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Effect of Microwave Treatment On Physical and Functional Properties of Orange (*Citrus Sinensis*) Peel and Leaves

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Abstract

The effect of microwave drying on drying characteristics of "Maltaise" peel and leaves was investigated. The effect of microwave power on color, total phenols and water and oil holding capacities was determined. By increasing microwave powers (100–850W), drying time decreased from 6960 to 420 s for peel and from 4800 to 210 s for leaves. Page model successfully described the drying kinetics. For microwave powers ranging from 100 to 850W, the values of r, SE and P are ranging respectively from 0.8636 to 0.9806, from 0.2292 to 0.4307 and from 15.0381 to 34.1190. The applied microwave powers affect significantly all color parameters of peel and leaves (P<0.001). Compared to the fresh state, functional properties of peel and leaves decreased after microwave drying except the water holding capacity of peel that increased. For both dried peel and leaves and at each applied microwave power, water holding capacity values were higher than oil holding capacity values. Microwave drying decreased total phenols of the dried leaves compared to the fresh ones. However, drying at 450W improved the extractible phenols amounts from peel (1.880 ± 0.050g caffeic acid/100g db).

Keywords: Orange peel and leaves; "Maltaise" variety; Microwave drying; Drying kinetics; Page model; Functional properties; Color; Total phenols

Introduction

Most of the conventional thermal treatments such as, hot-air drying, vacuum drying and sun-drying are used for food preservation primarily intended to inactivate enzymes, deteriorative microorganisms and reduce water activity. However, high temperatures and long drying periods usually reduce the quality of the final product [1]. It has been reported that many reactions can affect color during processing of fruits and their derivatives. Among them, the most common are pigment degradation, especially carotenoids and chlorophylls, and browning reactions such as Maillard condensation of hexoses and amino components, and oxidation of ascorbic acid [2]. One of the most important changes during drying is the volume reduction suffered by the sample. When water is removed from the material, a pressure unbalance is produced between the center of the material and the external pressure, generating contracting stresses that lead to material shrinkage and changes in shape which can lead to a cracking of the product. In some cases the mechanical equilibrium is reached when shrinkage of the material equals the volume of the removed water [3]. Functional properties have been correlated essentially with the quality of the dietary fiber. Processes, like drying and heating, could modify the physical properties of the fiber matrix and also affect the hydration properties [4]. The potential use of citrus by-products in different technological applications involves some type of processing, i.e., dehydration, which can alter their functional properties as the water holding capacity (WHC) and oil holding capacity (OHC). The end product should exhibit adequate physico-chemical properties after the dehydration process. Citrus by-products have high contents of bioactive compounds such as flavonoids and terpenes which exhibit interesting antioxidant properties and some authors have claimed that certain parts of what is considered as dietary fiber might also exert antioxidant effects [5,6]. Microwave (MW) drying has gained popularity as an alternative drying method for a variety of food products such as fruit, vegetable, snack food and dairy product. MW drying is rapid, more uniform, energy efficient and produces a high-quality end product compared to conventional hot-air drying. Several food products have been successfully dried by the microwave-vacuum application and/or by a combined microwave assisted-convection process [7].

The aims of this work were: to study the MW drying kinetics at different MW powers (100–850W) of "Maltaise" orange peel and leaves and to examine the effect of MW drying on the shrinkage and the WHC and OHC of the fibers and on total phenolic content.

Nomenclature

K, n: Page Model constants; r: Coefficient of Correlation; SE: Standard Error; P: Relative Percent Error (%); t: Time; X_{r} : Reduced moisture content; X_0 : Initial moisture content (kg water/kg db); X_r : Final moisture content (kg water/kg db); R_r : Reduced drying rate; N: Numbers of data points; n_r : Numbers of parameters

Subscripts

Cal: Calculated; exp: Experimental; j: Samples number.

Material and Methods

Material

Fresh oranges (*Citrus sinensis*) and leaves of "Maltaise" variety were picked in Manzel Bouzalfa (Nabeul, Tunisia) in an advanced stage

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of ripeness (Mature-orange). Harvest was achieved in March, 2009. The whole oranges and the leaves were stored 2–4 days at 4°C until processing. The average weight of the oranges was $125.14 \pm 12.22g$. The peel reached $28.0 \pm 3.3\%$ of the total weight of the fresh orange.

