Drying Kinetics and Color Properties of Lemon Balm (*Melissa officinalis*) Leaves Dried by Convective Hot Air Drying

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Abstract. Lemon balm (*Melissa officinalis*) leaves with the moisture content of 3.18 g water/g dry base were dried by convective hot air drying at 50, 60 and 70°C until the moisture content fell down to 0.15 g water/g dry base. Drying experiments were completed between 17 and 50 min. depending on drying temperatures. For the selection of the most suitable thin layer drying model, five mathematical models (Page, Modified Page, Logarithmic, Lewis, Henderson and Pabis) were applied to the drying treatments. The higher correlation coefficient (*R2*), and reduced root mean square error (*RMSE*), Chi square (*χ2*) were used to identify the excellence of fit model for drying of lemon balm leaves. As a result of the statistical tests, Page and Modified Page were considered to be the best models for 60 and 70°C hot air drying experiments when compared to the other models. In addition, Logarithmic model resulted in preferable statistical values than other thin layer models at 50°C. The color values such as L^*, b^*, C^*_{ab} and $h°$ decreased, while a^* value increased after drying. The effective moisture diffusivity (Deff) values of dried lemon balm leaves increased with the rise of drying temperatures and ranged between 2.03×10-8 to 7.13×10-8 m2/s. Total phenolic content and antioxidant capacity of dried lemon balm samples were both increased after drying. The total phenolic content and antioxidant capacity was obtained as the highest from 50°C treatment when compared with all cases.

INTRODUCTION

Lemon balm (*Melissa officinalis* L.), which is a member of *Lamiaceae* (formerly *Labiatae*) family,is grown as an ornamental plant in countries with a Mediterranean climate. It is native to southern Europe and northern Africa, and east as far as the Caucasus and northern Iran. Geographically, it has spread to countries such as France, Bulgaria, Germany and Romania. On the other hand it is widely grown in Aegean and Mediterranean Regions of Turkey and also Istanbul and Bursa provinces. The subspecies of *M. officinalis* are evaluated in domestic markets and they are also on the list of the exported medicinal and aromatic plants (Gasquet et al., 1993).

Lemon balm is a thin-leafed perennial herbaceous plant with yellow or whitish flowers at a height of 3-5 meters. Lemon balm, which has been known to have a calming effect since ancient times, is quite effective in the treatment of many diseases from stress to stomach disorders and it has a comforting feature due to its lemon like smell. It has also antispasmodic, antimicrobial and antimicrobial effects. Rosmarinic acid plays an important role in the chemicals obtained from melisa plant (Abad et al., 1997).
In a study, the essential oil of lemon balm was determined as 0.2%. In addition, the most important components were geranial (E-citral), neral (Z-citral), citronellal, Ecaryophylenne and geraniol respectively (Dias et al., 2012). Carnat et al. (1998), also studied aroma components in lemon balm tea infusion, by GC-MS and determined geranial, neral and citronellal compounds respectively in the ratios of 43.53%, 30.15% and 16.81%.

Material and Method

Material and drying process

Fresh lemon balm leaves supplied from a local market in Bursa were stored in the refrigerator at a temperature of 4 ± 0.5 °C until drying process. After the samples were washed, the water was removed from the surface of leaves by paper towel. The initial moisture content of samples was obtained by moisture analyzer (Sartorius MA150, Germany) and the average moisture content of lemon balm leaves was determined as 3.18 g water/g dry base.

Drying trials were performed in a hot air convective dryer which was produced by Yucebas Machine Analytical Equipment Industry (Y35, Izmir, Turkey) with the technical features of 220 V, 50-60 Hz, 200 W. 20 g lemon balm leaves were placed uniformly on an aluminum plate and dried at 50, 60 and 70°C with the constant 20% relative humidity. During drying samples were removed at intervals and weighed. The weight loss of samples was recorded by using a digital balance (Mettler Toledo, MS3002S, Greifensee, Switzerland) with the accuracy of 0.01 g. All weighing processes were completed in 10 s during drying process.

