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MICROWAVE DRYING of BLACK OLIVE SLICES: EFFECTS on TOTAL PHENOLIC CONTENTS and COLOUR

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Abstract

In this study, the drying behaviour of black olive slices using microwave was investigated and the effects of microwave power on total phenolic content and colour were determined. Black olive slices were dried at different power levels (180, 360, and 540 W) using a microwave cabin. The results showed that the drying time of the olive slices decreased considerably as the microwave power increased. The total phenolic content of the product dried at 540 W was found to be significantly lower than those dried at 180 or 360 W. For the microwave power range studied, the use of an irreversible first-order model adequately described the decrease in the total phenolic content. The effects of microwave power level on changes of the surface temperatures and weight loss ratios of the olive slices were also determined. The temperature increase and the weight loss were the highest at 540 W during drying. Changes in the colour values (L*, a*, and b*) of the black olive slices during drying were statistically different (*P<0.05*) for each microwave power level and the maximum colour change in brightness occurred drying at 360 W. It is thought that results obtained in this study will provide valuable data for the design of microwave drying systems used for drying of olive slices in pilot or industrial scales.

Keywords: Olive, microwave, kinetic, modeling, total phenolic content, colour.

SİYAH ZEYTİN DİLİMLERİNİN MİKRODALGA ile KURUTULMASI; TOPLAM FENOLİK MADDE ve RENK ÜZERİNE ETKİLERİ

Özet

Bu çalışmada, mikrodalga kullanılarak siyah zeytin dilimlerinin kuruma davranışları incelenmiş ve mikrodalga gücünün toplam fenolik madde ve renk üzerine etkileri belirlenmiştir. Siyah zeytin dilimleri üç farklı güç seviyesinde (180, 360 ve 540 W) mikrodalga kabin kullanılarak kurutulmuştur. Sonuçlar zeytin dilimlerinin kuruma süresinin mikrodalga gücü arttıkça önemli ölçüde azaldığını göstermiştir. 540 W gücünde kurutulan ürünün toplam fenolik madde içeriğinin, 180 ve 360 W güçlerinde kurutulanlarınkinden önemli ölçüde daha düşük olduğu belirlenmiştir. Çalışılan mikrodalga güç aralığı için, tersinmez birinci dereceden kinetik modelin kullanılması toplam fenolik madde içeriğindeki azalmayı başarıyla tanımlamıştır. Mikrodalga güç seviyesinin yüzey sıcaklığı ve ağırlık kaybı değişimi üzerine etkileri de incelenmiştir. 540 W gücünde kurutma sırasında sıcaklık artışı ve ağırlık kaybı en fazla olmuştur. Kurutma sırasında her mikrodalga güç seviyesi için siyah zeytin dilimlerinin renk değerlerindeki (L*, a*, b*) değişim istatistiksel olarak anlamlı bulunmuştur (*P<0.05*) ve en fazla renk parlaklık değişimi 360 W gücünde kurutmada gerçekleşmiştir. Bu çalışmada elde edilen sonuçların zeytin dilimlerinin pilot veya endüstriyel ölçekte kurutulması amacıyla kullanılacak mikrodalga kurutma sistemlerinin tasarımları için değerli veri sağlayacağı düşünülmektedir.

Anahtar kelimeler: Zeytin, mikrodalga, kinetik, modelleme, toplam fenolik madde, renk.

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INTRODUCTION

The olive tree (*Olea europaea L.*) is one of the most important fruit trees in the Mediterranean countries. Its products, olive oil and table olives, are important components of the Mediterranean diet and are consumed throughout the world (1, 2). The low incidence of cardiovascular diseases and cancer in the Mediterranean countries with a diet consisting of olive and olive products, which is thought to be the reason, has drawn the attention of investigators, and the Mediterranean diet consisting of olive and olive products has been (3).

The Kalamata olive is a large black olive with a smooth meaty texture named after the city of Kalamata in Greece. It is often used as a table olive and preserved in wine vinegar or olive oil (4). Phenolic compounds are of great importance for the olive fruit, being responsible for important characteristics and properties such as color, taste, and texture (5).

