



Convective drying of regular mint leaves: analysis based on fitting empirical correlations, response surface methodology and neural networks

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ABSTRACT. In the present work, an analysis of drying of peppermint (*Mentha x villosa* H.) leaves has been made using empirical correlations, response surface models and a neural network model. The main goal was to apply different modeling approaches to predict moisture content and drying rates in the drying of leaves, and obtaining an overview on the subject. Experiments were carried out in a convective horizontal flow dryer in which samples were placed parallel to the air stream under operating conditions of air temperatures from 36 to 64°C, air velocities from 1.0 to 2.0 m s⁻¹ and sample loads from 18 to 42 g, corresponding to sample heights of 1.4, 1.7 and 3.5 cm respectively. A complete 3³ experimental design was used. Results have shown that the three methodologies employed in this work were complementary in the sense that they simultaneously provided a better understanding of leaves drying.

Keywords: *Mentha x villosa* H, kinetic parameters, convective drying, moisture content.

Secagem convectiva de folhas de hortelã comum: análise com base no ajuste de correlações empíricas, da superfície de resposta e redes neurais

RESUMO. No presente trabalho foi analisada a secagem de folhas de hortelã (*Mentha x villosa* H.) por correlações empíricas, superfícies de resposta e de rede neural. Pretendeu-se com esta análise testar diferentes alternativas de modelos para prever o conteúdo de umidade e as taxas de secagem das folhas, obtendo-se por estes métodos uma visão mais completa da secagem das folhas. Os dados experimentais foram obtidos em um secador convectivo com escoamento horizontal, paralelo à amostra, variando-se a temperatura do ar no intervalo de 36 a 64°C, a velocidade do ar entre 1,0 a 2,0 m s⁻¹, com massa das amostras de 18 a 42 g, correspondendo a alturas de 1,3, 1,7 e 3,5 cm, respectivamente. Utilizou-se um planejamento fatorial completo 3³. Os resultados indicaram que a análise através dos três métodos foram complementares e para um material como as folhas é conveniente utilizá-los simultaneamente.

Palavras-chave: *Mentha x villosa* H, parâmetros cinéticos, secagem convectiva, teor de umidade.

Introduction

Mentha is the most important genus of the Lamiaceae family because it contains numerous essential oils of high economic value. Originated from Europe, regular mint (*Mentha x villosa* L.) is a hybrid from the crossing between *Mentha spicata* L. and *Mentha suaveolens* Ehrh, which is widely cultivated throughout Brazil, where it is used as traditional drug to treat amebiasis, giardiasis and schistosomiasis (SOUSA et al., 2009). The plant has many different pharmacological activities, namely antiparasitic and central nervous system depressant, tranquilizer, anti-hypertensive and bradycardizing and analgesic, explaining its popular use in stomach disorders and menstrual pain (MARTINS et al., 2007). The essential oil extracted from *Mentha x*

villosa leaves has an oxygenated monoterpene, the piperitenone oxide as a major constituent. Some other constituents including monoterpenoids, sesquiterpenoids, triterpenoids and steroids were also identified (MARTINS et al., 2007). Fresh and dry leaves are also used for cooking and to prepare infusions due to their aromatic properties.

In spite of their potential use in food, cosmetic and pharmaceutical industries, the production of these leaves on a commercial scale depends on effective techniques to preserve leaves and their constituents. Hot air drying is a widespread preserving technique, but the quality of dried herbs depends on the drying technology and drying conditions, and the temperature is a key parameter to be controlled (CHEN; MUJUMDAR, 2006). Since most active compounds in the leaves and in

their oils are heat-sensitive, a very high temperature may cause a loss of active ingredients, while a very low temperature may accelerate decomposition by promoting enzyme activity within the plant itself or even a microbial attack. Therefore, proper drying conditions must be determined for each specific species according to their particular characteristics and compounds to be preserved. Concerning convective drying of regular mint leaves, Radünz et al. (2006) observed that higher extraction yields of essential oils was obtained at an air drying temperature of 50°C. Nevertheless, drying fresh leaves in Brazil is often performed by local producers or small businesses with little knowledge of processing techniques. The lack of technical regulations and standardization procedures originates poor-quality dried products, which is a shortcoming in the industry production chain.

