

Research Article

Modeling the Sequential Extraction of Water and Oil Essential from *Cymbopogon Citratus* Acclimatized in the “Plateau des Cataractes”. Empirical Diffusion and Desorption Models

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Abstract

Drying and oil extraction from the same plant matrix have very often been treated as independent operations with different methodologies. The study of drying involves the numerical solution of mathematical models using regression methods and leads to adjustment parameters, often without obvious physical significance: the model parameters. Oil extraction favours empirical models constructed from simple physical representations and validated graphically, in particular by linear correlation lines. In this study, these two operations were considered as a linked pair, treated using empirical models based on the phenomenological approach to metabolite desorption and validated graphically. Such an approach, with few mathematical bases and computer inputs, corresponded to our objective: the production of essential oil from *Cymbopogon citratus*, on a small scale and in a rural environment. Indeed, it is mainly in rural areas that lemongrass cultivation is developing with the support of public authorities as part of the fight against poverty. Peleg's desorption model and the Avrami/Page and Fick diffusion models gave consistent results, and the parameters generated had physical significance. This study showed that drying could therefore be described by the empirical methods widely used in the literature to model the extraction of various metabolites from plant matrices.

Keywords

Modeling Extraction, *Cymbopogon Citratus*, Water, Essential Oil, Congo-Brazzaville

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Received: 27 June 2025; Accepted: 11 July 2025; Published: 4 August 2025



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1. Introduction

For three last decades, the Congo has implemented a program to promote the production of essential oils in rural areas to fight against poverty. Thus, with the support of International Tropical Timber Organization (ITTO), an experimental program of planting 300 ha of *Corymbia citriodora* (formerly *Eucalyptus citriodora*) was set up [1]. In parallel with this national initiative, the production of *Cymbopogon* essential oil has developed, particularly on the “Plateau des Cataractes” department of Pool, Congo- Brazzaville [2, 3]. *Cymbopogon nardus* acquired from West Africa and acclimatized on the “Plateau des Cataractes” in Congo Brazzaville presents a profile rather close to that of *Cymbopogon winteranius*. Two species produce citral essence (lemongrass): *C. flexuosus*, which has adapted very well, has two varieties that are difficult to discern; *C. flexuosus flexuosus*, rich in citral and *C. flexuosus albesens* rich in citronellal and therefore close to *C. nardus* and *C. winteranius* [2]. This leads to a very high variability in the composition of the oil produced, especially at the domestic level. *C. citratus* which reproduces only by cuttings (absence of flowers) gives an essential oil rich in citral with a constant quality. It is unfortunately the least suitable on the plateau, it is especially very sensitive to water stress in the dry season (May-September). This agronomic disadvantage can be circumvented by optimizing the distillation step on the production chain, particularly the drying and extraction steps.

The general tendency in the literature is to consider these steps as independent operations, while the first (drying) prepares the second (extraction). Better, one can imagine the valorization of a plant as a succession of linked operations of sequential extraction of metabolites, a kind of refining of the plant. In this exploratory work we consider the drying and the extraction of the essential oil as a linked steps, because of, in the phenomenological approach to the extraction of metabolites from solid plant matrices, the extraction process depends greatly on the quantity of cells broken during preliminary operations, particularly drying [4-6]. Modeling is creating a representation aimed at understanding, describing and/or predicting the behavior of a system under given conditions. When the system varies with time, the study is said to be kinetic. For the study of drying and/or extraction, kinetic prediction is an essential tool for modeling processes and optimizing yields. It makes it possible to determine the optimal conditions for these operations running and optimized installations designing.

Theoretical, empirical and semi-empirical mathematical models are often used to describe the behavior of plant matrices during drying as well as that of various metabolites during their extraction. The most relevant models are fitted by different criteria: “Mean Root Standard Error (MRSE)” as small as possible, coefficient of determination (R^2), close to 1; chi-square (χ^2) tending to zero.

Current studies on the modeling of drying and extraction

use the numerical resolution of the model in its mathematical form without necessarily a physical meaning of the results. Thus approach is unfortunately too theoretical for the users targeted at a domestic scale. It would therefore be better to consider empirical models with parameters having an obvious physical meaning [7, 8].

Moreover, for supporting small producers, it would be preferable to take ways with least mathematical prerequisites and computer inputs, in order to guarantee an effective transfer to these users, via simple charts.

The diffusional model, validated, after judicious approximations, by kinetics of order 1 and the Peleg model (pseudo order 2) gave satisfactory results for the drying of plant matrices, in particular those of fruits of *Dacryodes edulis* [9] and those of *Raffia Sese* fruits [10]. These same models also fitted the extraction of other metabolites.

