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MICROWAVE DRYING KINETICS OF THYME

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Abstract: Thyme (*Thymbra spicata* L.) leaves were dried in a domestic microwave oven to determine the effects of microwave output power on the drying time, drying rate, and the colour of the dried product. Seven different microwave output powers ranging from 360 W to 900 W were used in the experiments. Drying of thyme and mint leaves took place mainly in falling rate period with no constant rate period. Increasing the microwave output power resulted in a considerable decrease in drying time. The semi-empirical Page's equation used to describe the drying kinetics of leaf materials gave an excellent fit for all data points with values for the coefficient of determination (R^2) greater than 0.997 and the standard error of estimates (SEE) lower than 0.023. The value of the drying constant showed an increasing tendency with the increased microwave output power signifying that with increase in microwave output power, drying curves become steeper indicating faster drying of the products. Both the traditional in-shade drying and microwave drying techniques used in this study caused some undesirable effects on the colour of the thyme and mint leaves. The traditional in-shade drying produced a darker brownish green product. Based on the overall colour evaluation results, compared to traditional in-shade drying, more stable green colours similar to those of the original fresh materials were obtained by using microwave output powers of 810 and 900 W for thyme and 720, 810, and 900 W for mint.

Key words: thyme, mint, drying, microwave, drying time, drying rate, colour.

Notation

- α hue angle, °
- ΔE total colour difference, dimensionless
- a colour redness (+)/greenness (-) coordinate, dimensionless
- b colour yellowness (+)/blueness (-) coordinate, dimensionless
- C chroma, dimensionless

k	drying constant, min^{-1}
L	colour brightness coordinate, dimensionless
M_R	moisture ratio, decimal
n	exponent, dimensionless
R^2	coefficient of determination, decimal
SEE	standard error of estimate, decimal
t	drying time, min
X	moisture content (db), kg water/kg dry matter
X_0	initial moisture content (db), kg water/kg dry matter
X_e	equilibrium moisture content (db), kg water/kg dry matter

1. INTRODUCTION

The members of Labiatae family are common herbs mainly grown in the mountainous areas of the Mediterranean region of Turkey (Başer 1994). These spontaneous herb plants are largely collected from the wild and then exported to the world markets. Among them, thyme and mint are widely used as culinary, medicinal, and aromatic herbs. The fresh or dried leaves and flowering tops of these plants are used in the food, cosmetic, and pharmaceutical industries to produce spice, essential oils, and drugs.

Compared to solar and hot air drying, microwave or hybrid microwave drying techniques (microwave-hot air drying; microwave-freeze drying, microwave-vacuum drying; osmotic pre-treatment before combined microwave-hot air drying) can greatly reduce the drying time of biological materials without quality degradation (Funebo & Ohlsson, 1998; Nindo *et al.*, 2003).

There has been extensive research on microwave drying techniques, examining a broad spectrum of fruits and vegetables including: potato (Bouraout *et al.*, 1994), apple (Funebo & Ohlsson, 1998), mushroom (Torrington *et al.*, 2001), carrot (Litvin *et al.*, 1998), banana (Maskan, 2000), garlic (Sharma & Prasad, 2001), asparagus (Nindo *et al.*, 2003).

The introduction of a microwave drying technique which reduces drying time considerably and produces a high quality end-product could offer a promising alternative and significant contribution for the herb processing industry.

Therefore, the overall objective of this study was to improve the basic knowledge about the important parameters of the microwave drying of thyme to determine the influence of microwave output power on drying time and improve the product quality in terms of colour. Specific objectives were:

- (a) to evaluate the influence of microwave output power on drying kinetics and colour of thyme and
- (b) to describe the drying process by producing a thin layer drying model for the purpose of simulation and scaling up of the process.

2. MATERIALS AND METHODS

2.1. Material

The leaves of the fresh green thyme (*Thymbra spicata* L.) known as "black thyme" or "Za'tar" leaves used in the drying experiments were obtained from the local market in the Hatay province of Turkey. The whole samples were stored at $4\pm 0.5^\circ\text{C}$ before they were used. Prior to each drying experiment, the whole material samples were taken out of storage and leaves from stems were separated. The initial moisture content of the samples was measured individually using four 50 g leaf samples dried in an oven at 105°C for 24 h. The initial moisture contents (m.c.) of thyme and mint leaves were determined as 2.30 ± 0.29 and 4.95 ± 0.33 dry basis (db), respectively.

