MODELING OF THIN-LAYER KINETICS AND COLOR CHANGES OF APPLE SLICES DURING FAR-INFRARED VACUUM DRYING

by

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In this study far infra-red vacuum drying kinetics and change of color quality parameters of apple slices were analyzed. The experimental data set of drying kinetics was obtained on the experimental setup designed to imitate an industrial far infra-red vacuum dryer. For approximation of the experimental data with regard to the moisture ratio four well known thin-layers drying models from scientific and engineering literature and new developed model of Mitrevski et al., were used. The performed statistical analyzes show that the model of Mitrevski et al., has the best statistical performance than well-known thin-layer drying models. The estimated values of moisture diffusivity of apple obtained from this study are within the range from $2.80\cdot10^{-11}$ *to* $1.70\cdot10^{-10}$ $m^2\ s^{-1}$*. A negative effect on the total color change of far infra-red vacuum dried apple slices was observed with increasing of temperature of infrared heaters and vacuum pressure.*

Key words: *far infra-red vacuum drying, apple, color change*

1. Introduction

In recent few decades the consumers' demand evolving in the direction of dry food materials that keep their original characteristics to a higher degree i.e. to dry materials with high sensorial and microbiological quality. Convective drying is the most used method for the production of dried fruits and vegetables. The main disadvantages of this classical method of drying are [1]: the material is exposed at high temperature for a long time during the contact with hot air which is the reason for decreases on nutritive values and color change, shrinkage and low dehydration capacity of the dried material, structure and flavor changes during drying. In recent years, far-infra red drying is popular method for thin-layer drying of various food materials [2-4]. In comparison with convective drying the method of far infra-red drying has more advantage as: high energy efficiency, uniform heating of material, acceleration of drying time and improved dried product quality [5], minimization of color

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change and shrinkage of dried materials [6]. According to Nimmol fact is that infrared radiation accelerates the drying process, but heat sensitive materials as agricultural and foods materials could be damaged or degraded along with the quality decreasing, if radiation intensity is not properly applied [7]. The effects of far infra-red drying method can improve the quality and nutritional value of dried materials if was used in conjunction with vacuum drying. With combination on the advantages of both drying methods, energy efficiency of the drying processes is enhanced and degradation of dried product quality is reduced [8]. In scientific and engineering literature, several researchers investigated far-infrared vacuum drying of various fruits and vegetables [9-15]. But, limited study of drying kinetics and change of color parameters of far infra-red vacuum dried materials at high value of temperature of infrared heaters from 120 to 200 ^{o}C and vacuum pressure from 20 to 80 kPa were conducted. So, the objectives of this study were: (i) research drying kinetics of far-infrared vacuum drying apple slices and development of new thin-layer drying model, (ii) estimate to moisture diffusivity of apple slices and compared estimated values with the values published in scientific and engineering literature by other authors (iii) research of the influence of boundary conditions on total color changes of dried apple slices.

2. Material and methods

2.1. Sample preparation

Fresh apples variety Golden Delicious was used as raw material in the experimental part of the research. Prior to processing, the apples were stored in a cold chamber at a temperature of 4^{o}C and a relative air humidity of 75%. The apples were washed, peeled and sliced with a slicer machine in order to obtain uniform samples. The spherical samples with diameter 43±10^{-1} mm and thickness of 3±10^{-1} mm, obtained from the central medulla region where the cell structure is more uniform, were used in the drying experiments. Several measurements were made using a caliper, and only the samples with a tolerance of ±2 % were used in the experimental part of research.

The apple slices were dried in a far-infra red vacuum set-up [15]. The experimental conditions were chosen such as that of temperature of infrared heaters and vacuum pressure were in the range to be used on the industrial dryer. The effect of temperature of infrared heaters 120, 140, 160, 180 and 200^{o}C, and vacuum pressure 20, 40, 60, 80 kPa on the drying kinetics of apple slices were investigated from [15]. After each experiment the dried samples were stored in an air tight paper packet for further analysis. The transient temperatures of drying samples were measured with thee micro-thermocouples placed in each of the mid-plane of the drying samples. The measurement of samples mass changes with time was enabled with load cell type OMEGA LCL-040 (Omega, Inc., USA), which was connected to data acquisition system. The temperature and mass changes of dried samples were registered on the personal computer. The initial moisture content of the samples was obtained according to the AOAC method no. 934.06 (AOAC 1995). The experiments were carried out in triplicate and an arithmetic average value was used for data processing. The drying experiments were performed until obtaining the moisture content of dry apple samples of 0.04 kg kg^{-1}.

