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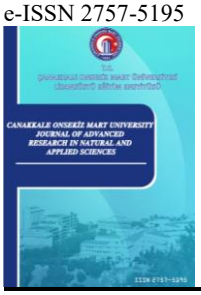

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Volatile Organic Compounds, Total Phenolic Content, Color, and Heating Uniformity of Lemon Peel as Affected by Rotational Speed of Turntable During Microwave Drying

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Research Article

Abstract − In this study, the effects of rotational speed of microwave turntable (0, 6.5, 9.5, and 12.5 rpm) on surface temperature distribution of lemon peels during drying and, on some quality attributes (color, water activity, total phenolic content (TPC), and volatile organic compounds) of dried lemon peel powders were investigated. The quality analyses were also performed in freeze-dried peels. During microwave drying with rotation, speed of turntable affected the surface temperature values and TPC depending on the power level but did not exert a clear effect on the homogeneity of the surface temperature distribution and the color parameters. The percentage amount of major monoterpene hydrocarbons (limonene, γ-terpinene, myrcene, *p*-cymene, and α-*p*-dimethylstyrene) detected in fresh lemon peel decreased after microwave and freeze-drying. At 600W, formation of furan compounds (furfural, furfuryl alcohol, tetrahydrofurfuryl alcohol, 2-acetyl furan, 5-methyl furfural, and HMF) were identified. Microwave drying without rotation (0 rpm) caused uneven heating which led to the production of peel powders with unacceptable dark color especially at 450 and 600W. Drying at 600W-0 rpm gave the darkest color, the highest amount of TPC (1730.7 mg GAE/100g d.b.) and furan compounds. Therefore, microwave drying at low power levels with rotation function preserved the quality of lemon peel powder better.

Keywords *–Lemon peel, microwave drying, turntable speed, uniformity, volatile compound, waste utilization*

1. Introduction

Citrus peel is the primary by-product of citrus juice processing. Citrus peel is a rich source of valuable phytochemicals, such as phenolic compounds, carotenoids, dietary fibers, ascorbic acid, and essential oils (Singh et al., 2020). Since it contains many valuable substances, it may be utilized as a source of healthy and functional ingredient in food industry. There are various studies in literature where dried powder of citrus by-products has been used in preparation of various foods like bread, sausage, yogurt, ice cream (Han et al., 2021; Yi et al., 2014; Crizel et al., 2014; Tomaschunas et al., 2013). Positive results have been obtained from the incorporation of citrus by-product powders into different food products depending on the dose and properties of powder. The use of this by-product in food formulations provides economic benefits to food industries without sacrificing the environment.

The quality of peel powder is important to obtain the desired food characteristics. It is well known that drying conditions during powder processing greatly influence the physicochemical or nutritional properties of the final product (Karam et al., 2016). So, the application of proper drying treatment is important to obtain high quality product while extending its storage life. Various studies have been performed to investigate the effects of different drying treatments on physicochemical, nutritional, and technological properties of citrus peels. Drying of citrus peels may promote important modifications affecting phenolic compounds (M'Hiri et al.,

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2017), induce significant changes in color parameters (Ghanem et al., 2012, 2020) depending on the method of drying and type of citrus. Drying process was shown to have remarkable effect on the compounds identified in essential oils extracted from citrus peels (Tekgül & Baysal, 2018, 2019; Zhang et al., 2018; Farahmandfar et al., 2020; Kamal et al., 2011).

Microwave drying has been used by some researchers for drying of citrus peels (Ozcan et al., 2020; Farahmandfar et al., 2019, 2020; Abou Arab et al., 2017; Mahmoud et al., 2015; Ghanem et al., 2012, 2020; Bejar et al., 2011b; Tekgül & Baysal, 2018; Tuncer et al., 2020; Assefa & Keum, 2017). During microwave drying, volumetric heat generation occurs within the product. This offers opportunity of higher drying rate, especially in the falling rate period compared to traditional drying methods. Microwave power level affects the rate of drying. The effect of microwave power level was studied by Ghanem et al. (2012, 2020) for lemon peel, by Bejar et al. (2011b) for orange peel and by Shu et al. (2020) for tangerine peel. They noted that microwave power level is an important factor influencing dehydration rate and some quality attributes of citrus peels. While the microwave drying method provides many advantages it also brings some disadvantages when used alone. The major drawback of using microwave drying alone is the non-uniform heating (Zhang et al., 2006). The turntable, one of the basic elements of a home type microwave oven, is one of the methods to increase temperature homogeneity (Geedipalli et al., 2007). To our knowledge, no studies have been conducted on the effect of rotational rate of the turntable on quality characteristics of a food material. Thus, the objective of the present study was to investigate the effect of the rotational rate of turntable on uniformity of surface temperature distribution of lemon peels during microwave drying and some quality properties (color, total phenolic content, and volatile organic compounds) of lemon peel powder.

2. Materials and Methods

Lemon fruit (*Citrus limon* (L.) var. Meyer) cultivated in Adana was obtained from a local grocery store in Giresun, Türkiye. Lemon fruits were kept in a refrigerator at 4±0.5°C. Before drying experiments, lemon fruits were taken out of the refrigerator and left for 2 hours to reach the ambient temperature. The fruits were washed with tap water and dried with tissue paper. Peels (containing flavedo and albedo) of the fruits were manually separated and cut into 10 mm-sided square slab shapes (~4 mm thickness).

2.1 Drying Process

The lemon peel samples were dried using a domestic microwave oven (MC32F604TCT, Samsung, Port Klang, Malaysia). The microwave oven was modified to adjust the speed of the turntable. Lemon peel samples were spread on a petri dish of 11.5 cm diameter as a single layer, with the albedo tissue at the top. Then, the petri dish was placed at the centre of the turntable. Microwave drying was performed at power levels of 180W, 300W, 450W, and 600W and at rotational rates of 0 rpm, 6.5 rpm, 9.5 rpm, and 12.5 rpm. Drying was continued until reducing moisture content of fresh lemon peel (78.4%) to the final moisture content of about 10% (Tekgül & Baysal, 2018). For each drying condition, preliminary experiments were performed to determine the drying time required to attain final moisture content. After drying, lemon peels were ground (SCM-2934, Sinbo, Türkiye) and sieved (40 mesh sieve) to obtain peel powder.

Freeze drying produces the highest-quality dried foods. Therefore, to have a basis of comparison for the quality parameters of peel powders, lemon peels were dried also by freeze drying in a lab-scale freeze dryer (FreeZone 2.5L 7670530, Labconco, Fort Scott, KS, USA, at -50°C and 0.1 mbar vacuum pressure).

2.2 Determination of Moisture Content

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

2.3 Infrared (IR) Thermal Imaging

Infrared (IR) thermal camera (PTi120, Fluke Corp., Everett, WA, USA) was used to obtain the surface temperatures of the samples. During drying, the sample was taken out of the microwave-oven at certain intervals, its thermal image was taken and placed back into the oven. The imaging process was repeated in 3 parallels. The IR images were analyzed using Fluke SmartView 4.4 software program (Everett, WA, USA).

2.4 Color

A colorimeter (Minolta CR-400, Minolta Co. Ltd., Osaka, Japan) was used to determine the L* (lightness-darkness), a* (redness-greenness), and b* (yellowness-blueness) color parameters of dried peel powders. For color determination, each drying treatment was replicated nine times and the results were averaged. The color parameters of a sample were obtained by taking the average of L*, a*, and b* readings from five different positions on its surface.

2.5 Total Phenolic Content (TPC)

TPC of samples was determined by a modified Folin–Ciocalteu method (Apak et al., 2008). For extraction of phenolic compounds, 0.5 g of peel powder was mixed with 5 ml of 90% ethanol solution. This mixture was thoroughly mixed in a shaker (Rocker-shaker MR-12 Biosan, Vetrotecnica, Padova, Italy) at 50 rpm for 60 min. Then, the extracts were centrifuged (D2012 Plus, ISOLAB Laborgeräte GmbH, Wertheim, Germany) at 15,000 x g for 10 min. The supernatant was taken and diluted with 90% ethanol solution (1:10). 60 µL of diluted extract solution was mixed with 3.48mL distilled water and 300 µL of Folin–Ciocalteu reagent (2 N). After waiting the mixture for 8 min at dark, 900 µl of 20% Na_2CO_3 solution was added to it. Then, the mixture was incubated in a water bath (WSB 18L, Daihan Scientific, Korea) at 40 °C for 30 min. The absorbance was determined at 760 nm in a spectrophotometer (UV mini-1240, Shimadzu, Kyoto, Japan). Results were expressed as milligram gallic acid equivalents (mg GAE/100g d.b.).