2.2. Drying equipment and procedure

A programmable domestic microwave oven (Galanz; Model: WP900AL23-Z1) with maximum output of 900 W at 2450 MHz was used for the drying experiments. The dimensions of the microwave cavity were 215x350x330 mm. The oven was fitted with a glass turntable (314 mm diameter) and had a digital control facility to adjust the microwave output power (by 10% decrements) and the time of processing.

Seven different microwave output powers (360, 450, 540, 630, 720, 810, and 900 W) were investigated at constant sample loading density. In each drying experiment, $60 (\pm 0.01)$ g of thyme and $30 (\pm 0.01)$ g of mint leaves were uniformly spread on a turntable fitted inside the microwave cavity for even absorption of microwave energy. Each drying experiment was replicated three times with a preset microwave output power and time schedule. Moisture loss was recorded by taking out and weighing the turntable on a digital balance periodically. For the mass determination, a digital balance with an accuracy of 0.01 g (Sartorius; Model: GP3202) was used. The microwave power was applied until the weight of the sample reduced to a level corresponding to a moisture content of about 0.10 db.

The common semi-empirical Page's equation (Eqn 1) was used to describe the thin layer drying kinetics of thyme and peppermint leaves (Ren & Chen, 1998; Sharma & Prasad, 2001):

$$M_R = \frac{X - X_e}{X_0 - X_e} = \exp(-kt^n) \quad (1)$$

where: M_R is the moisture ratio; X is the moisture content db; X_e is the equilibrium moisture content db; X_0 is the initial moisture content db; t is the time in min; k is the drying constant in min^{-1} ; and n is the dimensionless exponent. The equilibrium moisture content was assumed to be zero for microwave drying (Maskan, 2000).

In addition to the microwave drying experiments, thin layer of fresh thyme leaves were spread on a plastic mat and dried in-shade for seven days to determine the extent of the colour changes. Final moisture contents of traditionally dried thyme and mint leaves were then determined as 0.13 ± 0.41 and 0.14 ± 0.50 db, respectively. Colour properties of these materials were measured to describe the colour change during drying.

2.3. Colour measurement

Sample colour was measured before and after drying by using a colour meter (Minolta Co.; Model: Chroma CR-100). The colour meter was calibrated against a standard calibration plate of a white surface and set to CIE Standard Illuminant C. The display was set to CIE $L a b$ colour coordinates. Ten random readings per sample were recorded and the average values of colour parameters with standard deviation values were reported. The colour brightness coordinate L measures the whiteness value of a colour and ranges from black at 0 to white at 100. The chromaticity coordinate a measures red when positive and green when negative, while the coordinate b measures yellow when positive and blue when negative. Also, the chroma C (Eqn 2), hue angle α (Eqn 3), and the total colour difference from the fresh material ΔE (Eqn 4) were calculated from the values for $L a b$ and used to describe the colour change during drying;

$$C = \sqrt{(a^2 + b^2)} \quad (2)$$

$$\alpha = \tan^{-1}(b/a) \quad (3)$$

$$\Delta E = \sqrt{(L_o - L)^2 + (a_o - a)^2 + (b_o - b)^2} \quad (4)$$

where subscript "o" refers to the colour reading of fresh material which is used as the reference. Larger ΔE denotes greater colour change from the reference material (Maskan, 2000).

2.4. Data analysis

A multiple comparison test was conducted using the Sigma-Stat software (SPSS Inc., version 3.0) to establish the actually differing applications. Also, non-linear regression analysis was performed using Sigma-Plot software (SPSS Inc., version 8.03) to estimate the parameters k and n of the semi-empirical Page's equation (Eqn 1). Regression results include the coefficients for the equation, standard error of estimate (SEE), and coefficient of determination (R^2).

