

**Effect of Turntable Rotation Rate on Drying Kinetics and Functional Properties of Lemon Peel during Microwave Drying**

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**Article History Abstract –** The aim of this study is to investigate the effect of the rotational rate of the turntable on drying kinetics of lemon peels and some functional and flow properties of lemon peel powders. Lemon peels were dried by microwave drying using different rates of rotation (0, 6.5, 9.5, and 12.5 rpm) at different microwave power levels (180W, 300W, 450W and 600W), and dried by oven drying and freeze-drying methods. Drying time was shortened by 72- 95% by microwave drying compared to oven drying. Microwave drying with rotation provided 5.6-23.8% reduction in drying time of peels compared to drying without rotation. Effect of rotation rate on drying time of lemon peels depended on the microwave power level. Page model provided lower SSE, RMSE, and higher  $R<sup>2</sup>$  values within 5 different thin layer models. The effective moisture diffusivity value, ranging between  $1.7 \times 10^{-8}$  m<sup>2</sup>s<sup>-1</sup>-7.6x10<sup>-8</sup> m<sup>2</sup>s<sup>-1</sup>, vas higher during microwave drying with rotation. The activation energy ranged between 21.3-22.7 W/g. Microwave rying provided higher bulk density, similar or lower water holding capacity and oil retention capacity values compared to freeze drying and oven drying. Freeze dried lemon peel powder had the lowest bulk density due to its porous structure. Microwave drying without rotation and the highest power level caused lower bulk density. At higher power levels, influence of turntable rotation on water holding capacity was more notable. Microwave drying technique can be used as alternative drying techniques to obtain high quality dried lemon peel powder if appropriate processing conditions are selected.

*Keywords – Drying kinetics, functional properties, lemon peel, microwave drying, turntable,* 

# **1. Introduction**

Nowadays, there has been a growing interest in utilization of agro-industrial by-products (Trigo et al., 2020). Citrus fruits are one of the most cultivated crops worldwide. Citrus family includes economically important fruits such as orange, lemon, grapefruit, mandarin. World citrus fruit production was nearly 143.8 million tons in 2019 (FAO, 2021). During fruit juice processing, approximately 50-70% of the citrus fruits are discarded as waste (Zema et al., 2018). Globally, 110–120 million tons of citrus waste are produced from citrus processing industries yearly (Mahato et al., 2020). Citrus waste includes peel (flavedo+albedo), pulp (vesicular membranes), rag (core, carpellary membranes) and seeds. Citrus peel is a rich source of valuable phytochemicals, and dietary fibers and thereby may be utilized in preparation of new food products. However, it is prone to quick spoilage due to its high moisture content and thereby requires preservation for later use. Citrus peel powders obtained by drying can be used as an ingredient for innovative food formulations (Trigo et al., 2020).

Various drying techniques have been employed for drying citrus peels with the aim of achieving improved product quality in a shorter time. Among the artificial drying methods, hot-air drying is the most widely used

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method for drying of citrus peels. Microwave drying is known to offer opportunity of high drying rate, especially in the falling rate period. Some researchers studied on the effects of microwave drying on drying kinetics and various quality properties of citrus peels (Ozcan et al., 2020; Farahmandfar et al., 2019, 2020; Abou Arab, Mahmoud, & Abu-Salem, 2017; Ghanem et al., 2012, 2020; Bejar, Kechaou, & Mihoubi, 2011b; Tekgül & Baysal, 2018; Tuncer, Güler, & Usta, 2020; Kirbas et al., 2019) and noted that microwave drying may be used as a convenient and effective drying method for drying of citrus peels.

Drying kinetics is used to express the water removal process. The rate of water removal depends on many factors and changes with time during drying. The understanding of drying kinetics helps the control and optimization of drying processes. For fruits and vegetables, thin layer drying models have been found in wide application (Onwude et al., 2016). Some semi-theoretical and empirical models have been used to describe the relationship between the moisture content and the drying time of citrus peels. Bejar et al. (2011b) indicated that microwave drying kinetics of Maltaise orange peel was well described by Page model. 9 different thin layer models were used to describe the dehydration behavior of lemon peel during microwave drying by Ghanem et al. (2020). These studies indicated that dehydration rate and some quality characteristics of citrus peels were influenced by microwave power level.

In the microwave drying process, the homogeneity of the temperature distribution in the product is extremely important in terms of drying efficiency and product quality. Turntables used in microwave ovens are one of the frequently used methods to create a homogeneous temperature distribution. To the best of the author's knowledge, no study has been conducted to investigate the effects of rotational rate of turntable on drying kinetics or quality characteristics of foods during microwave drying. In this study, it was aimed to investigate the effect of the rotational rate of turntable (0, 6.5, 9.5, and 12.5 rpm) on drying kinetics of lemon peels during microwave drying at different microwave power levels (180W, 300W, 450W and 600W). In addition, SEM images, flow properties, and some functional properties of lemon peel powders dried under different microwave drying conditions were obtained and the results were compared with the powders prepared using freeze drying and oven drying methods.

# **2. Materials and Methods**

Fresh *Citrus limon* fruits were purchased from a local market in Giresun, Turkey and stored at 4±0.5°C until further use.

# **2.1 Drying Process**

Lemon fruits were taken out of the refrigerator and left for 2 hours to come to ambient temperature before the experiments. The fruits without blemishes or damage were chosen, washed, and dried with tissue paper. Peels (containing flavedo and albedo) of the cleaned fruits were manually separated and cut into slices (1x1 cm).

