color results are expressed as mean values of six determinations ± SD. Moisture content and water activity of date syrup are 61.81±0.08% and 0.92±0.01 respectively, these results are similar to Ganbi (2012) with values of 74.06±6.10% for water bath date fruits juice (Dibs) and Fennir *et al.* (2003) for Saidi date syrups with values of 0.881. The difference could be due to date varieties. Syrup ash content is 1.02±0.99 (g/100g), this result is lower than Raiesi Ardali and Akbarian (2014) who found a value of 1.69 (g/100g). Total soluble solids, titrable acidity and pH have values of 35±0.00°Brix, 0.58±0.20 (% citric acid) and 6.22±0.06 respectively, which are closer to values reported by Farahnaky *et al.* (2016); El-Sharnouby *et al.* (2014) and Ganbi (2012) respectively. Density has a value of 1.20±0.02 (g/ml) which is similar to Jamshidi Mokhber *et al.* (2008) with a value of 1.35 (g/ml). Color parameters (L*, a*, b) of syrup were shown in Table 3. Syrup was slightly orange (a*0.60±0.00) and slightly yellow (b*6.93 ±0.14). Similar L*, a*, and b* values were reported by Raiesi Ardali and Akbarian (2014); Fennir *et al.* (2003) for date syrup.

Table 4. Model constants and statistical parameters for date pulp and pomace

Date pulp					
Models	**Equations**	**Constants and coefficients**	R^2	χ^2	**RMSE**
Newton	MR=exp(-kt)	k= 0.00450	0.9403	0.000030	0.00152
Page	MR=exp(-kt^n)	k= 0.00231 n= 1.29878	0.9635	0.000021	0.00390
Henderson and Papis	MR=aexp(-kt)	a=1.00538 k= 0.00512	0.9613	0.000022	0.00401
Logarithmic	MR= aexp(-kt)+c	a=24.13641 k=0.00021 c=-23.13126	0.9619	0.000028	0.00398
Wang and Singh	MR= 1+at+bt^2	a=-0.00443 b=-0.00005	0.9630	0.000027	0.00392
Date pomace					
Models	**Equations**	**Constants and coefficients**	R^2	χ^2	**RMSE**
Newton	MR=exp(-kt)	k= 0.07042	0.6691	0.009080	0.08811
Page	MR=exp(-kt^n)	k= 0.25645 n= 0.37086	0.9774	0.007420	0.02307
Henderson and Papis	MR=aexp(-kt)	a=0.89177 k=0.05420	0.7824	0.007160	0.07152
Logarithmic	MR= aexp(-kt)+c	a=0.45683 k=0.41041 c= 0.54464	0.9987	0.000052	0.00546
Wang and Singh	MR= 1+at+bt^2	a=-0.10019 b= 0.00566	0.9688	0.001290	0.02710

## 3.5. Mathematical modeling of freeze drying kinetics

The freeze drying behavior was determined from the loss of samples weight. Total drying time was determined as 12 hours for the pulp and pomace samples. Similar results were obtained by Marques *et al.* (2011) for freeze dried pineapple, guava and mango pulp.

The experimental data were fitted using Origin Pro 2016 32 Bit software to five models (Newton, Page, Henderson and Papis, Logarithmic and Wang and Singh) (Table 4). The correlation coefficient (R^2) was used to define the best model of the drying process for the pulp and pomace date, with χ^2 having the lowest value (Togrul and Pehivan, 2002).

Figure 2 represents the experimental data of water content with the best mathematical model for pulp and pomace date.
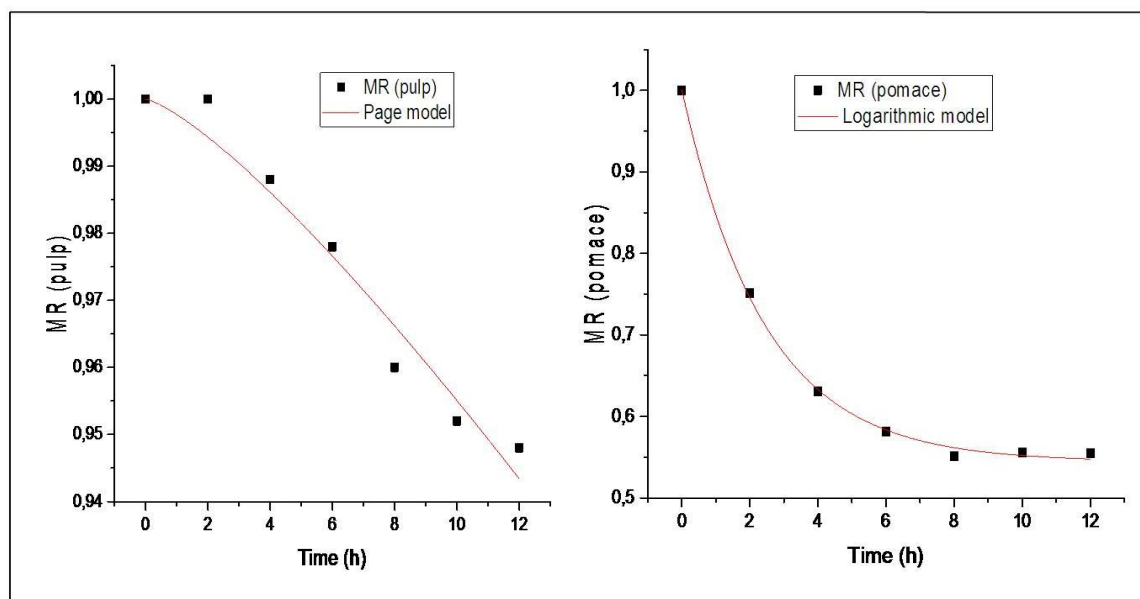
Figure 2. Plot of moisture ratio versus drying time for freeze dried pulp and pomace date (Page and Logarithmic models)

Diffusivity values were ranged between 9.74 × 10^{-11} and 5.15 × 10^{-11} m^2/s for pulp and pomace respectively. These values are within the range reported by Ergun *et al.* (2014) for the foods ranging from 10^{-12} to 10^{-6} (m^2/s).

4. Conclusions

This study investigated the production and characterization of pulp and pomace date powders as well as date syrup from Garn Ghzel variety. Powders were obtained using freeze drying process. The Garn Ghzel variety date shows interesting physicochemical and functional characteristics. The freeze drying process of pulp and pomace date was modeled and best fitted to Page and Logarithmic models respectively.

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