3. RESULTS AND DISCUSSION

3.1. Drying curves

The moisture content *versus* time curves for microwave drying of thyme as influenced by microwave output power are shown in *Fig. 1*. As the microwave output power was increased, the drying time of both thyme were reduced, considerably. Similar findings were reported by several authors (Drouzas & Schubert, 1996; Funebo & Ohlsson, 1998). By working at 900 W instead of 360 W, the drying time up to the moisture content of 0.09 db could be shortened by 3.0 fold for thyme leaves (*Fig. 1*). The microwave drying process which reduced the thyme leaves moisture content from 2.30 (± 0.29) db to 0.09 (± 0.01) db took 4.0-12.0 min, depending on the applied microwave output power. Since the initial moisture contents of the thyme leaves used in the drying experiments was relatively constant, the difference in drying time requirements was considered to be mainly due to the difference in the drying rates. The microwave drying can greatly reduce the drying time of thyme leaves when

compared to the published drying data for thyme by Balladin & Headley (1999) which states that about 13 h was required to reach the moisture content of 0.14 and 0.10 db for wire basket solar drying and oven drying at about 50°C, respectively.

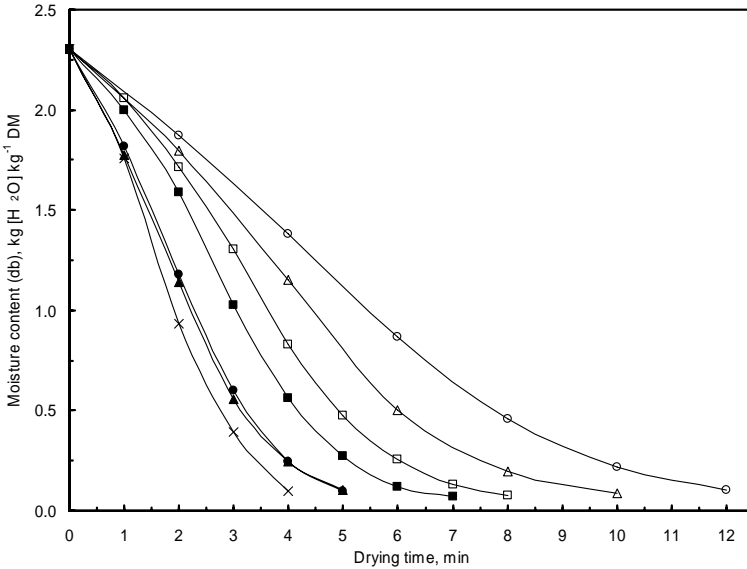


Fig. 1. Drying curves for thyme (*Thymbra spicata* L.) leaves under various microwave output powers; ×, 900 W; ▲, 810 W; ●, 720 W; ■, 630 W; ◻, 540 W; △, 450 W; ⊖, 360 W; DM, dry matter

The drying rate was calculated as the quantity of moisture removed per unit time per unit dry matter (DM) ($\text{kg [H}_2\text{O] kg}^{-1} \text{ DM min}^{-1}$). The drying rate curves for thyme dried with different microwave output powers are shown in Fig 2. Depending on the drying conditions, average drying rates of thyme leaves ranged from 0.18 to 0.55 $\text{kg [H}_2\text{O] kg}^{-1} \text{ DM min}^{-1}$ for the output power between 360 W and 900 W, respectively (Fig. 2). The moisture content of the material was very high during the initial phase of the drying which resulted in a higher absorption of microwave power and higher drying rates due to the higher moisture diffusion. As the drying progressed, the loss of moisture in the product caused a decrease in the absorption of microwave power and resulted in a decrease in the drying rate. Higher drying rates were obtained at higher microwave output powers. Thus, the microwave output power had a crucial effect on the drying rate of thyme leaves. Similar findings were reported in several previous studies (Funebo & Ohlsson, 1998; Maskan, 2000; Sharma & Prasad, 2001). The drying rates were relatively high in the beginning of drying and reached the peak value after 2 to 5 min drying (heating period). The length of this so called heating period increased with decreased microwave output power. After these peak values, drying rates decreased with decreasing moisture content, signifying that drying of thyme occurred mainly in falling rate period. The accelerated drying rates during the initial phase of drying may be attributed to internal heat generation and the liquid movement within the material when it is exposed to microwaves.