Moisture content

Moisture content (X) of "Maltaise" peel and leaves samples was determined before and after microwave treatment by dehydration during 24h in an oven at 105°C [8]. The sample weight was measured by an analytical balance (Metteler AT 400) having a precision of 0.0001g. Moisture content was expressed in dry basis (kg water/kg db). The difference of mass before (m) and after (m_d) drying in the oven gives the moisture content of the product.

$$X = \frac{m - m_d}{m_c} \tag{1}$$

m_d Microwave treatment

Microwave (MW) drying experiments were performed in a domestic microwave oven (TDS: Triple Distribution System, M 1714, Korea) with maximum output of 850W at 2450MHz. Before processing, the orange peels were cut as cubes of which surfaces are equal to 1cm². However, the orange leaves were treated whole. Seven MW output powers (100, 180, 300, 450, 600, 700 and 850W) were investigated in drying experiments. In each of drying experiments, twenty and ten grams respectively of orange peel and leaves samples were placed in the oven. Weight loss was recorded by a digital balance having a precision of 0.001g (GIBERTINI EUROPE). Three replications of each experiment were performed. Moisture loss was recorded by removing the plate from the microwave, and placing this, along with the sample on the digital balance periodically (every 30s) [9]. Time adjustment is done using a digital chronometer. The MW power was applied until the weight of the sample reduced to a level corresponding to moisture content of about 0.10kg water/kg db.

In order to solve the problem of the initial water content variation, the drying kinetics were represented by reduced moisture content (X_r) versus MW drying time (t) and reduced drying rate (R_r) versus reduced moisture content (X_r) .

Where

$$X_r = \frac{X - X_f}{X_0 - W_f} \tag{2}$$

X is the moisture content of the product at each moment of drying, X_0 is the initial moisture content of the product and X_f is the final moisture content corresponding at about 0.1kg water/kg db.

and

$$R_{r} = \frac{\left(-\frac{dX}{dt}\right)}{\left(-\frac{dX}{dt}\right)_{0}}$$
(3)

 $\left(-\frac{dX}{dt}\right)$ is the drying rate at each moment of MW drying and $\left(-\frac{dX}{dt}\right)_0$ is the initial drying rate.

Physical properties

Surface area and thickness: Determination of surface area and thickness of each peel orange cube was achieved by using an electronic digital slide gauge of 10⁻² accuracy. The volume value of each one was consequently determined.

Color measurement: Color values of fresh and dried orange peel and leaves at different MW power were directly read using a Minolta Chroma Meter CR-300, CIE, 1976. The calibration of the apparatus was done before the analysis using a white plate.

The color values were expressed using CIE Lab coordinates, where L* value is a measure of lightness, ranging from 0 (black) to 100 (white); the a* value ranges from -100 (greenness) to +100 (redness) and the b* value ranges from -100 (blueness) to +100 (yellowness). The chroma value (C) and the changes in lightness (Δ L) of each color parameters were calculated respectively as follows:

$$C = \sqrt{\left(a^2 + b^2\right)} \tag{4}$$

$$\Delta L = L - L_0 \tag{5}$$

whereas the total color difference (ΔE) was then determined using the following equation:

$$\Delta E = \sqrt{\left(L - L_0\right)^2 + \left(a - a_0\right)^2 + \left(b - b_0\right)^2} \tag{6}$$

The subscript "0" in both equations refers to the fresh samples for each orange by-product (either peel or leaves).

Functional properties

Functional properties measured included water holding capacity (WHC) and oil holding capacity (OHC). These functional properties were measured for all fresh and dried orange peel and leaves at different microwaves powers. For WHC and OHC measurements, all samples were grinded to a relative large particle size that not affects the functional characteristics [10].

Water holding capacity: WHC was measured after centrifugation of the water insoluble residues. Orange peel and leaves samples (0.1g) were hydrated in excess (24h) in a 50 ml centrifuge tube, prior to centrifugation at 2000g for 25min. Excess supernatant was decanted. Water retention was recorded as g water/g dry matter [11].

Oil holding capacity: Hundred milligrams of each sample were mixed with sunflower oil (10ml), centrifuged at 2000g for 20min and the excess supernatant was decanted. OHC was expressed as g oil/g dry matter [11].

