

Effects of Drying Methods on the Drying Kinetics and the Essential Oil of *Lippia multiflora* Moldenke Leaves

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Abstract: *Lippia multiflora* is widespread in Africa and America. Its leaves are consumed as tea and have various medicinal properties. To preserve the flavour and properties, the farmers dry the leaves in shade for six to ten days. The aim of this work on one hand, is to check the soundness of this traditional drying method by comparing its effects with those of the forced convective drying at 40, 45, 50, 55 and 60°C on the drying kinetics, the composition and content of the Essential Oil (EO) of dried leaves. On the other hand, to test the agreement of drying curves with the Page model, the Peleg model and the diffusional model. The drying duration passes from 7 days for shade-drying to 3 h for convective drying at 60°C, while the composition and the content of EO do not vary significantly. The three models simulate well the drying kinetics nevertheless the Page one is the best. Analysed in the framework of Avrami law, its constant values are in agreement with the unidirectional diffusion. The activation energy obtained from the Peleg model (47.8 kJ/mol) is almost equal to that deduced from the diffusional model (46.3 kJ/mol). The drying of lippia leaves between 45 and 55°C reduces significantly the drying length without degrading the EO. The results obtained from the three models turn out coherent

Key words: Drying kinetics, essential oil, *Lippia multiflora*, modelling, tea

INTRODUCTION

The genus *Lippia* (Verbenaceae) consists of about 200 species distributed in South America and tropical Africa (Pascual *et al.*, 2001). *Lippia multiflora* Moldenke (*LmM*) leaves have a pleasant flavour and are widely consumed in Africa as tea. They are now exported to Europe and pharmacological studies conducted on its Essential Oil (EO) revealed numbers of beneficial activities such as: antimicrobial, hypotensive, antioxidant, analgesic, antipyretic, and anti-inflammatory (Abena *et al.*, 2003; Pascual *et al.*, 2001; Valentin *et al.*, 1995). Its EO chemical composition varies with the harvest period, and the origin of the plant (Menut *et al.*, 1995; Pascual *et al.*, 2001). Besides, it was shown on other plants that the conditions of conservation and extraction influence the EO composition and content (Sefidkon *et al.*, 2006). According to our knowledge, such studies have not been yet carried on *LmM*. The conservation mode used for *LmM*'s leaves is the sun-drying. The reasons are the availability of the sun, the ease of the method, and the moderate cost of the installations. In order to limit the losses of the constituents, in most cases *LmM*'s leaves are drying in the shadow. It takes six up to ten days (depending on the sunshine) to complete the drying

process. Demir *et al.* (2004) even report longer durations for the bay leaves. Nevertheless, longer drying durations might expose the harvests to the attacks of mushrooms, requires large drying areas for large production quantities, and thus raise the costs.

Many parameters influence the drying kinetics such as: temperature, air velocity and humidity, nature and quantity of material. Nevertheless, the drying temperature is recognized as the most dominating factor (Demir *et al.*, 2004; Krokida *et al.*, 2003). The rise of the drying temperature reduces the drying duration; however, it generally involves the lowering of the yield and the quality of the EO (Di Scala and Crapiste, 2008; Omidbaigi *et al.*, 2004; Simal *et al.*, 2000). For example, Omidbaigi *et al.* (2004) report that the yield of the EO of the Roman chamomile flowers passes from 1.9% when flowers are shade-dried to 0.4% when they are sun-dried. But for certain plants reports revealed only small variations of both the yield and the composition up to 50°C while the drying duration is greatly reduced (Demir *et al.*, 2004; Sefidkon *et al.*, 2006; Venskutonis, 1997). These apparently contradictory results could be explained by the fact that the diffusion and the evaporation of the constituents depend on several parameters among which their nature and the structure of the plant. In any case, for

economic reasons, a compromise is to be found with drying speed and global or partial yield of the wanted constituents.

The first objective of this study is to check the soundness of the traditional approach by comparing its effects to those of forced convective drying on the drying kinetics, composition and content in the EO of these *LmM* leaves. In the second step the drying kinetics are modeled and kinetic parameters such as activation energy, the moisture effective diffusivity and avrami law constants are extracted. The obtained results are subsequently discussed in relation with the available literature data.