Convective drying of leaves involves heat and mass transfer in packed beds of deformable 'particles'. Besides, as the leaves usually lose up to 85% of their weight in drying (CHEN; MUJUMDAR, 2006), they shrink significantly. The interaction between gas and solid phases in this case is much more complex than observed in packed beds of rigid particles, as the packing structure changes over time, and the transfer properties change as well. An additional drawback to the analysis is the challenge to obtain reproducible packs, given the random nature of packing for such irregular type of material. Properties such as size, thickness, superficial texture, and shape, which are usually not uniform for biological particulates, are key factors in defining packed-bed properties such as permeability, local voidage and bulk density. In most studies focused on drying leaves published in the literature, the material is not fully characterized as a 'particulate' matter, and information on size or bed bulk density are often neglected, although it is expected that these properties do affect convective drying.

The high number of variables and parameters that play a role in the drying leaves is a major drawback in modeling the process using phenomenological equations. Therefore, most studies are based on empirical approaches, such as fitting of experimental data to kinetic empirical models. However, analysis only based on fitting drying parameters is limited because the fitted equations do not often extend for different materials or drying conditions. Recently, alternative modeling approaches have been proposed in the literature to describe drying processes. Dalpasquale et al. (2012) simulated fixed-bed drying of corn grains assuming

a constant enthalpy of air drying as a quantitative physical indicator for correcting the heat and mass exchange in each step of the process, in order to obtain more real evaluations in all drying stages, and in the results for final moisture of the grain. Techniques based on surface response methodology and on artificial neural networks have been successfully applied to model drying processes of different products, accordingly to Freire et al. (2012) and Karimi et al. (2011). Such empirical techniques may contribute to a better assessment of how many different variables affect drying of leaves.

Response Surface Methodology (SRM) is a series of experimental design, analysis, and optimization techniques originally proposed by Box and Wilson (1951). The aim is to optimize an unknown and noisy function by means of simpler approximating functions that are valid over a small region using design experiments (KARIMI et al., 2011).

Artificial Neural Networks (ANN) models are particularly suitable for modeling dynamic nonlinear systems, such as those typically obtained when describing drying processes (MATTEO et al., 1999; ZBICINSKI et al., 1996). ANNs are mathematical models inspired by the biological neural systems. They 'learn' from examples through interaction, without requiring *a priori* knowledge of relationships between variables under investigation. Input variables and responses obtained experimentally associated with alteration in these variables are supplied to the neural network at a stage known as 'training'. A trial and error process is used to develop patterns for the responses, until the network outputs are compatible with patterns provided to a certain specified level of accuracy (KARIMI et al., 2011).

The network design may be modified or adapted to meet particular requirements of each problem by varying the number of neurons and neuron layers, resulting in a variety of possible structures that allow constructing very flexible models.

Aiming at applying these methodologies in the analysis of drying leaves, the purpose was investigate the drying of packed-beds constituted by leaves of regular (*Mentha x villosa* H.) mint through different modeling approaches, namely fitting empirical correlations, modeling based on response surface methodology and developing of an ANN empirical model. The independent variables selected to analyze regular mint leaves drying are air temperature, air velocity and mass of the samples. Experiments were carried out in a convective dryer with parallel-air flow at temperatures from 36 to 64°C and velocities from 1.0 to 2.0 m s⁻¹.

Material and methods

Fresh leaves used in this work were obtained from a local dealer in São Carlos, São Paulo State, Brazil. Before processing, leaves were separated from the stalks by hand, selecting those of similar sizes (damaged or old leaves were discarded). The main dimensions of the leaves (the two longest intercepts) and their surface area were obtained using image analysis with software Image-Pro Plus 3.0 for three samples of ten selected leaves. The mean thickness was measured using a digital micrometer accurate to 0.01 mm. As for the fresh leaves, the thickness is not uniform along the whole length, the measurements were taken at four positions and an average value was adopted. To estimate the apparent density of leaves, their weight and volume were determined. A sample containing around 100 leaves was weighed using a precision scale (accurate to 10^{-4} g) and their volumes were obtained by the liquid displacement technique with hexane. The mass and volume of each leaf were estimated by dividing the values of mass and volume of a sample by the number of leaves. The assays were performed in triplicate, and average values were considered.