Total phenols

Total phenols content of the fresh and dried orange peel and leaves extracts was measured using the colorimetric Folin-Ciocalteu method [12]. The fresh and dehydrated orange peel and leaves samples were grinded to a very small particle size and then extracted using ethanol solution 95% (1:10) during an hour of continuous agitation. Appropriately diluted samples (0.5ml) were mixed with 0.5ml of Folin-Ciocalteu reagent. The reaction was neutralized with 10ml of a 1M sodium carbonate solution (Na_2CO_3). The absorbance of the resulting blue solution was measured spectrophotometrically at 750 nm and the concentration of total phenols was expressed as mg cafeic acid/100 g db.

Fitting of microwave drying kinetics

The experimental MW drying kinetics data were fitted by using the empirical Page's model (Equation 7) [13], the most used model in literature for describing drying kinetics of food products [7,14,15].

$$X_r = \exp\left(-k \cdot t^n\right) \tag{7}$$

To evaluate the ability of Page model to fit the experimental data,

the mean relative percent error (P), the standard error (SE) and the coefficient of correlation (r) between the experimental and predicted data were determined by using the following equations:

$$P = \frac{100}{N} \sum_{j=1}^{N} \left| \frac{y_{jcal} - y_{jexp}}{y_{jexp}} \right|$$
(8)

$$SE = \sqrt{\sum_{j=1}^{N} \frac{(y_{jcal} - y_{jexp})^2}{N - n_p}}$$
(9)

$$r = \sqrt{\frac{S_t - SCE}{S}}$$
(10)

where

$$S_t = \sum_{j=1}^{N} \left(\overline{y} - y_{jexp} \right)^2 \text{ being } \overline{y} = \frac{\sum_{j=1}^{N} y_j}{N}$$
(11)

and

$$SCE = \sum_{j=1}^{N} (y_{jcal} - y_{jexp})^{2}$$
(12)

where y_{jcal} and y_{jexp} are calculated and experimental values of y, respectively. The first criterion used to evaluate the quality of the fit was the mean relative percent error. It is generally considered that the lowest (P) values indicate the adequate fit for practical purposes [16, 17].

Statistical analysis

All measurements were carried out in triplicate. ANOVA test was performed in order to examine the effect of MW treatment on drying kinetics and quality parameters. The SPSS[®] version 11.0 (Statistical Package for Social Science) was used for statistical investigations. For all statistical analysis, the level of significance is fixed at 95%. Each factor having a P value ≤ 0.05 was considered significant.

Results and Discussion

Microwave drying procedure

Microwave drying kinetics of "Maltaise" peel an leaves: The initial moisture contents (X_0) of fresh orange peel and leaves were calculated: 3.789 ± 0.698 and 1.507 ± 0.008 kg water/kg db, respectively.

The orange peel is constituted of two parts: the flavedo and the albedo. The flavedo constitutes the colored external part. It shows a

very compact cellular structure, containing oil glands and covered with a layer of natural wax. However, the internal zone, the albedo, is the white, spongy and porous part of the peel (gas volume fraction) with large intercellular structure [18]. The effect of the orange peel face exposed (albedo or flavedo) to the MW on the drying kinetics was studied. At any MW power applied (high or low), the orange peel face exposed affect the processing time. In fact, the albedo drying time was revealed shorter than the flavedo drying time. For example, at 700W, the albedo and the flavedo drying times were respectively equal to 690 and 840s. This difference could be attributed to the difference in the structure and the composition of the flavedo and the albedo. In fact, as indicated previously, the peel porosity (gas volume fraction) is located in the albedo zone of the peel, whereas the flavedo zone shows a very compact cellular structure, containing oil glands and covered with a layer of natural wax. It was assumed that the natural waxes of the flavedo prevent the water losses through this slab side. As a consequence, the albedo face was chosen to be exposed to the MW for the next experiments.

The moisture ratios versus MW drying time and the reduced drying rates versus moisture ratio for "Maltaise" peel and leaves are shown in Figure 1. From Figure 1a and Figure 1c, it can be noted that, at the same moisture ratio, the MW drying time decreased with increasing applied MW power. The results indicated that mass transfer within the sample is more rapid during drying at higher MW power because more heat is generated within the sample, creating a larger vapor pressure differential between the center and the surface of product [19]. Similar findings were reported by [14].