3.2. Modelling drying data

The drying data were then used to describe the microwave drying kinetics of the thyme leaves. The parameters k and n of the semi-empirical Page’s equation (Eqn. 1) for a given drying condition were estimated using non-linear regression technique (Table 1) and the fitness were illustrated in Fig. 3. The model gave an excellent fit for all of the experimental data points with the coefficient of determination (R^2) values greater than 0.997 and the standard error of estimates (SEE) lower than 0.023. It is determined that the value of drying constant (k) increased with the increase in the microwave output power (Table 1). These results were in good agreement with the drying rate data which follows the similar trends and signified that with the increase in microwave output power, drying curves becomes steeper indicating faster drying of the leaves.

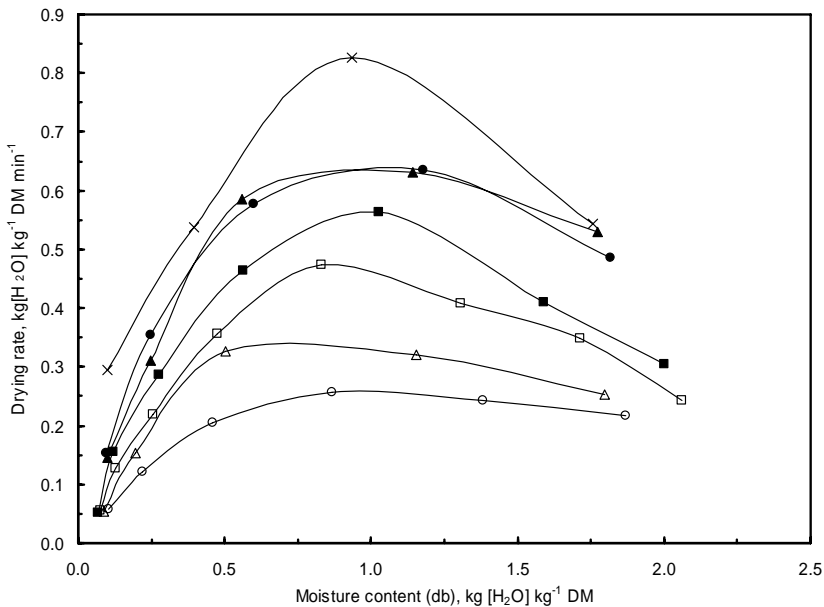


Fig. 2. Drying rate curves for thyme (*Thymbra spicata* L.) leaves under various microwave output powers; ×, 900 W; ▲, 810 W; ●, 720 W; ■, 630 W; ◻, 540 W; △, 450 W; ⊖, 360 W; DM, dry

Table 1. Non-Linear Regression Analysis Results Of Semi-Empirical Page’s Equation (Eqn. 1) for Microwave Drying Of Thyme (*Thymbra Spicata* L.) Leaves Under Various Microwave Output Powers; (K , Drying rate constant in min^{-1} ; n , exponent; SEE , standard error of estimate; R^2 , coefficients of determination)

Parameter	Microwave output power, W						
	900	810	720	630	540	450	360
k	0.2706	0.2451	0.2222	0.1122	0.0828	0.0704	0.0601
n	1.7282	1.5811	1.6409	1.8131	1.8142	1.6943	1.5742
$SEE (\pm)$	0.0063	0.0108	0.0091	0.0135	0.0138	0.0159	0.0145
R^2	1.000	0.999	1.000	0.999	0.999	0.999	0.999

3.3. Colour assessment

The colour of the dried product is an important quality indicator for the acceptance in the market. Dried thyme should have a bright green colour. The results of the colour measurements of fresh, microwave dried, and traditionally in-shade dried thyme are given in Table 2. Preferred colours are those closest to the original colour of the fresh samples.