Experiments were carried out at 36, 50 and 64°C, and air flow velocities of 1.0 1.5 and 2.0 m s⁻¹. The recipients for the leaves were made of metallic iron screens, with dimensions of 10 x 20 cm on the basis, and heights of 1.4 1.7 and 3.5 cm to accommodate the different masses of leaves used in the tests, which were 18, 30 and 42 g. Whole leaves were inserted into the recipients always in the same way, with the purpose of making the packed-beds as similar as possible to each other.

The experimental setup for convective drying consisted of a blower, a series of electrical resistances to heat the air and homogenizing plates, followed by a horizontal tunnel 0.56 m long with a transversal-cross section of 0.041 m², where the samples were placed, according to the layout presented in Figure 1. The temperature was adjusted using a variac that allows a variation of electrical power input for the electrical resistances. Samples were inserted into the dryer after reaching stable temperature and velocity conditions. Mass variation of the beds was measured by weighing the recipients at time steps of 2.5 minutes in the first 5 minutes, from 5 minutes onwards in the interval from 5 to 40 minutes and then using non-periodic intervals until constant mass. Initial moisture content was determined by keeping the samples in an oven at $105 \pm 3^\circ\text{C}$ for 24 hours. A semi-analytical scale GEHAKA model BK400, with a precision of $\pm 10^{-4}$

g was used to weigh the samples. The air temperature was measured using a thermo hygrometer and the air velocity was measured using an anemometer, both of a measuring kit from Cole-Parmer Instruments Co.

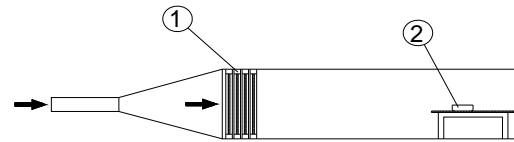


Figure 1. Horizontal convective dryer: (1) air distribution system (2) samples.

The moisture ratio (MR) was calculated using the equation:

$$MR = \frac{M - M_e}{M_0 - M_e} \quad (1)$$

where M is the moisture content at a drying time t, M₀ is the initial moisture content and M_e is the final moisture content, given in kg of moisture kg⁻¹ of dry matter.

The experiments were performed according to a full factorial design with 3³ experiments, in compliance with the levels listed in Table 1.

Table 1. Variables and levels used in the 3³ experimental design.

Code	T (°C)	V (m s ⁻¹)	M _A (g)
-1	36	1.0	18.0
0	50	1.5	30.0
+1	64	2.0	42.0

Experimental data of dimensionless moisture ratio over time were fitted to five empirical models, given in Table 2. Henderson and Pabis equation is considered a semi-empirical model, once it has been formulated based on the truncated solution of diffusion equation (SHARMA et al., 2011). Lewis and Page equations have been formulated based on analogy to the convective Newton's model (OJEDIRAN; RAJI, 2011; REIS et al., 2012). Equations (5) and (6) describe purely empirical models. The regression analysis to verify the goodness of fitting was performed using the Solver routine available in software Microsoft Excel®.

Table 2. Selected thin-layer drying models.

Model	Equation	
Lewis	MR = exp (-kt)	(2)
Page	MR = exp (-kt ⁿ)	(3)
Henderson & Pabis	MR = a exp (-kt)	(4)
Logarithmic	MR = a exp (-kt) + c	(5)
Two-terms	MR = a exp (-kt ⁿ) + b exp (-k ₂ t)	(6)

Results and discussion

Main dimensions and density of mint leaves are listed in Table 3.

Dimensions have varied significantly, with projected areas ranging from approximately 9 to 14 cm² and mean diameters from 3.4 to 4.1 cm. The apparent density of leaves was equal to 0.988 g mL⁻¹, a consistent value, since the initial moisture content of the leaves is very high, about 88% (w.b.).