The lemon peel samples were dried by 3 methods, which were oven drying (OD), freeze drying (FD), and microwave drying (MWD). Oven drying was performed in a fan oven (Ordel OC770) at 60°C which was selected based on the data from literature surveys about the drying of citrus peels (Garau et al., 2007; Ozcan et al., 2020). A domestic microwave oven (MC32F604TCT, Samsung) was used for microwave drying. The oven was modified so that the speed of the microwave's turntable can be adjusted. Lemon peel samples were spread in a single layer on a petri dish 115 mm in diameter. The petri dish was positioned at the center of the turntable. The drying process was carried out using 4 different microwave powers (180W, 300W, 450W, and 600W) as provided on the control panel of the microwave oven (Ghanem et al., 2020). Some preliminary tests exhibited that the samples were burned at the microwave power level higher than 600W. At each power level, the lemon peels were subjected to three different rotational rates of turntable (6.5, 9.5, and 12.5 rpm). Lemon peels were also dried using the same microwave oven without rotating the turntable at 180W, 300W, 450W, and 600W power levels. Dried lemon peels were ground into powder using a grinder for 30s (Sinbo, SCM-2934) and then sieved (40 mesh sieve). Sieved powder samples were used in the further analysis. Functional

and flow properties of microwave dried, and oven dried samples were compared with that of lemon peels dried by freeze drying in a lab-scale freeze dryer (FreeZone 2.5L 7670530, Labconco) at -50°C and 0.1 mbar vacuum pressure.

# **2.2 Determination of Moisture Content**

The determination of moisture content of lemon peel was done by drying in an oven at 105 °C to constant weight (AOAC, 1995).

#### **2.3 Drying Characteristics**

#### **2.3.1. Moisture Ratio (MR)**

Lemon peel samples were dried until the moisture content of samples dropped to 0.11  $g/g$  on dry basis. During microwave and oven drying processes, lemon peels were weighted at specified time intervals to obtain moisture content of samples. MR values were calculated from the experimental moisture content (kg water kg- $<sup>1</sup>$  dry matter) data. Each drying treatment was carried out two times. The arithmetic average of data was taken</sup> for the calculation of MR values by Equation (2.1).

$$
MR = \frac{X(t) - X_e}{X_0 - X_e} \tag{2.1}
$$

 $X(t)$ ,  $X_e$  and  $X_0$  represent the moisture content obtained at any drying time, equilibrium moisture content, and the initial moisture content, respectively.

# **2.3.2. Drying Rate**

The drying rates (DR) of lemon peels during the microwave and oven drying processes were determined using Equation 2.2. (Deng et al., 2019):

$$
DR = \frac{x_{t1} - x_{t2}}{t_2 - t_1} \tag{2.2}
$$

DR is the drying rate (kg water kg<sup>-1</sup> dry matter min<sup>-1</sup>),  $X_{t1}$  and  $X_{t2}$  are the moisture contents (kg water kg<sup>-1</sup> dry matter) at time  $t_1$ and  $t_2$ (min), respectively.

### **2.3.3. Kinetic Modelling**

Experimental MR data were fitted to five widely used single-layer drying models given in Table 1 (Ertekin & Firat, 2017). The model parameters were estimated by nonlinear least-squares procedure using MATLAB software (R2021b, Mathwork, Inc., MA, US). For evaluation of accuracy of fit, the statistical parameters, the determination coefficient  $(R^2)$ , the sum of square error (SSE), the root mean square error (RMSE) values were calculated using Eq. 2.3., 2.4, and 2.5, respectively.

$$
R^{2} = 1 - \left[ \frac{\sum_{i=1}^{N} (MR_{exp,i} \cdot MR_{pred,i})^{2}}{\sum_{i=1}^{N} (MR_{exp,i} \cdot MR_{mean})^{2}} \right]
$$
(2.3)

$$
SSE = \sum_{i=1}^{N} (MR_{exp,i} \cdot MR_{pred,i})^2
$$
 (2.4)

 $RMSE = \frac{SSE}{V}$  $\frac{3\pi}{v}$ 1 2 where  $v = N - m$  (2.5)

 $MR_{exp,i}$  and  $MR_{pred,i}$  are the ith experimental and predicted MR values, respectively;  $MR_{mean}$  is the mean of experimental MR values;. N is the number of experimental data points; m is the number of constants in drying model.

Table 1 Single-layer drying models

Model	<b>Equation</b>
Newton	$MR = exp(-kt)$
Page	$MR = exp(-kt^n)$
Henderson & Pabis	$MR = a \exp(-kt)$
Logarithmic	$MR = a \exp (+kt) + c$
Two-term	$MR = a \exp(-k_0 t) + b \exp(-k_1 t)$

#### **2.3.4. Effective Moisture Diffusivity (Deff)**

For most food materials, the internal diffusion occurring during the falling rate period is described by Fick's second law of diffusion. For a slab geometry, Crank (1979) provided the solution of diffusion equation (Equation 2.6) with the application of several boundary conditions (Onwude et al., 2016).

$$
MR = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp[-(2n-1)^2 \pi^2 \frac{D_{eff}}{4L^2} t] \tag{2.6}
$$

For long drying times, only the first term (setting  $n = 1$ ) of Equation 3.6 is used to estimate D<sub>eff</sub> in many cases. Thus, the simplified equation (Equation 2.7) can be written in logarithmic form as follows:

$$
\ln MR = \ln(\frac{8}{\pi^2}) - \frac{\pi^2 D_{eff}}{4L^2} t \tag{2.7}
$$

 $D_{\text{eff}}$  (m<sup>2</sup>/s) is the effective moisture diffusivity, and L is the half thickness of the slab (m). The  $D_{\text{eff}}$  can be determined from the slope of the plot of ln MR versus t.