2.6 Headspace - Solid Phase Microextraction (HS-SPME)

One gram of the finely crushed fresh peel or dried peel powder sample was weighed into a 15ml vial closed with a PTFE/Silicone septa cap. The samples were placed on the heating block at 60°C and equilibrated for 15 minutes. After equilibration, a carboxen/polydimethylsiloxane (CAR/PDMS) manual fiber (75 µm Fused Silica, Supelco Ltd., Bellefonte, PA, USA) was inserted into the vial. CAR/PDMS fiber was maintained in the headspace for 60 min at 60°C for solid-phase microextraction (SPME) of volatile organic compounds (VOCs) from the sample (Mazı et al., 2019).

2.7 Gas Chromatography – Mass Spectrometry (GC/MS) Analysis

The effect of different drying methods on the VOCs of lemon peel was determined using gas chromatography/mass spectrometry (GC/MS). The desorption and chromatographic separation of VOCs was performed in a gas chromatograph (GC) (GC-2010 plus, Shimadzu, Japan) equipped with a mass spectrometry (MS) detector, and a Restek Rxi-5 MS capillary column (30m x 0.25mm i.d. x 0.25 µm film thickness). The carrier gas was Helium at a flow rate of 1.44 ml/min; the column temperature program of GC was initially set at 40°C for 2 min and gradually increased (4°C/min) to 250°C, then kept there for 3 min. The temperatures of the injector and detector were 250°C. For GC/MS detection, electron ionization (EI) system was used with ionization energy at 70 eV. VOCs from the samples were identified by comparing their mass fragmentation pattern with those stored in the mass spectral libraries (NIST, Wiley and FFNSC) (Mazı et al., 2019).

2.8 Statistical Analysis

The data represent mean±standard deviation of triplicate determinations unless otherwise specified. The results were analyzed by analysis of variance (ANOVA) followed by Tukey's multiple comparison test ($p < .05$) (Minitab, version 17).

3. Results and Discussion

3.1 Surface Temperature Distribution

IR thermal image provides information about the surface temperature distribution. IR thermal images of lemon peels obtained during microwave drying at 180, 300, 450 and 600W power levels were presented in Figures 1, 2, 3, and 4, respectively.

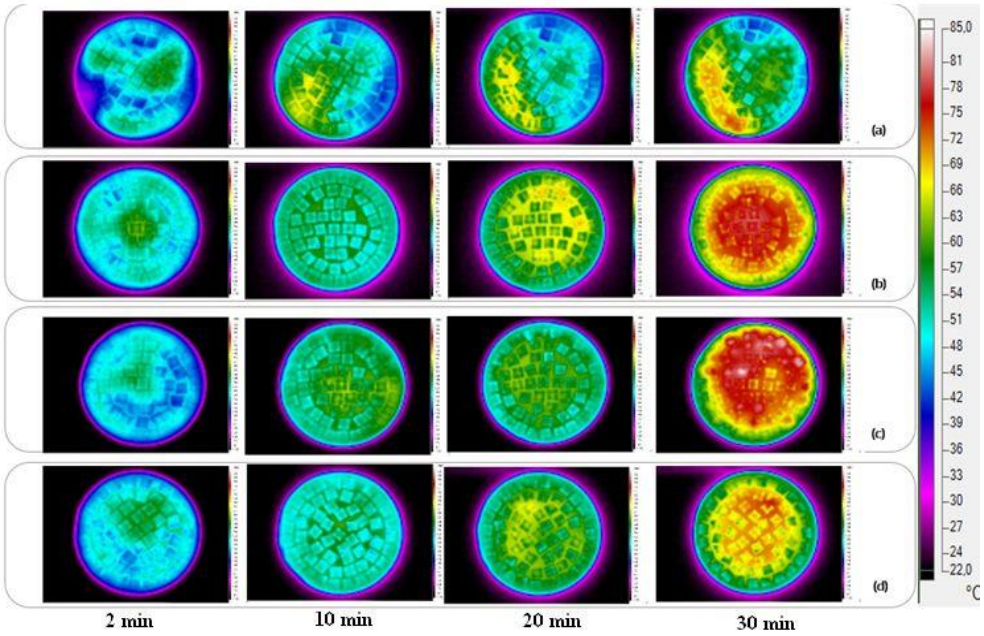

Figure 1. IR thermal images of lemon peels during microwave drying at 180 W and rotational rate of 0 rpm a), 6.5 rpm b), 9.5 rpm c), 12.5 rpm d)

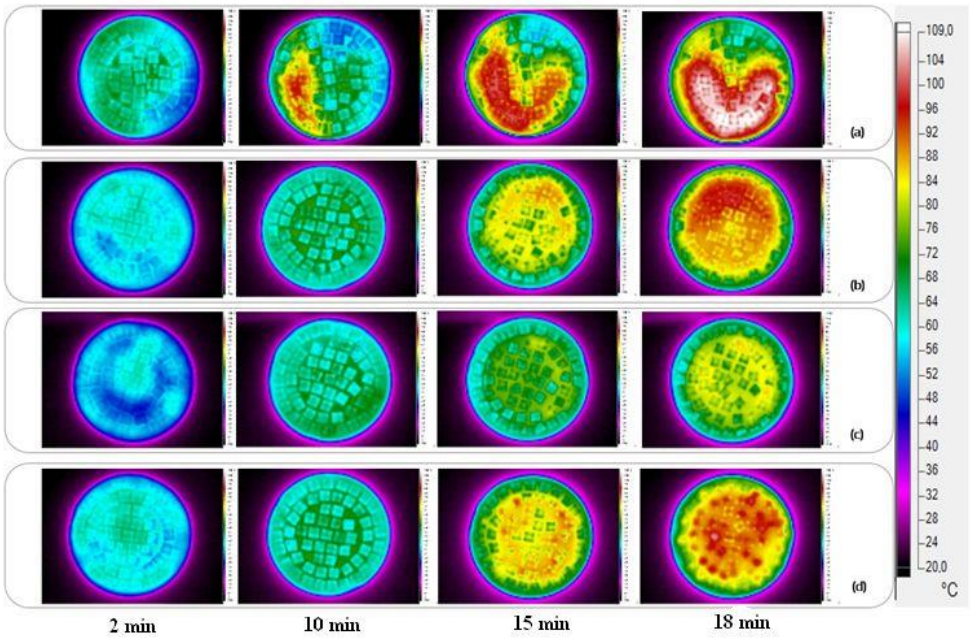

Figure 2. IR thermal images of lemon peels during microwave drying at 300 W and rotational rate of 0 rpm a), 6.5 rpm b), 9.5 rpm c), 12.5 rpm d)

As expected, the homogeneity of the temperature distribution during microwave drying without rotation (0 rpm) was lower compared to the microwave drying with rotation irrespective of the power level. During drying at 0 rpm, surface temperature raised to high levels in a certain region outside the center which caused non uniformity of temperature distribution in the product and, hence, inhomogeneous drying. In the case of microwave drying with rotation at high power levels (450, 600W), the surface temperature of samples located in the intermediate diameter were higher compared to the ones located at other regions. At lower power levels, microwave drying with rotation provided more uniform temperature distribution. During microwave drying, the drying time ranged from 32 to 36 min, 16 to 21 min, 9.0 to 11.3 min, and 7.0 to 8.5 min at power levels of 180, 300, 450, and 600 W, respectively. Longer drying time required at lower power levels may allow transfer of heat by conduction from hot regions to relatively colder regions and consequently formation of more uniform temperature distribution. Change of turntable rotation speed did not yield a noticeable effect on the homogeneity of the surface temperature distribution.