Drying kinetics

Some representative data of dimensionless moisture ratio versus time for different experimental conditions are shown in Figure 2. For the sake of concision, only the limiting conditions (maximum and minimum values) are presented since the intermediate values are contained in such analysis.

Figure 2a and b show that as expected, regardless of the values of air velocity and mass of sample, the increase of temperature from 36 to 64°C had shortened the time necessary to reach equilibrium moisture. The effect of temperature was remarkably strong, with very steep drops of moisture observed in the curves obtained at 64°C.

At a mass of 18 g, for instance, the time for reaching equilibrium moisture content was reduced in approximately 42% as the temperature increased from 36 to 50°C, and in about 77% as the temperature changed from 50 to 64°C.

The variation of air velocity on the other hand had little effect on the curves, as seen in Figure 3a

and b. In Figure 3a, an increase of air velocity from 1.0 to 1.5 m s⁻¹ reduced by 13% the time required for reaching the equilibrium moisture, and a further increase of air velocity to 2.0 m s⁻¹ had no effect on this time. The strong effect of temperature and the weak influence of air velocity observed in Figures 2 and 3 suggested that the major resistance to mass transfer is related to moisture migration from the inner packed-bed to surface. These results are compatible with those reported in the literature for the drying of leaves of different genera and species (DOYMAZ et al., 2006; PARK et al., 2002).

Figure 4a showed that the increase of sample mass had little effect on drying rates at a low temperature and air velocity (36°C and 1.0 m s⁻¹), but figure 4b illustrated that as the temperature was raised to 64°C and air velocity to 2.0 m s⁻¹, the moisture at a given time increased as mass increased from 18 to 30 or 42 g, suggesting a decrease in the drying rate. One should note that the sample thickness increased from 1.4 to 3.5 cm as the mass was raised from 18 to 42 g. At higher temperature and velocity, the rate of moisture removal from the sample surface was enhanced. With the increase in sample thickness, the moisture had not reached the surface at a high enough rate to replace the moisture removed.

Mathematical models

Experimental data at different drying air temperatures and size ratios were fitted to the models listed in Table 2.

Table 3. Physical properties of mint leaves.

Projected area (cm ²)	Perimeter (cm)	Maximum diameter (cm)	Minimum diameter (cm)	Mean diameter (cm)	Thickness (mm)	Density (g mL ⁻¹)
11.6 ± 2.2	14.8 ± 1.8	4.85 ± 0.53	2.86 ± 0.29	3.72 ± 0.35	0.168 ± 0.002	0.988 ± 0.232

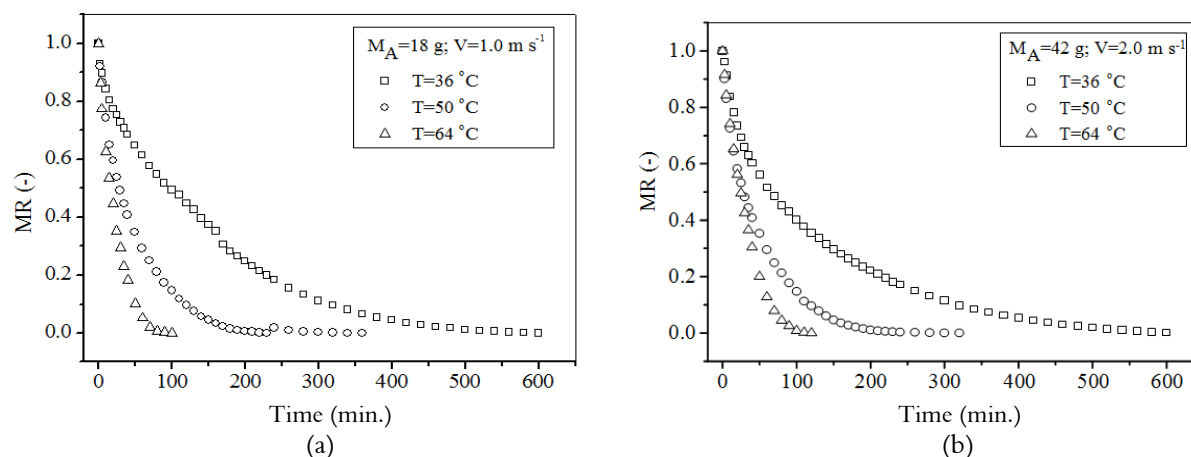


Figure 2. Dimensionless moisture ratio versus time for the drying of regular mint leaves at different air drying temperatures.