#### **2.3.5. Activation Energy (Ea)**

For microwave drying, the activation energy may be estimated by using an Arrhenius-type relationship given as below (Onwude et al., 2016).

$$
D_{eff} = D_0 \exp(-\frac{E_a m}{P})
$$
\n<sup>(2.8)</sup>

 $E_a$  (Wg<sup>-1</sup>) is the activation energy,  $D_0$  (m<sup>2</sup>s<sup>-1</sup>) is the preexponential factor of the Arrhenius equation, P (W) is the microwave output power and m (g) is the mass of the sample.

### **2.4 Bulk Densities**

2 gr of lemon peel powder was weighted into 10 mL graduated cylinder without touching the interior wall. The volume occupied by the peel powder was noted and used to calculate poured bulk density  $(\rho_p)$ . Then, the same cylinder containing powder was manually tapped until no further volumes change occurs, and the new volume was used to calculate tapped bulk density( $\rho$ <sub>t</sub>) (Farahmandfar et al., 2020). Bulk densities were expressed as grams per milliliter (g/ml).

# **2.5 The Hausner's ratio (HR) and Carr index (CI)**

Carr Index (Carr, 1965) and Hausner ratio (Hausner, 1967) values were calculated from the following equations using the obtained poured bulk density  $(\rho_p)$  and tapped bulk density( $\rho_t$ ) values (Seerangurayar et al., 2017).

$$
CI = \frac{\rho_t - \rho_p}{\rho_t} \times 100 \tag{2.9}
$$

$$
HR = \frac{\rho_t}{\rho_p} \tag{2.10}
$$

### **2.6 Water Holding Capacity (WHC)**

0.5 g of powder sample was hydrated (24 h) in 30 ml of distilled water. Then, it was centrifuged at 2000g for 25 min. After decanting the supernatant, sample was weighted and the water holding capacity was calculated as g water/g dry sample (Romdhane et al., 2015).

#### **2.7 Oil Retention Capacity (ORC)**

0.5 g of powder sample was mixed with 10 ml of sunflower oil. Then, it was centrifuged at 2000g for 20 min. The supernatant was decanted, and the oil retention capacity was calculated as g oil/g dry sample (Romdhane et al., 2015).

# **2.8 Microstructure**

The lemon powder samples were fixed with carbon tape on the sample holder plate and then coated with gold and the microstructure of powder samples were evaluated with scanning electron microscope (SEM) (SU1510; Hitachi, Tokyo, Japan) at 10kV.

#### **2.9 Statistical Analysis**

Data were presented as means  $\pm$  standard deviations. Mean comparisons were performed by Analysis of variance (ANOVA) at a 5% significance level. (Minitab 17). Multiple comparisons were carried out using Tukey test.

### **3. Results and Discussion**

## **3.1 Drying Process**

Drying curves of lemon peels obtained during oven drying, during microwave drying were presented in Figures 1 and 2, respectively. Drying processes were continued until the moisture content of samples was reduced to about 10% (w.b.). It took nearly 130 min to reduce the moisture content of lemon peel to 9.3% moisture content by oven drying at 60 °C. During freeze drying, the time required to drop the moisture content of lemon peel to 9.23% was 8 h. Freeze drying (FD) of lemon peel took a longer time compared to oven drying. Despite of many advantages, freeze drying has been known to require long processing time. In studies on drying of citrus peels, similar findings have been recorded by different authors (Mello et al., 2020; Kirbas et al., 2019; Xu et al., 2017). Abd Rahman et al. (2016) showed that the moisture content of flavedo and albedo parts of pomelo peels reduced to 5.15 and 5.88% after drying in a convection oven at 60 °C for 24 h and reduced to 9.33 and 8.63 % after freeze drying for 96 h, respectively.

For each microwave drying condition, the time required for the moisture content of lemon peel samples to decrease from its initial moisture content of 78.4% (w.b.) to below 10.0% (w.b.) were given in Table 2. Compared to oven drying, the drying time shortened by 72.3-75.4%, 83.8-87.7%, 91.3-93.1%, and 93.5-94.6%

at 180, 300, 450, and 600W power levels, respectively by the application of microwave drying. This is an expected result since microwave drying is known to offer opportunity of high drying rate, especially in the falling rate period. In a study, Ghanem et al. (2020) reported that microwave drying is more effective in shortening drying time of lemon peel compared to infrared drying and hot air drying. Similarly, Kirbas et al. (2019) dried albedo parts of pomelo peel by microwave, forced convection and freeze drying and indicated that microwave drying provided the shortest drying time. In a different study, Talens, Castro-Giraldez, & Fito, (2016) observed that the application of microwave during hot air drying (55 °C) increased the mass reduction rate of orange peels. The result about the decrease in drying time of lemon peel with increasing power level was in accordance with the literature (Bejar et al., 2011b; Ghanem et al. 2012, 2020; Shu et al. 2020). Increasing power level from 180W to 600W provided 76.4-90.6 % shorter drying times. This is an expected result since higher power level causes increased level of energy absorption by the sample. This yields higher volumetric heat generation and consequent higher levels of moisture vapor generation creating significant pressure inside the food material.