2.2. Drying kinetics

The experimental moisture content data obtained at different temperature of infrared heaters and vacuum pressure were converted to the moisture ratio, MR using the equation

$$MR = (u_\tau - u_{eq})/(u_0 - u_{eq}) \tag{1}$$

were MR is the dimensionless moisture, ratio, u_τ, u_0 and u_{eq} are moisture content at any time, initial moisture content and equilibrium moisture content. Because of the values of equilibrium moisture content, u_{eq} are relatively small compared to those of moisture content of material, u_τ or initial moisture content, u_0 the error involved in the simplification is negligible. Thus moisture ratio was then calculated as

$$MR = u_\tau / u_0 \tag{2}$$

2.3. Calculation of moisture diffusivity

The moisture diffusivity of foods is very often considered as an Arrhenius-type temperature function [16, 17]

$$a_m = a_{m0} \exp[-E_0/(R\,T)] \tag{3}$$

where: a_{m0} is the Arrhenius factor, E_0 is the activation energy for moisture diffusion, R is the ideal gas constant, and T is the absolute temperature. In this study the values of moisture diffusivity were estimated on the basis of an accurate and easy to perform single thermocouple temperature measurement by using an inverse approach [16, 17]. An arithmetical mean of the readings from the three thermocouples inserted in the mid-palate position separately of each of the three drying slices of apple was used as a transient temperature reading [16, 17].

The estimation methodology is based on the minimization of the ordinary least square norm

$$E(\mathbf{P}) = [\mathbf{Y} - \mathbf{T}(\mathbf{P})]^T[\mathbf{Y} - \mathbf{T}(\mathbf{P})] \tag{4}$$

Here, $\mathbf{Y}^T = [Y_1, Y_2, \ldots, Y_{imax}]$ is the vector of measured temperatures, $\mathbf{T}^T = [T_1(\mathbf{P}), T_2(\mathbf{P}), \ldots T_{imax}(\mathbf{P})]$ is the vector of estimated temperatures at time τ_i (i = 1, 2, …, imax), $\mathbf{P}^T = [P_1, P_2, \ldots P_N]$ is the vector ofunknown parameters, imax is the total number of measurements, and N is the total number of unknown parameters (imax ≥ N). For the minimization of E(**P**) representing the solution of the present parameter estimation problem the Levenberg-Marquardt method were utilized [16, 17].

2.4. Color measurement and kinetics on color changes

The colour changes in food materials during drying are due to degradation of pigments (chlorophylls, carotenoids, anthocyans, and betalaines), Maillard reactions or enzymatic browning [18]. In order to research the effect of temperature of infrared heaters and vacuum pressure on the color changes of dried apple, a three-filter colorimeter Konica Minolta CR-400 (Konica Minolta Sensing Americas, Inc., USA) was used. This instrument shows the quantitative parameters of color change in different systems. The measured values of the color parameters are represented in the CIE, L*, a*, b*, color space. In this color space, L* values are used as an indicator of lightness/darkness (ranges from 0 to 100), coordinate a* to indicate chromaticity on a green (-) to red (+) axis (ranges from -120 to 120), and coordinate b* to indicate chromaticity on a blue (-) to yellow (+) axis (ranges from -120 to 120). The color measurements were conducted before and after far infra-red vacuum drying. Five measurements were performed for each sample which is selected randomly. The values used for total color change calculations encompassed the average values of L*, a* and b* of each sample. The total color change, ΔE is calculated on the basis of the equation [19]:

$$\Delta E = \sqrt{(L^* - L_0^*) + (a^* - a_0^*) + (b^* - b_0^*)} \qquad (5)$$

where: L_0^*, a_0^*, b_0^*, are initial values for lightness, redness and yellowness.

In order to determine the rate of color changes during drying of far infra-red vacuum drying, the kinetics of the parameters of lightness, L*, redness, a* and yellowness, b*, were investigated. For approximation of the total color changes, ΔE data during far-infrared vacuum drying process new developed model were used

$$\Delta E = A + B/t_h + C \cdot p^2 \qquad (6)$$

where: A, B, C, are parameters, t_h, temperature of infrared heaters and p, vacuum pressure. In this study multiple regression relationship between dependent variable, ΔE and independent variables, temperature of infrared heaters and vacuum pressure were used.