Mathematical modelling of drying data

Moisture ratio (MR) and drying rate of lemon balm leaves during drying were calculated by employing the following equations (Eq.1, Eq. 2).
where, *MR* is moisture ratio, *M* is the moisture content at a certain time (g water/g dry base), *Mi* is the primary moisture content (g water/g dry base), *Me* is the equilibrium moisture content (g water/g dry base), *Mt* and *Mt+dt* are the moisture content at *t* and *t+dt* (g water/g dry base) respectively, and *t* is drying time (min) (Dadali et al., 2007).

$$MR = \frac{M - M_e}{M_i - M_e} \qquad (1) \qquad Drying\ rate = \frac{M_{t+dt} - M_t}{dt} \qquad (2)$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}(MR_{exp,i} - MR_{pre,i})^2\right]^{1/2} \qquad (3)$$

$$\chi 2 = \frac{\sum_{i=1}^{N}(MR_{exp,i} - MR_{pre,i})^2}{N-n} \qquad (4)$$

The equations in Table 1 were used to find most convenient model for explaining the drying curve of lemon balm leaves. Root mean square error (*RMSE*) gives deviation between the estimated and experimental values for the models. To identify the thin layer drying characteristics of lemon balm leaves, the model with the higher correlation coefficient (*R2*), and reduced *RMSE* and chi-squared ($\chi 2$) was selected as a best model (Ozbek and Dadalı, 2007). These parameters were calculated using the sequent equations (Eq 3, Eq 4):
where, *MRexp,i* is the empirically dimensionless moisture ratio for test *i*, *MRest,i* is the estimated dimensionless moisture ratio for test *i*, *N* is the count of observation and *n* is the count of constants in the model (Avhad and Marchetti, 2016).

Table 1. Mathematical models applied to drying curves of lemon balm leaves

Model no	Model name	Model	References
1	Page	MR = exp(-kt^n)	Wang et al. (2007)
2	Modified Page	MR= exp [(-kt)^n]	Toğrul (2006)
3	Logarithmic	MR = a exp(-kt) + c	Darıcı and Şen (2015)
4	Lewis	MR = exp(-kt)	Doymaz (2006)
5	Henderson and Pabis	MR = a exp(-kt)	Evin (2011)

Calculation of effective moisture diffusivity

Fick's second diffusion law has been widely used to explain the drying process of food products during the falling rate period (Doymaz, 2008). The solution of Fick's second law for an infinite slab is showed in Equation (5), assuming dimensional moisture movement volume change, constant temperature and diffusivity coefficients, and negligible shrinkage (Crank, 1975);
where, *Deff* is effective moisture diffusivity (m^2/s), *L* is the half thickness of the slab in samples (m), and *n* is a positive integer. In practice, only the first term Equation (5) is written in a logarithmic form as follows:

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)^2} exp\left(-\frac{(2n-1)^2\pi^2 D_{eff}t}{4L^2}\right) \qquad (5)$$

$$MR = \frac{8}{\pi^2} exp\left(-\frac{\pi^2 D_{eff} t}{4L^2}\right) \quad (6)$$

$$D_{eff} = -\frac{slope4L^2}{\pi^2} \quad (7)$$

The effective moisture diffusivity were determined using the method of slopes by plotting experimental drying data in terms of *lnMR* versus drying time, using the following equation (7).

Color analysis

Color measurements of the samples were determined over the outer surface of the samples by using a chroma meter (Konica Minolta CR-5, Bench-top, Japan). L*, a*, b* values were displayed as lightness/darkness, redness/greenness and yellowness/blueness respectively. Analyzed CIE-L, a and b values were used to calculate chroma and hue angle to characterize color changes during drying (Mujumdar, 2000; Demir, 2018).
Chroma (C*) changed from 0 (dull) to 60 (vivid) and was calculated with the first equation (1). Hue angle (h°) value, demonstrated in the second equation (2) is defined by the angles of 0, 90, 180 and 270°, representing the colors of red, yellow, green and blue, respectively (Karaaslan and Tuncer, 2008).

$$Chroma\ (C^*) = \sqrt{(a^*)^2 + (b^*)^2} \quad (8)$$

$$h^\circ = arctan\ \left(\frac{b^*}{a^*}\right) \quad (9)$$

Extraction of samples for total phenolic content and antioxidant capacity

Extractions were carried out according to Capanoglu et al. (2008). Extracts were prepared by adding 5 mL 75% aqueous methanol containing 0.1% formic acid in a cooled ultrasonic bath for 15 min and 10 min of centrifugation at 4°C and 2700 × g, after which the supernatants were collected. The extraction procedure was repeated three times, and all the extracts were stored at - 20° C until analysis.