Figure 1. Drying curve of lemon peel during oven drying



Figure 2. Drying curves of lemon peel during microwave drying

Rotation of turntable at different rates yielded 5.6-23.8% reduction in drying time of peels compared to drying without rotating the turntable. This reduction was the lowest at 180W (5.6-11.1%). For different power levels, the effect of rate of rotation on drying time did not follow a clear trend. At lower power levels (180, 300W), the highest rate of rotation (12.5 rpm) provided lower drying times. However, this is not valid for higher power levels. At higher power levels (450 W, 600W), 9.5 and 12.5 rpm gave similar drying times. Compared to 9.5 and 12.5 rpm, 6.5 rpm provided lower drying time at 450W, while higher drying time at 600W. These findings show that effect of rotation rate on drying time of lemon peels depended on the microwave power level.

# Table 2



Microwave drying times of lemon peels

\*M.C.: Moisture content (%wet base)

# **3.2 Drying Rate**

Drying curve includes three major stages which are transient period, constant rate period, and one or more falling rate periods. For lemon peels, a constant rate period was not observed during oven drying (Figure 3). The rate fell steadily with the decrease in water content. Similar to this finding, some researchers could not detect constant drying rate period during hot air drying of citrus peels (Deng et al., 2019; Mello et al., 2020; Garau et al., 2006; Romdhane et al., 2015; Xu et al., 2017). The absence of a constant drying rate suggests that diffusion was the dominant mechanism for removing of water from lemon peels during oven drying.

Microwave drying significantly enhanced the drying rate of lemon peels. During microwave drying of lemon peels, after the initial unsteady state warming-up period, both constant and falling rate periods were detected (Figure 4). After 20-24, 11-12, 5-6, and 4.5-5 minutes of drying at power levels of 180, 300, 450, and 600W, a rapid decrease in drying rate was seen. In literature, constant drying rate period have also been observed during microwave drying of mint leaves by Soysal, (2005), tomato slices by Abano et al., (2013) and amaranth leaves by Mujaffar & Loy (2016). Our results are also in agreement with the drying rate curves obtained for convective and microwave drying of onion slices (Demiray, Seker, & Tulek, 2017). In a study, Tasirin et al. (2014) observed both constant and falling rate periods during fluidized bed drying of orange peel.



Figure 3. Variation of drying rate with moisture content during oven drying



Figure 4 Variation of drying rate with moisture content during microwave drying

The rate of rotation did not create a clear effect on drying rate at the highest (600W) power level. At 600W power level, higher rate of heat generation within sample creates significant internal pressure which increases rate of moisture loss. Rapid rate of moisture removal at this power level may be a reason for not observing the effect of the rate of rotation on drying rate. At power levels of 180, 300, and 450 W, 9.5 rpm yielded lower drying rates compared to 6.5 and 12.5 rpm until reaching moisture contents of nearly 2.0, 1.0, and 0.4 kg water  $kg<sup>-1</sup>$  dry matter, respectively. When the moisture contents fell below these values, drying rates obtained at different rotational rates were quite similar. At 300W and 450W, 12.5 and 6.5 rpm rotational rates gave the highest initial drying rates, respectively.

In general, drying rate was higher at higher power levels during drying. When 450 and 600W powers were compared, this general trend showed some variations depending on the rate of rotation. At 6.5 rpm, both 450W and 600W power levels indicated similar drying rates during whole drying period. At 0 rpm and 12.5 rpm, 600W power level enhanced the drying rate of peels during initial period of drying compared to 450W, however, the drying rate curves almost overlapped under the moisture contents of 0.58 kg water kg<sup>-1</sup> dry matter (at 450 W) and 1.05 kg water  $kg^{-1}$  dry matter (at 600W).

### **3.3 Mathematical modelling of the drying curves**

In literature, some semitheoretical and empirical models have been used to describe the relationship between the moisture content and the drying time of citrus peels. In this study Page, Newton, Logarithmic, Henderson & Pabis and Two-term models were fitted to experimental microwave and oven drying data of lemon peels. The R<sup>2</sup>, SSE and RMSE values ranged between 0.984-0.997, 0.0058-0.0286, and 0.0184-0.0422, respectively for oven drying and between 0.928-0.997, 0.0064-0.2291, and 0.0199-0.0939, respectively for microwave drying (results not shown). Results of nonlinear regression analysis indicated that all the applied models were notable in description of the thin layer drying characteristics of lemon peel. Page model provided lower SSE, RMSE, and higher  $R^2$  values over the other models for all drying treatments. In a study, Shu et al., (2020) reported that Page model showed the best fit to experimental data within 9 different thin layer models during microwave vacuum drying (600W) of tangerine peels. It has been reported that the change of moisture ratios of lemon (Ghanem et al. 2020), orange (Bejar et al., 2011b), and pomelo (Tuncer et al., 2020). peels with microwave drying time were successfully described by the Page model. However, the most appropriate model for lemon and pomelo peels was found as the Midilli & Küçük and Logarithmic models, respectively. The statistical parameters and model coefficients (k and n) obtained from the fit of the Page model were reported in Table 3.