Here, we consider the aromatic plant drying and essential oil extraction of according phenomenological approach due to [4] and widely used in the literature for different metabolites: vegetable oils [11], polyphenols [12], essential oils [13, 14] with the same models, via correlation lines with physical meanings for the parameters generated by these models. This work could later be extended to the extraction of other metabolites: polyphenols, dyes, vitamins, mineral salts..., as well as to the constituents of these different metabolites: fatty acids, amino acids, terpene [15].

2. Materials and Methods

2.1. Plant Material

Cymbopogon citratus studied had harvested à Nkama on “Plateau des Cataractes” in Congo Brazzaville (2021).

2.2. Kinetics of Drying (Extraction of Water)

2.2.1. Term Meanings

The data relating to the drying are expressed in variation of the mass of the sample during the drying $m_t = f(t)$. Reduced mass (m_r) is also used in case of digital data processing.

$$m_r = m_t - m_f / m_0 - m_t \quad (1)$$

Empirical models generally use humidity and its derived expressions.

Moisture (X) represents the water content in the sample calculated on dry matter basis (db).

$$X_{t(spl)} = m_{w(spl)} / m_{spl} \text{ (db)} \quad (2)$$

with m_w : mass of extracted water; m_{spl} : dried mass of sample; X_t , X_0 , X_∞ : moisture in the sample at any time t, moisture at t =

0, moisture at equilibrium: end of the process (t_{∞}).

In a similar way, the humidity extracted is defined as the quantity of water extracted at t reduced to the mass of the sample in dry matter basis:

$$X_{t(\text{ext})} = m_{w(\text{ext})}/m_{\text{spl}} \text{ (db)} \quad (3)$$

A study [16] defines the moisture ratio (X_r) as:

(Residual moisture in the sample)/(total moisture in the sample a):

$$X_{r(\text{spl})} = (X_t - X_e)/(X_0 - X_e). \quad (4)$$

If $X_e \ll X_0$, X_t ,

$$X_{r(\text{spl})} = X_t/X_e. \quad (5)$$

In this study, extracted moisture ration $X_{r(\text{ext})}$ was the extracted moisture at time t ($X_{t(\text{ext})}$) versus extracted moisture at t_{∞} , the end of the process ($X_{e(\text{ext})}$).

$$X_{r(\text{ext})} = X_{t(\text{ext})}/X_{\infty} \quad (6)$$

We get rid of the humidity calculation basis (wet matter or dry matter of the sample) by taking the ratio of the masses of water extracted.

In effect

$$X_{r(\text{ext})} = X_{t(\text{ext})}/X_{\infty} = (m_{w(\text{ext})t}/m_{\text{spl}})/[m_{w(\text{ext})\infty}/m_{\text{spl}(\text{ext})}] \quad (7)$$

$$X_{r(\text{ext})} = m_{w(\text{ext})t}/m_{w(\text{ext})\infty} \quad (8)$$

Furthermore, it is shown the similarity of expressions of the reduced mass and that of reduced humidity.

Indeed

$$m_t = (m_t - m_f)/(m_0 - m_f), \quad m_0 - m_f = m_{w(\text{ext})\infty} \quad (9)$$

the quantity of water extracted at t_{∞} , (end of the process) and

$$m_t - m_f = m_{w(\text{ext})t} \quad (10)$$

the quantity of water extracted at the moment,

$$m_t = m_{w(\text{ext})t}/m_{w(\text{ext})\infty} = X_r \quad (11)$$

the reduced humidity, as we established above.

2.2.2. Modeling

Different models have been proposed in the literature to simulate the drying of plant matrices.

1) Diffusional model

The process is described by the sum equation [6]:

$$q_t/q_{\infty} = f \exp(-k_1 t) + (1-f) \exp(-k_2 t) \quad (12)$$

In no washing step assumption, the process is controlled by diffusion step and leads to:

$$q_t/q_{\infty} = 1 - \exp(-k_2 t) \quad (13)$$

dealing to following linear form:

$$\ln(1/(1-y)) = k_2 t \quad (14)$$

with

$$y = Y_t/Y_{\infty} \quad (15)$$

2) Sorption Model (Peleg)

Peleg model [17] was proposed to explain the behavior of the recovery of several natural metabolites from plant matrices. One assumes that the phenomenon runs as following type law:

$$q_t = q_0 \pm t/(k_1 + K_2 t) \quad (16)$$

with: $\pm$ sorption, adsorption (+) and desorption, (-); q_t : extracted metabolite quantity at time t (mt); q_{∞} : extracted metabolite quantity at t_{∞} ; $q_0 = 0$: extracted metabolite quantity at $t = 0$; k_1 : first order kinetic extraction constant, K_2 : constant of extraction capacity linked to equilibrium at the end of the process [18]. For a metabolite desorption, the linear form of this equation was used to fit Peleg's model is:

$$t/q_t = k_1 + K_2 t. \quad (17)$$

The validation criteria generally used was: theminimization of Mean Root Standard Error or The coefficient of determination (R^2 , around 1) of following straight line:

$$t/q_t = f(t) \quad (18)$$

[slope K_2 : Peleg extraction capacity constant (q^{-1}) and ordinate at the origin = k_1 : Peleg kinetic constant (order 2, $t \cdot q^{-1}$ unit)].