Fresh or dried olives are used in Mediterranean dishes including pizza, salads, and sauces and are also used as a snack or appetizer (6). In many countries, large quantities of food products are dried to improve shelf-life, enhance appearance, encapsulate the original flavour, and maintain nutritional value. In this respect, the development of new alternative products and tastes for table olives is under way (7).

Drying is a simultaneous heat and mass transfer in which the water activity of the material is lowered by removal of the water component to a specific level, at which microbial spoilage and deterioration chemical reactions are greatly minimized (8). It brings about substantial reduction in weight and volume, minimizes packing, storage and transportation costs, and enables storability of the product under ambient temperatures (9).

Different drying methods are used for the drying of fruits and vegetables. Hot-air drying is the most common method for drying of foodstuffs. However, this method leads to serious deteriorations in taste, colour, and nutritional content of the product, decreases the density and water absorbance capacity and shifting of the solutes from the internal part of the drying material to the surface, due to the long drying period and high temperature (10).

Microwave (MW) heating provides a possibility of shortening the drying time because of the volumetric dissipation of microwave energy and promoting heat and mass transport (11). Microwave energy is rapidly absorbed by water molecules which, consequently, results in rapid evaporation of water and thus higher drying rates. Microwave drying supplies uniform energy and high thermal conductivity towards the inner surfaces of materials and prevents any thermal degradation, therefore provides high quality of the finished products (12). Microwave drying offers significant energy savings, with a potential reduction in drying times of up to 50% in addition to the inhibition of surface temperature of the treated material (13, 14). MW drying can significantly shorten the drying process by virtue of the following unique advantages: [1] adjustment of energy absorption level by the wet products automatically-moisture-leveling effect of microwaves; [2] possible selective heating of the interior portions-microwave focusing effect; [3] rapid energy dissipation throughout the material; [4] relatively minor migration of water-soluble constituents; [5] lower product temperatures in combination with vacuum; and [6] more efficient drying in the falling rate period (15). In recent years, microwave drying is applied as an alternative method for drying a variety of foods (10,13-25).

Although applications of microwave drying have been evaluated in the food industry, there is limited information reported in literature about colour and total phenolic content changes during microwave drying of black olive slices. The main objectives of this work was to investigate the microwave drying characteristics of black olive slices by determining the colour characteristics during drying and characterizing the changes on the total phenolic contents and the weight loss by kinetic modeling.

MATERIAL and METHODS

Material

The olive fruits (*Olea europaea* L. var. Kalamata) were supplied from the Olive Research Institute in Izmir, Turkey. Olives were stored in a refrigerator at +4 °C before processing. After the seeds of olives were removed, they were sliced into thickness of 0.5 cm. The hallow olive slices having the average inside diameter of 0.9 cm and the average outside diameter of 1.6 cm were used. The total dry matter content, colour characteristics, and total phenolic content of the olives were determined before microwave drying.

Microwave Drying Equipment

MW drying experiments were carried out using a microwave oven (MW 595, Arçelik, Turkey) operated at 2450 MHz (a wavelength of 12.24 cm). The microwave cabin was the standard microwave unit that has the capability of operating at five different microwave power levels 180, 360, 540, 720, and 900 W. Samples were placed on a teflon drying tray with a diameter of 20 cm. The olive slices were then dried at three different microwave power settings (180, 360, and 540 W). The surface temperature of the olive slices were measured using an infrared thermometer (Testo, 830-T1, Germany) for 1 minute intervals during microwave drying.

Weight Loss

The olive slices were removed from the microwave oven periodically (every 1 min) during the drying process, and the weight loss was determined by weighting the olives using a digital balance (Ohaus, N315, China). All weighting processes were completed in 10 s during the drying process (26). Weight loss (%) was calculated according to Eq. (1):

Weight loss (%) = [(Initial weight after drying) ÷ Initial weight] x 100 (1)

Total Dry Matter Content

The total dry matter content of the olive slices was determined according to Anon (1990) in a vacuum oven (Nüve, EV 018, Turkey) at a constant temperature of 65 °C and a constant absolute pressure of 517.17 mmHg (27).