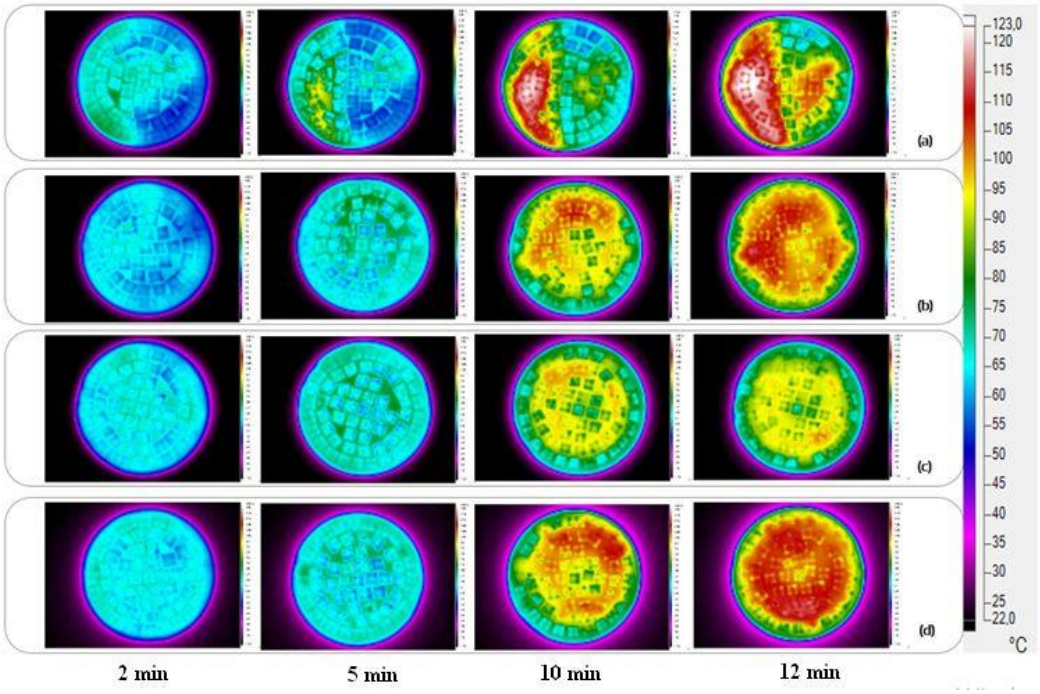

Figure 3. IR thermal images of lemon peels during microwave drying at 450 W and rotational rate of 0 rpm a), 6.5 rpm b), 9.5 rpm c), 12.5 rpm d)

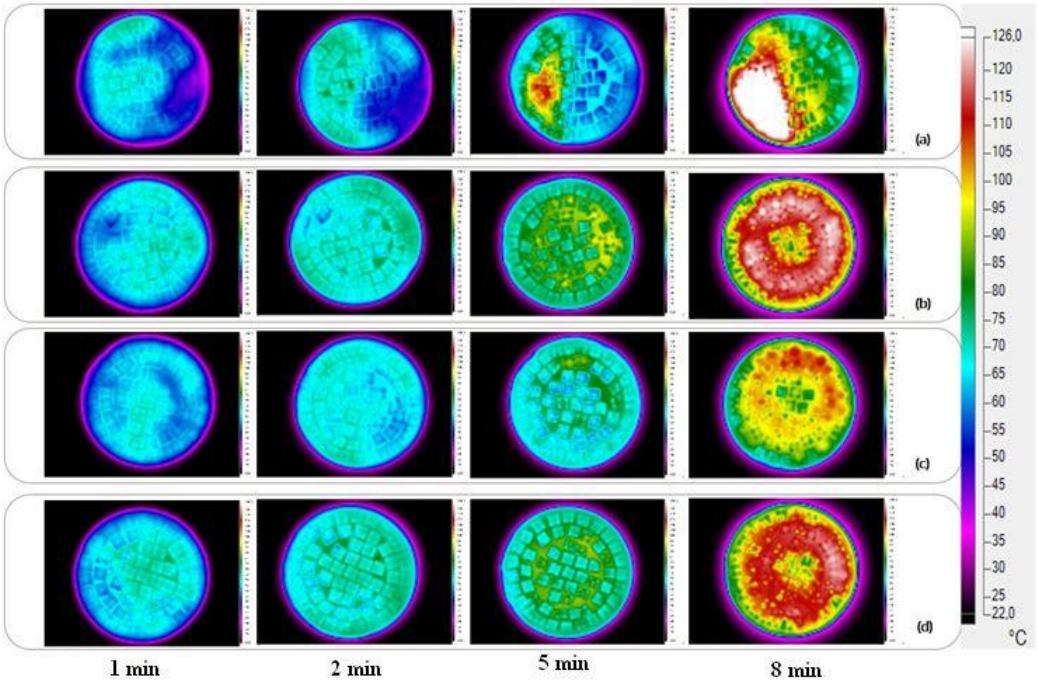

Figure 4. IR thermal images of lemon peels during microwave drying at 600 W and rotational rate of 0 rpm a), 6.5 rpm b), 9.5 rpm c), 12.5 rpm d)

Histograms of the thermal images were obtained using the SmartView (4.4.355.0) program of the Fluke camera (Figures 5-8). During initial period of microwave drying with rotation, a narrow single peak existed in histograms, but the number and width of the peaks increased in the later stages of drying process. This shows that there was a decrease in the homogeneity of the surface temperature distribution during the drying process. At power levels above 180 W, lower surface temperature values were reached at 9.5 rpm compared to 6.5 and 12.5 rpm rotation speeds at the end of the drying process. On the contrary, at 180 W power level, it was seen that the temperature values obtained at 9.5 rpm were higher. Acquisition of thermal images was performed in triplicate for each drying condition. The average of maximum temperature values obtained in samples during drying was calculated. At the end of the microwave drying without rotation, the maximum temperatures recorded were 71.4±2.7 °C, 109.7±2.4 °C, 127.6±2.9 °C and 153.5±9.5 °C at power levels of 180, 300, 450 and 600 W, respectively. The maximum temperatures recorded during microwave drying with rotation ranged between 74.2-82.0 °C, 90.4-103.8 °C, 104.5-116.4 °C, and 114.4-120.1 °C at power levels of 180, 300, 450 and 600 W, respectively. At 180 W power level, the temperature remained below 100 °C during drying. During microwave drying without rotation, the temperature in certain parts of the samples exceeded 100 °C in approximately 15 min at 300 W, within the first 10 min at 450 W and within the first 5 min at 600 W. Occurrence of overheating in these regions caused local burns. Similarly, during microwave drying with rotation, overheating was observed in certain areas at 450 W and 600 W power levels. The degree of local burns was less at 9.5 rpm than at 6.5 and 12.5 rpm.

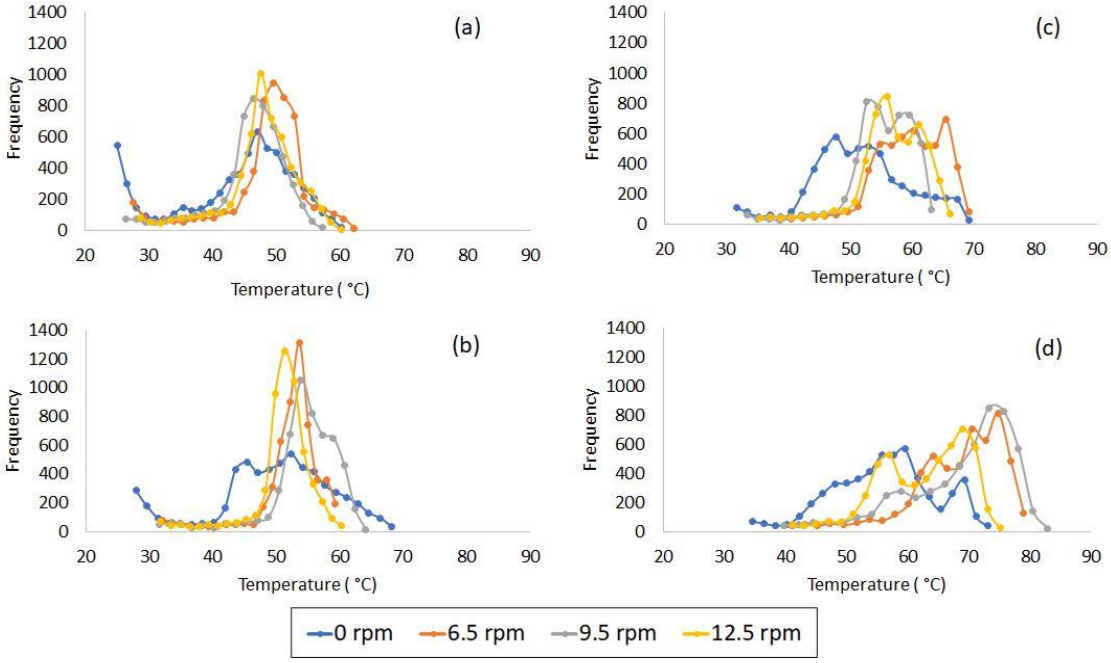

Figure 5. Histograms obtained from thermal images of lemon peels after 2 min a), 10 min b), 20 min c), and 30 min d) of microwave drying at 180 W

