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Modelling and characteristics of thin layer convective air-drying of thyme (*Thymus vulgaris*) leaves

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Abstract: Fruit and vegetable dehydration has been extensively studied for the improvement of food preservation. Effects of drying temperature on the drying kinetics of thyme were investigated and a suitable drying model was obtained to describe the drying process. Drying behaviour of thyme leaves at temperatures of 50, 60, 70 and 80°C was determined by using a conventional drying oven, and moisture ratio and drying rates were calculated. Four different thin layer drying models, namely Lewis, Henderson and Pabis, Page, and logarithmic models, were used to fit the experimental moisture ratio data. Three statistical parameters: coefficient of determination (R^2), chi-square (χ^2) and root mean square error (RMSE) were used to compare the goodness of fit of the drying models. Logarithmic model and Page model give the best description of the drying process kinetics of thyme leaves by comparing the experimental values and predicted values.

Keywords: activation energy; effective diffusivity; modelling; thin layer drying; thyme

Drying is one of the most commonly used preservation method which involves the removal of moisture from a food product while a simultaneous heat and mass transfer takes place (Onwude *et al.* 2016). It is applied to vegetables, fruits, herbs and other agricultural products to minimize microbial spoilage and deterioration reactions thus extending the shelf life (Chen *et al.* 2015). A hot air drying process is widely used in reducing the moisture content of agricultural products due to its low cost and ease of operation, but extended times of exposure to high temperatures cause a significant decrease in the product quality (Zielinska & Michalska 2016) microwave vacuum drying (MWVD). Therefore, it is required to control drying parameters in order to minimize chemical and physical changes in food material, and also to minimize nutritional losses. The drying rate during the operation is affected by the vapour pressures of air and food material, properties of air such as temperature and velocity, and properties of the food material such as thickness, surface area and moisture diffusion within the product (Muliterno *et al.* 2016).

Thyme (*Thymus vulgaris*) is a herb belonging to the family *Lamiaceae*, used for culinary and medicinal purposes. The plant was used for medicinal purposes in ancient times and currently it is used for its flavour as a cooking ingredient. Thyme also has anti-oxidative effects and its essential oils (thymol and carvacrol) show antimicrobial and inflammatory properties (Mandal & DebMandal 2016). The thyme plant blooms from June to July and is cultivated all around the world. Preservation of herbs is performed by two processes in general: drying and irradiation. The drying process is used to store seasonal plants all the year round, providing a high level of preservation (Lahnine *et al.* 2015).

Mathematical models that describe drying processes are used for improving drying systems or designing new systems. Models provide an improved understanding of the drying process parameters (Doymaz 2007). Information such as temperature and moisture

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is critical for drying process and equipment design, also for storage and handling procedures of the dried food material. This information can be provided by models that describe drying mechanisms (Özdemir & Devres 1999). Thin layer drying models are widely used in describing drying characteristics of food products. These models are classified into three groups, namely theoretical, semi-theoretical and empirical models. The most widely applied thin layer models are semi-theoretical and empirical models as these models consider external resistance to moisture transfer between food material and air, thus providing a better prediction of drying behaviour (Onwude *et al.* 2016). Empirical models neglect the fundamentals of the process and cannot show a clear view of the drying process, still may express the drying curve for the conditions of the experiment. Semi-theoretical models are derived by simplifying the general series solution of Fick's second law and they do not need any assumptions of geometry, diffusivity and conductivity of a food material (Özdemir & Devres 1999). Widely used semi-theoretical thin layer models are the Lewis model, Page model, Henderson and Pabis model, and a logarithmic model.

The objectives of the present paper were to determine the thin layer drying characteristics of thyme leaves at different drying temperatures, to obtain a suitable semi-theoretical thin layer drying model for the drying process and to determine the activation energy and effective diffusivity for the drying process of thyme leaves.

