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Thin layer drying models, antioxidative activity and phenolic compounds of rose petals (*Rosa damascena* Mill.) in tray dryer

Abstract: The drying behavior of the Damask rose (*Rosa damascena* Mill.) petals in a thin layer hot air drying at 35, 45 and 55°C and 0.4, 1.2 and 2 ($m \cdot s^{-1}$) air velocity levels, was investigated. The Midilli was the most suitable model among 14 different thin-layer models for showing the thin-layer drying characteristics. At the experimental temperature (35-55°C) and air velocity levels (0.4, 1.2 and 2 $m \cdot s^{-1}$) the effective moisture diffusivity was 2.02×10^{-12}- 11.3×10^{-12} ($m^2 \cdot s^{-1}$) and the activation energy varied from 56.3 to 64.5 ($kJ \cdot mol^{-1}$). Total anthocyanin content (TAC) ranged 77-240 ($mg \cdot L^{-1}$). The rose petal extracts showed higher DPPH (59.51%) and lower FRAP (1213.24 $\mu mol(Fe^{+2}) \cdot L^{-1}$), at an increasing temperature from 35 to 55°C. Results showed that the higher temperature caused less TAC. At 45-55°C and air velocities 0.4-1.2 ($m \cdot s^{-1}$) the number of anthocyanins increased significantly, but at air velocities, 1.2- 2 ($m \cdot s^{-1}$) a decrease was observed.
Key words: *Rosa Damascena* Mill., Thin layer dryer, Anthocyanin, Phenolic compounds, Antioxidative power.

Introduction

Damask rose (*Rosa damascena* Mill.) is an ornamental plant from the family Rosaceae with more than 200 species and approximately 18000 cultivars. Damask rose is widely cultivated in Iran, Turkey, Bulgaria, India, Morocco, France, China, Italy, Libya, South Russia and the Ukraine. It is used for producing rose oil, water, concrete (rose oil solid), and absolute, which are significant base materials for the medicine, food products, and cosmetic industry [1]. Some factors like genotype of Damask rose, environment conditions, the time of harvesting, and the technology of processing and distillation, substantially affect the amount, percentage and compounds of rose essential oil and extract [2]. Numerous compounds can be extracted from pistil, ovary and sepal of Rose flower such as flavonoids, glycosides, terpenes and anthocyanins. Studies shows that Damask rose and rosehip seed oils are significantly rich in unsaturated fatty acids (omega fatty acid), bitter principle, organic acids and tanning matter [3]. Dried flowers are also used as flavor and laxative agents [1].

The short harvest duration (40 days) of Rose flower and its high moisture make the transportation and storage a difficult task [4]. The destruction of high fresh petals occurs at high speed rates due to the growth of the living organisms such as bacteria, molds and yeasts. Therefore, drying at the appropriate moisture content is an important step in the process [5]. Drying rose petals would introduce new utilization opportunities such as decorative, herbal, aromatic, hydrotherapeutic and cosmetic uses. In addition, with an increased demand for using the roses in health and aroma therapy, the importance of preserving color, flavor, and essential oils during the drying process has been identified [6].

Drying method is one of the oldest processes for product preservation and protection. This technique decreases humidity from ~ 85% wb to ~% 8-12 wb. The drying process should be completed in a short period of time to prevent the decomposition and minimize the used energy for the business concerns [7]. The most common commercial technique of drying food and chemical products is air drying. Hot air drying in food processing decreases the drying

time and hence maintains the dried product quality [8]. Therefore, the temperature and humidity levels of drying air, drying speed, and drying duration should be optimized to achieve the highest product quality [9].

Thermal and physical characteristics such as heat and mass transfer, moisture diffusion, activation energy, and energy consumption are essential in the design of agricultural crops dryers [10]. The drying process and apparatus modeling are substantially important in order to optimize the operation parameters and improve the performance of the drying system. Thin layer drying removes the moisture from a porous material by evaporation, in which drying air is passed through a thin layer of the material until it reaches a moisture balance. The external factors of the process, the type and size of the product, and the internal diffusion mechanisms control the drying rate [11].