Some authors reported relations between the k value of Page model and the drying process variables like temperature, air velocity, microwave power density, sample thickness (Onwude et al., 2016). In this study, drying rate constant (k) values increased with increasing power level. It ranged between 0.014-0.0436 min<sup>-1</sup> and 0.1143-0.1306 min<sup>-1</sup> at power levels of 180W and 600W, respectively. During microwave drying of various foods, some authors similarly reported an increase in k value of Page model with increasing power level (Darvishi et al., 2014; Demiray et al., 2017). For microwave drying of orange slices, Alibas & Yilmaz (2021) noted an increase in k value (obtained from Modified Henderson model) with an increase in power density. A lower k value (0.00752 min<sup>-1</sup>) was estimated during oven drying compared to microwave drying. This was an expected result since microwave drying enhanced the drying rate of lemon peel as stated before. The change of k value with rate of rotation showed variance depending on the power level. In general, higher k values were obtained at 12.5 rpm compared to 6.5 and 9.5 rpm at all power levels.

<b>Power level</b>	<b>Rate of rotation</b>	<b>Parameters</b>		<b>SSE</b>	$\mathbb{R}^2$	<b>RMSE</b>	
	(rpm)	$k(1/\text{min})$ n					
Oven drying		0.00752	1.270	0.0058	0.9968	0.0184	
180W	$\overline{0}$	0.0124	1.556	0.0176	0.9934	0.0283	
	6.5	0.0170	1.494	0.0205	0.9919	0.0305	
	9.5	0.0214	1.423	0.0275	0.9887	0.0353	
	12.5	0.0436	1.253	0.0311	0.9856	0.0376	
300W	$\boldsymbol{0}$	0.0174	1.735	0.0170	0.9947	0.0261	
	6.5	0.0349	1.571	0.0173	0.9941	0.0263	
	9.5	0.0234	1.674	0.0203	0.9935	0.0285	
	12.5	0.0395	1.553	0.0149	0.9949	0.0244	
450W	$\boldsymbol{0}$	0.1022	1.533	0.0081	0.9960	0.0224	
	6.5	0.0946	1.704	0.0064	0.9970	0.0199	
	9.5	0.0700	1.723	0.0123	0.9943	0.0278	
	12.5	0.0961	1.637	0.0119	0.9942	0.0273	
600W	$\boldsymbol{0}$	0.1143	1.552	0.0134	0.9945	0.0259	
	6.5	0.1058	1.618	0.0182	0.9927	0.0301	
	9.5	0.0975	1.653	0.0234	0.9907	0.0342	
	12.5	0.1306	1.529	0.0188	0.9921	0.0307	

Table 3 Fitting parameters of the Page model.

### **3.4 Effective moisture diffusivity (Deff) for microwave drying**

The  $D_{\text{eff}}$  values at different microwave drying conditions were calculated by using Equation 2.7. The citrus peel was considered as homogeneous by neglecting the thickness of flavedo compared to albedo. The water loss through flavedo was assumed to be prevented due to waxy layer (cuticle) covering it (Garau et al., 2006; Garcia-Perez et al., 2009, 2012). Some researchers have observed that after hot air drying, the characteristic distribution of waxy components over the cuticular surface of orange peel disappeared and the stomatal pores become clogged (Garcia-Perez et al., 2012; Tamer et al., 2016). Garcia-Perez et al. (2012) stated that spread of waxy components over surface makes the moisture removal from this surface more difficult.

The  $D_{\text{eff}}$  values were listed in Table 4. The  $D_{\text{eff}}$  value was found to be 0.454 x  $10^{-8}$  m<sup>2</sup>s<sup>-1</sup> for oven drying while it ranged between  $1.688 \times 10^{-8} \text{ m}^2\text{s}^{-1}$  -  $7.595 \times 10^{-8} \text{ m}^2\text{s}^{-1}$  for microwave drying. These results are comparable with literature. For citrus peels subjected to various drying methods, D<sub>eff</sub> values ranging between 0.0495x10<sup>-8</sup>  $-7.295 \times 10^{-8}$  m<sup>2</sup>s<sup>-1</sup> have been reported in literature (Garcia-Perez et al. 2009; Deng et al. 2019; Tuncer et al. 2020). At similar rates of rotation, D<sub>eff</sub> values increased with increasing power level. This result agrees with the results presented by other authors (Darvishi et al., 2014; Alibas & Yilmaz, 2021; Demiray et al., 2017). Microwave drying performed without rotating the turntable caused lower D<sub>eff</sub> values at all power levels. No significant effect of rate of rotation on  $D_{\text{eff}}$  values was observed.

Table 4

Effective diffusivity  $(D_{\text{eff}})$  and activation energy (Ea) values obtained during microwave drying



# **3.5 Activation Energy**

For microwave drying, activation energy values calculated using Eq. 2.8. were reported in Table 4. The high values of  $R^2$  ( $>0.96$ ) are indicative of good fitness of empirical relationship to represent the relationship between D<sub>eff</sub> and the ratio of sample mass to microwave power level (m/P). Ea values estimated from D<sub>eff</sub> values ranged between 21.33 and 22.65 W/g. The values of  $D_{\text{eff}}$  obtained for lemon peel are consistent with the  $D_{\text{eff}}$  values for different types of fruits and vegetables (ranging between 5.54-24.22 W/g) reported in literature (Murthy & Manohar, 2012; Darvishi et al., 2014; Demiray et al., 2017). However, D<sub>eff</sub> values obtained in this study were lower than that presented by Tuncer et al. (2020) for the pomelo peel (124.36 and 115.03 W  $g^{-1}$  for 0.5 and 1 cm thick peels, respectively) subjected to microwave drying at 7 W.g<sup>-1</sup> power density.