Determination of total phenolic content and antioxidant capacity

Folin-Ciocalteu spectrophotometric method was used to determine total phenolic content as described by Spanos and Wrolstad (1990). Gallic acid was used for the calibration of the standard curve (R2=0.9835). The phenolic content was expressed as gallic acid equivalents of dry weight (mg of GAE/100g dw).

Antioxidant capacity of the fresh and dried lemon balm samples were measured with 2-diphenyl-1-picrylhydrazyl (DPPH), method (Katalinic et al., 2006). Trolox was used as the calibration of the standart curve (R2=0.9929). The results were given as µmol Trolox equivalent (TE) per g dry weight (µmol TE/g dw).

Statistical analysis

The experiment was conducted in a completely randomized design with three replications. The results were statistically evaluated by one-way analysis of variance

(ANOVA) using the JMP software package version 6.0 (SAS Institute Inc. NC, 27513). When significant differences were found ($P< 0.05$), the Least Significant Difference (LSD) test was used to determine the differences among means.

RESULTS AND DISCUSSION

Drying characteristics of lemon balm leaves

The lemon balm leaves were dried in a hot air dryer using different temperature until the moisture content reached 0.15 g water/g dry base. The changing of the moisture content versus drying time at various temperatures was given in Figure 1. The drying process took 50, 28 and 17 min at 50, 60 and 70°C, respectively. It was apparent that drying time decreased continuously with increasing temperature. This observation is in agreement with previous studies on drying of tomatoes (Doymaz, 2007), mint leaf (Therdthai and Zhou, 2009) and kiwifruit (Orikasa et al., 2008).

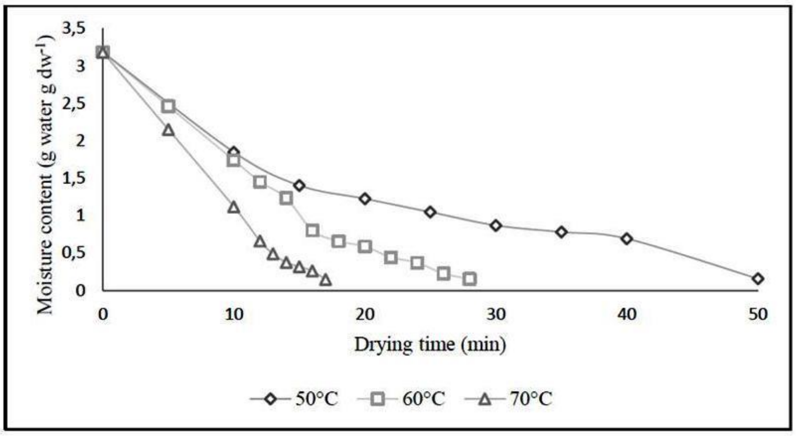

Figure 1
Moisture content of lemon balm leaves as a function of drying time at different drying temperatures

Results of drying rate during drying time, obtained in drying of lemon balm leaves carried out at three temperatures were presented in Figure 2. As can be seen from this figure, there is no constant rate period in drying curves, and all the drying processes occurred at a falling rate period. The results showed that moisture movement in the lemon balm leaves is governed by diffusion (Doymaz, 2005). Similar findings were reported on drying of various food products (Akpinar et al., 2003; Senadeera et al., 2003; Wang et al., 2007).

Mathematical modelling of drying curves

Table 2 shows drying model coefficients and comparison criteria (R^2, RMSE and $\chi 2$) of the five thin layer drying model. The statistical parameter estimations showed in all cases that R^2, RMSE and $\chi 2$

values ranged from 0.8837 to 0.9918, 0.005350 to 0.109539 and 0.000361 to 0.121489, respectively. As a result of the statistical tests, Page and Modified Page models provided higher R^2 and lower RMSE and $\chi2$ values when compared to the other models for 60 and 70°C hot air drying experiments. For a temperature of 50°C, Logarithmic model gave better statistical values than the other models. Madamba et al. (1996) and Toğrul and Pehlivan (2003) for Logarithmic model and Dadali et al. (2007) and Çakmak et al. (2016) for Page and Modified Page models working with the garlic, apricot, okra and strawberry, respectively also recorded similar results.