Determination of Colour Properties

The colour of the dried olive slices was measured by using the Hunter-Lab Colorflex (CFLX 45-2 Model Colorimeter; HunterLab, Reston, VA) based on three colour coordinates (L*, a* and b*). The cylindrical quartz cell containing the sample was placed directly into the colorimeter, and post-processing L*, a* and b* values were recorded. The determination of the colour properties of the olive slices was carried out in triplicate.

Determination of Total Phenolic Content

The total phenolic content of the dried olives was determined spectrophotometrically by the method of Sinhleton and Rossi (1965) (28). The results were expressed as gallic acid equivalents. The gallic acid calibration curve obtained by gallic acid standards at five different concentrations (0 to 2 mg/L) was used to determine the total phenolic content of the samples. Two grams of dried olive slices was dissolved in 50 mL of methanol 80%, extracted and filtered. Then 50 µl of filtrate was allowed to react with 250 µl of Folin-Ciocalteu reagent for 5 min in a volumetric flask. At the end of this period, 750 µl of saturated Na_2CO_3 solution was added and the volume was completed with 3.95 ml distilled water. After the mixture had been allowed to stand in the dark for 2 h at room temperature, the absorbance of the samples was measured at 760 nm using a spectrophotometer (Varian Cary 50 Scan, Victoria, Australia). The determination of total phenolic content in raw and dried products was carried out in triplicate.

Kinetic Modeling

A kinetic model was fitted to characterize the decrease of the total phenolic content of the black olive slices during microwave drying. The decrease in total phenolic content is assumed to be an irreversible first-order reaction.

The kinetic equation is described by İcier et al. (2006) (29) :

$$C = C_0 . e^{-kt} \quad (2)$$

which was the ratio of the instant (C) and initial (C_0; at zero holding time) values of total phenolic content during the microwave drying time (t) (29).

The coefficient k (min^{-1}) represented the first-order rate constant. Model adequacy was determined by non linear regression coefficient (R^2), correlation coefficient (r), and root mean square error (RMSE) (30).

The regression coefficient is one of the primary criteria to select the fit quality of models,

$$RMSE = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(V_n - V_e)^2} \quad (3)$$

$$r = \frac{N\sum_{i=1}^{N} V_n V_e - \sum_{i=1}^{N} V_n \sum_{i=1}^{N} V_e}{\sqrt{\left(N\sum_{i=1}^{N} V_n^2 - \left(\sum_{i=1}^{N} V_n\right)^2\right)\left(N\sum_{i=1}^{N} V_e^2 - \left(\sum_{i=1}^{N} V_e\right)^2\right)}} \quad (4)$$

ranging from 0 to 1. In addition to this, the root mean square error (RMSE) was also used to determine the quality of the fit. The higher values for R^2 and the lower values for RMSE indicate a better quality of fit (30, 31).

Determination of Drying Rates

The rate of microwave drying curves for constant-drying conditions was determined using Equation 5.

$$R = -\frac{L_S}{A}\frac{\Delta X}{\Delta t} \quad (5)$$

Where R is the drying rate in kg $H_2O/min.m^2$, Ls kg of dry solid used, A exposed surface area for drying in m^2, X in kg free water/kg dry solid, and t is drying time in minute (32).

Statistical Methods

The mean values and standard deviations were calculated using Excel (Microsoft Corp., USA). The effects of the power level on the quality characteristics investigated were statistically evaluated with analysis of variance (one-way ANOVA) and the Duncan test at a level of significance $P<0.05$ by using software SPSS 18 (SPSS Inc., Chicago, IL, USA). The linear or non-linear regression procedures were applied to determine the relations describing the changes on the rate constant of weight loss/total phenolic content or microwave drying rate as a function of microwave power level. Regression coefficient (R^2) were given to asses the adequacy of the relations obtained. Each experiment was performed triplicate at least.