MATERIAL AND METHODS

Material preparation. Fresh thyme (*Thymus vulgaris*) samples used in this study were purchased from a local farmer's market in Istanbul, where goods were sold directly after harvest. Samples were not stored, and experiments began just after they had been obtained. Initial moisture content was determined by using a standard method at 70°C under vacuum for 12 h until constant weight was achieved and calculated as 89.8% (AOAC 2002). Fresh samples were first washed in a centrifugal vegetable washer-dryer and excess washing water was removed. The remaining washing water was removed by gently wiping the sample at room temperature. After the cleaning process, the leaves of thyme samples were picked off from the branches to obtain the edible portion. 10 ± 0.5 grams of thyme leaves were spread on glass petri dishes of 10 cm in diameter, just before the drying operation. The thickness of the material should be reduced to dimensions that will provide uniform distribution of drying air and temperature over the material thus making lumped parameters suitable for a thin layer drying concept (Onwude *et al.* 2016). Due to the natural thickness of thyme leaves when spread as one layer on dishes, it can be considered as a slab having uniformly distributed moisture.

Drying procedures. A conventional drying oven (Memmert UNB 400; Memmert GmbH Co. KG, Germany) was used at four different temperatures (50, 60, 70 and 80°C). Three replicates of samples were prepared for each temperature. The dishes were placed in a pre-heated oven and weight differences were measured at particular time intervals. Measurements were done at 10-min intervals for the first 150 min of drying. After 2.5 h of drying, measurements were done at 30-minute intervals until constant weight was obtained by three consecutive measurements for each replicate.

Modelling. Drying curves were obtained by plotting moisture ratio (*MR*) versus time and drying rate versus moisture ratio at different temperatures. The moisture ratio of thyme leaves during drying was calculated using the following Equation 1:

$$MR = \frac{M - M_e}{M_0 - M_e} \tag{1}$$

where: M – moisture content at any time; M_e – equilibrium moisture content; M_0 – initial moisture content (kg moisture/kg dry matter)

The drying rate was calculated via Equation (2):

$$\text{Drying rate} = \frac{M_{t+dt} - M_t}{dt} \tag{2}$$

where: M_t – moisture content at time t; M_{t+dt} – moisture content at time $t+dt$; t – drying time (min); dt – differential time (min)

Drying curves were fitted to four well-known thin layer drying models, namely Henderson and Pabis model, Lewis model, Page model and a logarithmic model. The Henderson-Pabis model is the first term of the general series solution of Fick's second law of diffusion (Henderson & Pabis 1961). The analytical solution of Fick's second law for infinite slab or sphere is given below:

$$MR = A_1 \Sigma_{i=1}^{\infty} \frac{1}{(2i-1)} exp\left[-\frac{(2i-1)^2 \pi^2 D_{eff} t}{A_2}\right] \tag{3}$$

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where: D_{eff} – effective diffusivity (m^2/s), A_1 and A_2 – geometric constants

The first term of the series solution can be used for sufficiently long drying times (Erbay & Icier 2010) and food drying is especially very complex because of the differential structure of products. In practice, a food dryer is considerably more complex than a device that merely removes moisture, and effective models are necessary for process design, optimization, energy integration, and control. Although modeling studies in food drying are important, there is no theoretical model which neither is practical nor can it unify the calculations. Therefore the experimental studies prevent their importance in drying and thin layer drying equations are important tools in mathematical modeling of food drying. They are practical and give sufficiently good results. In this study first, the theory of drying was given briefly. Next, general modeling approaches for food drying were explained. Then, commonly used or newly developed thin layer drying equations were shown, and determination of the appropriate model was explained. Afterwards, effective moisture diffusivity and activation energy calculations were expressed. Finally, experimental studies conducted in the last 10 years were reviewed, tabulated, and discussed. It is expected that this comprehensive study will be beneficial to those involved or interested in modeling, design, optimization, and analysis of food drying. (Erbay & Icier 2010). Geometric constants have different values depending on the product geometry, which are given in Table 1.