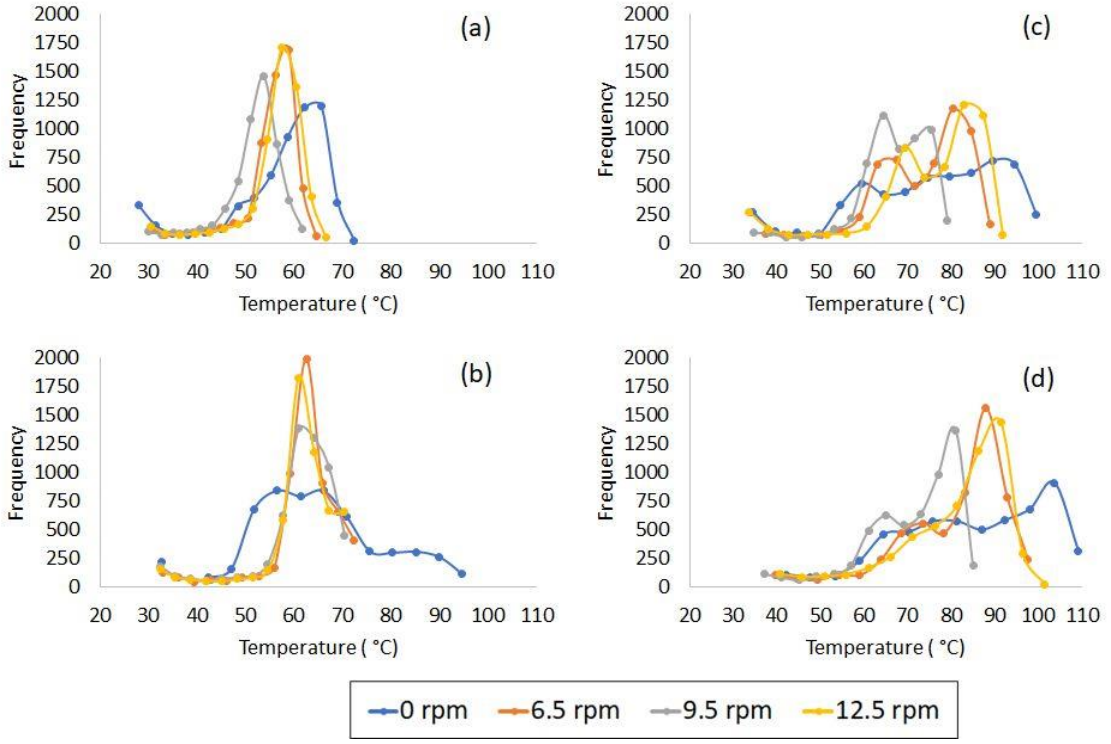

Figure 6. Histograms obtained from thermal images of lemon peels after 2 min a), 10 min b), 15 min c), and 18 min d) of microwave drying at 300 W

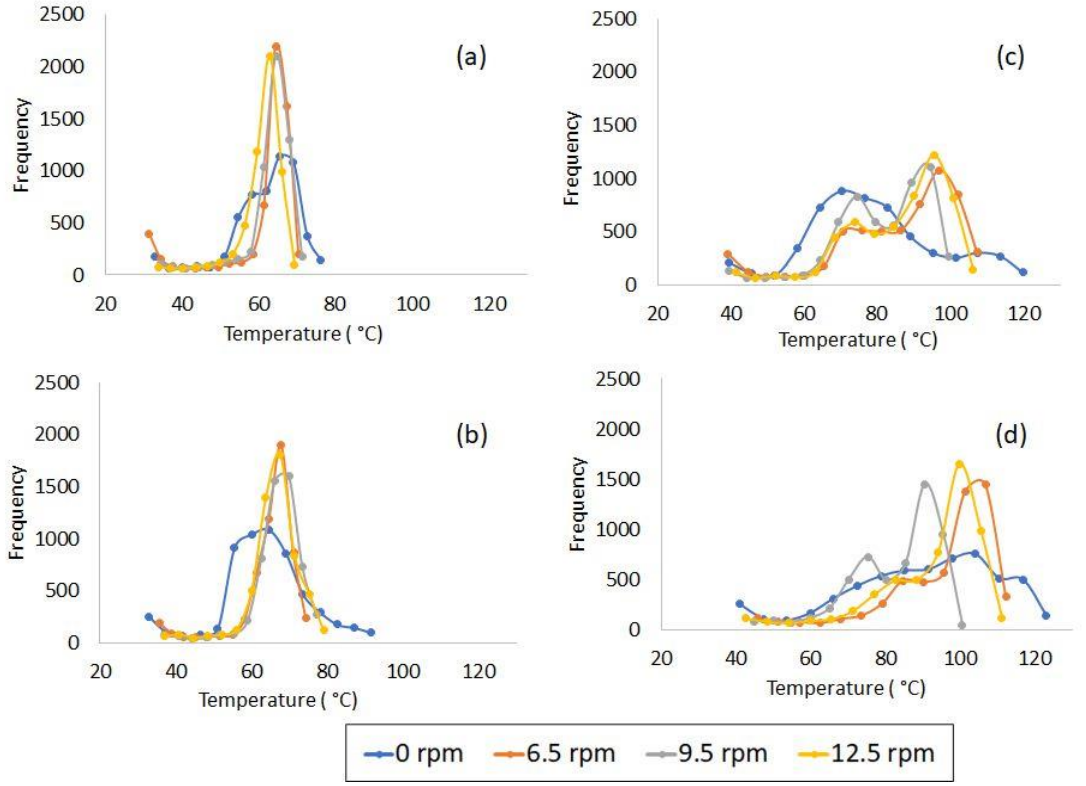

Figure 7. Histograms obtained from thermal images of lemon peels after 2 min a), 5 min b), 10 min c), and 12 min d) of microwave drying at 450 W