Table 2. Statistical results obtained from the selected models

Model no	Drying processes	Model coefficient	RMSE	$\chi2$	R^2
1	50°C	n=0.8958 k=0.0662	0.011510	0.001533	0.9090
	60°C	n=1.4383 k=0.0234	0.005350	0.000361	0.9918
	70°C	n=1.6657 k=0.0255	0.005582	0.000412	0.9913
2	50°C	n=0.8958 k=0.0483	0.011510	0.001533	0.9090
	60°C	n=1.4383 k=0.0736	0.005350	0.000361	0.9918
	70°C	n=1.6657 k=0.1105	0.005582	0.000412	0.9913
3	50°C	k=0.0423 a=0.8299 c=0.0494	0.018581	0.004661	0.9786
	60°C	k=0.1323 a=1.3423 c=0.0484	0.038613	0.023855	0.9078
	70°C	k=0.2052 a=1.4235 c=0.0459	0.056000	0.042336	0.9209
4	50°C	k=0.8837	0.109539	0.121489	0.8837
	60°C	k=0.0915	0.022209	0.006457	0.9308
	70°C	k=0.1513	0.027955	0.007912	0.9157
4	50°C	k=0.0500 a=1.0672	0.017598	0.003584	0.8855
	60°C	k=0.108 a=1.3991	0.035285	0.017929	0.9589
	70°C	k=0.1757 a=1.3985	0.047437	0.026039	0.9376

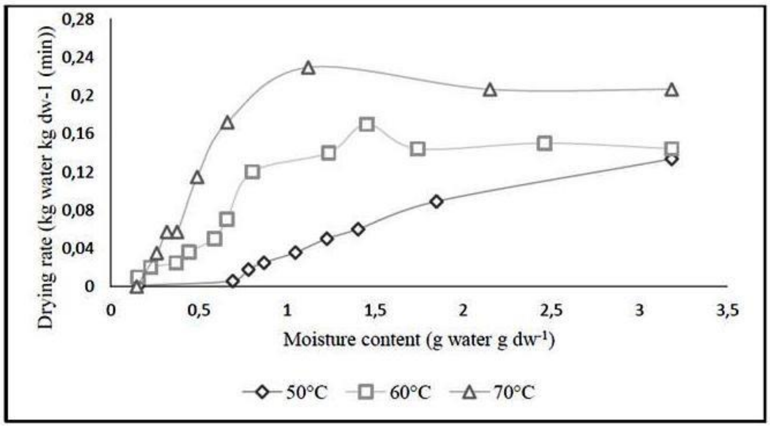

Figure 2
Drying rate of lemon balm leaves as a function of moisture content at different drying temperatures

Table 3: Values of effective moisture diffusivity obtained for lemon balm leaves at different temperatures

Drying processes	Deff (m^2/s)
50°C	2.03×10^{-8}
60°C	4.38×10^{-8}
70°C	7.13×10^{-8}

Effective moisture diffusivity (Deff)

The effective moisture diffusivity (Deff) values for different drying temperatures, calculated from Equation 7, ranged from 2033×10^{-8} to 7.13×10^{-8} m^2/s (Table 3). It can be seen that the values of Deff increased greatly with increasing temperature. This result can be explained by the easier evaporation of the product in high temperature and the increase in drying rate (Mengeş and Ertekin, 2006). The values of Deff in our study were within the general range 10-12-10-8 for drying of food materials (Demiray and Tulek, 2017). Our findings are in line with the results informed by Doymaz (2006) who also acquired an increase in Deff values of mint leaves from 3.067×10^{-9} m^2/s to 1.941×10^{-8} m^2/s with the rise in drying temperature.

