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The effect of hot air, vacuum and microwave drying on drying characteristics, rehydration capacity, color, total phenolic content and antioxidant capacity of Kumquat (*Citrus japonica*)

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Abstract

Sliced kumquats were dried by using three different drying methods, microwave (375 W), hot air (70 and 80 °C), and vacuum (70 and 80 °C with 100 and 300 mbar) to determine drying characteristics, antioxidant capacity and total phenolic content and color. All color parameters (*L*, *a*, *b*, C_{ab}, ΔE and *h*°) changed depending on the drying methods. Page and Modified Page models are the best fitted drying methods with the highest value of R^2 (0.9994) and the lowest values of RMSE (0.000635-0.000735) and χ^2 (0.000010-0.000013) compared to other models. Effective moisture diffusivity values for dried kumquats ranged from 1.54×10^{-8} to 8.24×10^{-8} in vacuum drying at 70 °C-300 mbar and microwave drying at 375 W, respectively. In comparison to the fresh sample, the dried samples showed an increase in both total phenolic content and antioxidant capacity. The total phenolic content (3095.71 ± 101.41 mg GA/100g d.w) and antioxidant activity (10.51 ± 0.19 μmol TE/g d.w) with DPPH assay showed the highest levels for the vacuum drying at 70 °C-100 mbar method. Microwave dried samples had the highest antioxidant activity with CUPRAC assay as (17.58 ± 0.63 μmol TE/g d.w.). This study indicated that microwave drying and vacuum drying at 70 °C-100 mbar were able to yield high-quality kumquat slices.

Keywords: kumquat; drying methods; drying characteristics; rehydration; antioxidant capacity.

Practical Application: Optimum drying conditions were evaluated and dried fruit and food ingredient were obtained.

1 Introduction

Kumquats (*Citrus japonica*) the smallest of the citrus fruits (Young, 1986), can be eaten as fresh, pickled, candied, marmalade or jelly. It has sweet rind and the acidic pulp. The nutrient profile of raw kumquat (exclude seeds) was determined and reported as: water 80.85 g, energy 71 kcal, carbohydrate 15.90 g, total sugars 9.36 g, total dietary fiber 6.5 g, protein 1.88 g, total lipid 0.86 g, ash 0.52 g, per 100 g edible portion. It is also rich in minerals and vitamins; it contains potassium 186 mg, calcium 62 mg, magnesium 20 mg, phosphate 19 mg, sodium 10 mg, vitamin C 43.9 mg per 100 g edible portion (United States Department of Agriculture Agricultural Research Service, 2016). Kumquat is planted as ornamental in gardens, parks and as ornamental house plant in patios and terraces. Cultivation of this fruit become widespread in south regions of Turkey as it is rich in several nutritious and bioactive compounds.

Citrus fruits have many health benefits including anticold, antiallergic, antiinflammatory, anticancer activity and antiviral activity (Attaway & Moore, 1992; Economos & Clay, 1999). Kumquats are also used to cure inflammatory respiratory disorders as a part of folk medicine.

Drying is one of the most important preserving methods used in the food industry. This process is mostly used to minimize chemical, microbiological and enzymatic reactions which limit shelf life of fresh fruit and vegetables. Dried foods can consume every season and low moisture content allows them to store longer time than fresh food. Many suitable drying methods present for kumquat such as convective drying, microwave drying and vacuum drying. Air drying is one of the most popular and effective method for drying of foods. It takes a long time with low energy performance to dried foods. Apart from this technique, alternative methods such as vacuum and microwave drying have more advantage such as lower drying temperature, higher drying rate, homogeneous energy delivery on the material, better space utilization, formation of suitable final product characteristics and giving better process control (Demiray et al., 2017; İncedayi et al., 2016). Mathematical models of drying processes, which are important in the design and optimization of those processes, have been widely utilized for the definition of drying characteristics of foods (Krokida & Marinos-Kouris, 2003; Babalis & Belessiotis, 2004; Alibas, 2012).

Several studies have been performed about the drying kinetics of orange skin (Garau et al., 2006), lemon slices (Darvishi et al.,

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2014), strawberry (Méndez-Lagunas et al., 2017), kiwi (Kaya et al., 2010). However there is very limited number of studies which focus on thin-layer drying of the kumquats (Mohamadi et al., 2012).

The aim of this study was to determine the hot air, vacuum and microwave drying characteristics of kumquat based on thin-layer drying models at different temperatures, select the best mathematical model for the drying curves and evaluate the effect of drying techniques' on rehydration capacity, color, total phenolic contents and antioxidant capacity of the kumquats.