RESULTS and DISCUSSION

Determination of the Weight Loss and the Increase in Surface Temperature of the Olive Slices

Changes in the weight loss and surface temperatures of the 0.5 cm thick olive slices for each power setting (180W, 360W, and 540W) of the microwave drying process is given in Figure 1. The weight loss of the olive slices were given as the weight loss of dried olive slices for an instant drying time to their initial weights.

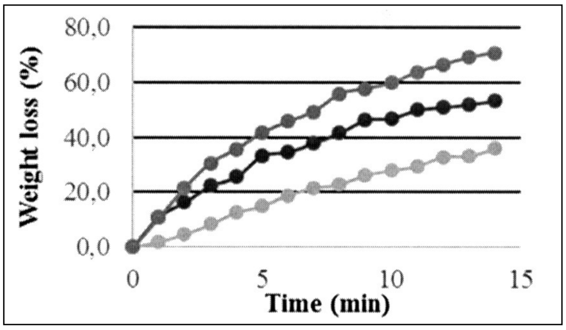

Figure 1. The changes in weight loss of black olive slices during microwave drying at different power levels

—•— 180 W —•— 360 W —•— 540 W

The weight loss of black olive slices increased decreased as the microwave power level increased. At the end of the microwave drying to nearly a surface temperature of 75 °C, weight loss at the 180 W microwave power level was lower than at 360 or 540 W. In addition, the surface temperatures of the olive slices increased as the microwave power level increased.

The weight loss change of black olive slices was explained by the first order kinetic model (Table 1). The regression coefficient was considered as the basis to select the appropriate combinations, which well described the first order reaction. On the other hand, the RMSE values were low for each power level.

The kinetic equations describing the changes in weight loss of the olives were found as follows in Table 1. The first-order rate constant (k_w) proved the increase in the weight loss of the olive slices during microwave drying, k_w increased linearly as the microwave power increased (Figure 2) and as the microwave power level was increased, the rate constant of the sample was apparently increased. The rate constant of weight change loss (R_w) during the drying (between 0 to 14 min) as a function of the microwave power level (P) were defined by the linear relation (R^2=0.9997) given in Equation 6.

$$R_w = 0.0012(P) + 0.0017 \qquad (6)$$

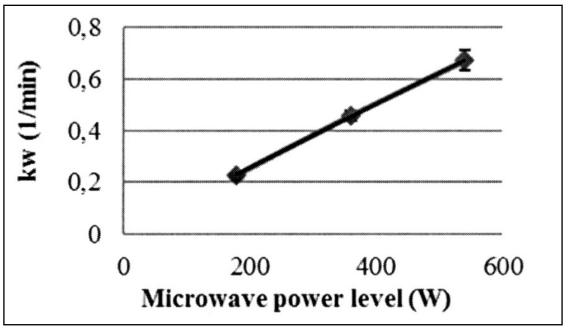

Figure 2. Kinetic constants for weight loss at different microwave power level

Microwave Drying Curves of Olive Slices

The time required to heat the olive slices to nearly 75 °C surface temperature at 180, 360, and 540 W microwave power levels are given in Figure 3. It was observed that the drying times decreased as the microwave power level increased. After 180, 360, and 540 W microwave drying treatments, the

Table 1. Kinetic equations and statistical results of the weight loss for microwave drying of olive slices

	Weight loss (WL)			
Microwave power (W)	Equation	R^2	r	RMSE
180	$WL = 1.0022e^{-0.033t}$	0.993	0.997	0.009
360	$WL = 0.9223e^{-0.054t}$	0.971	0.986	0.027
540	$WL = 0.9307e^{-0.086t}$	0.991	0.994	0.024

surface temperatures of olives slices reached nearly 75 °C in 11 min, 8 min, and 5 min, respectively. By working at 540 W instead of 180 W, the drying time was shortened by 54.5%. Similar effects of microwave power levels on surface temperature of parsley (33), carrot (34), and olive paste (35) have been previously reported.