MATERIALS AND METHODS

Plant material: The *LmM* leaves are harvested at random in the experimental field of the Faculty of Science at Brazzaville (Congo) in January 2008. A voucher specimen is deposited at the herbarium of the Centre d'Etudes des Ressources Végétales at Brazzaville. To determine the initial moisture content, three samples of 100 g each of fresh leaves are dried at 105°C for 24 h in an oven (Termosi SR 3000). The average value obtained is 3.37 g/g (db).

Drying: Drying experiments are performed on two methods: shade-drying at room temperature (22-27°C) as done by traditional farmers, and convective drying in a laboratory scale pilot with air velocity of 1m/s, at 40, 45, 50, 55 and 60°C. The mean air humidity was 60%. For each test, immediately after the harvest, 200 g of leaves are weighed and dried. This mass is chosen in order to have a sufficient EO quantity for chemical analysis without stacking layers during drying. Moisture loss is periodically measured by use of balance with a precision of 0.001g. The duration of the drying is the time to reach the equilibrium mass. Each experiment is performed three times. The reproducibility is within 7%.

Essential oil extraction: After drying, the leaves are subjected to hydrodistillation in a Clevenger-type apparatus for 3 h. The volatile oils are extracted from the distillation water with diethyl ether, dried at room temperature over anhydrous sulphate of sodium and refrigerated in sealed vials till time of analysis. The EO yield is given by the following relation.

$$R = \frac{M_0}{M_1} 100 \quad (1)$$

Essential oil analysis: The Gas Chromatography-Mass Spectrometry (GC-MS) data acquisitions are performed through a Hewlett-Packard HP 6890 equipped with a mass selector detector HP 5973. The capillary column is DB5 (30 m × 0.25 mm i.d., film thickness 0.25 μm). Helium is

used as the carrier gas with a flow rate of 1 mL/min. The ionization energy is 70 eV (EI mode), injector and detector temperatures are 280°C, and the split ratio is 1/10. The initial temperature is set at 50°C for 5 min, then from 50 to 300°C at 5° per min, and then holds 5 min. The volatile constituents are identified by comparison of their Retention Indexes (RI) and mass spectra with authentic reference compounds and MS electronic data (NBS 98K and Wiley 275) as well as with the published data from literature.

The quantification of the constituents is made with a GC HP 5890 equipped with a detector with a Flame Ionization Detector (FID) provided with a data acquisition software HP chemstation. The split ratio is 1/20. Other experimental conditions are the same. Percentages are calculated by peak areas normalization measurements.

Drying kinetics modeling: The air drying of the plant materials is known to be a very complex phenomenon in which intervene heat and mass exchanges, structural and composition modifications (Ramos *et al.*, 2003; Zogzas *et al.*, 1994). This is one the reasons why empirical or simplified theoretical models developed many years ago are often applied. The Table 1 presents three of these models which we used because their constants have a physical meaning. In the Page model, the coefficients k and n depend on the drying conditions but they are considered without physical meaning (Karathanos and Belessiotis, 1999). This model is identical to that known as Kolmogorov-Jhonson-Mehl-Avrami (KJMA) model or Avrami's law (Avrami, 1939; Avrami, 1940; Piorkowska *et al.*, 2006). It was established by making the following assumptions: the nuclei are randomly distributed in the sample, the volume remains constant, germination mode and growth geometry all remain constant during the phase transformation. Avrami's constant n is an integer or a half integer. It depends on the mode of germination and on the geometry of growth of the new phase. This model is usually used for modeling the isothermal crystallization kinetics, the development of droplets in a gas or the bubbles in a liquid, and drying kinetics((Demir *et al.*, 2004; Karathanos and Belessiotis, 1999; Piorkowska *et al.*, 2006; Simal *et al.*, 2005) . In practice, the n values measured are lightly different from those foreseen. The reason is that the basic hypotheses are hardly practicable.

Table 1: Drying models used in this study

Model name	Model
Page or KJMA	$M_r = \exp(-kt^n)$
Diffusional (Infinite Plate)	$M_r = \sum_{i=1}^{\infty} \frac{8}{(2i-1)^2 \pi^2} \exp(-i - \frac{1}{2})^2 \pi^2 \frac{D_c}{L^2} t)$
Peleg	$M(t) = M(0) - \frac{t}{a+bt}$

Furthermore, given that the same value of n can correspond to different situations, it is not easy to deduct the parameters of the transformation from the value of n alone.