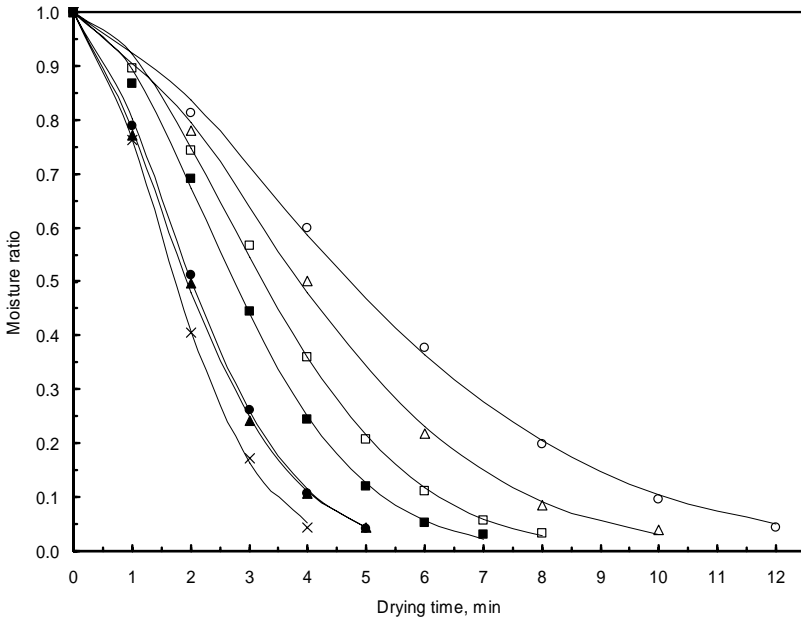


Fig. 3. Moisture ratio versus time, comparing experimental curve with the predicted one (–) through semi-empirical Page’s equation (Eqn 1) for thyme (*Thymbra spicata* L.) leaves under various microwave output powers; ×, 900 W; ▲, 810 W; ●, 720 W; ■, 630 W; □, 540 W; △, 450 W; ○, 360 W

Table 2. Effect of Microwave Output Power on the Colour of Thyme (*Thymbra spicata* L.) Leaves; L, brightness of a colour; a, greenness of a colour when negative; b, yellowness of a colour when positive; C, chroma of a colour; α, hue angle of a colour in °; ΔE, total colour difference

Microwave output power, W	Colour parameters					
	L	a	b	C	α	ΔE
Fresh	41.60 (2.02)*	-11.08 (1.37)	19.07 (1.65)	22.07 (2.00)	120.10 (2.11)	0.00
In-shade dried	39.94 (2.88)	-2.33 (0.89)	15.34 (0.94)	15.54 (1.02)	98.50 (2.92)	9.66
900	41.15 (1.65)	-4.71 (0.53)	18.38 (0.53)	18.97 (0.63)	104.34 (1.21)	6.43
810	40.02 (1.03)	-3.68 (1.00)	17.66 (0.97)	18.06 (1.14)	101.67 (2.48)	7.69
720	39.06 (1.36)	-3.40 (0.81)	17.03 (1.10)	17.37 (1.19)	101.22 (2.26)	8.34
630	38.24 (0.89)	-2.42 (0.79)	16.28 (0.81)	16.47 (0.88)	98.37 (2.44)	9.70
540	37.82 (0.99)	-1.82 (0.49)	15.72 (0.74)	15.83 (0.78)	96.57 (1.51)	10.54
450	37.79 (1.04)	-1.74 (0.59)	15.50 (1.06)	15.61 (1.07)	96.41 (2.15)	10.70
360	36.41 (0.69)	-1.27 (0.39)	14.66 (0.39)	14.72 (0.40)	94.96 (1.50)	11.94

* Values in parenthesis indicate the standard deviation

It is clear from Table 2 that the colour of in-shade dried thyme differed significantly from the values of fresh thyme leaves ($P < 0.05$). The L , b , a , C , and α value of in-shade dried thyme leaves were significantly decreased compared to the fresh leaf materials. Such changes in the colour of the in-shade dried thyme resulted darker brownish green leaf colour. Therefore traditional in-shade drying had a crucial effect on the colour of the thyme.

On the other hand, compared to fresh thyme, a significant decrease in L and b values of the leaf material dried at 360, 450, 540, and 630 W microwave output powers was observed ($P < 0.05$). Compared to fresh material, no significant difference was found in L and b values of the leaf material dried at 720, 810, and 900 W microwave output powers ($p > 0.05$) (Table 2). The a and C values of the leaf material dried at 360, 450, 540, 630, and 720 W microwave output powers were also differed significantly from the fresh material ($P < 0.05$). The chroma (C) value indicates colour saturation which varies from dull (low value) to vivid colour (high value) and is proportional to strength of the colour. Compared to fresh product, little change was found in C and a values of the products dried at 810 W and 900 W, and they were not statistically different ($p > 0.05$). A significant decrease was found in hue angle (α) values among the fresh and microwave dried leaves ($P > 0.05$) (Table 2).