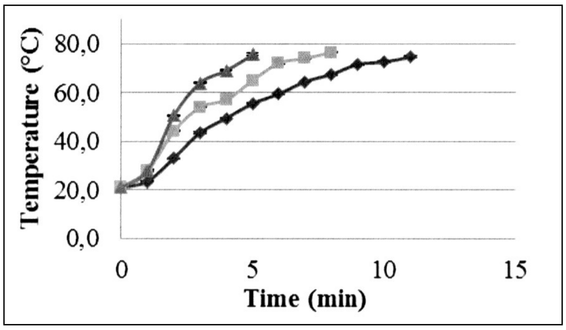

Figure 3. Temperature increase during microwave drying of black olive slices at different power levels

180 W — 360 W — 540 W

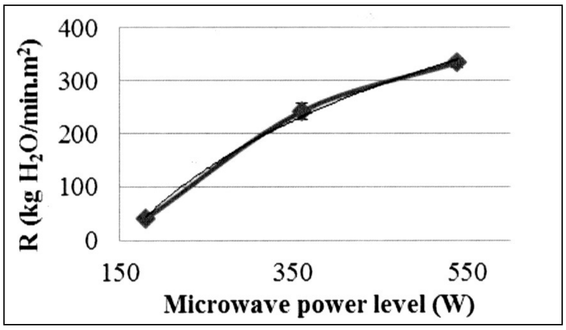

Figure 4. Rates of microwave drying for different power levels

Figure 4 shows the rates of microwave drying as a function of microwave power level. As seen in this figure, the drying rate increases with the increase in microwave power level. The rate of microwave drying (R) as a function of microwave power level (P) were defined (R^2=0.996) by Equation 7.

$$R = 269.72\ln(P) - 1356.2 \quad (7)$$

The predicted values of drying rate, which were calculated based on the average of 1-4 min, were found as 44.44, 231.40, and 340.76 kg $H_2O/min.m^2$ for 180, 360, and 540 W respectively.

Effects of Microwave Drying on Quality Characteristics Colour Parameters

The colour change of the raw material and microwave dried black olive slices are given in Table 2. As shown in Table 2, the value of L^*/L^*_{raw} decreased during microwave drying.

In this study, the statistical analysis (ANOVA and Duncan tests) showed that there were significant differences between the colour values (*P<0.05*) for each microwave power level. The colour parameters were given as the values calculated by dividing the colour parameters to the responding colour values of the raw material in order to reduce the margin of error. For the microwave power level studied, L* values of the dried olive slices decreased as the drying time increased. It was determined that significant changes of a* values were obtained in the olive slices dried at 360 and 540 W. Similarly, Maskan (2001) found that parameters L* and b* decreased and a* value increased during drying of kiwifruits (16). Moreover, the reduction in brightness value L* of the dried spinach leaves (21) and dried onion slices (36) was obtained during microwave drying. Akbudak and Akbudak (2013) reported that significant reduction in color change compared to fresh samples was obtained by microwave drying and the change in product color was highly dependent on its temperature (37). In addition Fang et al. (2010) and Çelen and Kahveci (2012) reported that the brightness decreased and the change in the colour increased significantly as the microwave power increased (38, 39). They

Table 2. The changes in colour of black olive slices during microwave drying at different powers

Time (min)	180 W			360 W			540 W		
	L*/L*rm	a*/a*rm	b*/b*rm	L*/L*rm	a*/a*rm	b*/b*rm	L*/L*rm	a*/a*rm	b*/b*rm
1	1.03± 0.02^{bc,Y}	0.71± 0.02^{d,Y}	0.47± 0.02^{c,Z}	1.22± 0.04^{a,X}	1.26± 0.07^{a,X}	1.27± 0.10^{a,X}	1.02± 0.01^{a,Y}	1.31± 0.03^{a,X}	0.84± 0.05^{c,Y}
2	1.00± 0.02^{c,X}	0.82± 0.01^{c,Z}	0.45± 0.02^{c,Z}	0.99± 0.01^{b,XY}	0.88± 0.04^{b,Y}	0.67± 0.01^{c,Y}	0.98± 0.01^{b,Y}	1.25± 0.04^{b,X}	1.20± 0.02^{b,X}
3	1.09± 0.01^{a,X}	0.86± 0.01^{b,Y}	0.60± 0.02^{b,Y}	0.94± 0.01^{c,Y}	0.93± 0.02^{b,X}	0.85± 0.03^{b,X}	0.74± 0.02^{e,Z}	0.58± 0.02^{e,Z}	0.53± 0.01^{d,Z}
4	1.06± 0.04^{ab,X}	0.71± 0.03^{d,Y}	0.58± 0.01^{b,Y}	0.88± 0.01^{d,Y}	0.71± 0.01^{c,Y}	0.54± 0.02^{d,Z}	0.89± 0.02^{c,Y}	0.85± 0.02^{d,X}	0.82± 0.05^{c,X}
5	0.97± 0.02^{d,X}	1.14± 0.02^{a,X}	1.33± 0.02^{a,Y}	0.71± 0.02^{e,Z}	0.75± 0.02^{c,Z}	0.65± 0.02^{c,Z}	0.87± 0.00^{d,Y}	0.94± 0.01^{c,Y}	1.39± 0.05^{a,X}