3. Results and discussion

3.1. Fitting of drying curves

In the most number existing thin-layer drying models which is used to for approximation experimental data of drying kinetics the effect of boundary conditions on the empirical parameters it is not take into account. It that reasons in this study the new generalized thin-layer drying model was developed. For approximation of experimental data on the drying kinetics of apple slices five thin-layer mathematical models from scientific literature and new developed model of Mitrevski *et al*. were used (Table 1).

Table 1. Thin-layer drying models

Model	Equation	Name of model	References
M01	$MR = A\exp(-k_1\tau^m)+B\tau$	Midilli	[20]
M02	$MR= A\exp(-k_1\tau)+(1-A)\exp(-k_1B\tau)$	Diffusion approach	[21]
M03	$MR = A\exp(-k_1\tau)+(1-A)\exp(-B\tau)$	Verma	[22]
M04	$MR = A\exp(-k_1\tau+B\tau^{0.5})+C$	Jena and Das	[23]
M05	$MR = A\exp(-k_1B\tau)/(1/p^C)(1/t_h^D)$ $+(1-A)\exp(-k_1B\tau)^{**}(1/p^E\ 1/t_h^F)$	Mitrevski *et al*.	This study

$MR = M_\tau/M_0$ moisture ratio, A, B, C, D, E, F - parameters, k_1 - drying rate constant [min^{-1}],
τ - drying time [min^{-1}], t_h - temperature of infrared heatres [^oC], p - vacuum pressure [kPa]

The method of multiple indirect non-linear regression and estimation methods of: Quasi-Newton, Simplex, Simplex and quasi-Newton, Hooke-Jeeves pattern moves, Hooke-Jeeves pattern moves and quasi-Newton, Rosenbrock pattern search, Rosenbrock pattern search and quasi-Newton, Gauss-Newton and Levenberg-Marquardt from computer program StatSoft Statistica (Statsoft Inc., Tulsa, OK, http://www.statsoft.com), were used to for calculation of the statistical parameters and estimate the parameters in the given models. Because the regression method, estimation method, the initial step size, the start values of parameters, convergence criterion and form of the function have significant influence on accuracy of estimated parameters [24], a large number of numerical experiments were performed. On the basis of thin-layer data and each model from (Table. 1), the values of: coefficient of determination, R^2, root mean squared error, RMSE and mean relative deviation, MRD, were calculated. When the value for coefficient of determination obtained from different estimation methods was different, the greatest value was accepted as relevant. In order to estimate and select the best thin-layer drying model the value of performance index, ϕ and chi-squared, χ^2 were calculated

$$\phi = \frac{R^2}{RMSE \cdot MRD} \quad (7)$$

$$\chi^2 = z_1^2 + z_2^2 \quad (8)$$

where: z_1 and z_2 are individual statistics for testing of the population of skewness and kurtosis [25].

The best model that is describing the thin-layer drying characteristics apple slices has to be chosen on the basis of higher, ϕ and χ^2 the value lower than tabled critical 0.05 chi-square value $\chi^2_{0.05} = 5.99$ for degrees of freedom, df = 2 (Table 2).

Table 2. Statistic summary of the regression analysis

Model	R^2	RMSE	MRD	ϕ	χ^2	Rank
M01	0.9393	0.1170	1.8162	4.4197	4.6435	5
M02	0.9394	0.1166	1.7277	4.6620	4.5585	3
M03	0.9394	0.1166	1.7197	4.6836	4.5413	2
M04	0.9394	0.1166	1.8089	4.4512	4.6225	4
M05	0.9942	0.0363	0.8602	31.869	1.1539	1

R^2 - coefficient of determination, RMSE - root mean squared error, MRD - mean relative deviation, ϕ - performance index, χ^2 - chi-squared value

From Table 2 it is evident that model M05 i.e. new developed model, has the highest value of performance index, ϕ = 31.869, (rank 1) in comparison with the other models. Also it can be seen that all models have smaller average value of, χ^2 than tabled critical chi-square value. In accordance with statistical criteria, the new developed model of Mitrevski *et al.*, is able to correlate the experimental values of drying kinetics of apple slices with 3.63% root mean squared error. The estimated values of parameters in new generated model of Mitrevski *et al.*, are presented in Table 3.