Economic justification, quality, and quantity of the dried petals are dependent on the proper drying methods. Therefore, designing an effective operating system to minimize the drying process time and optimize energy consumption is required. The objectives of this paper are to 1) investigate the drying behavior of Damask rose petals through thin-layer drying 2) determine the best mathematical model for describing the drying process kinetics and 3) evaluate the effects of hot air velocity and temperature on qualitative properties of petals.

Materials and methods

Sample Preparation

Fresh rose flowers were picked up early in the morning, April to May 2018, from Gonabad, Khorasan Razavi province, Iran. Samples were transported to the laboratory and petals were separated before drying.

Chemicals

The applied chemicals and solvents were analytical reagent grade and were supplied by Merck (Darmstadt, Germany) and Sigma–Aldrich (St. Louis, MO) Chemical Companies.

Experimental Design

A laboratory-model tray dryer, equipped with installed instrumentations was used for the drying test. Different parts of dryer were air inlet and outlet parts, control unit and drying chamber (in which perforated trays are placed horizontally). The specifications of the fabricated dryer along with instrumentations are all explained in detail in [12]. Before the start of the drying process, the desirable constant temperature was obtained by the electrical heaters. To execute a drying run at each temperature, the dryer turned on 30 minutes so that it would stabilize at the specified temperature and air velocity [12]. About 10 g of fresh petals were spread on the shelves as a thin layer and kept in the drying chamber. Drying experiments were conducted at the temperature of 35, 45 and 55°C and hot air velocities of 0.4, 1.2 and 2 $m \cdot s^{-1}$. The reduced moisture content of petals was recorded every 2 minutes to achieve a constant weight and obtain the drying curves.

Mathematical modeling

Moisture ratio (MR) of the petal samples was obtained using equation (1):

$$MR = (M_t - M_{te})/(M_0 - M_e) \quad (1)$$

where, M_t is the last moisture content, M_0 is the initial moisture content, and M_e is the equilibrium moisture content. In practice, M_e value is rather less than M_t or M_0. Thus, the equation can be simplified as follows [13]:

$$MR = M_t/M_0 \quad (2)$$

Moisture ratio data were consistent with 14 thin-layer models that were mainly used to assess their suitability for thin layer drying kinetics of foods. MATLAB R2013 tool was used to fit the experimental data to the 14 thin-layer models. Three statistical parameters including coefficient of determination (R^2), root mean squares error (RMSE), and sum square error (SSE) were used to determine the performance of the models. For quality fit, R^2 value should be closed to one while SSE and RMSE values should be closed to zero.

Moisture Loss Kinetics

Fick's second law of unsteady state diffusion can describe the transport of moisture during the drying process that occurs in the falling rate period for most food materials [14]. It is calculated using the equation (3):

$$\frac{\partial M}{\partial t} = D_{eff}\nabla^2 M \quad (3)$$

where, D_{eff} is effective moisture diffusion ($m^2\ s^{-1}$) and M is moisture content

Research shows that D_{eff} is related to temperature and the kind of material being dried, including its texture and structure [15]. For

sufficiently long period of drying, using the first term in the series in the equation is significant and therefore [16]:

$$MR = \frac{8}{\pi^2} \exp\left[\frac{-D_{eff}\pi^2 t}{4L^2}\right] \quad (4)$$

Taking the natural logarithm of the equation gives:

$$\ln(MR) = \ln\left(\frac{8}{\pi^2}\right) - \frac{\pi^2 D_{eff} t}{4L^2} \quad (5)$$

where, L is the half thickness of *Rosa damascena* petal (RDP). Hence, the effective moisture diffusivity (D_{eff}) is obtained by plotting the experimental drying data in terms of ln (MR) against drying time (s).