2 Materials and methods

2.1 Materials

Fresh kumquats (*Citrus japonica, Fortunella japonica Swingle*) were purchased from wholesale food market in Antalya, Turkey and stored at 4 ± 0.5 °C. Kumquats were washed, blanched in boiling water for 30 sec, cooled down in cold water and sliced. The average diameter of slices was 20.00 ± 0.25 mm and thickness of sliced pieces was 4.00 ± 0.08 mm. Moisture contents of kumquats were determined by using moisture analyzer (Sartorius MA150, Germany). The initial moisture content of kumquats was 3.01 g water/g dry base.

2.2 Drying processes

Oven drying, vacuum drying and microwave drying methods were used. All experiments were performed in triplicate to obtain the reproducibility in the results of the experiments.

Hot air drying

Hot air drying treatments were performed with a cabinet type laboratory dryer which was produced by Yucebas Machine Analytical Equipment Industry (Y35, Izmir, Turkey) with the technical features of 220 V, 50-60 Hz, 200 W. The temperature and relative humidity in the dryer was measured by temperature sensor (± 2 °C) and relative humidity sensor (± 2%). 50 g of kumquats slices were placed uniformly as a thin layer on an aluminum plate with a 300 mm diameter. Drying experiments were performed at temperatures of 70 and 80 °C and a constant 20% relative humidity. During drying, the samples were weighed at 30 min intervals for 2 hours and followed by every 15 min. The loss of moisture was determined by weighing the plate using a digital balance (Mettler Toledo, MS3002S) measuring to accuracy of 0.01 g.

Vacuum drying

The drying experiments were performed in a vacuum dryer (Memmert VO400, Germany, 49 L volume) at temperature of 70 and 80 °C and absolute pressures of 100 and 300 mbar. 50 g of samples were put down the aluminum plate as it was in convective drying. The moisture loss of samples during drying was recorded at 30 min intervals for 3 hours and followed by every 15 min.

Microwave drying

For this method a domestic digital microwave oven (Hotpoint Ariston, MWHA 2824 B, Italy, 28 L volume) with technical features of 230V~ 50 Hz and maximum output of 900 W was used. The dimensions of the microwave cavity were 520 cm × 479 cm × 341 cm in size and consisted of a rotating glass plate of 315 mm diameter at the base of the oven. Drying treatments were performed at 375 W microwave power level. In the experiments, 50 g of kumquats slices were put down in a thin layer on a rotating glass plate in the microwave oven. During drying, the rotating glass plate was removed from the microwave oven at predetermined intervals (2 min) and weighed using a digital balance (Mettler Toledo, MS3002S). All drying experiments continued until the moisture content of kumquats fell down to about 0.10 g water/g dry base. Every weighing process was carried out in maximum 10 s during drying treatment.

2.3 Mathematical modelling of drying curve

Five different thin-layer drying models were used to select the best model for describing the drying curve of kumquat slices in Table 1. The moisture ratio (MR) of kumquats slices during drying were calculated using the following Equation 1:

$$MR = \frac{M - M_e}{M_i - M_e} \quad (1)$$

where: MR is moisture ratio; M is the moisture content at a specific time (g water/g dry base); M_i is the initial moisture content (g water/g dry base); M_e is the equilibrium moisture content (g water/g dry base) (Arslan & Musa Özcan, 2010).

RMSE gives deviation between the estimated and experimental values for the models. The higher correlation coefficient (R^2), reduced root mean square error (RMSE) and reduced Chi square (χ^2), were used to determine the goodness of fit model in the oven, vacuum and microwave drying curves of kumquats slices. These parameters could be calculated using the following Equations 2 and 3:

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}(MR_{\exp,i} - MR_{pre,i})^2\right]^{1/2} \quad (2)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{\exp,i} - MR_{pre,i})^2}{N - n} \quad (3)$$

where: $MR_{\exp,i}$ is the experimentally dimensionless moisture ratio for test *i*; $MR_{pre,i}$ is the predicted dimensionless moisture ratio for test *i*; N is the number of observation; and n is the number of constants in the model (Avhad & Marchetti, 2016).

Table 1. Mathematical models applied to drying characteristics of kumquats slices.