Table 3. The values of estimated parameters

A	K_1	B	C	D	E	F
-0.0004	0.0054	0.0052	-0.0872	1.3645	0.0253	-1.2991

The analysis of variance (ANOVA) results indicated that the temperature of heatres ($p \leq 0.001$), vacuum pressure ($p \leq 0.05$), and the interaction of the temperature of heatres and vacuum pressure ($p \leq 0.05$) significantly affected the drying time of apple slices.

In fig. 1, the experimental and predicted values of moisture ratio with drying time at temperature of infrared heaters 120, 140, 160, 180 and 200°C for apple slices at vacuum pressure of 20 kPa are shown.

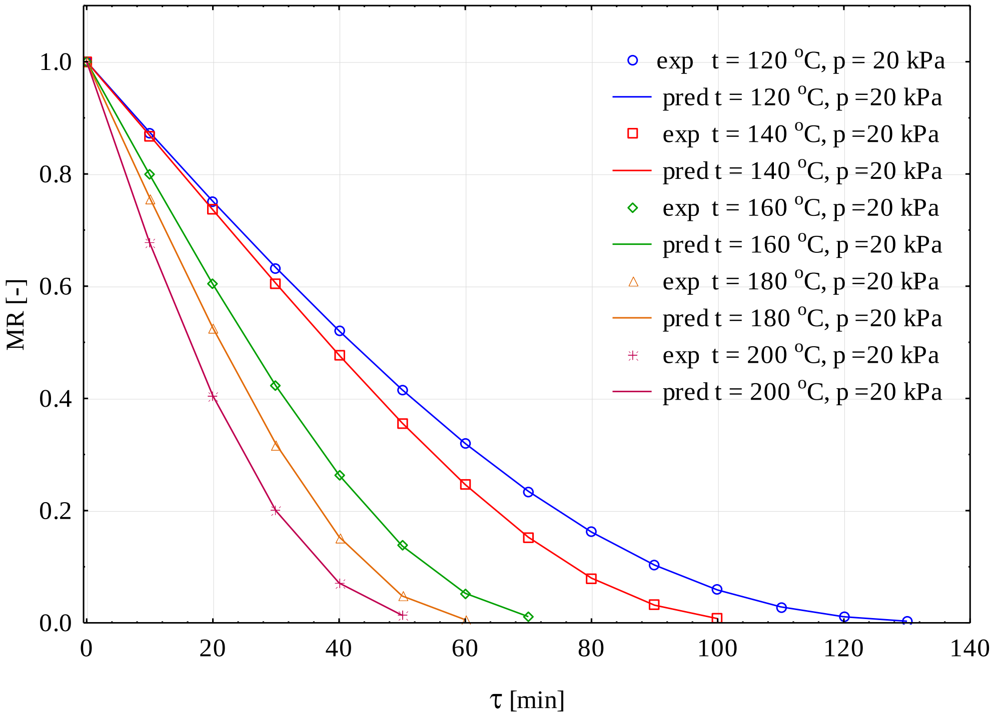

Fig. 1. Experimental and predicted moisture ratio values by the Mitrevski et al. model for different temperatures of infrared heaters at vacuum pressure 20 kPa

From fig.1 is evident that has a good agreement between the experimental and predicted values of moisture data of apple slices.

3.2. Moisture diffusivity

The values of the parameters of effective moisture diffusivity, a_{m0}, and activation energy E_0 are determined by application of inverse approach. In Table 4 the initial guesses of parameters and computationally obtained values of parameters using the Levenberg-Marquardt method and RMS-error for the one of real realized experiment E17 (t_h = 180 °C, p = 40 kPa, $2L_0$ = 3 mm, u_0 = 5.24 kg/kg and t_0 = 24.82 °C), [26] are shown.

Table 4. Estimated values of unknown parameters and RMS-error

	$a_{m0} \cdot 10^3$ [$m^2 s^{-1}$]	E_0 [kJ mol^{-1}]	RMSE
Initial guess	0.274	38.50	7.660
Estimated values	0.085	36.40	0.710

a_{m0} - Arrhenius factor, E_0 - Activation energy, RMSE - root mean squared error

The estimated values of moisture diffusivity of apple obtained from this study are within the range from $2.80 \cdot 10^{-11}$ to $1.70 \cdot 10^{-10}$ m^2 s^{-1}. These values are comparable with values for other dried food materials which values generally were reported in scientific literature, from 10^{-13} to 10^{-6} $m^2 s^{-1}$ [27, 28]. The determined values of moisture diffusivity in this study showed that the higher values of temperature of infrared heaters and lower value of vacuum pressure is better to dry the apple slices in the selected experimental domain, because the obtained values of moisture diffusivity are relatively higher.