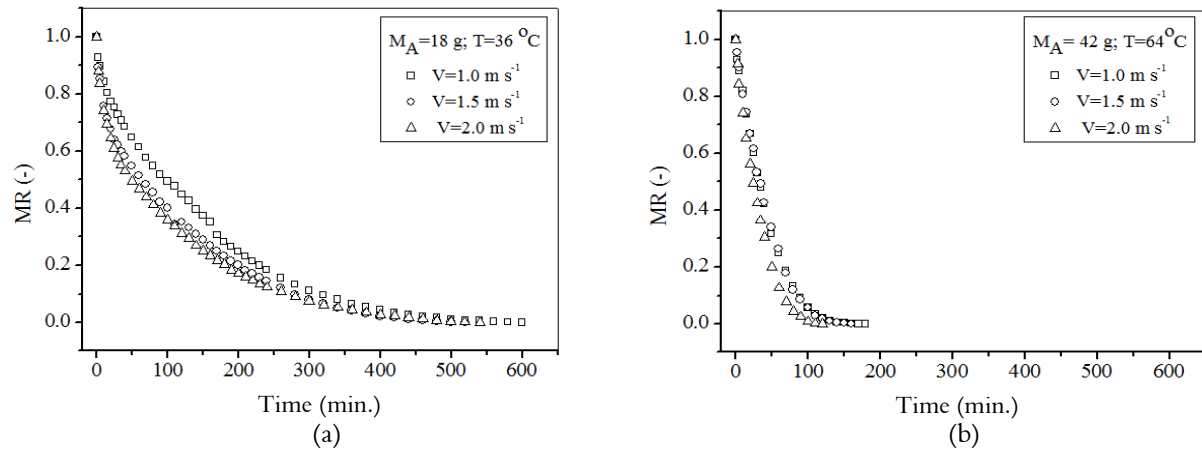


Figure 3. Dimensionless moisture ratio versus time for the drying of regular mint leaves at different air velocities.

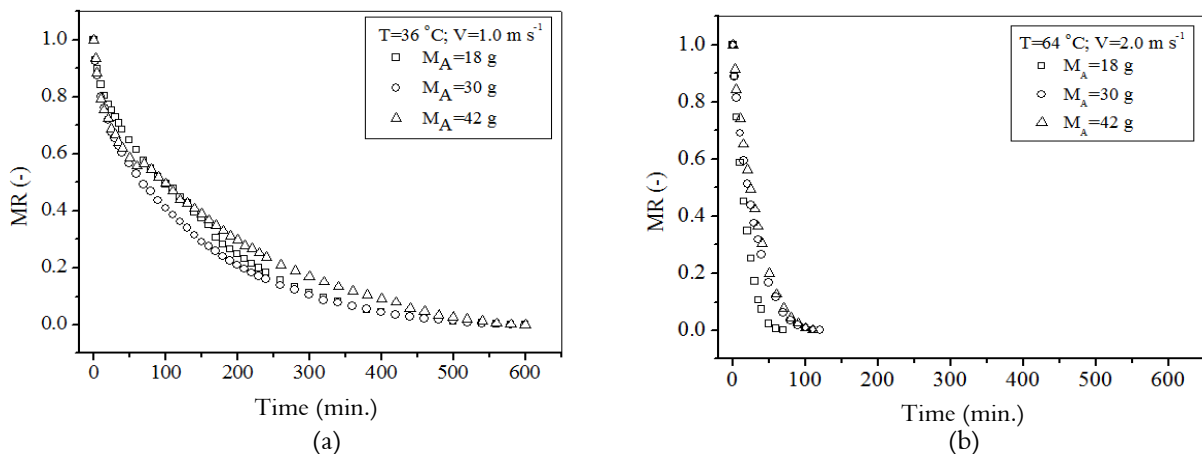


Figure 4. Dimensionless moisture ratio versus time for the drying of regular mint leaves at different sample masses.