One deduced: (i) the kinetic desorption constant (first order) at the start of the process:

$k = K_2/k_1$, (t^{-1} unit) and (ii) extracted metabolite quantity at equilibrium (the end of the process): $q_{\infty} = 1/K_2$ (q^{-1} unit). Peleg model could be used to simulate drying as desorption of moisture (X_t).

2.3. Essential Oil (EO) Extraction Kinetics

The kinetic modeling of the metabolite extraction from a solid plant matrix is very active field of research. It is an important step to scale-up metabolite extraction yields. The models used are based on physical laws (formal kinetics, Fick's diffusion law...) or on empirical laws [11, 19].

We presented in detail the modeling of the hydrodistillation of the essential oil of the fruits of *Xylopia aethiopica* previ-

ously [20] with the first order diffusion models [6, 11, 19, 21]; and the desorption model of Peleg [17]. We will use these two models already widely used in the literature for the hydrodistillation of *C. citratus* at the laboratory scale.

2.4. Statistics

Descriptive statistics and graphical representations were produced using Microsoft Excel 2019. The models were validated graphically by regression lines with a coefficient of determination $R^2 > 0.90$.

3. Results and Discussion

3.1. Drying Modeling

3.1.1. Dry in the Open Air and in the Shade

The raw data collected on drying is generally expressed as the loss of mass versus drying time $m_t = f(t)$. Table 1, Figures 1 and 2 gathered results on drying in the open air under shade of *C. citratus* leaves. The sample mass m_t and sample moisture $X_{r(spl)}$ decreased and mass of extracted moisture increased ($m_{w(ext)}$ and $X_{t(ext)}$, g/g). In the literature, the exponential decreasing shape was explained by the Fick diffusional model, and the hyperbolic increasing shape by the Peleg model.

Table 1. Variation of masses and moisture of *C. citratus* leaves versus drying time.

t(d)	0	1	2	3	4	5	6	7	8	9
m_t (g) run 1	24,8	13,5	11,82	7,6	9,31	9,25	8,83	8,72	8,52	8,31
m_t (g) run 2	17,73	7	6,61	3,31	5,76	5,68	5,83	5,78	5,65	5,41
$m_{w(ext)}$ (g) run 1	0	11,3	12,98	17,2	15,49	15,55	15,97	16,08	16,28	16,49
$m_{w(ext)}$ (g) run 2	0	10,73	11,09	14,42	11,97	12,05	12,05	11,95	12,32	12,32
X_r run 1	0	0,685	0,787	1,04	0,96	0,944	0,968	0,975	0,987	1
X_r run 2	0	0,87	0,9	1,17	0,97	0,98	0,98	0,97	1	1
X_t (g/g) _{ext} run 1	0	1,36	1,602	2,07	1,864	1,871	1,922	1,935	1,959	1,984
X_t (g/g) _{ext} run 2	0	1,98	2,04	2,66	2,21	2,23	2,23	2,21	2,28	2,28
$X_{t(spl)}$ (g/g) run 1	1,984	0,624	0,422	0,085	0,12	0,11	0,062	0,05	0,025	0
$X_{t(spl)}$ (g/g) run 2	2,28	0,98	0,22	0,1	0,1	0,08	0,08	0,1	0	0

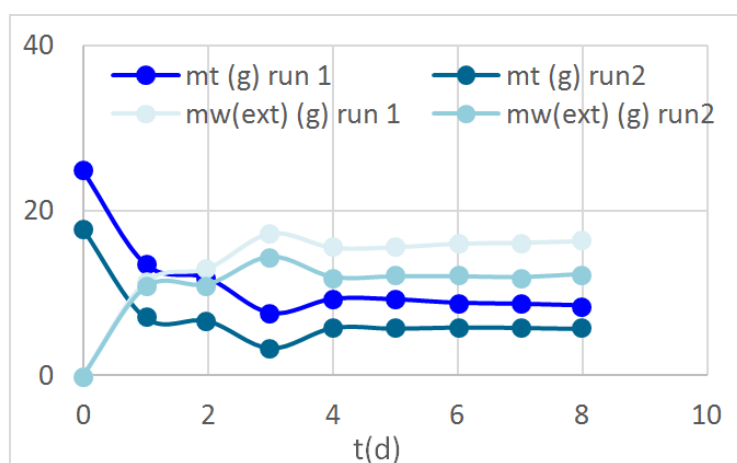


Figure 1. Curve sharps of mass losses and water desorption during *C. citratus* air drying: $m_t = f(t)$; $m_{w(ext)} = f(t)$.