3.3. Color change

In Table 5 the average value changes in lightness, L*, redness, a*, yellowness, b* and total color changes, ΔE, of apple slices for different value of temperature of infrared heaters and vacuum pressure were presented.

Tableb 5. The effect on drying conditions on changes of color parameters

t_h	p	a*	b*	L*	ΔE
120	20	2.41±3.81	22.23±9.36	81.07±2.09	8.67
140	20	2.98±3.84	22.29±9.78	73.01±3.26	13.10
160	20	4.14±3.60	23.40±10.66	71.73±3.09	14.85
180	20	4.98±3.84	23.50±19.40	70.03±3.26	16.67
200	20	5.56±4.16	23.95±11.36	68.99±3.19	17.87
120	40	2.87±3.76	22.66±9.40	80.15±3.26	9.32
140	40	3.41±3.60	23.11±10.36	71.12±3.39	14.78
160	40	4.61±3.84	23.67±10.78	70.66±3.56	15.98
180	40	5.33±3.94	24.41±11.32	69.16±3.36	17.66
200	40	5.99±4.60	24.89±11.56	67.22±3.89	19.62
120	60	3.13±3.92	22.91±10.36	77.42±3.09	10.56
140	60	3.89±3.84	23.35±11.40	69.68±3.26	16.22
160	60	4.98±3.60	23.97±11.36	68.09±3.49	18.19
180	60	5.88±4.84	24.59±11.40	67.30±3.56	19.44
200	60	6.34±3.73	25.32±11.66	66.34±4.09	20.58
120	80	3.41±3.64	23.22±10.40	73.44±3.26	13.13
140	80	4.24±3.60	23.66±11.36	66.32±3.39	19.17
160	80	5.21±3.84	24.44±12.40	65.21±3.46	20.70
180	80	6.11±4.14	25.24±13.40	64.89±3.66	21.58
200	80	6.67±4.60	25.67±13.56	63.32±4.49	23.32

t_h - heaters temperature, p - vacuum pressure, a* - redness, b* - yellowness, L* - lightness,
ΔE - total color change, mean ± standard deviation

From Table 5 it is evident that, the L* value of dried apple slices decreased during drying. The, L* decreased from initial value Lo* = 83.32 to L* = 63.32, when apple slices are dried at 200°C and 80 kPa. The changes in L* parameter value was less at lower temperature of infrared heaters and lower vacuum pressure. As temperature of infrared heaters increased from 120 to 200°C and vacuum pressure changes from 20 to 80 kPa the lightness of apple slices decreased from 81.07 to 63.32. The decrease in L* value was in correlation with non-enzymatic browning reactions which accelerates at high temperatures. The reduction in L* value may be attributed to intense browning reaction and increase crust formation due to exposure to high temperature [29]. Because the lightness is a very important color quality parameter, lower temperature of infrared heaters and lower vacuum pressure are preferable to preserve of dried apple slices. The temperature of infrared heaters and vacuum pressure also have an effect on the redness parameter, a*. From instance, a* value increased from initial value a_0* = -6.16 to a* = 6.67 at the end point when apple slices were dried at 200°C and 80 kPa. As shown in Table 5 the yellowness of dried slices increased during drying from 2.41 to 6.67. The values of yelowness, b* increased from initial value b_0* = 21.86 to b* = 25.67 when apple slices were dried at 200°C and 80 kPa. As the temperature of infrared heaters increased from 120 to 200°C and value of vacuum pressure varied between 20 to 80 kPa the yellowness of apple slices increased from 22.23 to 25.67. Similar effects on the drying kinetics on changes of color parameters were reported for banana [10].

From Table 5 obviously that ΔE values, increased during drying apple slices and color changes intensity is more intense at higher temperature of infrared heaters and higher vacuum pressure. The total color change, ΔE values increased from 8.67 to 23.32, when apple slice are dried at temperature of heaters from 120 to 200 °C and vacuum pressure from 20 to 80 kPa. Similar results were reported from far infra-red vacuum drying of peach [6] and kiwi [12].

For estimation of the statistical parameters in the model of total color change (eq.5), the method of multiple indirect non-linear regression and estimation method of Quasi-Newton, from computer program StatSoft Statistica (Statsoft Inc., Tulsa, OK, http://www.statsoft.com), were used. In Table 6 the estimated values of parameters from the mathematical model with equation (5) are presented.