The first term of the equation can be written in a simplified form as below, which is referred to as the Henderson and Pabis model:

$$MR = a\ exp\ (-\ kt) \quad (4)$$

where: a – model constant (dimensionless); k – drying constant (h^{-1}); t – time (h)

The Lewis model is a special case of the Henderson and Pabis model where the food material is assumed to be thin enough and the air conditions are kept constant during operation (Lewis 1921). A model equation is given below:

$$MR = exp\ (-\ kt) \quad (5)$$

The Page model is an empiric modification of the Lewis model, where the constant n was added to the equation to increase accuracy (Page 1949):

$$MR = exp\ (-\ kt^n) \quad (6)$$

The logarithmic model is the modified form of the Henderson and Pabis model with the addition of an empirical constant (Yagcioglu *et al.* 1999):

$$MR = a\ exp\ (-\ kt) + c \quad (7)$$

where: c – dimensionless model constant

Table 1. Geometric constants according to the product geometry

Product geometry	A_1	A_2
Infinite slab	$8/\pi^2$	$4L^2$
Sphere	$6/\pi^2$	$4r^2$

L – thickness of the slab when the drying occurs on one side

The model parameters were evaluated by non-linear regression analysis with the Levenberg-Marquardt procedure in Statistica software (StatSoft Inc., USA). The goodness of fit of the curves was evaluated by using the coefficient of determination (R^2) primarily. Other criteria used to determine the most suitable drying model were root mean square error (RMSE) and reduced chi-square (χ^2). These values were calculated by using the equations below. The higher R^2 values and lower χ^2 and RMSE values indicate the goodness of fit (Doymaz 2006).

$$\chi^2 = \frac{\Sigma_{i=1}^{N} (MR_{exp,i} - MR_{pre,i})^2}{N - z} \quad (8)$$

$$RMSD = [\frac{1}{N} \Sigma_{i=1}^{N} (MR_{exp,i} - MR_{pre,i})^2]^{1/2} \quad (9)$$

where: N – number of observations; z – number of constants in the model; $MR_{exp,i}$ – experimental moisture ratio; $MR_{pre,i}$ – predicted moisture ratio at the i^{th} observation

Calculation of effective diffusivity and activation energy. Diffusion of moisture in solids during drying is a complex process. Surface diffusion, capillary flow, molecular diffusion and/or Knudsen flow take place along with drying. So, these phenomena are combined into one term, with a lumped parameter concept, namely effective moisture diffusivity (D_{eff}) (Erbay & Icier 2010)and food drying is especially very complex because of the differential structure of products. In practice, a food dryer is considerably more complex than a device that merely removes moisture, and effective models are necessary for process design, optimization, energy integration, and control. Although modeling studies in food drying are important, there is no theoretical model which neither is practical nor can it unify the calculations. Therefore the experimental studies prevent their importance in drying and thin layer drying equations are important tools in mathematical modeling of food drying. They are practical and give sufficiently good results. In this study first, the theory of drying was given briefly. Next, general modeling approaches for food drying were explained. Then, commonly used or newly developed thin layer drying equations were shown, and determination of the appropriate model was explained. Afterwards, effective moisture diffusivity and activation energy calculations were expressed. Finally, experimental studies conducted in the last 10 years were reviewed, tabulated, and discussed. It is expected that this comprehensive study will be beneficial to those involved or interested in modeling, design, optimization, and analysis of food drying (Erbay & Icier 2010). The first term of the diffusion equation (Equation 3) is used to calculate the effective diffusivity for a slab:

$$MR = \frac{8}{\pi^2} exp\left[-\frac{\pi^2 D_{eff} t}{4L^2}\right] \qquad (10)$$