These results indicate that the changes in the colour values of microwave dried thyme were highly depend on the microwave output power. Although the microwave drying at relatively low output powers resulted in some darkening in the leaf colour (lower L value), compared to fresh and in-shade dried thyme leaves, a good green colour in the leaves dried at 810 and 900 W microwave output powers was maintained. The total colour change (ΔE) values, which takes into account the changes in the greenness (a) and yellowness (b) supports these results (Table 2). The microwave output powers below 810 W lead to increased colour deterioration possibly due to the lengthy drying time and associated internal heat generation during microwave drying. The lower colour degradation of the dried thyme leaves at higher microwave output powers (810 and 900 W) may, therefore, be due to the substantial reduction in the drying time.

5. CONCLUSION

Results showed that in the main, microwave drying of thyme leaves took place in the falling rate period with no constant rate period. Relatively high drying rates in the beginning of drying were observed depending on the dried material and applied microwave output power. After 1 to 5 min heating periods, drying rates decreased with decreasing moisture content, signifying that drying of thyme leaves occurred mainly in falling rate period. Higher drying rates were obtained with higher microwave output powers.

Drying time decreased considerably with increased microwave output power. This suggests that the microwave output power had a crucial effect on the drying rate. Compared to the drying data for thyme using wire basket solar drying and oven drying method and mint using oven drying method published before, microwave drying technique used in this study can greatly reduce the drying time of thyme.

The semi-empirical Page's equation used to describe the drying kinetics of thyme gave an excellent fit for all of the data points with higher coefficient of determinations and lower standard error of estimates. The value of the drying constant showed an increasing tendency with the increased microwave output power signifying that with an increase in microwave output power, drying curves become steeper indicating faster drying of the products.

It is determined that both the in-shade drying and the microwave drying techniques used in this study caused some undesirable effects on the colours of the thyme. The traditional in-shade drying produced a darker brownish green product. The change in the colour values depend highly on the microwave output power. Lower microwave output powers up to 720 W for thyme lead to considerable colour changes in dried products. Based on the overall colour evaluation results, compared to traditional in-shade drying, more stable green colours close to that of the original fresh materials were obtained by using microwave output powers of 810 and 900 W for thyme.

As an overall conclusion, microwave drying technology can greatly reduce the drying time and successfully be used to produce dried thyme leaves with desirable colours.

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KINETIKA MIKROTALASNOG SUŠENJA *Thymbra cpicata-e*

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Sadržaj: Lišće *Thymbra cpicata-e* je sušeno u mikrotalasnoj sušari kako bi se odredio uticaj snage sušare na vreme sušenja, stepen sušenja i boju osušenog proizvoda. U eksperimentu je korišćeno sedam tipova mikrotalasnih sušara snage od 360 W do 900 W. Povećanje snage sušare rezultiralo je značajnim smanjenjem vremena sušenja. Poluempirijska Pejdžova formula (Page's) koja se koristi za opis kinetike sušenja lisnatih materijala, pokazala je dobro uklapanje za sve tačke sa vrednostima koeficijenta determinacija (R^2) većeg od 0.997 a standardna greška je bila niža od 0.023. Vrednost konstante sušenja pokazala je tendenciju porasta sa povećanjem snage sušare ukazujući da sa povećanjem snage sušare krive sušenja postaju strmije ukazujući na brže sušenje proizvoda. I tradicionalno sušenje proizvoda u senci i korišćenje mikrotalasne sušare su tokom ovog istraživanja pokazali određene negativne posledice na boju listova *timbre* i mente. Tradicionalno sušenje u senci je uzrokovalo pojavu tamnije braon boje na proizvodu. Obzirom na izvršenu analizu boje i upoređenjem sa proizvodima sušenim na tradicionalni način, postojanija zelena boja je uočena kod lišća *timbre* kod sušara snage 810 i 900 W, i 720, 810 i 900 W za mentu.

Ključne reči: *timbra*, *menta*, *sušenje*, *mikrotalasna sušara*, *vreme sušenja*, *stepen sušenja*, *boja*.