Model name	Model	References
Page	$MR = \exp(-kt^n)$	Sarsavadia et al. (1999)
Modified Page	$MR = \exp[(-kt)^n]$	Overhults et al. (1973)
Logarithmic	$MR = a\exp(-kt) + c$	Yagcioglu (1999)
Lewis	$MR = \exp(-kt)$	Doymaz (2006)
Handerson ve Pabis	$MR = a\exp(-kt)$	Westerman et al. (1973)

2.4 Calculation of effective moisture diffusivity

Fick's diffusion equation has been widely used to describe the drying process of biological products during the falling rate period (Dadalı et al., 2007a). The solution of Fick's second law in slab geometry is given by Crank (1975) as shown in Equation 4, assuming moisture change being only by diffusion, constant temperature and effective moisture diffusivity, and negligible shrinkage (Demiray et al., 2017).

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)^2}\exp\left(-\frac{(2n-1)^2\pi^2 D_{eff} t}{4L^2}\right) \quad (4)$$

where: Deff is effective moisture diffusivity (m^2/s); L is the half thickness of the slab in samples (m); and n is a positive integer. In practice, only the first term Equation 5 is written in a logarithmic form as follows:

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

The effective moisture diffusivity can be calculated by plotting experimental drying data in terms of ln MR versus drying time, using the following Equation 6:

$$D_{eff} = -\frac{slope 4L^2}{\pi^2} \quad (6)$$

2.5 Rehydration capacity

Dried kumquats with different methods were rehydrated at 25 °C by using water bath (Memmert, WNE14, Germany) to measure moisture content recovery during rehydration. Five grams of samples added to 150 mL distilled water, in a 250 mL flask beaker. The rehydration process continued for 24 hours, weight increments measured the first 7 hours every 60 minutes and at the end of 24 hours with digital balance (Mettler Toledo, MS3002S). Rehydration capacity was expressed as moisture content over rehydration time. Determinations were made in triplicate (Demiray & Tulek, 2017a).

2.6 Color analysis

The color of fresh and dried kumquats was determined by using a Hunter Lab MiniScan EZ4500L (Virginia, USA) calorimeter. The calibration was done with a black and white ceramic plate before the experiments. The Hunter *L, a, b* values were displayed in lightness, redness and yellowness, respectively. The Hunter *L, a, b* values were used to calculate total color difference (ΔE), Chroma (C_{ab}) and hue angle (h°) to describe color changes during drying (Dadalı et al., 2007b; Suna et al., 2014). C_{ab} changes from 0 (dull) to 60 (vivid) and was calculated by using the following Equation 7:

$$Chroma\ (C_{ab}) = \sqrt{(a)^2 + (b)^2} \quad (7)$$

The color of food samples generally characterizes by calculating Hue angle (h°) value as shown in the Equation 8. This value is explained in angles of 0°, 90°, 180° and 270°, which represent the color of red, yellow, green and blue, respectively (Karaaslan & Tuncer, 2008).

$$h^{\circ} = \arctan\left(\frac{b}{a}\right) \quad (8)$$

Total color difference (ΔE) which indicates the saturation of color and was evaluated by using Equation 9 (Šumić et al., 2013).

$$\Delta E = \sqrt{(L-L_0)^2 + (a-a_0)^2 + (b-b_0)^2} \quad (9)$$

where: L_0, a_0, b_0 indicate the value of fresh kumquats color and *L*, *a*, *b* are the individual readings at each processing time.

2.7 Extraction of samples for total phenolic content and antioxidant capacity

The extracts of fresh and dried kumquats were prepared according to Vitali et al. (2009) with some modifications. 2 g of grinded (Moulinex, China) kumquat samples was mixed with 20 mL HCl/methanol/water (1:80:10) mixture and shaken by using a rotary shaker (JB50-D; China) at 250 rpm for 2 h at 20 °C. Then the mixture was centrifuged at 3500 rpm for 10 min at 20 °C in a centrifuge (Sigma 3K 30, Germany). The supernatants were stored in falcon tubes at -20 °C until used.

Determination of total phenolic content and antioxidant capacity

Folin-Ciocalteu spectrophotometric method was used for the determination of total phenolics as defined by Spanos & Wrolstad (1990). Total phenolic content was described as mg gallic acid equivalents (GAE) per 100 g dry weight (mg GAE/100g d.w.). Antioxidant capacity of the fresh and dried kumquat slices were measured according to CUPRAC (Apak et al., 2004), DPPH (Katalinic et al., 2006) and FRAP (Benzie & Strain, 1996) methods and the results were given as μmol Trolox equivalent (TE) per g dry weight (μmol TE/g d.w.) in all assays. All reagents were used in analytical grade.

2.8 Statistical analysis

The experiment was conducted in a completely randomized design with three replications. The results were statistically evaluated by one-way analysis of variance (ANOVA) using the JMP software package version 6.0 (SAS Institute Inc. NC, 27513). When significant differences were found ($p < 0.05$), the Least Significant Difference (LSD) test was used to determine the differences among means.