At any applied MW power, the total drying times to reach the final moisture content of 0.10 kg water/kg db are higher for orange peel than leaves (Figure 1a and Figure 1c). In fact, peel is thicker and more humid than leaves. At 100, 180, 300, 450, 600, 700 and 850W, orange peel needed respectively 6960, 4320, 1650, 1320, 840, 810 and 420s for reaching 0.10kg water/kg db, whereas, leaves needed shorter processing times of about 4800, 2460, 480, 420, 480, 210 and 210s; respectively.

As can be observed in Figure 1b and Figure 1d, when drying the orange peel and leaves, a constant rate period followed by a falling rate period were observed for all drying conditions. This observation is in agreement with previous reports on MW drying of biological products by [14,20,21].

Sample	MW powers (W)	Parameters		05		P
		k	n) SE	r	۲ ۲
Peel	100	0.0020	0.8874	0.3110	0.9589	24.6487
	180	0.0012	0.9977	0.3263	0.9670	26.0751
	300	0.0014	1.0692	0.2974	0.9683	24.0931
	450	0.0021	1.0503	0.3372	0.9536	28.1830
	600	0.0039	0.9907	0.2555	0.9478	20.6859
	700	0.0040	1.0134	0.3158	0.9620	25.6748
	850	0.0019	1.2605	0.4697	0.9229	35.7474
Leaves	100	0.1327	0.3606	0.3772	0.9023	31.2228
	180	0.0921	0.4405	0.3657	0.9042	29.6929
	300	0.3572	0.2726	0.4307	0.8636	34.1190
	450	0.2563	0.4278	0.3555	0.9406	24.0734
	600	0.3973	0.3381	0.2292	0.9688	15.8554
	700	0.2285	0.4946	0.2322	0.9806	15.0381
	850	0.6732	0.2880	0.3081	0.9633	20.3573

Fitting data: Fitting the MW drying behavior is important for

Table 1: Estimated parameters and fitting criteria of the Page model applied to experimental MW drying kinetics data of "Maltaise" orange peel and leaves.

Page 3 of 7

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investigation of drying characteristics of *Citrus sinensis* peel and leaves. In this study, the MW experimental drying data of the orange peel and leaves at different MW power levels were fitted to the empirical Page's model (Equation 7). The mean relative percent error (P), the standard error (SE) and the coefficient of correlation (r) for the studied MW powers were calculated. Results are tabulated in Table 1. As can be seen, for MW powers ranging from 100 to 850W, the values of r, SE and P are ranging respectively from 0.8636 to 0.9806, from 0.2292 to 0.4307 and from 15.0381 to 34.1190. In Figure 1a and Figure 1c, the adjustment of experimental data with the Page model is also shown. A good agreement between experimental and predicted data was found. Page model was successfully applied to several agricultural products like apple [15], spinach [21] and parsley [14].

Effect of microwave treatment on physical properties

Surface area and thickness: Figures 2a, b and c illustrate the effect of MW treatment at different powers (100–850W) on surface area, thickness and volume of "Maltaise" peel samples. MW drying power has a significant effect on surface area (0.01<P<0.05) and no significant effect on thickness (P>0.05) and volume (P>0.05) of peel samples. Orange peel samples undergoing MW drying did not shrink isometrically when the moisture content decreased up to 0.10kg water/kg db. Shrinkage of the surface area was most important at low MW power (100W) and less important at high MW power (600W), whereas shrinkage of the thickness was most considerable at high MW power (100W) and less considerable at low MW power (100W). As a consequence, shrinkage of the volume was lowest at low MW power (180W) and highest at high MW power (700W) and it was mainly due to the shrinkage of the thickness (Figure 2b). Shrinkage of the

MW dried orange peel samples was essentially due to the quantity of evaporated moisture.

Color: The effect of MW treatment at different powers (100–850W) on color parameters of the orange peel and leaves (L*, a*, b*, C and ΔE) was shown in Figure 3a and Figure 3b. MW powers affect significantly all color parameters of both peel and leaves (P<0.001). ΔE decreased significantly and reached a lowest value at 600W for both peel and leaves, when, its highest value was obtained at 700W for peel (after 810s) an at 850W for leaves (after 210s). 600W was then found to be the adequate MW power for preserving the global color of both peel and leaves.