$Ln(MR)$ versus time curve is plotted according to the equation, and the effective diffusivity can be calculated via the slope of the curve (k) as below:

$$k = -\frac{\pi^2 D_{eff}}{4L^2} \qquad (11)$$

Temperature has a significant effect on the effective diffusivity. This effect can be described by the Arrhenius equation:

$$D_{eff} = D_0\, exp\left(-10^3\ \frac{E_a}{R(T + 273.15)}\right) \qquad (12)$$

where: D_0 – Arrhenius factor; E_a – activation energy for diffusion (kJ/mol); R – universal gas constant (kJ/kmol·K). The slope of the $ln(D_{eff})$ versus $[1/(T + 273.15)]$ straight line is equal to $(-10^3 \times E_a/R)$ according to the equation.

RESULTS AND DISCUSSION

Drying curves. Thin layer drying operations were carried out at four different temperatures (50–80°C), moisture loss values were calculated after constant weight was obtained. The change of moisture ratio with time and the change of drying rate with moisture ratio were expressed. Moisture ratio and drying rate changes during the drying period of thyme leaves are given in Figures 1 and 2. As can be seen in figures, the drying rate increases significantly as the temperature rises from 50 to 80°C, and the drying time declines proportionally. It is also seen that the constant rate drying period does not exist. Drying rate continuously decreases with time. Similar results were reported for agricultural products such as red pepper (Akpinar *et al.* 2003), mint leaves (Doymaz 2006) and pumpkin (Hashim *et al.* 2014). The falling rate behaviour of drying rate indicates that diffusion is a dominant physical mechanism for the movement of moisture during the drying process (Onwude *et al.* 2016). Moisture ratio and drying rate deviations for three replicates are given as error bars in the figures.

As shown in Figure 2, the drying rate increases with an increase of drying temperature and the highest rate of drying was reached at 80°C as expected. Also the drying rate decreases with time as the moisture ratio drops. The duration of drying operation to reduce the moisture content to a desired level is directly

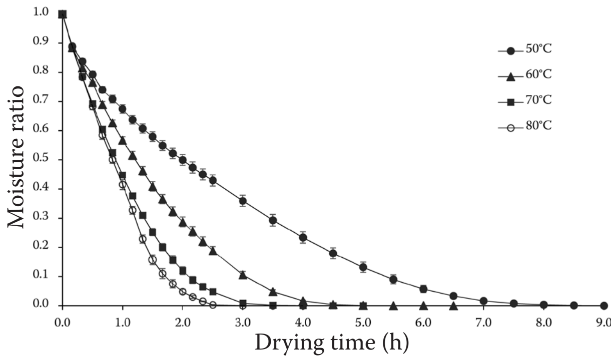

Figure 1. Change of moisture ratio with time at different temperatures

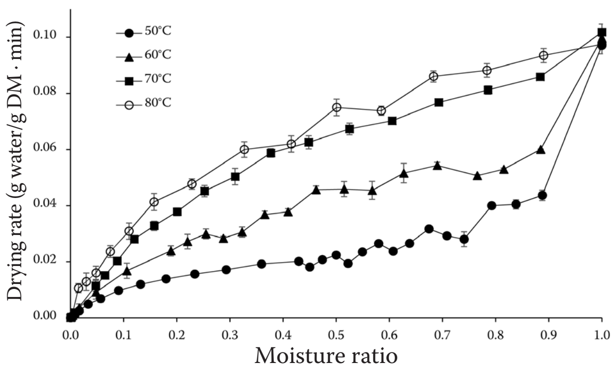

Figure 2. Change of drying rate as a function of moisture ratio

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dependent on drying temperature. Many researchers suggested corresponding results for food materials such as gundelia (Evin 2011), araticum (Botrel *et al.* 2016) and pumpkin (Hashim *et al.* 2014).