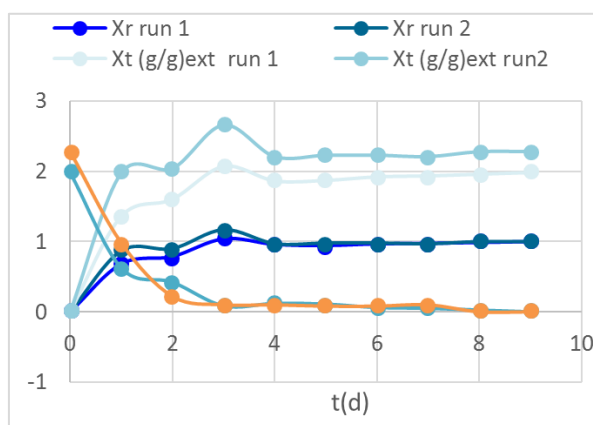


Figure 2. Curve sharps of variation of moisture versus drying time during *C. citratus* air drying: $Xt(ext) = f(t)$, $Xr = f(t)$ and $Xt(spl) = f(t)$.

These two models were tested for the drying in open air and under shade of *C. citratus* leaves. Only Peleg model fitted experimental data using its linear form:

$$t/Xt = k_1 + K_2 t$$

This curve is a straight line with slope = K_2 : the extraction capacity constant and the ordinate at the origin k_1 , the 2nd order kinetic constant (Figure 3):

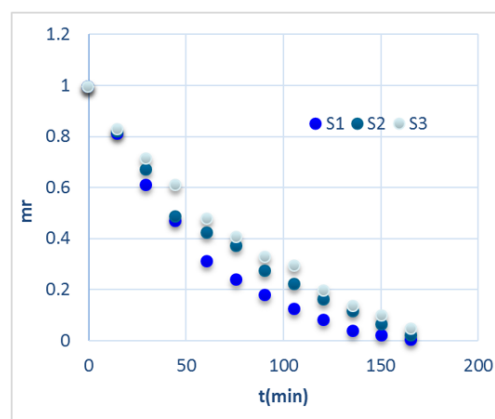
$k_1 = 0.6047 \text{ d (g/g)}^{-1}$ and $K_2 = 1.4435 \text{ (g/g)}^{-1}$ with $R^2 = 0.9955$

We deduce from these two quantities:

$k = K_2/k_1 = 1.4435/0.6047 = 2.4049 \text{ d}^{-1}$, the kinetic drying constant of order 1

$t_{1/2} = 0.25 \text{ d}$, the duration of the half-process

The end of drying could be estimated at $t_{1/2} = 1.43 \text{ d}$.



(a)

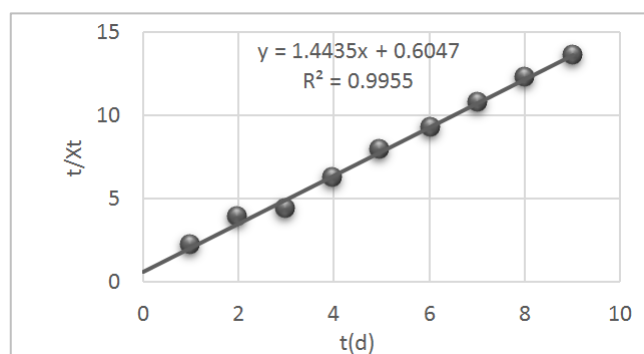
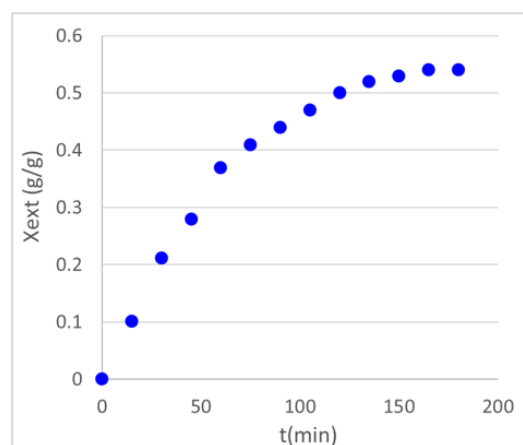


Figure 3. Peleg model validation straight line: $t/X_t = k_1 + K_2 t$.