The diffusional model was given by Crank (1975) in resolving the Fick's second equation in the case of a plane sample and assuming that the product is uniform and without structural modifications. This model is largely used for products whose drying curve has not constant drying rate period (Ghazanfari *et al.*, 2006; Simal *et al.*, 2005). To increase the agreement with the experimental results several terms of the series must be considered. But, in first estimate, this model can be reduced to the expression:

$$M_r = A \exp\left(-\frac{\pi^2 D_e}{L^2} t\right) \quad (2)$$

A is an adjustable parameter.

The Peleg model (1988) is empirical too, but its constants have a physical meaning:

$$M(t) = M(0) - \frac{t}{a + bt} \quad (3)$$

$$a^{-1} = -\left(\frac{dM}{dt}\right)_{t=0} \quad (4)$$

$$b^{-1} = M(0) - M_e \quad (5)$$

This model was used for the water sorption and desorption of mint leaves, papaya and small peas (Palou *et al.*, 1994; Turhan *et al.*, 2002). To compare it with the other models, we deduced from the original expression of Peleg, the expression of M_r which we call the modified Peleg model:

$$M_r = 1 - \frac{t}{c+dt} \quad (6)$$

where, c is the inverse of the initial drying rate:

$$c^{-1} = -\left(\frac{dM_r}{dt}\right)_{t=0} \quad (7)$$

Statistical analysis: The statistical analysis of the results is made with the software Origin Pro 7.5. The fit's quality of the models is estimated by the means of the correlation coefficient (R^2) and the reduced Chi-squared (χ^2).

RESULTS AND DISCUSSION

Effect of the temperature of drying: Figure 1 and 2 present the drying curves of samples dried respectively in shade at Room Temperature (RT) and in the convective dryer at various drying temperatures. The drying duration is considerably reduced with the increase of the drying temperature. It passes from 7 days for shade-drying to

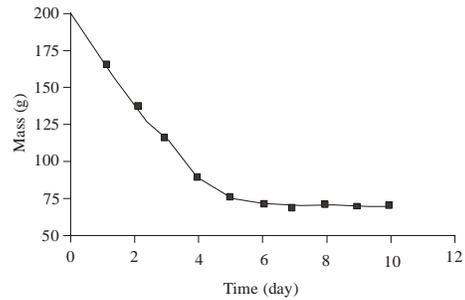


Fig. 1: Shade drying curve

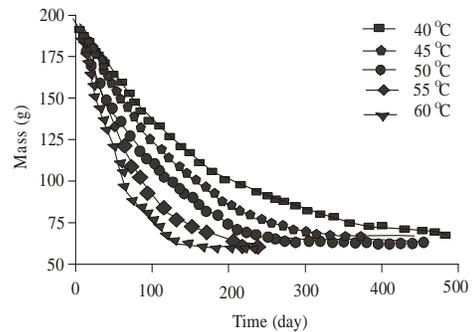


Fig. 2: Convecting drying curves at different temperatures

8 h for convective drying as from 45°C. This shows that sun-drying or drying on beforehand warmed surface could reduce the drying duration to 2 days at the very most. Furthermore, drying at high temperature reduces the equilibrium moisture content and then decreases the risks of mushroom attack. Besides, the constant rate period seems well marked while it disappears for drying temperatures above 40°C. Such absence of constant rate period was already reported for other vegetables (Demir *et al.*, 2004; Jin Park *et al.*, 2002; Krokida *et al.*, 2003).

Evaluation of the models: The drying curves at all drying temperatures with the theoretical curves of the three models are reported on the Fig. 3. All the models fit well enough the experimental curves. Indeed, the Page's model is the best as shown by the values of the statistic parameters displayed in Table 2. It is followed by the diffusional model and the modified Peleg model. As listed in Table 3, the Avrami constant n's values obtained are between 1 and 1.3. According to the theory, this could be explained by a continuous but decreasing germination, a transformation controlled by the diffusion and a rod-like development of the vapor germs. These values are in agreement with those reported in the literature for other vegetables (Demir *et al.*, 2004; Doymaz, 2006; Doymaz and Pala, 2002; Karathanos and Belessiotis, 1999). As already reported by several authors, the fluctuation of n in this drying temperature range does not seem significant