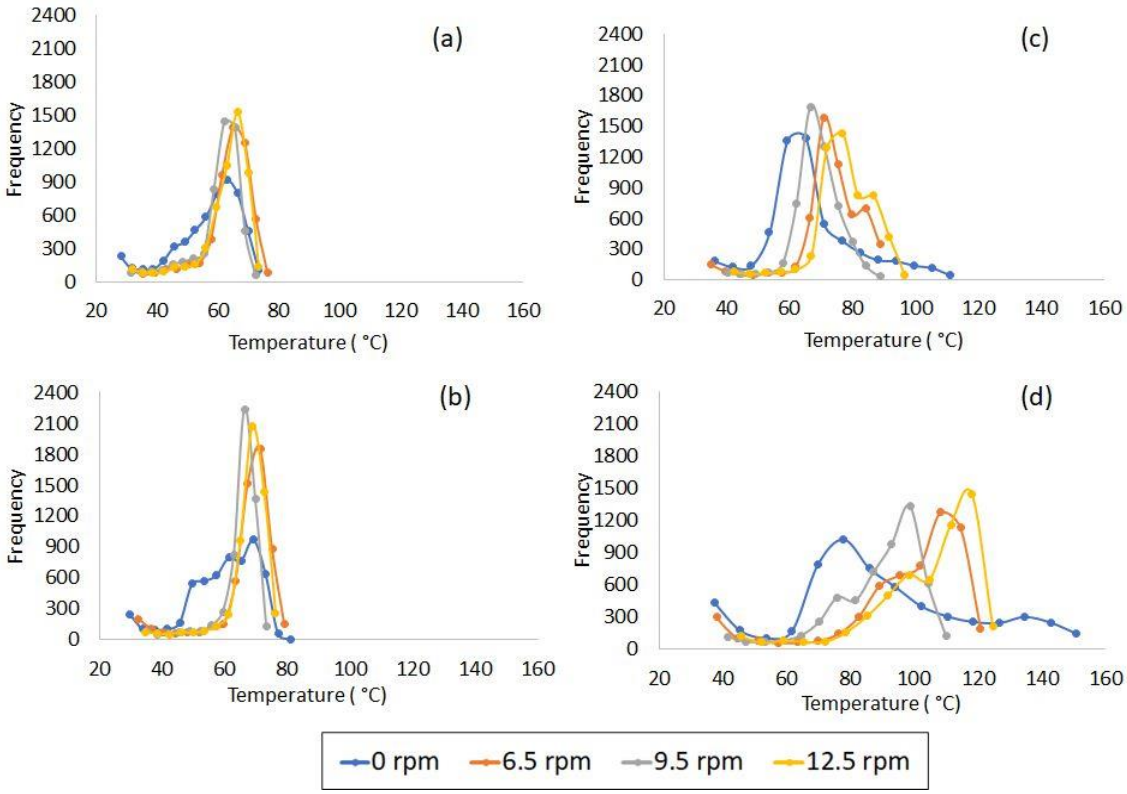

Figure 8. Histograms obtained from thermal images of lemon peels after 1 min a), 2 min b), 5 min c), and 8 min d) of microwave drying at 600 W

3.2 Color and Water Activity

The pictures of microwave dried lemon peel powders were given in Figure 9. Lemon peels were dried until reaching moisture content of 10%. Water activity (a_w) of dried powders were given in Table 1. Water activity of peel powders ranged between 0.528 and 0.545. All a_w values were below 0.6 which is the limit for microbial growth (Tapia et al., 2020).

Color is one of the most important quality attributes of dried foods. The L*, a, and b* values of peel powders varied between 42.03-83.66, 10.65-17.29, and 26.86-49.35, respectively (Table 1). The highest L* and the lowest a* value belonged to the freeze-dried sample. The decrease in L* is generally attributed to the formation of brown pigments. A lower L* value means darker color. During microwave drying, the occurrence of Maillard reactions due to high temperatures may contribute to darkening of sample. Freeze dried lemon peel powder had similar or higher b* values compared to microwave dried ones depending on the microwave drying conditions. During drying, apart from the Maillard reactions, color of lemon peel is influenced by many factors such as degradation of carotenes, ascorbic acid oxidation, the removal of water and its replacement with air, the increase in the dry matter and color substance concentration, and the change of the surface and internal structure of the material (Pathare et al., 2013). Carotenoids are the primary pigments responsible for the color of the peel of most mature citrus fruits. The proportion of each carotenoid in peel is the determining factor on the color of peel. During microwave drying, high temperatures may produce higher degree of deterioration of color pigments which gave lower b* values. While the temperature is high during microwave drying, the drying time was very short compared to freeze drying. This may, at least in part, limit the color degradation during microwave drying.

In general, an increase in power level yielded a reduction in L* and b* values. This may be related with the higher temperatures reached in samples at higher power levels. Lower b* value indicates lower yellowness. The reduction in yellowness was attributed to the higher degree of deterioration of color pigments. In a study searching the effect of drying on total carotenoid content of kinnow peel, Rafiq et al. (2019) detected a decrease in carotenoid content and yellowness of peel after drying and interpreted that reduction in yellowness of peel is associated with the degradation of carotenoids. During microwave drying with rotation, 450 and 600 W provided similar L*, a*, and b* color parameters irrespective of the rate of rotation. Changing the rate of rotation between 6.5 and 12.5 rpm did not cause a marked influence on color parameters of lemon peel powders. Microwave drying without rotation created lower L* and b* values compared to microwave drying with rotation. The effect of rotation function on L* and b* values was notable at high power levels. At 450 and 600 W power levels, microwave drying without rotation caused local temperature rise in sample. At 0 rpm, the maximum surface temperatures detected at power levels of 450 and 600W was about 128 and 154°C, respectively. This resulted in local burns within samples and consequently formation of unacceptable dark powder. Microwave drying at 600 W -0 rpm gave the darkest sample. At 180 W, all lemon peel powder samples had similar color parameters.

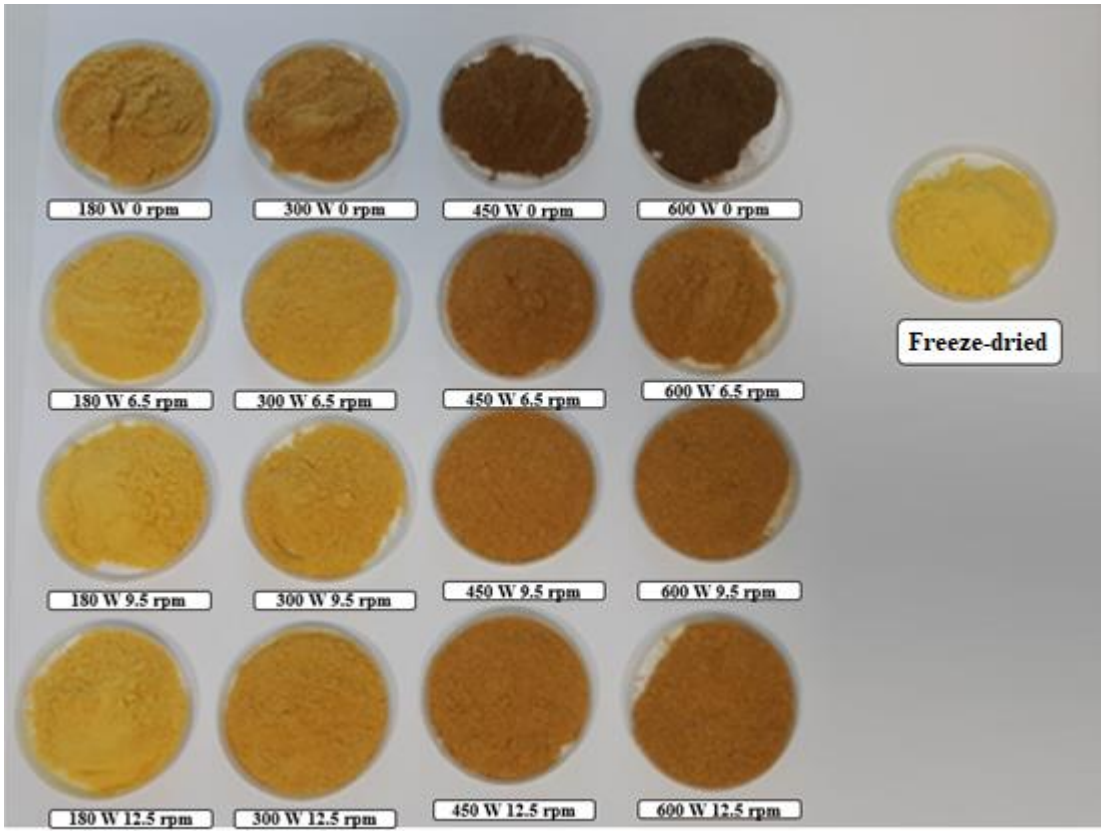

Figure 9. Microwave dried and freeze-dried lemon peel powders

3.3 Total Phenolic Content (TPC)

TPC of fresh peel was 896.5 mg GAE/100g d.b. TPC of dried lemon peels were presented in Table 1. Total phenolic content of dried lemon peel samples ranged between 554 and 1731 mg GAE/100 g d.b. TPC of samples dried by freeze-drying and microwave drying at 180 W were found to be lower when compared to fresh sample. For citrus peels, a decrease in TPC after drying has been reported by several authors (Bejar et al., 2011a; Ghanem et al., 2020; Romdhane et al., 2015; Zhang et al., 2018; Li et al., 2020; Assefa & Keum, 2017). In these studies, the reduction level in TPC of citrus peels ranged between 10 and 70%. In our study, freeze-drying caused 36.8% reduction in TPC and microwave drying at 180 W resulted in a reduction ranging between 21.6 and 33.7%.