(b)

3.1.2. Oven Drying

Drying in the oven gave more regular curves, and therefore more easily readable diagrams (Figure 4).

Figure 4. Curve sharps of oven drying of 3 samples of *C. citratus* leaves (70 °C).

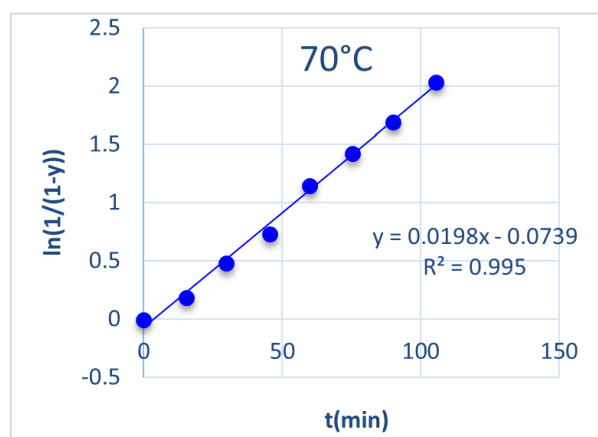
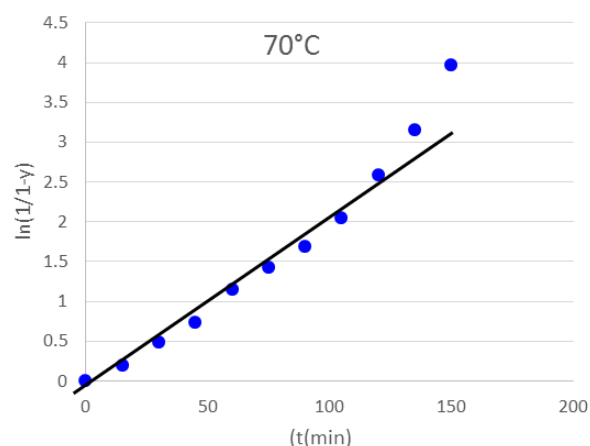


Figure 5. Validation of the diffusional model: oven drying straight line of *C. citratus* leaves.

The observed shapes of the drying curves in the oven suggest an exponential evolution of the loss of mass (diffusional model, figure 4a) and a hyperbolic evolution of the moisture extracted (Peleg sorption model Figure 4b).

These two models could be validated by regression lines. The diffusional model postulates a process taking place on two sites (extra and intracellular) in two stages running at different rates: the fast stage for the “extracellular” metabolite which is extracted almost spontaneously; the slow step which depends on the intracellular diffusion of the metabolite before extraction.

In no washing step assumption, the process is controlled by diffusion step and leads to following linear form:

$$\ln(1/(1-y)) = k_2 t$$

with $y = X_t/X_\infty$

Moisture extraction from *Cymbopogon citratus* leaves follows first-order kinetics for, at least, the first 100 minutes of drying in the oven at 70 °C ($R^2 = 0.995$), with a rate constant $k = 0.0198 \text{ min}^{-1}$ and a half process time $t_{1/2} = 0.69/0.0198 = 34.8 \text{ min}$. The overall extraction time could be evaluated at $5t_{1/2} = 174 \text{ min}$ (Figure 5).

Results on drying at 90 and 105 °C were gathered in Table 2.

Table 2. Kinetic constant, k ; half-process time $t_{1/2}$ and overall drying time of *C. citratus* leaves.

Drying temperature (°C)	$k \text{ (R}^2\text{)min}^{-1}$	$t_{1/2} \text{ (min)}$	Duration of the process (min)
70	0.0198 (0.9950)	34.8	174.0
90	0.0262 (0.9860)	26.3	131.5
105	0.0225 (0.9946)	30.67	153.3