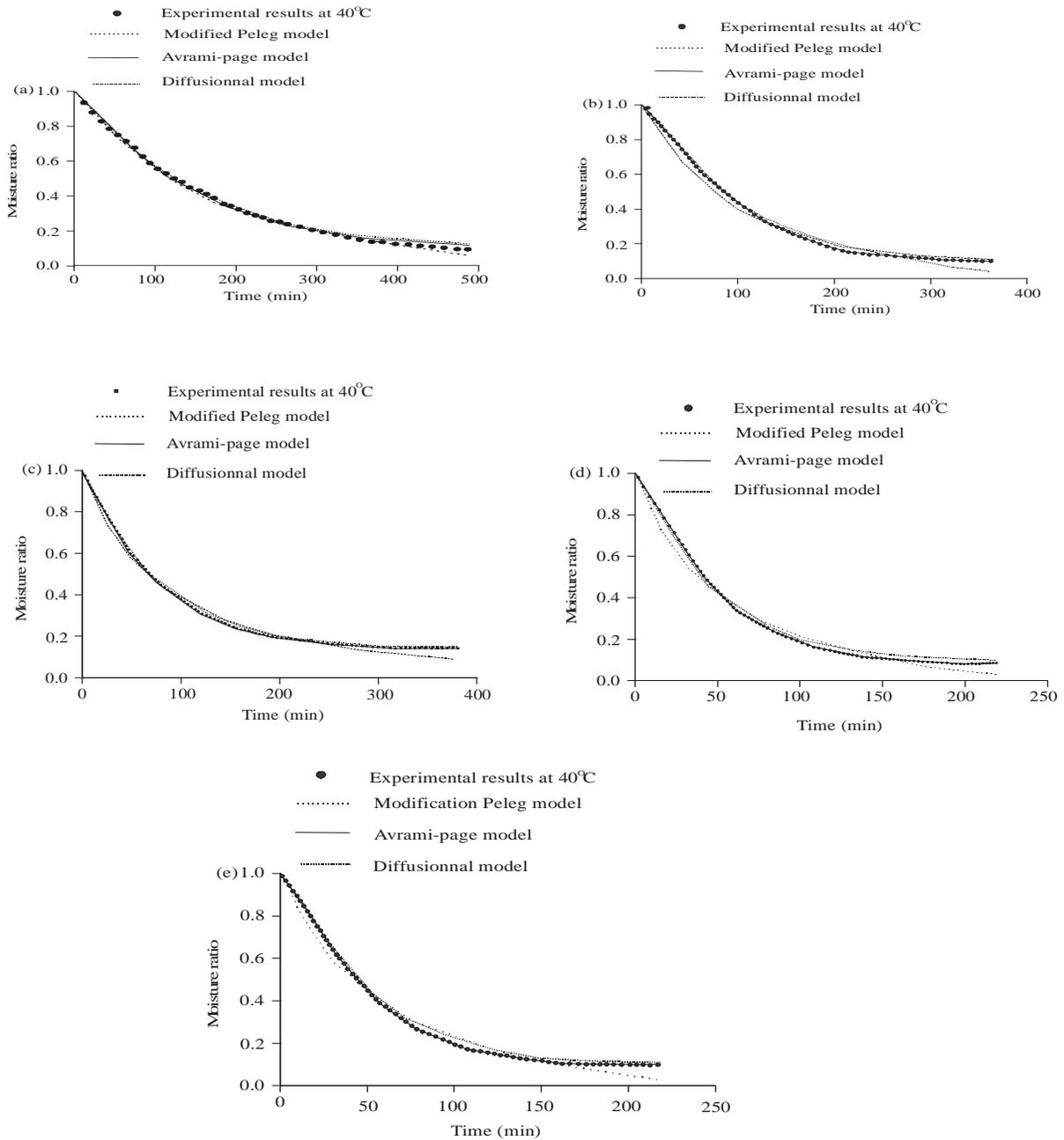


Fig. 3: Modeling convecting drying kinetics of lippia leaves at 40, 45, 50, 55 and 60°C

Table 2: Comparison of the drying kinetics models

Temperature (°C)	Page-JMAK model		Diffusionnal model		Peleg model	
	R ²	χ ²	R ²	χ ²	R ²	χ ²
40	0.998	0.00015	0.996	0.00034	0.997	4.19
45	0.998	0.00015	0.992	0.00088	0.984	25.48
50	0.995	0.00038	0.994	0.00052	0.988	17.37
55	0.998	0.00020	0.991	0.00011	0.981	32.65
60	0.997	0.00032	0.990	0.00012	0.983	30.56