The criterion for suitability of the models were based on the values of coefficients of determination, R^2 the reduced chi-square (SQR) and the root mean square error (RME). The higher the values of the R^2 , the lower the values of SQR and RME, the better the quality of fitting (AKPINAR, 2010). The statistical analysis carried out in the models listed in Table 2 provided coefficients of determination $R^2 > 0.975$, and the values of SQR and RME remained within a range considered adequate in most cases, suggesting that any model could be applied to estimate the moisture content of mint leaves over time. Considering only the two-parameter models, which are most practical for use, Henderson-Pabis' model provided good fitting to data obtained at a mass of 18 g, and Page's model performed well for masses of 30 and 42 g. For this reason, these models are considered adequate to represent the kinetic curves and their fitted parameters are presented in Table 4.

Analysis of surface responses

In this study, effects of air temperature, air velocity and mass of the sample on moisture content were evaluated. Each drying experiment performed at given values of T , V and M yielded several response variables because the moisture changes along time. The analysis was then performed at $t = 100\text{ min.}$, for which all the tests, even the shortest ones could be analyzed. The regression models were evaluated at a confidence interval of 95% and the regression coefficients were obtained. Based on the probability values (p -values < 0.05 indicate that a parameter is statistically significant), the significant regression coefficients were selected. The effect of the linear factors air velocity and air temperature was found to be highly significant on the moisture content of mint leaves after 100 min. drying. The quadratic effect of air temperature, and interaction effects between air temperature and velocity, air temperature and mass and also temperature square and mass were also significant.

Table 4. Fitted parameters and estimated values of R^2 , SQR and RME.

Model	T (°C)	V (m s ⁻¹)	Coefficients		R ²	SQR	RME
Henderson Pabis	M _A = 18 g						
	36	1.0	a = 0.8565	k = 0.0056	0.9818	0.0661	0.0014
	36	1.5	a = 0.8491	k = 0.0058	0.9898	0.0415	0.0008
	36	2.0	a = 0.8899	k = 0.0076	0.9871	0.0511	0.0011
	50	1.0	a = 0.9498	k = 0.0112	0.9916	0.0372	0.0009
	50	1.5	a = 0.9382	k = 0.0145	0.9975	0.0094	0.0002
	50	2.0	a = 0.9251	k = 0.0201	0.9948	0.0153	0.0005
	64	1.0	a = 1.0240	k = 0.0236	0.9952	0.0171	0.0007
	64	1.5	a = 1.0343	k = 0.0236	0.9940	0.0206	0.0009
64	2.0	a = 1.0095	k = 0.0311	0.9953	0.0122	0.0007	
Page	M _A = 30 g						
	36	1.0	k = 0.0313	n = 0.7425	0.9947	0.0225	0.0005
	36	1.5	k = 0.0001	n = 1.9987	0.9279	0.8982	0.0180
	36	2.0	k = 0.0481	n = 0.6994	0.9972	0.0112	0.0002
	50	1.0	k = 0.0409	n = 0.7963	0.9976	0.0084	0.0002
	50	1.5	k = 0.0339	n = 0.8749	0.9972	0.0092	0.0003
	50	2.0	k = 0.0379	n = 0.8542	0.9979	0.0070	0.0002
	64	1.0	k = 0.0358	n = 0.9551	0.9928	0.0165	0.0008
	64	1.5	k = 0.0209	n = 1.1009	0.9952	0.1116	0.0006
64	2.0	k = 0.0331	n = 1.0137	0.9971	0.0067	0.0004	
Page	M _A = 42 g						
	36	1.0	k = 0.0296	n = 0.7154	0.9811	0.0722	0.0015
	36	1.5	k = 0.0301	n = 0.7185	0.9868	0.0593	0.0011
	36	2.0	k = 0.0305	n = 0.7448	0.9969	0.0133	0.0003
	50	1.0	k = 0.0141	n = 0.9627	0.9890	0.0493	0.0012
	50	1.5	k = 0.0249	n = 0.8924	0.9966	0.0141	0.0004
	50	2.0	k = 0.0435	n = 0.8287	0.9979	0.0072	0.0002
	64	1.0	k = 0.0123	n = 1.1624	0.9983	0.0055	0.0002
	64	1.5	k = 0.0109	n = 1.1897	0.9979	0.0065	0.0003
64	2.0	k = 0.0207	n = 1.1109	0.9969	0.0073	0.0004	