Energy of activation (E_a) in a sample is the minimum amount of energy required for the initiation of a drying process to cause moisture diffusion through the sample. Therefore, the activation energy was calculated using Arrhenius equation [10] as shown in Eq. (6):

$$D_{eff} = D_0 . exp\left(-\frac{E_a}{R_g T_{abs}}\right) \quad (6)$$

E_a: the energy of activation, D_0: the pre-exponential factor of Arrhenius equation ($m^2\ s^{-1}$), R_g: the universal gas constant (8.3143 $kJ \cdot mol^{-1} \cdot K^{-1}$), and T_{abs}: the absolute air temperature (°K). D_0 and the corresponding E_a were determined by plotting $\ln(D_{eff})$ versus 1/T [14].

Total Anthocyanin Content

Anthocyanin pigments undergo reversible structural transformations with a change in pH manifested by strikingly different absorbance spectra. The colored oxonium form predominates at pH 1.0 and the colorless hemiketal form at pH 4.5. The pH-differential method is based on this reaction, and permits accurate and rapid measurement of the total anthocyanins, even in the presence of polymerized degraded pigments and other interfering compounds. Briefly, transfer 1 mL extracted solution into 10 mL volumetric flask for preparing two dilutions of the sample, one adjust volume with potassium chloride buffer, pH 1.0, and the other with sodium acetate buffer, pH 4.5, diluting each. Let these dilutions equilibrate for 15 min. Measure the absorbance of each dilution at the 510 and 700 nm (to correct for haze), against a blank cell filled with distilled water [17]. TAC was expressed as cyanidin-3-glucoside (%w/w) equivalents and measured by the following equation (7):

Total anthocyanin content

$$(\%\, W/W) = \frac{A}{\varepsilon \times l} \times MW \times DF \times \frac{V}{W} \times 100\% \quad (7)$$

where,

$A = (A_{520nm} - A_{700nm})_{PH1.0} - (A_{520nm} - A_{700nm})_{PH4.5}$;

MW (molecular weight): 449.2 for cyanidin-3-glucoside; DF: dilution factor; W: sample weight (mg); l: diameter of spectrophotometer cell (cm); ε: 26,900 M extinction coefficient in $L \cdot mol^{-1} \cdot cm^{-1}$ for cyd-3-glu; and 10^3: factor for conversion from g to mg.

Determination of Antioxidant Activity

DPPH and FRAP assays were used to evaluate the total antioxidant activity.

The DPPH method: The radical scavenging activity of RPE (Rose petals extract) was tested according to the method described in [18]. RPE was mixed with 1 ml of 0.5 mM free radical 2, 2-diphenyl-1-picrylhydrazyl (DPPH) methanolic solution, stored in the dark for 30 min, and then the absorbance was measured at 517 nm. The radical scavenging activity (%RSA) of RPE was calculated by the following equation:

$$\%RSA = \frac{A_{control} - A_{sample}}{A_{control}} \times 100 \quad (8)$$

where A_{sample} is the absorbance of RPE at a particular level and $A_{control}$ is the absorbance of the DPPH solution.

The FRAP method: The FRAP (Ferric reducing-antioxidant power) assay was performed by the 2, 4, and 6-Tripyridyl-S-triazine (TPTZ) following the method of Benzie and Strain [19]. FRAP reagent was prepared by mixing acetate buffer (0.3 M, pH=3.6), TPTZ solution (10 mM) and $FeCl_3$ (20 mM) in the ratio of 10:1:1. Briefly, 90 µl RPE (10 gr L^{-1}), 2700 µl of freshly prepared FRAP reagent and 270 µl distilled water were mixed and warmed to 37°C in a water bath and absorptions were determined at 595 nm. A standard curve was prepared using different concentrations of (200–2000 Fe (II) $\mu mol \cdot L^{-1}$).