The reduction in TPC with drying has been attributed to various factors such as activation of oxidative enzymes, thermal degradation of polyphenols, changes in the chemical structures of polyphenols, binding of polyphenols to proteins and lower extraction yields of phenolics (Deng et al., 2019; Li et al., 2020). Except 180 W, lemon peels subjected to microwave drying had similar or higher TPC compared to fresh peel. Although most of the studies reported a decrease in TPC of citrus peels, in some cases, depending on the drying conditions and the type of citrus, an increase in TPC with drying has been detected (Tekgül & Baysal, 2018, 2019). The release of bound phenolics or some cell wall phenolics due to the high pressure and temperature generated within the tissue during microwave drying may be a possible reason for the increase in TPC (Ghanem et al., 2020; Xu et al. 2017). Phenolics are heat sensitive compounds. However, while some phenolic components are inactivated by heat treatment, some phenolic components are released by the degradation of the cell wall and matrix, resulting in an increase in TPC content. Ghanem et al. (2020) detected that, the TPC of lemon peel subjected to microwave drying between power levels of 300 and 600 W degraded following first-order kinetics during initial period but increased after a certain period. It is well known that freeze-drying is an effective method to obtain high quality dried products. However, in this study, freeze-drying did not

provide the best results in terms of TPC of lemon peel powder. At all conditions, TPC of microwave dried peels were higher than that of freeze-dried one. This may be at least in part due to the increase in free fraction of phenolic acids. Hayat et al. (2010) subjected the mandarin peel powders to microwave treatment and observed an increase in the contents of free phenolic acid, flavanol, flavanone, flavonol compounds but a decrease in bound phenolic acid content. This study's finding agrees with those obtained by Chen et al. (2011) who showed that drying of the orange peel over 70 °C provided higher TPC compared to the freeze-drying and by Papoutsis et al. (2017) who found higher TPC in hot air dried (70-110 °C) lemon peels compared to freeze-dried one. They attributed this to the release of some bound phenolics and the decrease of the polyphenol oxidase enzyme by the heating effect.

Table 1
Water activity (a_w), color parameters, and total phenolic content (TPC) of lemon peels

Drying treatment		**a_w**	**Color**			**TPC (mg GAE/100g d.b.)**
Power	Rate of rotation (rpm)		L*	a*	b*	
Freeze-drying		0.528 ± 0.000 ^{e*}	83.66 ± 1.38 ^{a}	10.65 ± 0.80 ^{f}	46.77 ± 2.91 ^{abcd}	566.30 ± 29.2 ^{j}
180W	0	0.538 ± 0.003 ^{bcd}	76.33 ±0.95 ^{bc}	13.64 ± 1.20 ^{de}	46.52±3.23 ^{abcdefg}	675.01 ± 16.1 ^{ij}
	6.5	0.535 ±0.000 ^{de}	77.42 ± 0.77 ^{b}	13.82 ± 0.91 ^{cde}	48.96 ± 2.10 ^{a}	703.22 ± 10.4 ^{i}
	9.5	0.539±0.003 ^{abcd}	77.70 ± 1.08 ^{b}	13.88±0.74 ^{bcde}	49.35 ± 1.74 ^{a}	594.60 ± 29.8 ^{ij}
	12.5	0.542±0.002 ^{abcd}	77.50 ± 0.87 ^{b}	14.04±1.19 ^{bcde}	49.01 ± 2.29 ^{a}	670.29 ± 8.2 ^{ij}
300W	0	0.535 ±0.002 ^{de}	63.35 ± 3.57 ^{d}	16.35 ± 0.91 ^{ab}	41.70 ± 4.36 ^{bcdefg}	1101.07 ± 11.4 ^{de}
	6.5	0.537 ±0.001 ^{cd}	72.25 ± 2.78 ^{c}	15.57±1.57 ^{abcd}	47.76 ± 3.86 ^{abc}	878.00 ± 17.4 ^{gh}
	9.5	0.538 ±0.003 ^{bcd}	72.68 ± 2.94 ^{c}	15.49±1.64 ^{abcd}	48.12 ± 4.26 ^{ab}	841.80 ± 25.7 ^{h}
	12.5	0.538 ±0.001 ^{bcd}	71.61 ± 2.52 ^{c}	16.11±1.51 ^{abcd}	46.97±3.83 ^{abcdefg}	967.30 ± 41.0 ^{fg}
450W	0	0.540±0.001 ^{abcd}	48.37 ± 3.69 ^{e}	15.39±1.77 ^{abcd}	31.30 ± 2.66 ^{h}	1105.00 ± 54.4 ^{de}
	6.5	0.539±0.001 ^{abcd}	64.02 ± 4.10 ^{d}	16.32 ± 1.05 ^{abc}	40.48 ± 4.51 ^{efg}	1189.80 ± 29.3 ^{cd}
	9.5	0.543 ±0.001 ^{abc}	62.33 ± 2.85 ^{d}	17.17 ± 0.94 ^{a}	41.42 ± 4.01 ^{defg}	1060.30 ± 94.5 ^{ef}
	12.5	0.537 ±0.003 ^{bcd}	61.58 ± 3.70 ^{d}	17.17 ± 1.05 ^{a}	41.78 ± 4.91 ^{bcdefg}	1115.00 ± 60.8 ^{de}
600W	0	0.541±0.003 ^{abcd}	42.03 ± 2.76 ^{f}	12.71 ± 1.43 ^{e}	26.86 ± 4.21 ^{h}	1730.71 ± 12.9 ^{a}
	6.5	0.543 ±0.000 ^{abc}	60.29 ± 3.21 ^{d}	17.05 ± 0.82 ^{a}	41.06 ± 4.85 ^{fg}	1402.90 ± 29.9 ^{b}
	9.5	0.546 ±0.001 ^{a}	62.10 ± 2.86 ^{d}	17.11 ± 0.81 ^{a}	41.11 ± 4.74 ^{g}	1152.10 ± 42.9 ^{de}
	12.5	0.544 ±0.001 ^{ab}	61.35 ± 3.45 ^{d}	17.29 ± 0.64 ^{a}	41.56 ± 4.10 ^{cdefg}	1261.40 ± 24.1 ^{c}

*Means ± standard deviation within a column followed by different letters is significantly different ($p < 0.05$)

TPC of the samples increased with increasing power level. Ghanem et al. (2012) and Bejar et al. (2011b) also observed similar trend during microwave drying of orange peel. This is probably due to the higher temperatures reached in sample during drying at higher power levels (Table 1). The highest TPC (1730.71±12.9 mg GAE/100 g d.b.) belonged to the sample undergoing microwave drying without rotation (0 rpm) at 600 W. It was seen that the maximum surface temperature recorded under this drying condition was quite high (150 °C). Several researchers subjected the citrus peels to elevated drying temperatures and noted similar findings. Papoutsis et al. (2017) detected higher TPC in lemon peels subjected to hot air and vacuum drying treatments at 110 °C compared to the ones dried at 70 °C. In their study, increasing drying temperature provided significant increase in gallic acid content. Chen et al. (2011) dried orange peels in an oven at different temperatures ranging between 50 and 100 °C and obtained the highest flavonoid and phenolic acid contents in the peels dried at 100 °C. For 300 and 600 W, the highest TPC was determined at 0 rpm. The sample dried at 600 W-0rpm conditions was found to have the highest TPC content among all samples. This was attributed to

the respectively higher temperatures reached in this sample under these drying conditions. According to the Tukey's pairwise comparison test, the group average obtained at 0 rpm rotational speed was the highest and that obtained at 9.5 rpm was the lowest. Considering the surface temperatures, it was seen that, at 0 rpm, the temperature rises to relatively higher values due to local overheating and the surface temperature values obtained at 9.5 rpm are lower compared to those obtained at 6.5 and 12.5 rpm.