## **3.6 Bulk Density**

Citrus peels are rich source of dietary fibers (DFs). DFs are commonly classified as soluble dietary fibers (SDFs) including non-cellulosic polysaccharides, oligosaccharides, pectin, β-glucans, gums, and insoluble dietary fibers (IDFs) including cellulose, hemicellulose, lignin. Drying process may lead to the modification of the microstructures of the dietary fibers of citrus peel powders and consequently their physical properties (Abou-Arab et al., 2017, Romdhane et al., 2015, Ghanem et al., 2020). Bulk density is an important physical property which is related with the interparticle voids in the sample. Lower proportion of air volume in sample give higher bulk density (Barbosa-Canovas et al., 2005). Greater bulk density is desirable for reducing packaging and transportation costs. Bulk density values of the powder samples were given in Table 5.

Table 5

Poured bulk density  $(\rho_p)$ , Tapped bulk density  $(\rho_t)$ , Hausner ratio (HR) and Carr index (CI) of lemon peel powders

<b>Drying</b> <b>Treatment</b>	Rate of rotation	$\rho_{\rm p}$ (g/ml)	$\rho_t (g/ml)$	HR	<b>Cohesiveness</b> **	CI(%)	<b>Flowability</b> ***
Freeze drying		$0.212 \pm 0.029$ d <sup>*</sup>	$0.335 \pm 0.055$ c	$1.576 \pm 0.117$ a	High	36.184±4.975 a	Bad
Oven drying		$0.374 \pm 0.018$ c	$0.495 \pm 0.020$ b	$1.327 \pm 0.068$ b	Intermediate	24.448±3.852 b	Fair
180W	$\mathbf{0}$	$0.468 \pm 0.016$ ab	$0.603 \pm 0.029$ a	$1.290 \pm 0.071$ b	Intermediate	22.276±4.073 b	Fair
	6.5	$0.468 + 0.020$ ab	$0.615 + 0.042$ a	$1.313 + 0.070$ b	Intermediate	$23.623 + 4.042$ h	Fair
	9.5	$0.466 \pm 0.026$ ab	$0.615 \pm 0.040$ a	$1.322 \pm 0.085$ b	Intermediate	$24.076 + 4.878$ b	Fair
	12.5	$0.460 \pm 0.022$ ab	$0.617 \pm 0.041$ a	$1.340\pm0.075$ b	Intermediate	25.193±4.105 b	Fair
300W	$\boldsymbol{0}$	$0.452 \pm 0.040$ ab	$0.594 \pm 0.048$ a	$1.318 \pm 0.093$ b	Intermediate	23.808±5.419 b	Fair
	6.5	$0.472 \pm 0.025$ a	$0.616 \pm 0.041$ a	$1.308 \pm 0.093$ b	Intermediate	$23.198 \pm 5.541$ b	Fair
	9.5	$0.468 \pm 0.031$ ab	$0.617 \pm 0.038$ a	$1.321 \pm 0.099$ b	Intermediate	$23.884\pm5.546$ b	Fair
	12.5	$0.467 \pm 0.030$ ab	$0.621 \pm 0.042$ a	$1.332\pm0.090$ b	Intermediate	24.659±4.913 b	Fair
450W	$\mathbf{0}$	$0.463 + 0.025$ ab	$0.608 + 0.028$ a	$1.317 + 0.051$ h	Intermediate	23.958±2.920 b	Fair
	6.5	$0.470 \pm 0.032$ a	$0.614 \pm 0.031$ a	$1.311\pm0.072$ b	Intermediate	23.515±4.167 b	Fair
	9.5	$0.465 \pm 0.019$ ab	$0.622 \pm 0.029$ a	$1.338\pm0.084$ b	Intermediate	25.009±4.840 b	Fair
	12.5	$0.471 \pm 0.022$ a	$0.618 \pm 0.036$ a	$1.315 \pm 0.075$ b	Intermediate	23.753±4.309 b	Fair
600W	$\overline{0}$	$0.428 \pm 0.013$ b	$0.571 \pm 0.030$ ab	$1.334\pm0.074$ b	Intermediate	$24.851 \pm 4.193$ b	Fair
	6.5	$0.444\pm0.028$ ab	$0.603 \pm 0.031$ a	$1.363\pm0.091$ b	Intermediate	26.326±4.688 b	Fair
	9.5	$0.448 + 0.024$ ab	$0.614\pm0.035$ a	$1.373 \pm 0.072$ b	Intermediate	27.016±3.745 b	Fair
	12.5	$0.455 \pm 0.018$ ab	$0.611\pm0.044$ a	$1.344\pm0.079$ b	Intermediate	25.343±4.285 b	Fair

\*Values represent mean  $\pm$  standard deviation. Different small letters in the same column shows significant difference ( $p < .05$ ).