Total phenolic content (TPC) determination

The total phenolic content was determined espectrophotometrically using the Folin Ciocalteu reagent described by Singleton and Rossi [20]. The calibration curve was plotted by absorbance measurements of various concentrations of Gallic acid (0.04-0.4 $mg \cdot ml^{-1}$) at 760 nm.

Results and discussion

Drying Model Investigation

The Model constants, R^2, RMSE, and SSE for 14 thin layer drying models (at three different drying temperatures with three levels of hot air velocities) consistent with the moisture ratio are presented in Table 1.

Table 1 – Coefficient of determination range for some thin-layer drying models applied to *Rosa damascena* petals (RDP)

T (°C)	V (m.s^{-1})	Model name	Model equation	Coefficients a b c d h g k n						R^2	X^2	RMSE
35	0.4	Midilli	MR=a exp (-kt^n)+bt	0.9836	-0.00104			0.0062	1.24	0.999	0.00003	0.00522
45	0.4	Midilli	MR=a exp (-kt^n)+bt	0.9727	-0.00085			0.0185	1.256	0.999	0.00007	0.00780
55	0.4	Two term	MR=a exp (-bt)+c exp (-dt)	-14.02	0.1611	15.01	0.1544			0.999	0.00004	0.00585
35	1.2	Midilli	MR=a exp (-kt^n)+bt	0.9277	-0.00005			0.0012	1.733	0.995	0.00053	0.02218
45	1.2	Midilli	MR=a exp (-kt^n)+bt	0.9777	-0.00056			0.0320	1.146	0.999	0.00010	0.00946
55	1.2	Midilli	MR=a exp (-kt^n)+bt	0.9815	-0.00034			0.0428	1.33	0.998	0.00018	0.01208
35	2	Midilli	MR=a exp (-kt^n)+bt	0.9598	-0.00085			0.0043	1.315	0.999	0.00012	0.01056
45	2	Two term	MR=a exp (-bt)+c exp (-dt)	-17.74	0.1111	18.73	0.1073	0.0645		0.999	0.00005	0.00654
55	2	Jena&Das	MR=a exp (-kt+at^2)+c	-0.1729	0.6838	1.176				0.999	0.00005	0.00606

R^2, RMSE and SSE values varied from 0.995 to 0.999, 0.00522 to 0.02218, and 0.0006 to 0.0246, respectively. Compared to other models, Midilli drying model had the highest R^2 and the minimum RMSE and SSE values, at 35 °C air temperature and 0.4 (m·s^{-1}) while the two term model had the best performance for air velocity of 0.4 m·s^{-1} at 55°C and 2 m·s^{-1} at 45°C. Jena & Das was the best model for air velocity of 2 m·s^{-1} at 55 °C. For all the nine drying conditions, the Midilli was the most suitable model to show the thin-layer drying characteristics of the petals. Similar results were reported for *Rosa damascena* petals drying by Karimi and Bankar [21]. The Midilli model has been also successfully used to study the drying characteristics of agricultural products such as savory leaves and eggplant [22].

Effective Moisture Diffusivity and Activation Energy

Effective moisture diffusivity is influenced by temperature, air velocity and the kind of substance. The higher temperature and air velocity cause the shorter drying time due to increased thermal gradients and mass transfer and as a result, drying rate increases [23]. The values of temperature, air velocity and effective moisture diffusivity are showed in Table 2.