3.4 Volatile Organic Compounds

The list of volatile compounds in lemon peel and their percentages were given in Table 2. A total of 65 volatile compound was detected in fresh lemon peel. The major volatile compounds in fresh peel were limonene (57.91%), γ-terpinene (6.40%), myrcene (5.59%), p-cymene (3.52%), and α-p-dimethylstyrene (3.45%) which are monoterpene hydrocarbons. Thymol, a monoterpene phenol, was another main compound identified in lemon peel, with percentage of 2.58%. Drying process caused a decrease in percentage amount of these major monoterpene hydrocarbons. The percentage amount of limonene dropped to 34.9 % after freeze drying and to a value ranging between 25.55% and 35.10% after microwave drying. Previous studies showed that, the effect of drying treatment on volatile components varies depending on the method of drying (Farahmandfar et al., 2020) and citrus species (Kamal et al., 2011).

In general, the rate of rotation did not exert a clear effect on the amount of major monoterpene hydrocarbons detected in lemon peel. Depending on the power level and rotation function, microwave drying caused appearance of some derivatives of furan compounds (furfural, furfuryl alcohol, tetrahydrofurfuryl alcohol, 2-acetyl furan, 5-methyl furfural, and hydroxy methyl furfural (HMF)). Of these compounds, furfuryl alcohol is classified as Group 2B (possibly carcinogenic to humans) by International Agency for Research on Cancer (IARC, 2019). In the samples dried at 600W, all these compounds existed. In the samples dried at 180 W power level with rotation, formation of furfural, 2-acetyl furan, and 5-methyl furfural was observed but in a lower level compared to 600 W. Microwave drying at 600 W without rotation (0 rpm) caused also appearance of 3-furfural. Furan and its derivatives were identified in a variety of heat-treated foods through various mechanisms. Akyıldız et al. (2021) detected formation of furfural and HMF in orange juice subjected to thermal treatment (70-90 °C). Lemon peel contains soluble sugars (glucose, sucrose, fructose, raffinose) and ascorbic acid (Aung et al., 1998). Thus, during drying, derivatives of furan compounds in lemon peel may be formed by Maillard reaction or ascorbic acid degradation. In a study, Randhawa et al., (2020) recorded formation of furfuraldehydes (HMF, 2-furfural, 5-methyl furfural) during storage of orange juice at different temperatures (0-40 °C). They noted a significant correlation between vitamin C loss and HMF accumulation in orange juice during storage and stated that the formation of HMF is mainly due to the ascorbic acid degradation. Garcia-Salas et al., (2013) detected formation of HMF and furfural in freeze-dried and vacuum-dried whole lemon powder. In our study, freeze-dried lemon peel powder contained furfural (0.07 %) but not HMF. Total amount of furan derivates identified in peels dried at 0 rpm, 6.5 rpm, 9.5 rpm, and 12.5 rpm was 7.683%, 5.048%, 4.071%, and 4.714%, respectively at 600 W and 0.745 %, 0.402 %, 0.484 %, and 0.737 %, respectively at 180 W. Caramelization, which requires high temperature and sugar, may also lead the formation of furfural and HMF (Agcam, 2022). This might contribute to the comparatively higher level of furfural obtained at 600 W.

Table 2
Percentage composition (%) of volatile compounds in fresh and dried lemon peels

Peak No	RT(min)	Compound Name	CAS #	Fresh	FD	MD at 180W				MD at 600W			
						0 rpm	6.5 rpm	9.5 rpm	12.5 rpm	0 rpm	6.5 rpm	9.5 rpm	12.5 rpm
1	4.977	Capronaldehyde	66-25-1	0.20	0.13	0.02	0.02	0.02	0.02	-	0.01	0.01	0.01
2	5.788	3-Furfural	498-60-2	-	-	-	-	-	-	0.28	-	-	-
3	5.884	Furfural	98-1-1	-	0.07	0.48	0.27	0.35	0.57	4.71	3.53	2.83	3.56
4	6.497	*E*-2-Hexenal	6728-26-3	0.85	1.03	-	-	-	-	-	-	-	-
5	6.630	Furfuryl alcohol	98-0-0	-	-	0.07	-	-	-	0.61	0.54	0.45	0.34
6	7.550	Tetrahydrofurfuryl alcohol	97-99-4	-	-	0.01	-	-	-	0.14	0.08	0.08	0.10
7	7.769	Styrene	100-42-5	0.02	0.04	0.06	0.07	0.08	0.09	0.06	0.06	0.08	0.08
8	8.497	2-acetyl Furan	1192-62-7	-	-	0.08	0.04	0.05	0.07	0.17	0.17	0.15	0.17
9	9.095	α- Thujene	2867-5-2	0.32	0.11	0.08	0.07	0.09	0.08	0.09	0.09	0.08	0.10
10	9.323	α- Pinene	80-56-8	1.23	0.32	0.24	0.22	0.25	0.25	0.30	0.26	0.27	0.29
11	10.173	*E*-Hept-2-enal	18829-55-5	-	-	0.02	0.01	0.01	0.01	0.03	0.09	0.10	0.09
12	10.282	Benzaldehyde	100-52-7	1.04	0.36	0.47	0.54	0.42	0.37	0.21	0.31	0.29	0.32
13	10.431	5-methyl Furfural	620-2-0	-	-	0.10	0.09	0.09	0.09	1.48	0.60	0.47	0.45
14	10.839	Sabinene	3387-41-5	0.20	0.08	0.06	0.05	0.06	0.06	0.08	0.05	0.05	0.07
15	10.933	β- Pinene	127-91-3	0.77	0.24	0.23	0.21	0.24	0.24	0.33	0.22	0.25	0.25
16	**11.563**	**Myrcene**	**123-35-3**	**5.59**	**1.35**	**0.97**	**0.80**	**0.84**	**0.95**	**1.07**	**0.95**	**0.89**	**1.26**
17	12.019	α- Phellandrene	99-83-2	0.25	0.06	0.07	0.06	0.10	0.06	0.05	0.05	0.05	0.05
18	12.507	α- Terpinene	99-86-5	0.40	0.16	0.15	0.14	0.14	0.15	0.14	0.14	0.13	0.15
19	**12.817**	***p*- Cymene**	**99-87-6**	**3.52**	**0.96**	**0.94**	**0.80**	**0.84**	**0.94**	**1.03**	**0.92**	**0.84**	**0.86**
20	**13.039**	**Limonene**	**138-86-3**	**57.91**	**34.90**	**32.72**	**25.55**	**29.88**	**31.53**	**34.78**	**33.55**	**31.10**	**35.10**
21	13.101	Eucalyptol	470-82-6	1.28	0.27	0.38	0.34	0.27	0.27	0.36	0.28	0.28	0.26
22	13.394	*(Z)*-. β- Ocimene	3338-55-4	0.49	0.07	0.12	0.08	0.07	0.08	0.11	0.13	0.09	0.10
23	13.791	*(E)*-. β- Ocimene	3779-61-1	0.56	0.14	0.20	0.18	0.20	0.18	0.16	0.18	0.16	0.16
24	**14.182**	**γ- Terpinene**	**99-85-4**	**6.40**	**4.23**	**5.23**	**4.20**	**4.55**	**5.04**	**4.66**	**5.05**	**4.60**	**4.85**
25	**15.354**	**α-** ***p*- Dimethylstyrene**	**1195-32-0**	**3.45**	**1.84**	**2.76**	**2.43**	**2.41**	**2.63**	**2.02**	**2.60**	**2.29**	**2.06**
26	15.787	Linalool	78-70-6	0.51	0.86	0.91	0.87	0.95	0.85	0.46	0.51	0.56	0.62
27	15.957	Pelargonaldehyde	124-19-6	0.25	0.16	0.21	0.20	0.21	0.20	0.12	0.17	0.17	0.16
28	16.231	Phenethyl alcohol	60-12-8	0.03	0.04	0.05	0.04	0.04	0.05	0.04	0.05	0.04	0.04
29	17.325	*trans*-3-Caren-2-ol	0-0-0	0.11	0.22	0.44	0.35	0.39	0.41	0.42	0.64	0.56	0.48
30	17.844	Isopulegol	89-79-2	0.56	1.07	1.65	1.22	1.51	1.50	1.15	1.37	1.43	1.30
31	18.757	Terpinen-4-ol	562-74-3	0.15	0.15	0.21	0.20	0.22	0.21	0.14	0.20	0.18	0.18
32	19.271	α- Terpineol	98-55-5	0.26	1.12	0.85	0.86	0.96	0.86	0.46	0.53	0.54	0.63
33	19.476	Dihydrocarveol	38049-26-2	0.05	0.12	0.21	0.18	0.21	0.20	0.11	0.18	0.18	0.15
34	19.601	γ- Terpineol	586-81-2	0.34	0.98	1.81	1.47	1.69	1.56	0.86	1.43	1.45	1.27
35	19.825	Capraldehyde	112-31-2	0.16	0.21	0.31	0.29	0.32	0.29	0.23	0.29	0.29	0.26
36	20.192	Linalyl formate	115-99-1	0.09	0.15	0.23	0.24	0.24	0.24	0.31	0.20	0.21	0.15
37	20.279	Nona-2(E).4(E)-dienal	5910-87-2	0.08	0.19	0.20	0.20	0.21	0.20	0.33	0.29	0.26	0.22
38	20.570	Hydroxy methyl furfural	67-47-0	-	-	-	-	-	-	0.30	0.12	0.08	0.08
39	20.676	*L*- Citronellol	7540-51-4	0.24	0.62	0.45	0.40	0.43	0.43	0.53	0.56	0.52	0.64
40	20.921	Methylthymol	1076-56-8	0.51	0.69	0.98	0.88	0.98	0.91	0.68	0.81	0.85	0.72