3 Results and discussion

3.1 Drying kinetics

The experimental results indicated that the time required for significant reduction in the moisture content was related with the drying techniques. While vacuum drying at 70 °C-300 mbar had the longest drying duration (315 min), microwave of 375 W (42 min) had the shortest one. According to data, total drying time was shortened (86.67%) by microwave drying comparing to vacuum drying at 70 °C-300 mbar. The drying time of kumquats which were hot air dried were respectively 195 and 190 minutes at drying air temperatures of 70 and 80 °C at a constant relative humidity (20%) while vacuum drying at 70 °C-100 mbar, vacuum drying at 80 °C-100 mbar, vacuum drying at 80 °C-300 mbar were respectively 285, 210 and 300 minutes (Figure 1).

When air temperature raised from 70 °C to 80 °C, the average total drying time decreased 2.56, 26.32, 4.76% under the condition of hot air, 100 mbar vacuum and 300 mbar vacuum, respectively. Moreover, total drying time also decreased (9.52% at 70 °C, 30.00% at 80 °C) when the vacuum increased. Increase in vacuum and temperature allowed decrease in the drying time by accelerating moisture migration from the center to the outside (Kingsly & Singh, 2007). Similar results were attained at air dried orange peel by Garau et al. (2006), hot air dried apples by Vega-Gálvez et al. (2012), and vacuum oven dried tomato slices by Azeez et al. (2017).

3.2 Modelling of drying curves

Statistical results of the different thin-layer drying models, including the suitability of models, drying model coefficients of determination R^2, root mean square error (RMSE) and Chi square (χ^2), are used to evaluate the quality of dried kumquats (Table 2). The statistical parameter predictions exhibited that R^2 values varied between 0.8925 to 0.9994, RMSE values varied between 0.000635 to 0.042941 and χ^2 values varied between 0.000010 to 0.026342. The suitable drying methods with the highest value of R^2 (0.9994) and the lowest values of RMSE (0.000635-0.000735) and χ^2 (0.000010-0.000013) were obtained from Page and Modified Page models. For this reason, Page and Modified Page models were chosen as the most appropriate models to show the thin-layer drying characteristics of the kumquats when a decision was made between the five models. Akdaş & Başlar (2015) also determined the best fitted mathematical model as Page for mandarin slices under oven and vacuum drying conditions. Figure 2 shows the moisture content determined by Page's equation.

3.3 Effective moisture diffusivity

The effective moisture diffusivity (D_{eff}) values for different drying methods, calculated from Equation 6, ranged from 1.54×10^{-8} to 8.24×10^{-8} m²/s in vacuum drying at 70 °C-300 mbar and microwave drying at 375 W respectively. It was observed that D_{eff} values increased with the rise in temperature of air drying from 2.32×10^{-8} (70 °C) to 2.67×10^{-8} (80 °C) and vacuum drying from 1.54×10^{-8} (70 °C-300mbar) to 2.42×10^{-8} (80 °C-100mbar). The values of D_{eff} in our study were within the general range 10^{-12}-10^{-8} for drying of food materials (Demiray & Tulek, 2017b) and comparable to 1.87-3.59 × 10^{-8} m²/s for dried lemon slices (Darvishi et al., 2014), 9.44×10^{-8}-2.56×10^{-7} m²/s for onion slices (Demiray et al., 2017) and 0.35-0.87 × 10^{-8} m²/s for watermelon pomace (Oberoi & Sogi, 2015).

3.4 Rehydration capacity

The rehydration characteristics are usually considered an important quality parameter of dried products (Lewicki, 1998). The moisture content against rehydration time was shown in Figure 3. Moisture content was significantly increased within the beginning period of 3 hours whereas water absorption slowed as the curve reached the equilibrium state. The high rate of water absorption at the initial period of rehydration may be clarified by the quick rehydration of capillaries and cavities near the surface, which are rapidly filled up with water (García-Pascual et al., 2006; Markowski et al., 2009). The increase of moisture content during rehydration ranged from 2.09 to 2.58 g water/g dry matter in all dried kumquat samples and the highest moisture content obtained from microwave dried samples. Similar results indicating that microwave energy causes the highest rehydration capacity, were reported by some authors for banana (Maskan, 2000), apple (Askari et al., 2006) and sour cherry (Horuz et al., 2017). According to Askari et al. (2006), the intercellular gaps caused by microwave energy resulted in the absorption of a high amount of water which concludes an increment in rehydration capacity of dried fruits.