Table 2 – Variations effective moisture diffusivity at different temperatures and hot air velocities

Temperature (°C)	Hot air velocity (m·s^{-1})	R^2	D_{eff} (m^2 s^{-1})
35	0.4	0.95	2.2×10^{-11}
45		0.96	4.85×10^{-11}
55		0.99	7.68×10^{-11}
35	1.2	0.97	2.42×10^{-11}
45		0.98	4.9×10^{-11}
55		0.98	11.3×10^{-11}
35	2	0.94	2.02×10^{-11}
45		0.99	5.25×10^{-11}
55		0.98	9.29×10^{-11}

An increase in the temperature at constant air velocity increased the effective moisture diffusivity. Moisture diffusivity for the petals showed a minimum value of 2.02×10^{-12} $m^2\cdot s^{-1}$ at 35°C for air velocity of 2 $m\cdot s^{-1}$ and a maximum value of 11.3×10^{-12} $m^2\cdot s^{-1}$ at air velocity of 1.2 $m\cdot s^{-1}$ and 55°C. The results demonstrate that at a constant air velocity, the higher temperature caused the more effective moisture diffusivity. The most effective moisture diffusivity was observed at 55°C. Similarly, Sharayei et *al*. [24] found that the maximum value of effective moisture diffusivity is at the highest temperature.

Drying temperature affects the internal mass transfer during drying, and ultimately the moisture diffusivity [14]. This is due to a higher heating energy that increases the water molecules' activities and leads to a higher moisture diffusivity when samples are dried. Also, lower energy is required to remove the moisture at higher temperature because the water molecules are lightly bound to the food complex [25].

The Arrhenius equation was calculated for the activation energy (E_a) of air velocity values (9):

$$D_e = D_0 . exp\left(-\frac{E_a}{RT}\right) \quad (9)$$

E_a: The energy of activation ($kJ\cdot mol^{-1}$), R: universal gas constant (8.3143 $kJ\cdot mol^{-1}$), T: absolute air temperature (°K), D_0: The pre-exponential factor of the Arrhenius equation ($m^2\cdot s^{-1}$).

Figure 1 shows the plot of Ln(D_{eff}) versus 1/T at various treatments.