Table 6. The values of estimated parameters (eq.5)

A	B	C
32.264	-2.838.4	0.0009

In fig.2 the total colour change as functions of temperature of infrared heaters at different vacuum pressure was presented. The high value of coefficient of determination, R^2 = 0.9821 and small value of root mean squared error, RMSE = 0.062 showed that the total color change, ΔE during far infra-red vacuum drying apple slices could be modeled by new generated model of Mitrevski *et al.*

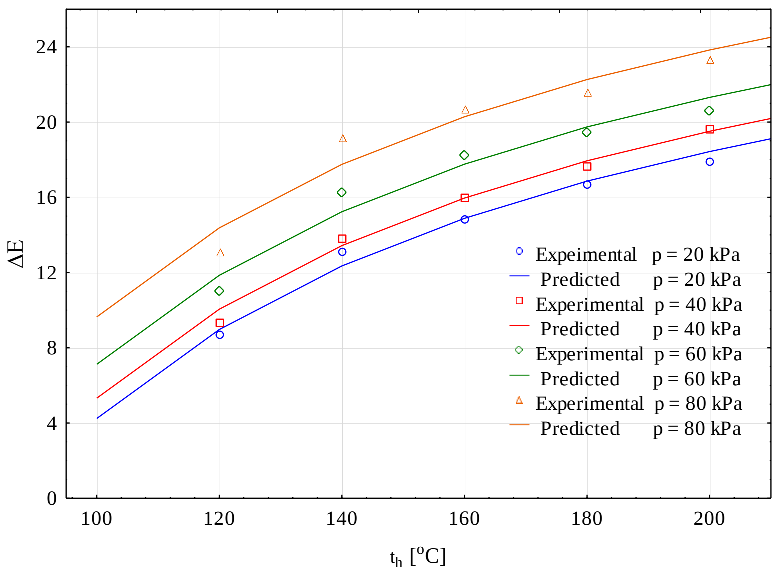

Fig. 2. Effect of temperature of infrared heaters on total color change at different vacuum pressure

4. Conclusions

In the present study the effects of temperature of infrared heaters and vacuum pressure on the drying kinetics and color quality of apple slices were examined. The experimental drying data in terms of moisture ratio were approximated with four thin-layer drying models from scientific literature and new generated model of Mitrevski *et al*. According to the statistical evaluation, the new developed model of Mitrevski *et al*., has the best statistical performances than other literature thin-layer drying models. The estimated values of moisture diffusivity for apple obtained by inverse approach are in the range from $2.80 \cdot 10^{-11}$ to $1.70 \cdot 10^{-10}$ $m^2\ s^{-1}$ and are comparable with values for other dried food materials. This study verified that the boundary conditions on the surface of dried apple slices (the temperature of infrared heaters and vacuum pressure) have a strong effect on the change of color parameters (L*, a*, b* and ΔE). The high temperature of infrared heaters and high value of vacuum pressure has a negative effect on the total color change of dried apple slices. For the preserved nutritional values of dried apple slices the optimum drying conditions for combined far infra-red vacuum drying is 120 °C and 20 kPa. The new developed thin-layer drying model and the results of kinetics on color changes presented in this study will find the technological application on far infra-red vacuum drying for preservation of other food materials.

Nomenclature

A,B,C,D,E,F	- parameters
a*	- redness
a_m	- moisture diffusivity, [$m^2\ s^{-1}$]
a_{m0}	- Arrhenius factor, [$m^2\ s^{-1}$]
b*	- yellowness
E_0	- activation energy, [$J\ kg^{-1}$]
k_1	- drying rate constant, [min^{-1}]
L*	- lightness
MR	- moisture ratio [-]
MRD	- mean relative deviation
P	- vector of unknown parameter
R	- absolute gas constant, [$J\ K^{-1}\ mol^{-1}$]
R^2	- coefficient of determination
RMSE	- root mean squared error
p	- pressure, [Pa]
t	- temperature, [°C]
T_k	- temperature, [K]
T	- vector of estimated temperature, [°C]
Y	- vector of measured temperature, [°C]
u	- moisture content, [$kg^{-1}\ kg^{-1}$ db]
z_1, z_2	- individual statistics for testing of the population of skewness and kurtosis

Greek symbols

ΔE	- total color change
ϕ	- performance index
τ	- time, [s]
χ^2	- chi-squared value

Subscripts

eq	- equlibrium
h	- heater
0	- initial

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