\*\* The cohesiveness is low (HR<1.2), intermediate (1.2<HR<1.4) and high (HR>1.4) [\(Hausner, 1967\)](https://www.sciencedirect.com/science/article/pii/S0260877417303084#bib21)

\*\*\*The flowability is very good (CR<15), good (15<CR<20), fair (20<CR<35), bad (35<CR<45%) and very bad (CR>45) (Carr, 1965)

The poured and tapped bulk densities of the freeze dried and oven dried powder samples was lower than the microwave dried ones. Microwave dried peel powders had tapped bulk densities ranging between 0.571 and 0.622 g/ml. Freeze drying yielded the lowest poured and tapped bulk densities. This was thought to be due to the more porous structure of the freeze-dried powders. This result is in line with those reported by different authors (Farahmandfar et al. 2019, 2020; Tekgül & Baysal, 2018, Liu et al., 2017; Lee et al., 2012). Liu et al., (2017) obtained the scanning electron micrographs of orange peel dietary fiber powders and reported that the

three-dimensional structures of soluble and insoluble dietary fiber particles were affected by the drying method. They noted that the increase of the porosity and surface area of the fibers during freeze drying lead to lower bulk density compared to hot air drying. Tekgül & Baysal (2018) obtained lower bulk density for freeze dried (0.17 g/ml) lemon peel powder compared to open-air, hot air and microwave dried samples (0.24-0.25 g/ml). Farahmandfar et al. (2019, 2020) obtained bulk density values ranging between 1.32-and 3.20 g/ml for powder of orange peels dried by various drying methods (shade, sun, oven, vacuum oven, microwave, and freeze-drying) and similarly indicated that the freeze-dried samples had the lowest bulk density. This was attributed by the authors to the protection of structure of freeze-dried sample with minimal shrinkage.

For microwave drying, the data were analyzed using general linear model and Tukey's pairwise comparison test. The mean of the bulk densities obtained at 600W was found to be lower compared to those obtained at lower power levels. However, this difference was not statistically significant for tapped bulk density. The other power levels provided similar bulk density values. Microwave drying without rotation caused lower mean value of tapped density compared to microwave drying with rotation. Bulk densities of lemon peels dried at rotational rates of 6.5, 9.5 and 12.5 rpm were similar. Microwave drying without rotation (0 rpm) causes non uniform heating. This may create variation in the degree of peel matrix destruction during drying which could affect the grinding performance resulting in relatively non uniform particle size distribution in powder. This may be one of the reasons for the lower density of the samples dried at 0 rpm. When the SEM images were examined, no clear difference was observed between the samples dried at 600W and the ones dried at 180W. However, during drying at 600W, local burns occurred due to the high temperatures reached in the sample. This may be a reason for the observation of the lower bulk density obtained at 600W as explained above. Non uniform heating was not observed at 180W.

# **3.7 Hausner ratio (HR) and Carr index (CI)**

Hausner ratio (HR) and Carr index (CI) are generally used to assess flowing properties of powders. Higher HR value means that this sample was relatively more cohesive resulting in decreased flowability of the powder. Freeze drying caused the highest HR (1.576) and CI (36.184) values which means freeze dried peel powder had bad flowability (Seerangurayar et al., 2017). Oven dried and microwave dried samples had similar HR and CI values. HR and CI of the microwave dried lemon peel powders ranged from 1.29 to 1.37 and 22.28 to 27.02, respectively. According to the reference values given in Table 5, oven dried, and microwave dried powders had fair flowability. Lee et al. (2012) showed that the Hausner ratio values of powders from pressed cake of citrus "Hallabong" ranged between 1.105 and 1.150 depending on frying method and particle size. In their study, for the samples having similar particle size, freeze drying yielded higher HR values. These results are in good agreement with the results of this study. Power level and the rotational rate of turntable were not significant factors effecting HR and CI of the samples.

## **3.8 Functional Properties of Powder**

Drying may have positive or negative effects on some functional properties of citrus peels depending on citrus variety and drying treatment. Water holding capacity (WHC) and oil retention capacity (ORC) are the functional properties of powders that have been widely studied. The effect of various drying treatments on WRC and OAC of citrus peels are presented in Table 6. In literature, the WHC and ORC of the dried citrus peel powders ranged between 2.7-13 g water/ g DM and 0.52-5.1 g oil/g DM, respectively.

In this study, dried lemon peel powder had the WHC and ORC values ranging between 6.34-9.32 g water/ g DM and 0.96-3.03 g oil/g DM, respectively (Table 7). Microwave drying at 180W irrespective of rate of rotation and at 450 and 600 W without rotation yielded significantly lower WHC values compared to oven drying. Except those samples, microwave drying yielded lower WHC values compared to freeze drying and oven drying, however the difference was not statistically significant. This is at least in part may be due to the higher temperatures reached in samples during microwave drying. Drying temperature is known to influence the functional properties of powders. The functional properties of citrus peel powders have been shown to be negatively affected by the higher drying temperatures (≥60 °C) (Garau et al., 2007; Romdhane et al., 2015;

Ghanem et al., 2020; Deng et al., 2019). Deng et al. (2019) reported no significant change in WHC of orange peel dried at 50 and 60 °C, however, further increase in temperature exhibited a reduction in WHC. Ghanem et al. (2020) noted that formation of more rigid structure resulting from higher temperature may lead to a lower WHC in lemon peel. Similar finding was reported by Bejar et al. (2011a) for orange peel undergoing infrared drying. Garau et al. (2007) recorded that high (90 °C) and low (30 °C) drying temperatures caused significant losses of pectic polymers during air-drying of orange peels. Degradation of pectic substances during processing is known to influence the functional properties. Higher temperature and the internal pressure created within material during microwave drying may cause deformation of cellular tissue resulting loss in WRC (Bejar et al., 2011a; Romdhane et al., 2015). However, our result differed from the results presented by Abou-Arab et al. (2017) who showed that microwave (900W) dried citrus (orange, tangerine, and lemon) peels had better functional properties compared to solar (40 °C) and oven (40 °C) dried ones.