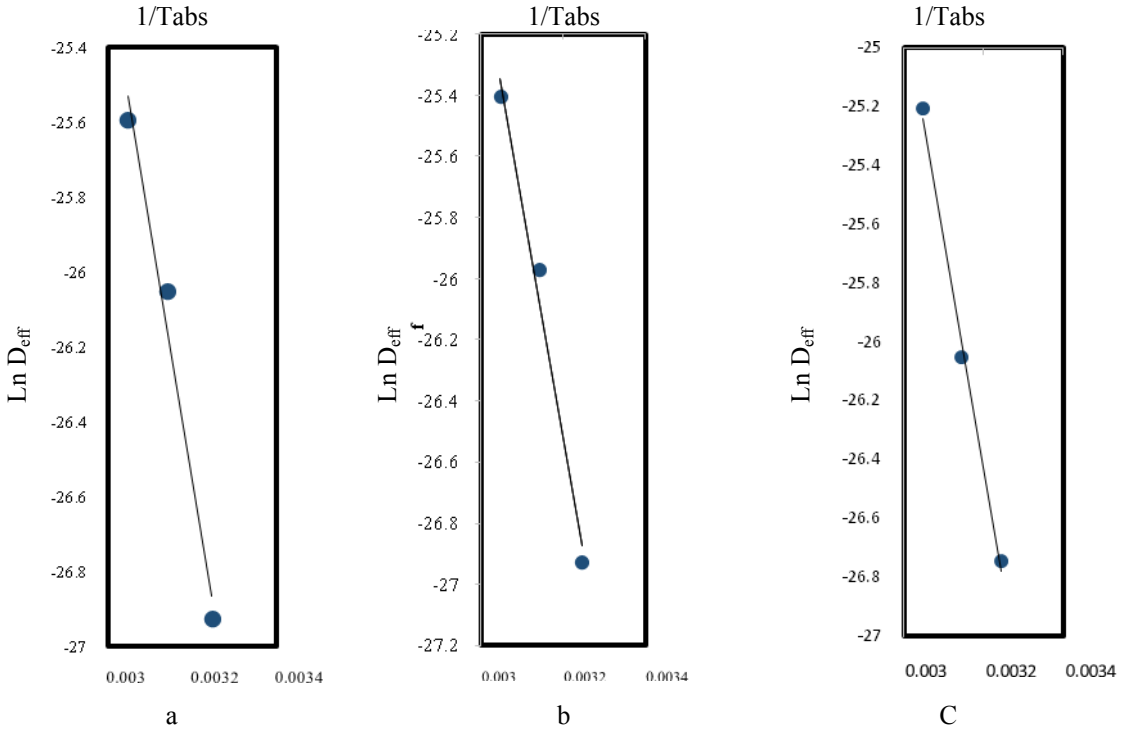

Figure 1 – Plotting experimental data for measuring of E_a: a: 0.4 $m\cdot s^{-1}$, b:2 $m\cdot s^{-1}$ c:1.2 $m\cdot s^{-1}$

$$lnD_e = lnD_0 - \frac{E_a}{RT} \quad (10)$$

The slope of the line (equation 10) was used for the calculation of the Activation energy (E_a) as follows

$$E_a = -(slope\ x\ R) \quad (11)$$

In this study, the values of E_a were in the ranges of 56.3 to 64.5 kJ·mol^{-1}, a result also reported by Sharayei et *al*. [24] for saffron petals. Aghbashlo et *al*. [10] and Doymaz [16] reported Ea of barberry and tomato varied from 110.837 to 130.61 and 17.40 to 32.94 kJ·mol^{-1}, respectively.

Values of E_a and D_0 at air velocity are represented in Table 3. The maximum and minimum values of E_a (64.5 kJ·mol^{-1} and 56.3 kJ·mol^{-1}) were at air velocity of 1.2 and 0.4 (m·s^{-1}), respectively.

Table 3 – E_a and D_0 at different hot air velocities

Hot air velocity (m.s^{-1})	E_a (KJ mol^{-1})	D_0
0.4	56.3	7.5×10^{-3}
1.2	64.5	2.2×10^{-1}
2	64.3	1.7×10^{-1}

Temperature and Air Velocity Effects on Drying Time Duration

The fresh petals were dried in the air temperatures of 35, 45 and 55°C and different air velocity levels (0.4, 1.2 and 2 m·s^{-1}). Figure 2 shows the decreasing trend of moisture content percentage over time due to temperature and air velocity.