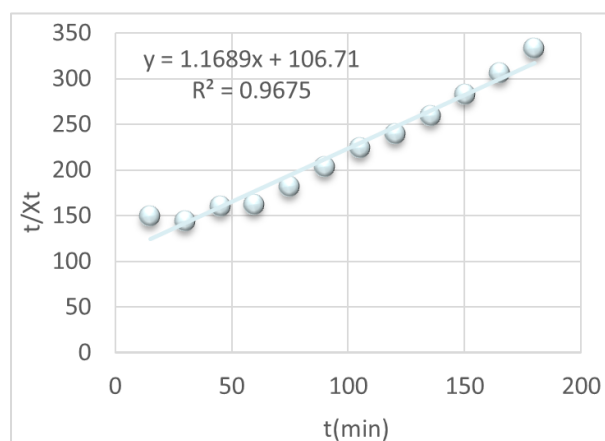
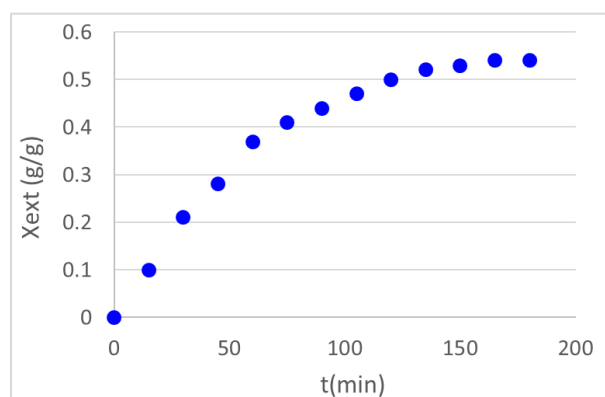


Figure 6. Peleg model validation line for oven drying at 70 °C.

It emerges from this preliminary work that between 70 and 105 °C moisture was extracted with a kinetic constant $k = 0.0198 - 0.0262 \text{ min}^{-1}$ corresponding to $t_{1/2} = 30 \text{ min}$ and therefore a drying time of about $5t_{1/2} = 2 \text{ h } 30 \text{ min}$.

The validation line of the Peleg desorption model $t/X_{t(\text{ext})} = f(t)$ was given in Figure 6 for oven drying at 70 °C. It fitted the experimental results and leads to a second order extraction

rate constant $k_1 = 106.71 \text{ min (g/g)}^{-1}$ and an extraction capacity constant $K_2 = 1.1689 \text{ (g/g)}^{-1}$ ($R^2 = 0.9675$). Finally the extraction constant of order 1, $k = K_2/k_1 = 1.1689/106.71 =$

0.0110 min^{-1} and the maximum moisture extracted is $X_{w\infty} = 1/K_2 = 1/1.1689 = 0.855 \text{ g/g (db)}$.

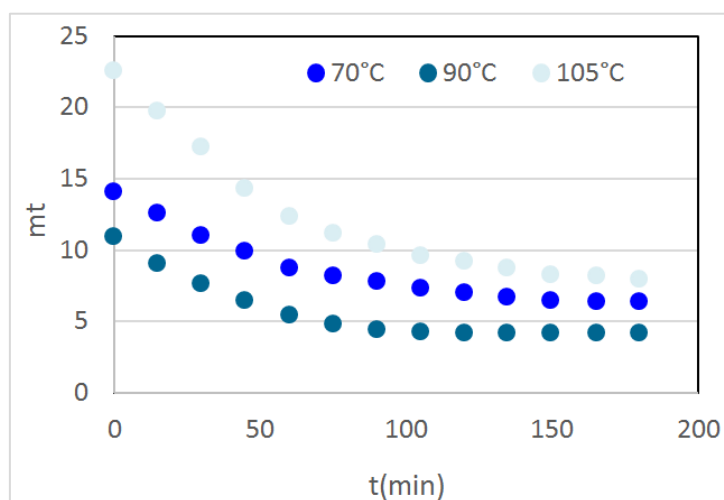


Figure 7. Drying curves $mt = f(t)$ of *C. citratus* leaves at 70, 90 and 105 °C.

Figure 7 gathered the curves of the mass variation versus drying time at different temperatures. Drying data at 70, 90 and 105 °C were gathered in Table 3.

Table 3. Drying data at 70, 90 and 105 °C of *C. citratus* leaves.

Drying temperature (°C)	$k_1 (R^2) \text{ min. (g/g)}^{-1}$	$K_{2\%}^{-1} \text{ (g/g)}^{-1}$	$K \text{ min}^{-1}$	$t_{1/2} \text{ (min)}$	$X_{w\infty} \text{ g/g db}$
70	106.71 (0,9675)	1.1689	0.0110	62.7	0.855
90	55.164 (0,9714)	1,2284	0.0223	30.9	0.818
105	61.141 (0,9929)	1,1812	0.0193	35.8	0.847

The Peleg model leads to a 2nd order kinetic constant $k_1 = 55.164 - 106.71 \text{ min (g/g)}^{-1}$. We deduce a 1st order drying constant $k = 0.110 - 0.0223 \text{ min}^{-1}$ and $t_{1/2} = 31-63 \text{ min}$. The maximum moisture extracted was $X_{w\infty} = 0.818 - 0.855 \text{ g/g (db)}$.