3.5 Color analysis

Color is an important part of food quality, because the color of food is consumers' first appraise when making purchasing decisions. The results of color changes in fresh sample for all drying conditions were given in Table 3. Yildiz Turgut et al. (2015) reported the *L* (lightness) value of fresh kumquat as 61.56 ± 0.24 similar to our findings. The *L* value were significantly affected by different drying treatments ($p < 0.05$) and resulted with a 2.05-70.77%

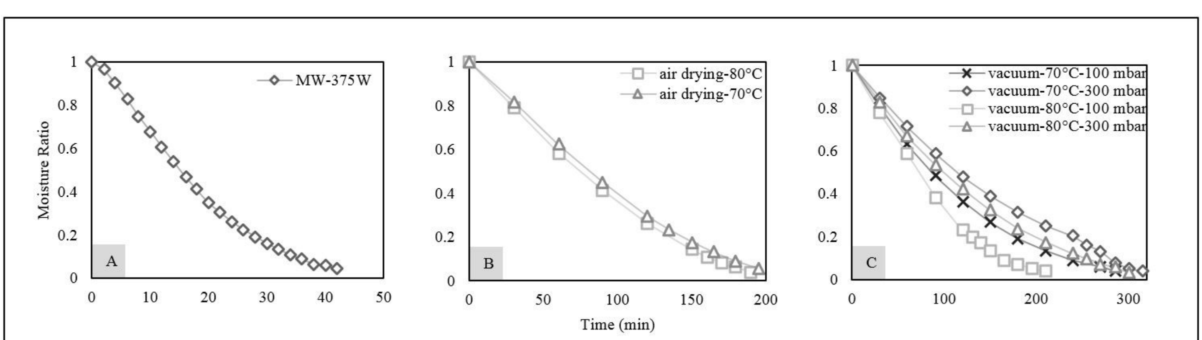

Figure 1. Moisture ratio of kumquat slices versus drying time at microwave (A), hot air drying (B) and vacuum drying (C) conditions determined by Page's equation.

Table 2. Statistical values obtained from the modelling of dried kumquats.

Model name		Model coefficients		R^2	RMSE	X^2
Page	MW-375 W	n	1.4539	0.9994	0.000635	0.000010
		k	0.0136			
	air drying 70 °C	n	1.4084	0.9940	0.004870	0.000296
		k	0.0015			
	air drying 80 °C	n	1.4117	0.9912	0.006326	0.000500
		k	0.0017			
	vacuum 70 °C-100 mbar	n	1.1870	0.9975	0.002709	0.000099
		k	0.0036			
	vacuum 70 °C-300 mbar	n	1.2334	0.9779	0.006542	0.000699
		k	0.0021			
	vacuum 80 °C-100 mbar	n	1.3524	0.9954	0.003739	0.000201
		k	0.0023			
	vacuum 80 °C-300 mbar	n	1.2304	0.9925	0.004301	0.000284
		k	0.0026			
Modified page	MW-375 W	n	1.4553	0.9994	0.000735	0.000013
		k	0.0514			
	air drying 70 °C	n	1.4084	0.9940	0.004870	0.000296
		k	0.0100			
	air drying 80 °C	n	1.4117	0.9912	0.006326	0.000500
		k	0.0110			
	vacuum 70 °C-100 mbar	n	1.1870	0.9975	0.002891	0.000112
		k	0.0087			
	vacuum 70 °C-300 mbar	n	1.2334	0.9779	0.006803	0.000756
		k	0.0069			
	vacuum 80 °C-100 mbar	n	1.3524	0.9954	0.003782	0.000206
		k	0.0112			
	vacuum 80 °C-300 mbar	n	1.2304	0.9925	0.004426	0.000301
		k	0.0079			
Logarithmic	MW-375 W	k	0.0496	0.9701	0.007194	0.001318
		a	1.0661			
	air drying 70 °C	k	0.0141	0.9473	0.038968	0.021693
		a	1.3013			
	air drying 80 °C	k	0.0164	0.9512	0.042941	0.026342
		a	1.3633			
	vacuum 70 °C-100 mbar	k	0.0114	0.9726	0.028996	0.012717
		a	1.2442			
	vacuum 70 °C-300 mbar	k	0.0095	0.9125	0.031448	0.017621
		a	1.3425			
	vacuum 80 °C-100 mbar	k	0.0160	0.9723	0.032961	0.017383
		a	1.3408			
	vacuum 80 °C-300 mbar	k	0.0105	0.9634	0.027636	0.012907
		a	1.2872			
Lewis	MW-375 W	k	0.0445	0.9598	0.005966	0.000820
	air drying 70 °C	k	0.0123	0.9291	0.024350	0.006588
	air drying 80 °C	k	0.0263	0.9323	0.026902	0.008041
	vacuum 70 °C-100 mbar	k	0.0103	0.9604	0.016112	0.003141
	vacuum 70 °C-300 mbar	k	0.0082	0.8925	0.018321	0.005061
	vacuum 80 °C-100 mbar	k	0.0141	0.9556	0.019578	0.005018
	vacuum 80 °C-300 mbar	k	0.0094	0.9493	0.016757	0.003955

Table 2. Continued...