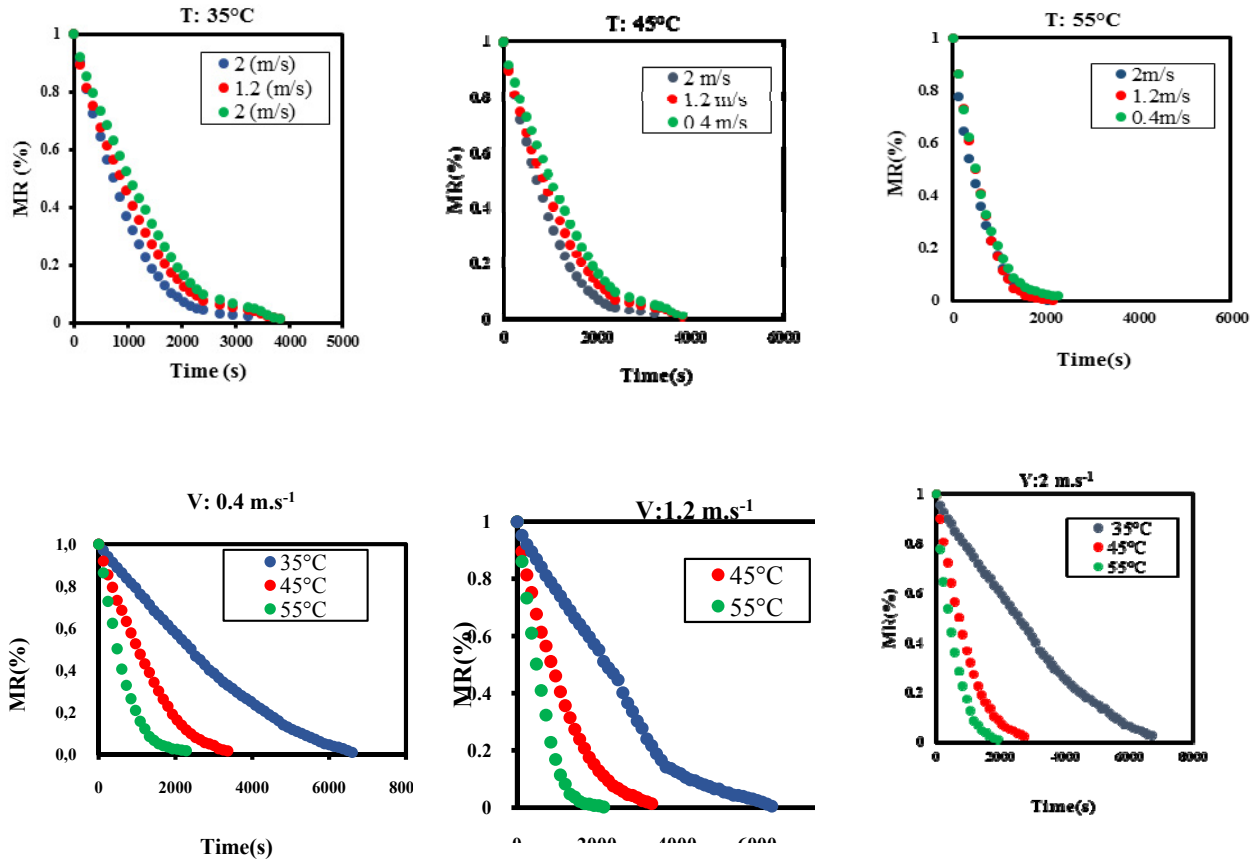

Figure 2 – Trend of variations of moisture content percentage with time for drying conditions

As shown in Table 4, the maximum (6720 s) and minimum (2040 s) drying times were at temperature of 35°C with air velocity of 0.4 m·s^{-1} and temperature of 55°C with air velocity of 2 m·s^{-1}, respectively. Drying time durations were 2000 – 7000 s at the 35, 45 and 55°C air temperatures and 0.4, 1.2 and 2 m·s^{-1} air velocity levels. The longest drying time was 3.5 times higher than those of the

shortest one. Figure 2 also demonstrates that the overall time of drying at 55°C was shorter than all the air velocities, suggesting that the temperature of drying was more effective than the air velocity. The increase in the temperature of drying increases the energy rate, the thermal gradient inside the product and also the accessible energy in the substrate for water transfer from the RDP. Therefore, higher temperatures lead to the faster drying rate and shorter drying time [26]. These results are similar to the findings of Kumar et *al*. [27] who dried different vegetables by the thin-layer dryer.

Table 4 – Time needed for removing moisture to 10% D.B. level for a combination of drying conditions

Temperature (°C)	Hot air velocity (ms^{-1})	Time (s)
35	0.4	6720
	1.2	6600
	2	6480
45	0.4	3360
	1.2	3240
	2	3240
55	0.4	2280
	1.2	2160
	2	2040

Increasing the hot air velocity reduced the moisture content of the RDP, and hence enhanced the drying rate. At first, RDP bulk water can be easily transferred to the surface and evaporated. With increasing the time of drying a significant decrease in the moisture was observed (Fig. 1). The internal moisture transfer rate is constant and does not evaporate easily. Therefore, the drying time is decreased with the increase of air velocity [28].

Influence of the Temperature and Air Velocity on RPE Properties

Anthocyanins that are natural dyes and food coloring agents are generally present in fruits and vegetables. They have beneficial health effects and possess strong antioxidative and antimicrobial activities. The presences of anthocyanins in RPE as quercetin-3-O-glucoside, kaempferol-3-O-rhamnoside, and kaempferol-3-O-arabinoside have been reported by several researchers [29]. The antioxidative activities are affected by drying process such as air temperature and velocity [30].