The two models converge towards the same value of the 1st order drying rate constant:

$k = 0.0110 - 0.0262 \text{ min}^{-1}$ with $t_{1/2} = 30-60 \text{ min}$ and a drying time $t = 130-300 \text{ min}$.

For drying under shade and in the open air, these quantities were respectively.

$k_1 = 0.6047 \text{ d. (g/g)}^{-1}$; $X_{w\infty} = 1/K_2$; $k = K_2/k_1 = 2.387 \text{ d}^{-1}$; $t_{1/2} = 0.289 \text{ d}$; $t = 1.45 \text{ d}$

3.2. Extraction Modeling

The Table 4 presents the data necessary to test the two selected models.

Table 4. Data on validation of two studied models for the hydrodistillation of *C. citratus* leaves.

Crushed leaves $Y_{\infty}=0, 28$								
Time (min)	0	15	30	45	60	75	90	105
EO mass m(t)	0	0.08	0.1	0.12	0.14	0.14	0.14	0.14

Crushed leaves $Y_{\infty}=0,28$

Y_t ($m_t/m_{\text{éch}}$) 100	0	0.16	0.2	0.24	0.28	0.28	0.28	0.28
t/Y_t		93.75	150	187.50	214.29	267.86	321.43	375.00
$\ln(1/(1-y))$	0	0.847	1.252	1.945				
Cut leaves (6 cm) $Y_{\infty}=0,76$								
Time (min)	0	15	30	45	60	75	90	105
EO mass $m(t)$	0	0.65	0.75	0.76	0.76	0.76	0.76	0.76
Y_t ($m_t/m_{\text{éch}}$) 100	0	0.65	0.75	0.76	0.76	0.76	0.76	0.76
t/Y_t		23.08	40.00	59.21	78.95	98.68	118.42	138.16
$\ln(1/(1-y))$	0	1.932	4.330					

3.2.1. Shapes of Essential Oil Extraction Curves

There was a very speedy essential oil extraction rate, especially with the leaves cut into 6 cm pieces (15min) which have retained their thin layer structure compared to the finely ground leaves, the oil of which was extracted more slowly

(60min), probably due to the settlement phenomenon (Figure 8).

The virtual absence of the diffusion step on the extraction curve suggests a localization of the oil to be extracted on the surface of the leaf. It is therefore in contact with the extraction solvent which drags it by simple washing.

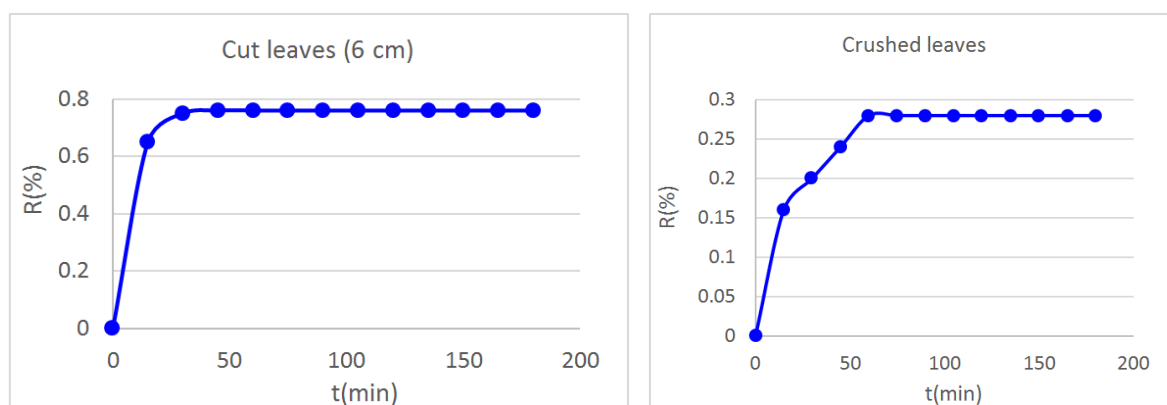


Figure 8. Extraction curves of the essential oil of *C. citratus* by hydrodistillation.

3.2.2. Peleg's Desorption Model

The most probable Peleg model was therefore tested and fitted the experimental data (with very good coefficients of determination ($R^2 > 0.99$)) (Figure 9).

The two studied samples gave very different extraction

yields (0.28 and 0.76% at 105min). One notes a high variability in the essential oil content of the "all-comers" plant material used.

From Figure 9, Table 5 was constructed and the first-order extraction constant $k = K_2/k_1$ was calculated as follows.