Modelling of drying curves. It is a matter of great importance to effectively model the drying behaviour of thyme leaves in order to determine the moisture content as a function of drying time. Widely used semi-theoretical thin layer drying model equations were fitted to the experimental data to obtain the most accurate model. The values of the coefficient of determination were calculated to evaluate the goodness of fit. Statistical criteria to evaluate the goodness of fit: coefficient of determination (R^2), root mean square error (RMSE) and chi-square (χ^2), and the drying model constants *k*, *a*, *n* and *c* are given in Table 2.

The drying models presented high coefficients of determination (R^2), ranging from 0.9644 to 0.9976. It can be said that all the models applied in this study can describe the characteristics of drying. The lowest values were observed at 80°C, and with the Lewis model.

As presented in Table 2, the values of R^2 obtained from Page and logarithmic models were higher than the others. The logarithmic model showed higher R^2 values at lower temperatures (50 and 60°C) while the Page model had higher values of R^2 at higher temperatures (70 and 80°C). Comparing the RMSE and chi-square values, identical conclusions can be made for R^2 values. As stated above, the Page model showed a higher χ^2 value at 50°C and the logarithmic model showed a higher χ^2 value at 80°C, which means a deviation from the model. Experimental moisture ratios were compared with the calculated values obtained from the models and are shown in Figures 3 and 4 to visualize the fit of the models. Comparison of the calculated values of the Page model with the experimental data is shown in Figure 3, and the comparison of the calculated values of the logarithmic model with the experimental data is documented in Figure 4. As seen in the figures, both models show a good fit to the experimental values. Considering higher temperatures, the Page model shows a better fit to actual data while the logarithmic model

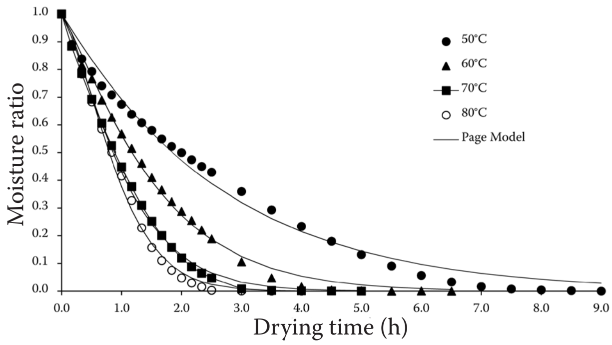

Figure 3. Comparison of experimental data and calculated moisture ratios via Page model

Table 2. Statistical criteria and model constants for different models

Model	Temperature (°C)	Model constants				Statistical parameters		
		k	*a*	*n*	*c*	R^2	RMSE	χ^2
Lewis	50	0.37883				0.9879	0.0335	0.00117
	60	0.62701				0.9892	0.0323	0.00109
	70	0.90250				0.9836	0.0401	0.00169
	80	1.03823				0.9644	0.0618	0.00403
Henderson and Pabis	50	0.36892	0.97974			0.9886	0.0327	0.00115
	60	0.64781	1.02972			0.9902	0.0306	0.00102
	70	0.95418	1.05813			0.9872	0.0354	0.00139
	80	1.12155	1.08788			0.9722	0.0547	0.00334
Page	50	0.36691		1.03353		0.9883	0.0330	0.00117
	60	0.57377		1.17399		0.9957	0.0204	0.00045
	70	0.84466		1.27706		0.9976	0.0155	0.00026
	80	0.97639		1.46334		0.9954	0.0221	0.00055
Logarithmic	50	0.28559	1.05839		–0.10993	0.9966	0.0178	0.00035
	60	0.55690	1.07554		–0.06567	0.9959	0.0199	0.00045
	70	0.83065	1.09909		–0.05898	0.9928	0.0266	0.00083
	80	0.90416	1.15851		–0.09706	0.9840	0.0414	0.00204