Table 2 (Continued)

Peak No	RT(min)	Compound Name	CAS #	Fresh	FD	MD at 180W				MD at 600W			
						0 rpm	6.5 rpm	9.5 rpm	12.5 rpm	0 rpm	6.5 rpm	9.5 rpm	12.5 rpm
41	21.133	Neral	106-26-3	0.13	0.29	0.41	0.35	0.41	0.37	0.25	0.34	0.35	0.35
42	21.238	Carvone	99-49-0	0.04	0.12	0.08	0.07	0.08	0.07	0.07	0.08	0.07	0.08
43	21.634	Geraniol	106-24-1	0.04	0.13	0.06	0.04	0.06	0.05	0.05	0.05	0.05	0.06
44	21.873	*(E)*-2- Decenal	3913-81-3	0.01	0.03	0.06	0.05	0.05	0.06	0.05	0.07	0.07	0.06
45	22.215	Geranial	141-27-5	0.32	1.04	0.99	0.93	1.01	0.91	0.62	0.74	0.79	0.80
46	22.342	Perillaldehyde	2111-75-3	0.08	0.64	0.27	0.27	0.28	0.24	0.19	0.20	0.22	0.19
47	**23.014**	**Thymol**	**89-83-8**	**2.58**	**8.58**	**6.61**	**6.85**	**6.80**	**6.33**	**4.79**	**5.44**	**5.10**	**6.05**
48	23.525	Undecanal	112-44-7	0.13	0.35	0.38	0.41	0.42	0.39	0.32	0.36	0.35	0.35
49	23.753	4-vinyl Guaiacol	7786-61-0	-	0.01	0.07	0.04	0.03	0.05	0.59	0.87	0.68	0.56
50	24.658	δ- Elemene	20307-84-0	0.10	0.44	0.47	0.63	0.55	0.56	0.52	0.52	0.61	0.57
51	25.082	α- Cubebene	17699-14-8	0.07	0.18	0.26	0.31	0.29	0.26	0.37	0.25	0.29	0.25
52	25.136	Citronellyl acetate	150-84-5	0.08	0.35	0.37	0.35	0.38	0.37	0.27	0.32	0.35	0.27
53	25.526	Neryl acetate	141-12-8	0.19	0.83	0.98	1.00	1.04	0.94	0.75	0.79	0.85	0.76
54	26.002	α- Copaene	3856-25-5	0.17	0.49	0.49	0.56	0.55	0.51	0.44	0.44	0.52	0.46
55	26.178	Geranyl acetate	105-87-3	0.03	0.13	0.13	0.13	0.14	0.13	0.11	0.12	0.12	0.10
56	26.559	β- Elemene	33380-83-9	1.36	4.78	4.94	6.05	5.37	5.49	6.20	6.05	6.72	6.06
57	27.128	β- Caryophyllene	87-44-5	0.04	0.25	0.37	0.43	0.40	0.38	0.31	0.33	0.34	0.28
58	27.313	α- Cedrene	469-61-4	0.07	0.28	0.32	0.39	0.36	0.33	0.27	0.27	0.32	0.27
59	27.496	β- Cedrene	546-28-1	0.72	2.06	1.97	2.32	2.23	2.12	1.67	1.75	2.06	1.91
60	28.001	α-*trans*- Bergamotene	64727-43-1	1.10	3.78	4.27	5.04	4.78	4.44	3.50	3.75	4.22	3.65
61	28.640	*(E)*- β- Farnesene	18794-84-8	0.73	5.50	5.27	6.73	5.97	5.61	4.57	4.87	5.29	4.47
62	28.812	β-Santalene	511-59-1	0.08	0.23	0.23	0.36	0.26	0.25	0.21	0.17	0.27	0.19
63	28.922	Cadina-1(6).4-diene	20085-11-4	0.06	0.20	0.22	0.35	0.23	0.24	0.29	0.17	0.29	0.18
64	29.531	Germacrene D	105453-16-5	0.68	3.93	4.71	5.81	5.26	4.84	2.61	4.16	4.85	4.57
65	29.700	β- Selinene	17066-67-0	0.38	0.79	0.73	1.13	0.82	0.83	1.04	0.57	0.75	0.51
66	29.989	α- Bulnesene	3691-11-0	0.82	1.41	1.22	2.08	1.45	1.72	2.25	1.04	1.45	0.89
67	30.146	Bicyclogermacrene	24703-35-3	0.16	0.80	0.79	1.13	0.89	0.84	0.71	0.67	0.84	0.67
68	30.358	β-Bisabolene	495-61-4	1.55	7.90	7.85	10.38	8.82	8.33	6.12	6.93	7.66	6.39
69	30.573	γ- Cadinene	39029-41-9	0.07	0.20	0.23	0.32	0.26	0.26	0.33	0.21	0.24	0.21
70	30.717	β- Sesquiphellandrene	20307-83-9	0.04	0.09	0.10	0.15	0.12	0.12	0.12	0.08	0.09	0.08
71	30.845	δ- Cadinene	483-76-1	0.26	0.88	0.85	1.20	0.96	0.95	1.01	0.81	0.90	0.80
72	31.298	Lilial	80-54-6	0.03	0.05	0.05	0.09	0.06	0.08	0.12	0.07	0.08	0.06
73	31.404	Neryl butyrate	999-40-6	0.02	0.15	0.13	0.22	0.16	0.17	0.13	0.14	0.17	0.13
74	31.941	Germacrene B	15423-57-1	0.04	0.20	0.17	0.26	0.21	0.20	0.13	0.16	0.19	0.18

Abbreviations: -, not detected; RT,Retention time; FD, Freeze drying; MD, microwave drying. Major volatile compounds were shown in bold text

4. Conclusion

During microwave drying, application of rotation function or the change in rotational speed of turntable did not create a clear influence on homogeneity of surface temperature distribution, color and TPC of lemon peels at 180W. However, at 450 and 600 W, microwave drying without rotation (0 rpm) caused localized overheating which led to the production of peel powders with unacceptable dark color. The maximum temperatures recorded on the surface of the peels during microwave drying, except at 180W, was slightly lower at 9.5 rpm compared to 6.5 and 12.5 rpm. At these power levels, variation in rotational speed of turntable significantly

affected the TPC of lemon peel powders. Freeze-dried peel and peels dried by microwave drying at 180 W had lower TPC while those dried at 450 and 600W had higher TPC compared to fresh peel. Drying caused loss of main volatile compounds detected in fresh peel and formation of some furan compounds depending on the drying conditions. Formation of furfuryl alcohol which is possibly carcinogenic to humans was detected in peels dried at 600W irrespective of speed of rotation and at a relatively lower level in the peel dried at 180W without rotation but not in other samples. Lemon peel powder dried at 600W contained higher percentage concentration of total furan compounds compared to ones dried at 180W. Results showed that good quality lemon peel powder may be produced by microwave drying with rotation at low power levels.

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Author Contributions

Sevilay San: Collected data and performed the analysis.

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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