Model name		Model coefficients		R^2	RMSE	X^2
Henderson and Pabis	MW-375 W	k	0.0508	0.9808	0.004735	0.000543
		a	1.0826			
	air drying 70 °C	k	0.0143	0.9524	0.035940	0.016146
		a	1.3335			
	air drying 80 °C	k	0.0165	0.9541	0.041634	0.021667
		a	1.3876			
	vacuum 70 °C-100 mbar	k	0.0114	0.9726	0.024535	0.008093
		a	1.2442			
	vacuum 70 °C-300 mbar	k	0.0095	0.9148	0.030401	0.015096
		a	1.3573			
	vacuum 80 °C-100 mbar	k	0.0149	0.9749	0.022479	0.007276
		a	1.2483			
	vacuum 80 °C-300 mbar	k	0.0106	0.9650	0.026219	0.010562
		a	1.3011			

Table 3. Color values of fresh and dried kumquat.

Drying conditions	*L*	*a*	*b*	ΔE	C_{ab}	*h*°
fresh	61.04 ± 0.15^{a}	16.27 ± 0.03^{e}	63.52 ± 0.18^{cd}	-	65.57 ± 0.16^{d}	75.63 ± 0.06^{a}
MW-375 W	17.84 ± 0.57^{e}	8.60 ± 0.14^{f}	26.36 ± 0.65^{g}	7.79 ± 0.11^{d}	27.73 ± 0.65^{g}	71.92 ± 0.21^{e}
air drying 70 °C	51.61 ± 1.37^{d}	20.80 ± 0.71^{b}	57.16 ± 0.55^{f}	8.45 ± 0.19^{c}	60.83 ± 0.27^{f}	70.00 ± 0.81^{g}
air drying 80 °C	58.74 ± 0.13^{b}	19.02 ± 0.06^{d}	63.70 ± 0.03^{c}	3.44 ± 0.10^{e}	66.48 ± 0.01^{c}	73.37 ± 0.05^{cd}
vacuum 70 °C-100 mbar	59.11 ± 0.20^{b}	20.57 ± 0.05^{bc}	72.19 ± 0.22^{a}	8.62 ± 0.44^{c}	75.06 ± 0.23^{a}	74.09 ± 0.03^{b}
vacuum 80 °C-100 mbar	53.19 ± 0.24^{c}	18.65 ± 0.07^{d}	61.84 ± 0.26^{e}	3.24 ± 0.17^{e}	64.59 ± 0.27^{e}	73.22 ± 0.01^{d}
vacuum 70 °C-300 mbar	59.42 ± 0.48^{b}	20.19 ± 0.14^{c}	69.84 ± 0.27^{b}	35.47 ± 0.57^{a}	72.70 ± 0.29^{b}	73.88 ± 0.05^{bc}
vacuum 80 °C-300 mbar	53.62 ± 0.19^{c}	21.63 ± 0.08^{a}	62.93 ± 0.24^{d}	10.76 ± 0.15^{b}	66.54 ± 0.25^{c}	71.03 ± 0.02^{f}

[a-g]Different letters in the same column display that significant difference ($p < 0.05$).

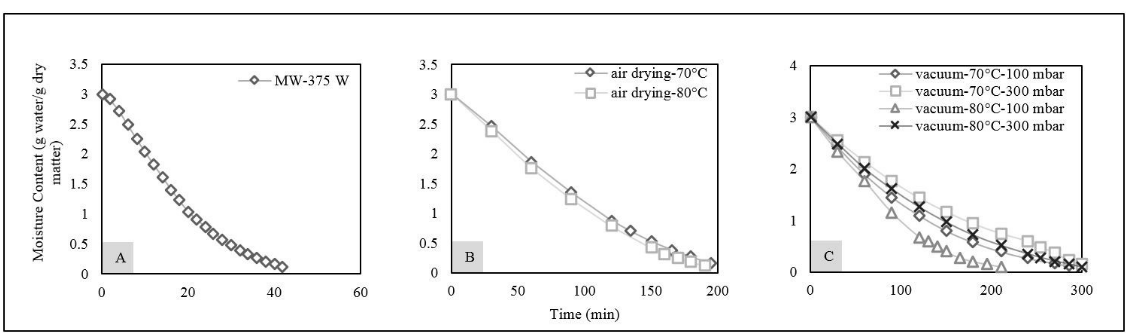

Figure 2. Drying curves of kumquat samples at microwave (A), hot air drying (B) and vacuum drying (C).