For the orange peel, MW drying has not a significant effect on L^{*} (P>0.05) and b^{*} (P>0.05), on the other hand, it has a significant effect on a^{*} (P=0.05) and C (0.01<P<0.05). In fact, after MW drying, L^{*}, a^{*} and C reached their lowest values at 700W. This can be explained by the degradation of carotenoids and flavonoids, pigments responsible of the orange peel color, and also by Maillard and the non-enzymatic browning reactions occurred essentially during MW drying at higher MW powers. On the contrary, 600W gave the highest values of L^{*}, a^{*}, b^{*} and C. In this way, this MW power seems to be very interesting for preservation of the orange peel color.

For the leaves, MW drying has a highly significant effect on L*, a* and C (P<0.001). However, MW drying effect on b* value is significant (0.01<P<0.05). From Figure 3b, it can be noted that, after MW drying at 850W, L*, b* and C reached their highest values, whereas, a* reached its lowest value. In other words, greenness, yellowness and lightness of the orange leaves increased at this high MW power. Finally, MW drying improved the color of the leaves, thereafter; pigments responsible of the leaves color (chlorophylls) were preserved.



Figure 1: Drying curves corresponding to "Maltaise" orange peel (a and b) and leaves (c and d) at different applied MW powers (X,: reduced moisture content; R; reduced drying rate; Dots: experimental data; Line: predicted data; X₀ of peel: 3.789 ± 0.698 kg water/kg db; X₀ of leaves: 1.507 ± 0.008 kg water/kg db).

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In conclusion, when high powers were used, drying was performed in a shorter time so that color was much more preserved. [21] found similar results with MW dried carrots. Several authors also studied the effect of MW drying on color of many biological products such as kiwifruits and parsley [23,14].

Effect of microwave treatment on functional properties

The functional properties, as WHC and OHC, of the fresh and MW dried orange peel and leaves samples at different MW powers (100 -850W) were measured. The obtained results for the fresh and dried samples are shown in Figure 4a and Figure 4b. As can be observed, fresh orange leaves possess higher WHC and OHC compared to the fresh peel (respectively 7.862 \pm 0.679 and 6.118 \pm 0.215 g/g db for leaves against 4.687 \pm 0.642 and 4.993 \pm 0.203 g/g db for peel). This can be due to the lower initial moisture content of leaves in comparison to the peel permitting the adsorption of more water and oil. Besides, we found that the total dietary fibers of the leaves was higher than that of the peel (56.991 \pm 0.435 against 42.129 \pm 0.147 g/100g db; respectively). Difference in the dietary fibers content promoted difference in the functional properties of these by-products. However, after MW drying, WHC and OHC of dried peel became higher than those of dried leaves. It can be also noticed that, compared to the fresh state, all functional properties of peel and leaves decreased after MW drying except the











WHC of peel that increased. According to these results, it seems that dried orange peel exhibits a higher capacity to absorb water than other dehydrated fruits, being this property one of the most desirable characteristics for the functionality of dietary fiber [6]. MW drying has a highly significant effect on WHC and OHC of peel (P<0.001), whereas it has a significant effect on OHC (0.01<P<0.05) and no significant effect on WHC (P>0.05) of leaves. When observing the effect of MW power on functional properties of orange peel and leaves, it can be noted that, applied MW power affected significantly both WHC and OHC (P<0.001). The highest WHC and OHC values of peel were obtained

Page 5 of 7

respectively at 180 and 700W (10.252 \pm 0.651 and 3.795 \pm 0.208 g/g db, respectively). However, for the leaves, the highest WHC and OHC values were obtained respectively at 850 and 180W (6.857 \pm 0.406 and 2.703 ± 0.245 g/g db, respectively). According to [24], water could be held in capillary structures of the fiber as a result of surface tension strength, and also water could interact with molecular components of fiber through hydrogen bonding or dipole form. However, OHC depends on surface properties, overall charge density, thickness and hydrophobic nature of the fiber particle. WHC obtained for the MW dried peel and leaves were higher than that presented by [6] for dried lemon by-products obtained after juice extraction (~ 6 g water/g db). Also, for both dried peel and leaves and at each MW power applied, the WHC values were higher than OHC values. Similar trends were found by [25]. MW drying affects the fibrous matrix modifying the structural characteristics and the chemical composition of the fiber (water affinity of its components) and promoting water retention to the detriment of oil retention. The high WHC of these MW dried by-products suggest that they can be used as functional ingredients to reduce syneresis, modify texture and viscosity and reduce calories of foods.