In this study, the TAC was determined by the pH-differential method. The anthocyanin content in RPE was 77-240 $mg \cdot L^{-1}$ (as cyanidin-3-glucoside) (Table 5). Similar results were reported for *Rosa damascena* petals by Ginova et *al*. [31]. TAC was in the same range (179-314 $mg \cdot L^{-1}$) that has been previously reported by Bayram et *al*. [18] for samples from seven industrial-scale plantations. TAC of extracts obtained from edible flowers (Poppy, Red tulip, Rose and Roselle) in the same study were found within the range of 10 to 405 $mg \cdot kg^{-1}$ dry extract. The TAC in the dried rose petals is more than fruits such as red grape (26.7 $mg \cdot L^{-1}$) and apple (1.3-2.3 $mg \cdot L^{-1}$) and less than of red fruit juices such as Sweet cherry (256.6 $mg \cdot L^{-1}$) Blackberry (739.93 $mg \cdot L^{-1}$), Sour cherry (369.39 $mg \cdot L^{-1}$), Black currant (1543.8 $mg \cdot L^{-1}$) and Chokeberry (3042.2 $mg \cdot L^{-1}$) [32-33]. Our results show that the temperature had a significant impact on the TAC, the lower temperature caused the more TAC (Table 5). Increasing the air velocity from 0.4 to 1.2 ($m \cdot s^{-1}$), at the temperature of 45-55°C, caused an increasing the amount of anthocyanins increased. However, at the air velocity of 1.2-2 ($m \cdot s^{-1}$), a decrease was observed showing that the air velocity at the 35°C did not have a significant impact on the total anthocyanin.

Antioxidant activity of RPE were examined by applying the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging method and FRAP assay. The methanol extracts showed higher DPPH (30.01 vs 59.51%) scavenging power and lower FRAP (992.27 vs 1213.24 μmol $(Fe^{+2}) \cdot L^{-1}$) with increasing temperature from 35 to 55°C (Table 5). The DPPH depends on structural conformation, whereas FRAP assay is less sensitive toward hydrophilic antioxidants and the interaction of antioxidant compounds. Thus, DPPH may not show the accurate antioxidant capacity of compounds compared to the latter [34].

Table 5 – Effect of drying air temperature and velocity on total anthocyanin content (TAC) and antioxidant activity

Temperature (°C)	Velocity of air (ms^{-1})	TAC (mg.c3g/L of extract)	RSA (%)	Folin (mg of GA/g of extract)	FRAP (µmol $(Fe^{+2})L^{-1}$)
55	2	160.10±9.09b	59.51±1.96a	661.33±2.11a	992.27±12.47d
	1.2	180.59±4.76b	58.37±2.70a	643.33±5.03a	1001.78±4.11c
	0.4	77.40±14.62c	54.71±2.60a	658.33±4.98a	1119.39±8.19b
45	2	135.06±8.78c	31.97±11d	612.33±8.05b	837.24±7.53d
	1.2	239.02±9.07a	34.77±0.66cd	618.33±6.66b	1006.53±7.58c
	0.4	158.59±5.15b	52.08±1.75ab	624±9.88b	1261.15±6.36a
35	2	223.08±9.83a	43.69±1.16bc	537.33±8.7d	1206.71±5.99a
	1.2	220.05±9.47a	44.26±2.68bc	581.66±5.44c	1092.06±3.41b
	0.4	229.91±8.20a	30.01±1.21d	577.2±7.67c	1213.24±10.22a

There is a positive relationship between total phenolic content and Radical scavenging capacity (DPPH); and some research have observed a high correlation. But some other researchers reported negative correlation between total phenolic content and antioxidant activity because total phenolic compounds do not contain all the antioxidants present in the extract [35].

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