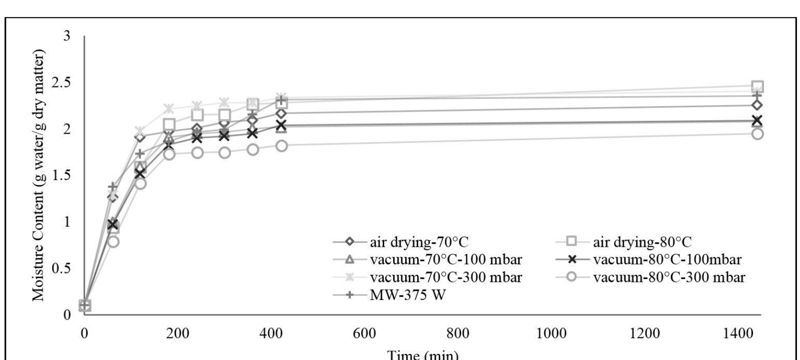

Figure 3. Moisture content uptake against rehydration time of dried kumquats with different methods.

decrease. The lowest *L* value obtained from microwave dried samples which had darker color than other drying methods. Compared to the fresh sample, *a* (redness) values significantly increased ($p < 0.05$) with vacuum and air drying methods whereas this value reduced in microwave dried kumquats. The increase of *a* value might be due to the Maillard reaction and degradation of pigments such as carotenoids (Maskan, 2001; Lavelli et al., 2007; Xiao et al., 2012). *b* values of dried kumquats with vacuum drying at 70 °C-100 mbar, 70 °C-300 mbar and air drying at 80 °C increased with respect to fresh kumquat samples. However, compared to fresh sample, microwave drying caused 58.50% decrease in *b* value and this was closely followed for Chroma (C_{ab}) (57.71%). (C_{ab}) values were used to comprehend intensity of color. Vacuum dried samples at 70 °C-300 mbar showed the highest C_{ab} value (72.70 ± 0.29) as compared to other treatments. The lowest ΔE value (3.24 ± 0.17) was obtained from vacuum drying at 80 °C-100 mbar sample while the highest value (35.47 ± 0.57) was obtained from vacuum drying at 70 °C-300 mbar. Hawlader et al. (2006) explained that the reduction in *h°* values is an expression of more darkening color. Vacuum drying at 70 °C-100 mbar caused a smaller reduction of *h°* values. Besides pigment decompositions, non-enzymatic and enzymatic reactions are responsible for the formation of browning pigments (Albanese et al., 2013).

3.6 Total phenolic content

The total phenolic contents (TPC) of fresh and dried kumquats were given in Figure 4. Fresh kumquats had 266.68 ± 14.57 mg GA/100 g d.w. TPC. Lou et al. (2015) reported the total phenolic content of hot water extracts of fresh immature kumquats as approximately 1500 GAE mg/100 g dry extract which is higher than our findings. Hot water extraction might lead destruction of the cell wall structure which may allow an increase in extracted phenolic constituents.

Different drying techniques provide a variety of TPC. The highest TPC (3095.71 ± 101.41 mg GA/100g d.w.) was attained by vacuum drying at 70 °C-100 mbar ($p < 0.05$) (Figure 4). Several studies reported that vacuum drying technique is allow minimum degradation in phenolic content compared to hot air drying (Karaman et al., 2014; Ruiz et al., 2014). The results indicated that degradation of phenolic components in kumquats during hot air drying at 70 °C was highest, with lowest TPC (2181.32 ± 52.16 mg GA/100 g d.w.) determined ($p < 0.05$).

Ramful et al. (2011) measured TPC of freeze dried kumquat pulp powders, extracted with 80% methanol, as 1412 ± 16 and 1694 ± 19 μg g^{-1} f.w. Ishiwata et al. (2004) investigated the total polyphenol content in dried kumquats bought from local market in Japan. According to their findings, TPC content was 530 ± 4 mg GAE/100g d.w. which was much lower than our results.

The similar increment in total phenolic content was obtained by Türkmen et al. (2005) and Priecina & Karklina (2014).