Effect of microwave treatment on total phenols

Figure 5 illustrates the effect of MW treatment at different MW powers (100 - 850W) on the total phenolic content of the orange peel and leaves. The fresh orange peel shows higher phenolic content than the fresh orange leaves $(1.125 \pm 0.041g \text{ caffeic acid}/100g \text{ db against})$ 0.441 ± 0.013 g caffeic acid/100g db). Similar result was found by [26] for the phenolic content of the fresh leaves (0.322g gallic acid/100g db). Whereas, the phenolic content obtained for the fresh peel was higher than that found for the fresh Citrus aurantium peels (0.522g gallic acid/100g db). MW power affected significantly the total phenolic content of both orange peel and leaves (P<0.001). The highest value obtained was 1.880 ± 0.050 g caffeic acid/100g db for the peel and 0.430 \pm 0.004g caffeic acid/100g db for the leaves at 450W. As can be noticed, 450W improved the extraction of phenol compounds from peel. At this MW power, structure of the fiber matrix could become larger and looser and thereafter facilitate the extraction with solvent. The lowest values were obtained at 180W for both peel and leaves (1.064 \pm 0.016 and 0.308 \pm 0.003g caffeic acid/100g db; respectively). 180W was revealed unfavorable for extraction of phenol compounds because of the longer drying times that could destroy some of them [27]. In any case, total phenolic content of MW dried peel was found higher than that of MW dried leaves. This fact can be due to the higher initial phenolic content of orange peel than leaves.



Figure 5: Effect of MW treatment at different powers on the total phenolic content of "Maltaise" orange peel and leaves; FM: fresh matter.

In comparison to the fresh state, the statistical study of the effect of MW drying on the total phenolic content of the orange peel and leaves showed that it has no significant effect for peel (P>0.05), but it has a significant effect for leaves (0.01<P<0.05). Effectively, MW drying decreased the total phenolic content of the dried leaves compared to the fresh one. At any MW power, dried orange peel presented higher phenolic content than that found by [28] in the air dried orange peel (0.25 gallic acid/100g db). [29] developed an optimized microwaveassisted extraction (MAE) method of phenolic acids from citrus mandarin peels. Compared with the traditional methods, MAE showed many advantages, such as shorter time, less solvent, higher extraction rate, savings of energy and better products with lower cost.

Page 6 of 7

In conclusion, MW drying at 450W seems to be very interesting to improve and to preserve the highest total phenolic content respectively for orange peel and leaves. Consequently, antioxidant activity of these phenolic compounds could be improved and preserved.

Conclusion

In this work, the MW drying kinetics of "Maltaise" orange peel and leaves were studied. Results showed that MW drying of both peel and leaves took place in two periods: the constant rate period followed by the falling rate period. The total drying times to reach the final moisture content of 0.1kg water/kg db were higher for "Maltaise" peel than leaves. The empirical Page's equation was used to describe the MW drying kinetics of orange peel and leaves. A good agreement between experimental and predicted data was found for both peel and leaves.

The effect of different MW power levels on physical, functional properties and on total phenolic content was also investigated. Applied MW powers affect significantly all color parameters (L*, a*, b*, C and ΔE) of both peel and leaves (P<0.001). When high powers were used, drying was performed in a shorter time so that color was much more preserved. MW drying has also a highly significant effect on WHC and OHC of peel (P<0.001), whereas it has a significant effect on OHC (0.01<P<0.05) and no significant effect on WHC (P>0.05) of leaves. For both dried peel and leaves and at each applied MW power, the WHC values were higher than OHC values. Total phenolic content of fresh and MW dried peel was found higher than that of fresh and MW dried leaves. MW drying decreased the total phenolic content of the dried leaves compared to the fresh ones. However, drying at 450W increases the content of extractible phenols from peel.

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Page 7 of 7