Color analysis

The results of color changes in fresh sample for all drying conditions were given in Table 4. The L^* value were significantly affected by different drying treatments ($p < 0.05$) and resulted with a 2.05-26.62% decrease. The lowest L^* value obtained from hot air 70°C dried samples which had darker color than other drying methods. Compared to the fresh sample, *a* (redness) values significantly increased ($p < 0.05$) with all hot air drying treatments. The increase of a^* value could be a result of the Maillard reaction and degradation of pigments such as carotenoids (Maskan, 2001; Xiao et al., 2012). b^* values of dried lemon balm were decreased with respect to fresh samples between the ratios of 41.70-65.60%. This decrement was closely followed for Chroma (C^*_{ab}) values, which were used to comprehend intensity of color. Hawlader et al. (2006) reported that, the reduction in $h°$ values is an expression of darker color. Hot air drying at 70°C caused a smaller reduction of $h°$ values. Additionally, pigment decompositions, non-enzymatic and enzymatic reactions are responsible for the formation of browning pigments (Albanese et al., 2013).

Total phenolic content and antioxidant capacity

The total phenolic content and antioxidant capacity of fresh and dried lemon balm samples were given in Table 5. The highest total phenolic content was attained by 50°C treatment with 2308.26±26.74 mg GAE/100 g dw ($p < 0.05$). Generally, total phenolic content was increased with drying treatments between the ratios of 5.85-255.07% when compared to fresh sample. Similar increment in total phenolic content was reported by Priecina and Karklina (2014) and Türkmen et al. (2005).

Table 4: Color values of fresh and dried lemon balm samples

Drying processes	L^*	a^*	b^*	C^*_{ab}	$h°$
Fresh sample	34,52±0,44[a]	-8,89±0,84[c]	19,71±2,62[a]	21,62±2,74[a]	114,38±0,91[a]
50°C	33,81±1,52[a]	-1,61±0,16[b]	6,79±2,42[c]	6,99±1,69[c]	104,26±5,09[b]
60°C	28,79±1,02[b]	2,11±0,41[a]	11,49±0,09[b]	11,69±0,14[b]	79,62±1,97[c]
70°C	25,33±1,25[c]	1,83±0,47[a]	6,78±1,30[c]	7,04±2,42[c]	74,44±4,04[c]

[a-c] Different letters in the same column display significant difference ($P < 0.05$)

Table 5: Total phenolic content and antioxidant capacity of fresh and dried lemon balm samples Total phenolic content

Drying processes	Total phenolic content (mg GAE/100g dw)	Antioxidant capacity (µmol TE/g dw)
Fresh sample	650.09±51.49[b]	1.00±0.34[d]
50°C	2308.26±26.74[a]	33.55±0.28[a]
60°C	795.97±60.68[b]	17.32±0.90[b]
70°C	688.59±53.52[b]	4.48±0.49[c]

[a-d] Different letters in the same column display significant difference ($P < 0.05$)

Antioxidant capacity of fresh lemon balm was significantly lower compared to dried samples ($p < 0.05$). The highest antioxidant capacity was obtained from hot air-50ºC treatment with 33.55±0.28 µmol TE/g dw while the lowest antioxidant capacity was determined in hot air drying at 70°C (4.48±0.49 µmol TE/g dw). Vega-Galvez et al. (2012) determined an increase in DPPH free radical scavenging activity of hot air dried peppers. Additionally, Priecina and Karklina (2014) reported an increment in antioxidant activity of some vegetables.

CONCLUSION

This study determined the effects of different hot air drying temperatures on drying characteristics, color, total phenolic content and antioxidant capacity of lemon balm leaves. Our results showed that, the fastest and the shortest drying times were obtained from 70 and 50°C, respectively. Among all applied mathematical models, Page, Modified Page and Logarithmic models were found to be the best models to describe the drying characteristics of lemon balm leaves. Fick's model of moisture diffusion fitted all experimental data with acceptable correlation coefficients. In the evaluation of color parameters, L^*, b^*, C^* and h^* values decreased while a^* value increased after drying. Total phenolic content and antioxidant capacity of dried lemon balm samples were both increased after drying. Among all samples, the total phenolic content and antioxidant capacity was obtained as the highest from 50°C treatment. In consequence, between hot air drying treatments, "50°C" was found applicable for lemon balm related with the enhanced bioactive properties.

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