$^{a\ to\ e}$Different letters within columns are significantly different (*P*<0.05)

$^{X\ to\ Z}$Different letters within rows for each power level of L, a, b values are significantly different (*P*<0.05) suggested that the darkening developed in the product due to the burning for microwave powers of 360 and 600 W. Thus, İzli et al. (2014) determined that microwave treatment at 160 W yielded the best product colour values which were closest to the L*, a* and b* values of the fresh sample. Similarly, the surface temperatures were higher at high power levels used in the present study, and it might have directly affected the colour changes in olive slices dried (40).

The experimental data for the change in the parameters L*, a*, and b* during drying period could not be fitted to any kinetic models. Because there is no ordered increase or decrease in the colour values of the microwave dried olive slices.

Total Phenolic Content

The total phenolic content of the raw material and microwave dried black olive slices are given in Table 3. The total phenolic content decreased as the microwave power increased. The total phenolic content was found to be significantly lower at 540 W than at 180 or 360 W. The total phenolic content was fitted to a first-order reaction kinetics model, in which the microwave power dependence of the rate constant is modeled by the Arrhenius-type relationship.

Table 3. The changes in the total phenolic content of the black olive slices during microwave drying at different power levels

	Total phenolic content (mg GAE/g dry matter)		
Time (min)	180 W	360 W	540 W
0	$0.95±0.02^{a,X}$	$0.95±0.02^{a,X}$	$0.95±0.02^{a,X}$
1	$0.93±0.00^{b,X}$	$0.88±0.00^{b,Y}$	$0.81±0.01^{b,Z}$
2	$0.92±0.01^{b,X}$	$0.82±0.02^{c,Y}$	$0.72±0.01^{c,Z}$
3	$0.86±0.01^{c,X}$	$0.79±0.01^{d,Y}$	$0.61±0.01^{d,Z}$
4	$0.84±0.01^{c,X}$	$0.76±0.01^{e,Y}$	$0.47±0.02^{e,Z}$
5	$0.80±0.01^{d,X}$	$0.68±0.01^{f,Y}$	$0.46±0.0^{1e,Z}$

$^{a\ to\ e}$Different letters within columns are significantly different (*P*<0.05)
$^{X\ to\ Z}$Different letters within rows are significantly different (*P*<0.05)

It was observed that there were significant differences between the total phenolic contents of olives dried at different microwave power levels (*P<0.05*). For the microwave power range studied, the use of an irreversible first-order model for the decrease in the total phenolic content proved adequate. Changes in the phenolic contents of black olive slices over the time for each microwave power are given in Figure 5.

Kinetic equations describing the changes on the phenolic content of olives were found as follows in Table 4. The first-order rate constant (k_p) proved the decrease in the total phenolic content of the olive during microwave drying. k_p increased parabolically as the microwave power increased (Figure 6). The rate constant of the total phenolic content (R_{FN}) during drying changes as a function of the microwave power level (P) were defined by the polynomial relation (R^2=1) given in Equation 8.