Table 3: Avrami constants for the different convecting drying temperatures

Drying method Temperature (°C)	Shade drying	Convecting drying				
	25	40	45	50	55	60
Yield (%)	1.14	1.13	1.11	1.12	1.13	1.10
n	-	1.08	1.20	1.06	1.22	1.20
k.10 ⁻⁴ (min ⁻ⁿ)	-	42	34	86	82	83

Table 4: The activation energy and the effective diffusivity coefficient of *lippia m.M.* leaves compared to those of other vegetables reported in the literature

Vegetable	E _a (kJ/mole)	D _{eff} (m ² /s)	Temperature (°C)	Reference
Mint	82.93	4.8×10 ⁻¹³ - 2.9×10 ⁻¹²	30-50	Jin Park <i>et al.</i> (2002)
	62.96	3.067×10 ⁻⁹ - 1.941×10 ⁻⁸	35-60	Doymaz (2006)
Okra	51.26	4.27×10 ⁻¹⁰ - 1.30×10 ⁻⁹	50-70	Doymaz (2005)
Olive leaf	52.15-83.6	2.95×10 ⁻¹⁰ - 3.60×10 ⁻⁹	40-60	Nourhène <i>et al.</i> (2008)
Kale	36.115	14.89-55.94×10 ⁻¹⁰	30-60	Mwithiga (2005)
Aloe vera	24.4	5.64×10 ⁻¹⁰ - 18.1×10 ⁻¹⁰	30-70	Simal (2000)
Black tea	406.03	1.141×10 ⁻¹¹ - 2.985×10 ⁻¹¹	80-120	Panchariya <i>et al.</i> (2002)
Lippia m. M	47.8 (Peleg)	7.1×10 ⁻¹⁰ -21×10 ⁻¹⁰	40-60	This study
	46.3 (diffusion)			

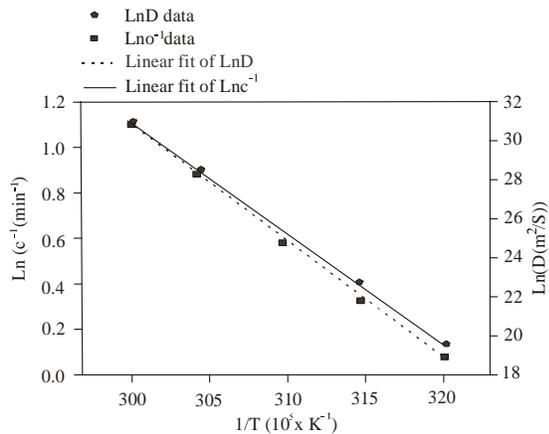


Fig. 4: Deduction of the activation energy by the modified Peleg and the diffusional models

(Demir *et al.*, 2004; Jin Park *et al.*, 2002; Karathanos and Belessiotis, 1999). This could mean that the germination mode and the growth geometry would not change a lot in this range. Besides, it should be noticed that these values also in agreement with the assumption widely accepted that the diffusion is one-dimensional for such large and thin samples. On average, the k values increase with the temperature within the studied range (Table 3). But their scattering does not allow to assert if this evolution is linear ($R^2 = 0.82$ in this study) as reported for Kiwis or of the Arrhenius type ($R^2 = 0.81$ in this study) as in the case of black tea (Panchariya *et al.*, 2002; Simal *et al.*, 2005).

The Peleg model describes better the first drying period than the last one. In agreement with the experimental results, the initial drying rate deduced from the model increases with the temperature. As reported in the Fig. 4, this evolution follows the Arrhenius law:

$$c^{-1}(\text{min}^{-1}) = 93.10^6 \exp(-5751/RT) \quad (7)$$

From this equation, the deduced activation energy is 47.8 kJ/mol. This value is similar to those of other vegetable leaves in the same drying temperature range, except the black tea (Table 4). The values of the effective diffusivity deduced from the diffusivity coefficient deduced from the diffusional model are also reported in the table 4. These values in agreement with those of many vegetables as listed in this table.