Table 6

	Effect of drying treatments on some functional properties of citrus peels			



WHC: Water holding capacity; OAC: Oil absorption capacity; T: Temperature

The power level did not exert a clear effect on WHC of citrus peels during microwave drying. For the microwave drying with rotation, 600W provided slightly higher WHC values (7.6-7.9 g water/ g DM). The maximum WHC (7.9 g water/ g DM) obtained in this study was similar to that presented by Ghanem et al., (2012) for lemon peels (8.15 g water/ g DM) dried by microwave drying at 450W power level. In their study, the change of WHC did not follow a clear trend with increasing power level (100-600W) and the citrus peels from different citrus varieties had the maximum WHC values at different power levels (100-600W). At higher power levels, influence of turntable rotation on WHC was more notable. At 600W, microwave drying with rotation provided 20-25% higher WHC values compared to microwave drying without rotation. The level of increase was 9-14%

Table 7

at 450W. In the case of microwave drying with rotation, 9.5 rpm provided slightly higher WHC values compared to 6.5 and 12.5 rpm at all power levels. However, differences were not significant. Variation of rotational rate yielded 1-5.5 % change in WHC values.





\*Values represent mean  $\pm$  standard deviation. Different small letters in the same column shows significant difference ( $p < .05$ ).

Microwave dried lemon peels had ORC values ranging between 1.359 and 1.692 g oil/ g DM. These values were similar to ORC of microwave dried citrus peels (1.16-1.95 g oil/ g DM) (Abou Arab et al., 2017) and lemon peels (around 1.8 g oil/ g DM) (Ghanem et al., 2012) but lower than ORC of microwave dried orange peel measured by Bejar et al. (2011b). Freeze drying provided the highest ORC. Microwave dried samples except the one dried at 180W (12.5rpm) had similar ORC values compared to oven dried ones. Some authors noted the opposite direction of the changes in WHC and ORC which was explained by the alteration in water affinity of components due to modification of the structural characteristics and change in the composition of fiber after microwave drying process (Ghanem et al., 2012; Bejar et al., 2011b). No such effect was observed in the results obtained in this study. The highest power level (600W) provided relatively higher ORC values. Influence of turntable rotation on ORC did not follow a clear trend. In general, 12.5 rpm provided slightly higher ORC values compared to 6.5 and 9.5 rpm at all power levels.

### **3.9 Microstructure**

SEM images of the lemon peel powders dried by different drying treatments were obtained to examine the microstructures (Figure 5). It is clear that the freeze-dried powder had large pores. It is widely known that freeze drying protects the structure and shape of the materials with minimal shrinkage. Dehydration via sublimation creates pores within material during freeze drying. The larger size and more porous particle structure of freeze-dried peel powders explains why the bulk densities of the freeze-dried samples was lower than the others. This result is consistent with the results of Lee et al. (2012) who obtained larger pore size for the freezedried powders obtained from pressed cake of citrus "Hallabong" compared to hot air-dried ones.



Figure 5. SEM images of lemon peel powder subjected to (a)freeze drying, b) oven drying, and microwave drying at c) 180W-0rpm, d)180W-12.5rpm, e)600W-0rpm, f)600W-12.5 rpm.

Oven dried and microwave dried samples had irregular shapes and rough surface. It can be seen that the particle size distribution differs between the samples. However, it is not possible to talk about a clear effect created by the higher microwave power level or rotation of turntable.

#### **4. Conclusion**

Lemon peels with high moisture content were dried with microwave drying to obtain a powder product. Microwave drying with rotation provided lower drying time compared to drying without rotation. Rate of rotation did not exert a clear effect on drying time of lemon peel at 300 and 450W power levels. Rate of rotation influenced initial drying rate of peels at 300 and 450 W power levels. Page model best described the drying characteristics of lemon peels at all drying conditions. Microwave drying at 600W gave higher Deff values. Microwave dried lemon peels had higher bulk density values and similar or lower water holding and oil retention capacities compared to freeze dried and oven dried ones. Variation of rotational rate from 6.5 to 12.5 rpm did not yield a significant change in WHC and ORC values. The findings showed that, when the appropriate parameters are selected, microwave drying may be an alternative method that can be used for drying of lemon peels. Higher power levels gave better results regarding drying time and the investigated quality parameters. Microwave dried lemon peel powder has the potential to serve as a functional component in the food processing industry in terms of its flow properties and functional features. However, apart from the functional properties, the physical properties and the chemical composition of the lemon peel powder need to be evaluated to determine the most suitable microwave drying parameters in respect of preserving the desired quality attributes.

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#### **Author Contributions**

Sevilay San: Performed the analyzes

Işıl Barutçu Mazı: Planned the study, performed statistical analysis, and wrote the paper.

### **Conflicts of Interest**

The authors declare no conflict of interest.

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