Table 4. Kinetic equations and statistical results of the total phenolic content for microwave drying of olive slices

	Total phenolic content			
Microwave power (W)	Equation	R^2	r	RMSE
180	$C/C_0 = 1.0306e^{-0.041t}$	0.949	0.972	0.013
360	$C/C_0 = 0.9806e^{-0.058t}$	0.948	0.975	0.015
540	$C/C_0 = 1.0079e^{-0.157t}$	0.961	0.985	0.025

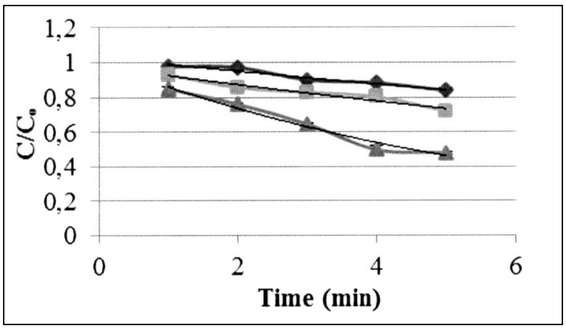

Figure 5. Change in total phenolic content of black olive slices during microwave drying at different microwave power levels
180 W — 360 W — 540 W

$$RFN = 3 \times 10^{-6}P^2 - 8 \times 10^{-4}P + 0.1577 \quad (8)$$

The kinetic constants predicted by this relation for 5 min drying at different microwave power levels were calculated as 0.11, 0.26, and 0.60 for 180, 360, and 540 W, respectively.

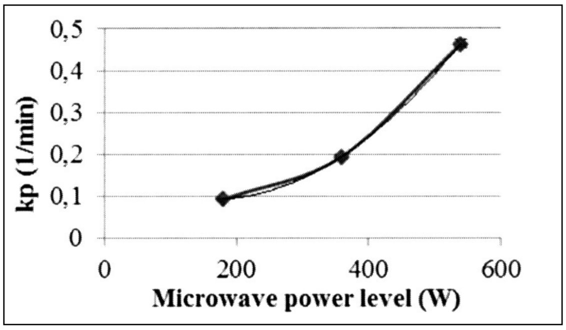

Figure 6. Kinetic constants for total phenolic content changes at different microwave power level

Erbay and Icier (2009) reported that a long term effect of temperature should cause a complete damage of the phenolics, so both the degree of intense heat and heat treatment time were important (3). Similarly, the increase in the power level significantly accelerated the loss of phenolics in the present study.

Arslan and Özcan (2010) found that oven dried samples had lower phenolic contents than the microwave oven dried samples onion slices (1664 mg/100 g and 1624 mg/100 g for 210 and 700 W, respectively). The short time required for microwave drying might have increased the phenolic content of microwave oven dried samples. The increase in total phenolics is possibly due to the liberation of phenolic compounds from the matrix during the drying process. Drying might have accelerated the release of bound phenolic compounds during the breakdown of cellular constituents (36). İzli et al. (2014) reported that after drying treatments (convective, microwave and microwave-convective), the phenolic content decreased by 64–75%. The fresh sample Goldenberry fruit had significantly ($P \leq 0.05$) higher phenolic content than the dried samples. Also, there was no significant ($P > 0.05$) difference between the phenolic contents of dried samples (40).

CONCLUSION

Microwave power can provide a very effective drying method for some foods. The effects of three microwave power settings on the drying of black olive slices were investigated. Microwave drying at 540 W presented the shortest drying time. The surface temperature and the weight loss of olive slices dried at 540 W were higher than 180 and 360 W. The greatest loss in brightness was determined drying at 360 W for 5 min, whereas the higher value was obtained in the drying at 180 W. As the power of the microwave oven increased, the total phenolic content of the dried black olive slices decreased. The surface temperature and the weight loss of olive slices dried at 540 W were higher than 180 and 360 W. The first order kinetic model was found to adequately describe the changes of the total phenolic content of the olive slices during the microwave drying time in the range of 180-540 W. The greatest loss in brightness was determined drying at 360 W for 5 min, whereas the higher value was obtained in the drying at 180 W. It can be concluded that the effects of the microwave process conditions including power level and the duration should be further investigated to determine the optimum conditions giving minimum changes on quality and also shortest process time.

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