Figure 4 also represents the Arrhenius law applied to the moisture effective diffusivity. From it, we deduce the following expression:

$$D_e = 626.10^{-6} \exp(-5567/RT) \quad (8)$$

The value of the activation energy obtained is 46.3 kJ/mol. This value is in agreement with that already found from the Peleg model. This agreement is coherent with the absence of the constant rate period which means that the drying is controlled by the diffusion.

Drying temperature effect on essential oil: As reported in the table 3, the essential oil yield remains nearly constant. Besides, the variation of the chemical composition (Table 5) with the drying temperature is not significant compared with that observed for *LmM* leaves collected in different regions (Agnaniet *et al.*, 2005; Bassole *et al.*, 2003; Menut *et al.*, 1995). Only α and β -selinene present in shade-dried leaves are absent in leaves dried at high temperatures. This result is identical to that obtained for bay leaves and *Satureja hortensis* (Demir *et al.*, 2004; Sefidkon *et al.*, 2006).

CONCLUSION

The objectives of this study were on the one hand to compare the effects of the traditional drying method to those of the convecting drying on the drying kinetics and the EO composition and yield, and on the other hand

Table 5: Composition of the essential oil of shade dried and convecting dried leaves.

RI	Constituents*	Percentage of total oil				
		Shade-drying	Convecting drying			
			40°C	45°C	55°C	60°C
989	Myrcene	0.6	0.7	0.0	0.9	0.8
995	octan-3-one	0.0	0.0	0.0	0.0	0.0
1014	α - terpinene	0.0	0.6	0.0	0.6	0.6
1020	p-cymene	12.3	12.1	2.6	13.0	12.4
1030	1,8 cineole	0.0	0.0	0.0	0.4	0.0
1056	γ -terpinene	1.1	3.0	0.6	3.4	3.3
1067	Sabinene	0.3	0.0	0.3	0.3	0.3
1100	Linalool	0.3	0.4	0.5	0.5	0.3
1185	Terpinen-4-ol	0.4	0.5	0.5	0.5	0.4
1202	Estragol	0.0	0.4	0.3	0.0	0.0
1303	Thymol	39.2	39.6	45.5	43.3	38.0
1310	Carvacrol	7.7	7.2	9.0	8.0	7.1
1356	Thymyl acetate	28.2	25.4	29.4	29.1	26.3
1363	Carvacryl acetate	2.5	2.8	3.1	0.0	3.0
1427	β -caryophyllene	2.7	4.1	4.2	0.0	4.6
1460	α -humulene	2.4	2.5	3.1	0.0	0.7
1458	(E)- α -farnesene	0.0	0.0	0.0	0.0	1.3
1490	β -selinene	0.3	0.0	0.0	0.0	0.0
1497	α -selinene	0.5	0.0	0.0	0.0	0.0
1574	β -caryophyllene oxide	1.1	0.0	0.0	0.0	0.0
1599	B-humulene oxyde II	0.6	0.7	0.9	0.0	1.0

*: According to the order of elution on DB-5

to model the drying kinetics based on previous semi-empirical models. Our results reveal that:

- Drying at 55°C, instead of shade-drying, decreases the drying duration from 168 to 3 h
- The yield and the chemical composition of the essential oil do not vary in a significant way up to 60°C. Thus, the craft producers have no interest to dry the *Lippia multiflora* moldenke leaves in the shadow
- The Page-JMAK model is the best on all the drying temperature range studied. The obtained values of the Avrami constant's n are between 1 and 1.3 in agreement with those reported in the literature and the assumption that the diffusion is unidirectional in that case. The diffusional and the Peleg models fit well the initial phase but deviate from experimental values in the final phase
- The value of the energy of activation obtained from Peleg's model (47.8 kJ/mol) corresponds to the diffusional one (46.3 kJ/mol). This agreement is coherent with the absence of the constant rate period observed

NOMENCLATURE

M(t)	Sample mass at time t
Mr	Moisture ratio
t	time
M ₀	Mass of the volatile oils

M ₁	Mass of dried leaves
M(0)	Initial sample mass
M _e	Equilibrium sample mass
n,k	Avrami paramaters
A,	a,b, c, d constants
D _e	Effective diffusivity coefficient
L	Average half-thickness of a leave
R	Gas constant, 8.3142 kJ/mol K
E _a	Activation energy
db	Dry basis

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