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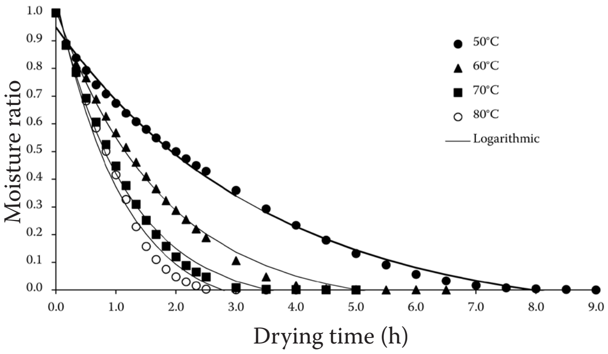

Figure 4. Comparison of experimental data and calculated moisture ratios via logarithmic model

shows a slightly better fit at lower temperatures. Consequently, it can be said that the Page and the logarithmic models can present the thin layer drying behaviour of thyme leaves.

Various studies show the goodness of fit of the Page model and the logarithmic one for different food materials. A study conducted on pumpkin showed that both the Page model and the logarithmic model had a good fit to the experimental values; where also the Page model had a better fit at higher temperatures (60 and 70°C) and the logarithmic model had a slightly better fit at lower temperatures (40 and 50°C) considering the R^2 values (Guiné *et al.* 2011). Also, the Page model showed a good fit for the drying of olive cake at different temperatures except at 50°C (Akgun & Doymaz 2005) and both the Page and logarithmic models were found to be suitable for the drying of banana slices (Doymaz 2010).

Effective diffusivity and activation energy. Effective diffusivities were calculated for the thin layer drying of thyme leaves and are given in Table 3. Values of D_{eff} ranged between 2.59×10^{-9} and 1.28×10^{-8} m^2/s for 50–80°C. Studies conducted on the thin layer drying kinetics of fruits and vegetables show that effective diffusivity varies between 10^{-12} and 10^{-6} m^2/s, and for most samples (80%) D_{eff} value is in the range of 10^{-11} to 10^{-8} m^2/s (Onwude *et al.* 2016). As shown in Table 3, D_{eff} values increased highly with increasing temperature, as reported in literature. An increase in the effective diffusivity with increasing temperature was observed in various studies conducted on different food materials, such as carrot (Kumar *et al.* 2012), jackfruit (Saxena & Dash 2015) and green peas (Pardeshi *et al.* 2009).

Table 3. Effective diffusivity values for drying of thyme leaves at different temperatures

Temperature (°C)	Effective diffusivity (m^2/s)
50	2.59×10^{-9}
60	5.18×10^{-9}
70	7.69×10^{-9}
80	1.28×10^{-8}

As presented above, the temperature has a significant effect on effective diffusivity and this effect is described by the Arrhenius equation (Equation 12). From this statement, the activation energy for the thin layer drying of thyme was calculated as 21.40 kJ/mol. The activation energy shows the sensibility of effective diffusivity to temperature, where higher E_a indicates higher sensibility to air temperature. The activation energy for the drying of thyme was found to be lower than in most of the other food products studied, such as basil (33.21 kJ/mol) (Kadam *et al.* 2011), mint (82.93 kJ/mol) (Jin Park *et al.* 2002) and pumpkin (35.6 kJ/mol) (Perez & Schmalko 2009).

CONCLUSIONS

Drying is one of the most important steps in food processing, thus it is critical to reveal the characteristics during the drying period of materials. Thin layer drying of thyme leaves was investigated in this study. Drying curves were obtained to explain the drying process, and the temperature seemed to be largely influential on the process. The drying operation occurred in the falling rate period. Widely used drying models were studied, while the Page model and the logarithmic model showed a better fit to the experimental data regarding the high coefficient of determination. Effective diffusivities were calculated for the drying of thyme leaves, and they were found to be in the range of 2.59×10^{-9} to 1.28×10^{-8} m^2/s for different temperatures and D_{eff} values were dependent on the temperature change. The relation of effective diffusivity with temperature was defined by the Arrhenius equation and the activation energy was found as 21.40 kJ/mol.

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