3.7 Antioxidant capacity

The antioxidant capacity of the fresh and the dried kumquats were given in Figure 4. Antioxidant capacity can be measured by several methods which have different mechanisms Antioxidant capacity of the fresh sample was found as 1.84 and 2.50 μmol TE/g d.w. respectively in DPPH and CUPRAC methods.

Antioxidant capacity of fresh kumquat was significantly lower compared to dried kumquats ($p < 0.05$). The highest antioxidant capacity was obtained from vacuum drying at 70 °C-100 mbar (10.51 ± 0.19 μmol TE/g d.w.) and vacuum drying at 80 °C-300 mbar (10.30 ± 0.49 μmol TE/g d.w.) in DPPH assay and microwave drying (17.58 ± 0.63 μmol TE/g d.w.) in CUPRAC assay. The lowest antioxidant capacity was determined in hot air drying technique at 70 °C both in DPPH (5.13 ± 0.07 μmol TE/g d.w.) and CUPRAC (6.47 ± 0.04 μmol TE/g d.w.) assays. Vega-Gálvez et al. (2012)

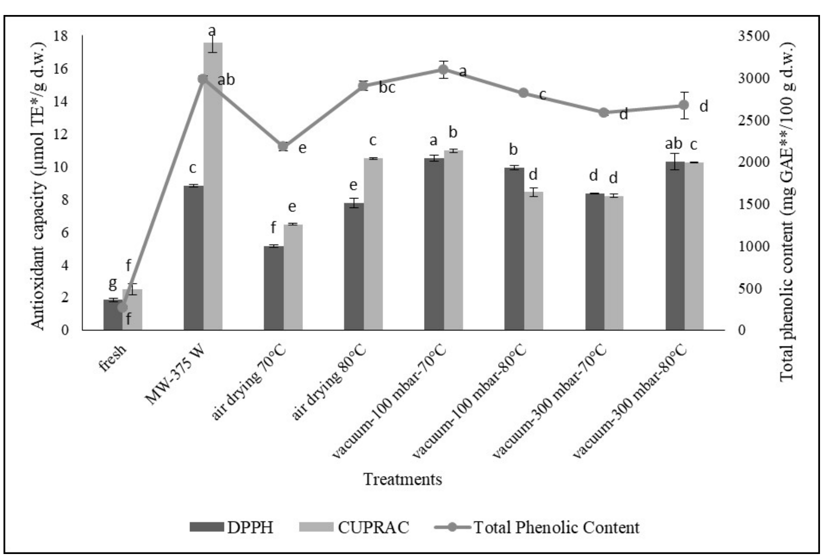

Figure 4. The effect of drying treatments on kumquats' antioxidant capacities and total phenolic content. *TE = trolox equivalent; **GAE = gallic acid equivalent; d.w. = dry weight.

determined an increase in DPPH free radical scavenging activity of hot air dried pepper slabs. Türkmen et al. (2005) found an enhancement in antioxidant activity as a result of cooking methods (boiling, steaming and microwave) in pepper, green beans, broccoli, and spinach. In line with our study, Priecina & Karklina (2014) also identified increment in antioxidant activity of some vegetables.

Drying with microwave technique, which had the lowest *L* value, allowed more browning reactions compared to the other techniques. In this situation, the highest measured antioxidant capacity with CUPRAC could be explained by formation of Maillard reaction products which have high antioxidant properties (Manzocco et al., 2000).

4 Conclusion

Drying kinetics, rehydration capacity, color, TPC and antioxidant capacity of kumquats dried with hot air, vacuum and microwave were investigated. Microwave drying significantly shortened the drying duration in proportion to hot air and vacuum drying. Also the highest effective moisture diffusivity was observed by microwave drying. Among the mathematical models, the Page and Modified Page models were considered to be the best models to describe the drying characteristics of kumquats. While *L* and *h°* values decreased, *a* value increased in dried samples except microwave dried one. Microwave dried kumquats had the lowest *L*, *a*, *b* and C_{ab} values. In addition, the highest rehydration capacity was obtained by microwave dried samples. Vacuum drying was defined as the best method for preserving color values. Total phenolic content and antioxidant capacity of dried kumquats were increased after drying. The total phenolic content and antioxidant activity with DPPH assay showed the highest levels for the vacuum drying at 70 °C-100 mbar method. Microwave dried samples had the highest antioxidant activity with CUPRAC assay. In consequence, microwave drying was found applicable for kumquats in order to reduce the drying time as well as enhancing bioactive content, but color of dried kumquats were not preferable. This was the first study not only investigate the effect of different drying methods on kumquat quality but also reveal the bioactive contents of dried fruit. More studies are need for further comparison.

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