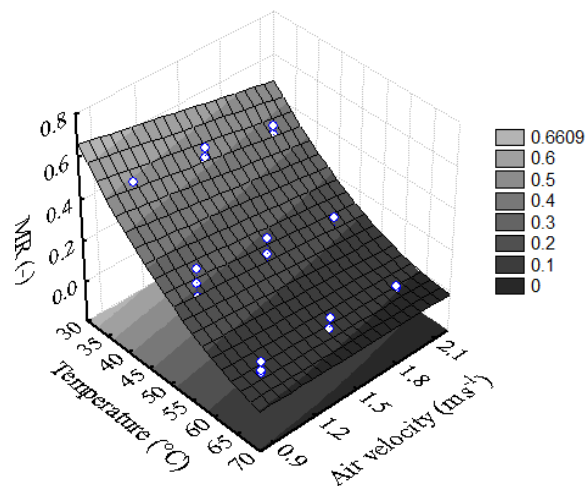
Table 5. Regression coefficients and p-value.

Factor	Regression coefficient	Standard Error	t(23)	p-value	Confidence limits	
					-95%	+95%
Mean	0.2010	0.0051	39.0056	0.0000	0.1902	0.2117
Temperature	-0.2001	0.0063	-31.8414	0.0000	-0.2141	-0.1878
Temperature ²	-0.0275	0.0055	-5.0333	0.0001	-0.0389	-0.0161
Air Velocity	-0.0380	0.0063	-6.0133	0.0000	-0.0511	-0.0248
Mass	0.0334	0.0063	5.2968	0.0000	0.0203	0.0466
T*V	0.0221	0.0077	2.8559	0.0098	0.0060	0.0382
T ² M	0.0217	0.0067	3.2371	0.0041	0.0077	0.0356

$R^2 = 0.98249$.

$$MR = 0.20 - 0.20T - 0.03T^2 - 0.04V + 0.03M + 0.02TV + 0.02T^2M \quad (7)$$

All the necessary information for the analysis of drying in the range investigated is provided in Equation 7. The fitted model demonstrated that the moisture content depends linearly on the air temperature (T), air velocity (V) and mass of sample (M), and also depends on the interaction between some factors, such as TV and T^2M , and on quadratic effects, such as T^2 . To illustrate the effects of single variables, (T, V or M), and of interacting effects on the moisture content, surface responses for some selected conditions are shown in Figures 5 and 6, which are representative of the remaining conditions investigated. The curves indicated that the temperature was the factor with major influence on the moisture content.

**Figure 5.** Dimensionless moisture ratio as a function of air velocity and air temperature (30 g).

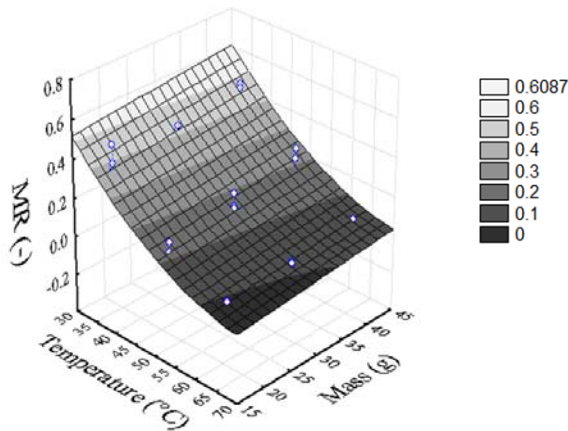


Figure 6. Dimensionless moisture ratio as a function of sample mass and air temperature ($V=1.5 \text{ m s}^{-1}$).

Neural network

Due to the reproducibility difficulties found in the drying experiment of mint leaves, a trained neural network was used instead of a mechanistic approach that would have required the estimation of physical parameters such as the effective diffusivity. Concerning the use of neural networks in this case it simulates the process by learning how to predict moisture kinetics from a purely experimental database. From a mathematical point of view, the neural network for the drying kinetics of mint leaves may be regarded as a fitting model that interpolates information in the range of the experimental database.

The design of the neural network was done in the Neural Network Toolbox for use with MatLab. MatLab Neural Network Toolbox allowed for fast model creation and validation in a single framework. Standard Bayesian regularization back propagation training algorithm was used for training the network. This training algorithm updated the weights between adjacent neurons according to the Levenberg–Marquardt algorithm. It minimizes a linear combination of squared errors multiplied by weights to produce a network that best fits the experimental data. The number of neurons in the hidden layer was chosen by trial and error, as suggested by Himmelblau (2008) starting with 2 neurons and adding up some more until the network performance in estimating the correct output is satisfactory. A reasonable number of neurons for this application was found to be 5. Figure 7 illustrated together with experimental points the simulated estimations from the neural network for $m = 30 \text{ g}$, $T = 50^\circ\text{C}$ and the three different air flow velocities indicated in the figure legend. It can be seen that a good agreement between measured and estimated results was

obtained by using a neural network. Similar results were obtained for the remaining operating conditions investigated, with the additional remark that measurements were more accurate for larger values of sample loads, which is consistent with the fact that a thicker layer of leaves had characteristics closer to that of a conventional or ‘well-behaved’ particulate medium.

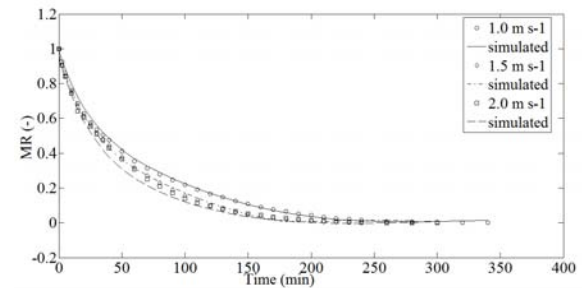


Figure 7. Drying kinetics for 30 g of leaves.

Values of measured moisture content against estimated moisture content are shown in Figure 8 with the correlation factor (R^2). It can be observed in Figure 8 that one single neural network was capable of interpolating information within the range of operating conditions used in this work. This was a great advantage of neural networks in comparison to fitting models, such as the Page model, that requires a specific set of fitting parameters for each particular operating condition.

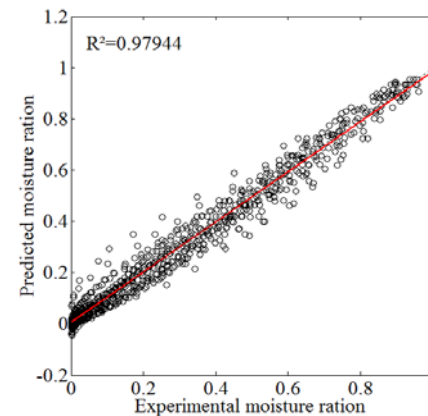


Figure 8. Comparison of predicted and experimental values using ANN.

Conclusion

From the analysis of each of the methods used in this study, it can be said that drying of leaves in the evaluated setup is affected mainly by air temperature, while the influence of air velocity is very weak, although it may be relevant depending on the operating conditions. The resistance to mass

transfer during the drying of peppermint leaves is essentially internal. Among the different fitting models for thin layer experiments, the best fit was obtained by the Henderson Pabis model for samples of 18 grams and by the Page model for samples of 30 and 42 grams.

Even though the samples of peppermint leaves consisted of a very heterogeneous particulate medium for masses from 18 to 64 g, one single neural network was satisfactory in the estimation of moisture content within the range of operating conditions investigated.

In relation the response surface analysis, an optimized function was obtained that well represented the process, conveniently correlating process variables with the moisture content.

Finally, the main conclusion that can be taken from this study is that for drying of heterogeneous and irregular shaped "particles" such as leaves, it is convenient to perform a combined analysis using the three methodologies proposed, as they complement to each other.

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