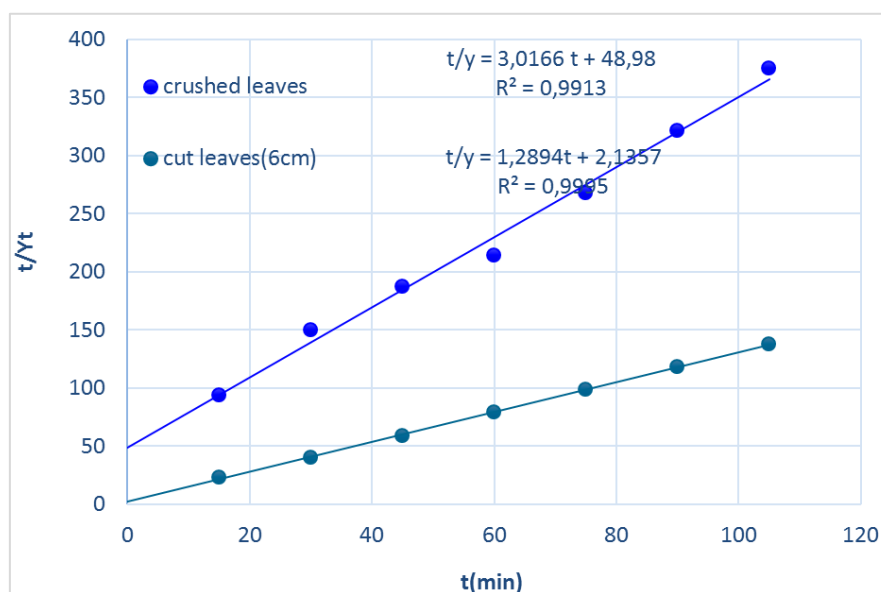


Figure 9. Peleg model validated straight lines for *C. citratus* leaves.

Table 5. Kinetic parameters data for *C citratus* hydrodistillation.

	$k_{1min} \cdot \%^{-1}$	$K_2 \%^{-1}$	R^2	$k = K_2/k_{1min}^{-1}$	$Y_{\infty} \%$
Crushed leaves	48.98	3.0166	0.9913	0.0616	0.53
Cut leaves	2.1357	1.2894	0.9995	0.6037	0.78

One notes a high difference on constant values, but in the same magnitude.

3.2.3. Diffusional Model of Sovova-Milojevic

The diffusional model was validated only the first moments of the extraction ($t < 60$ min) which corresponds for the cut leaves to more than 90% of the total quantity of essential oil extracted. Straight lines $\ln(1/(1-y))$ in Figure 10 validates the first order kinetic and leads to:

$$k_1 (\text{crushed leaves}) = 0.1443 \text{min}^{-1}$$

$$k_1 (\text{cut leaves}) = 0.0416 \text{min}^{-1}$$

Essential oil was extracted more rapidly in structured leaf (thin layer) than in crushed leaf, maybe by settling of plant material (fine powder).

4. Conclusion

Drying in the open air and under shade (25-35 °C) takes place according to a 2nd order kinetic with rate constant of $0.6047 \text{ d (g/g)}^{-1}$ and leads to a residual content of $0.6928 \text{ (g/g)}^{-1}$.

A first-order drying rate of 2.4 d^{-1} was deduced. The 1st

order diffusional model was not validated here, whereas it was during oven drying (70-105 °C) with a rate constant $k_1 = 0.0198 \text{min}^{-1}$ and a half of the process time $t_{1/2} = 34.8 \text{min}$. The overall extraction time $5t_{1/2} = 174 \text{min}$. Oven drying (70-105 °C) also fitted the Peleg model with a 2nd order kinetic constant $k_1 = 55.16 - 106.71 \text{min. (g/g)}^{-1}$ and a residual moisture content $X_{w\infty} = 0.818 - 0.855 \text{ g/g (db)}$ leading to a 1st order drying constant $k = 0.0110 - 0.0223 \text{min}^{-1}$, a half-process time $t_{1/2} = 30.9 - 62.7 \text{min}$ an overall process time $5t_{1/2} = 154.5 - 313.5 \text{min}$. In the selected temperature intervals, solar drying, considered as a moisture desorption, proceeds more than 10 times slower than oven drying, following a second-order kinetic according to the Peleg model. The extraction of essential oil by hydrodistillation is validated by two models studied, with perfectly consistent results: a 1st order kinetic constant of hydrodistillation $k_1 = 0.0616 - 0.6037 \text{min}^{-1}$ and a maximum extraction yield $Y_{\infty} = 0.53 - 0.78\%$. The essential oil was extracted with a little more difficulty than moisture from the leaves of *C. citratus* but at similar magnitude, probably according to very similar mechanisms.

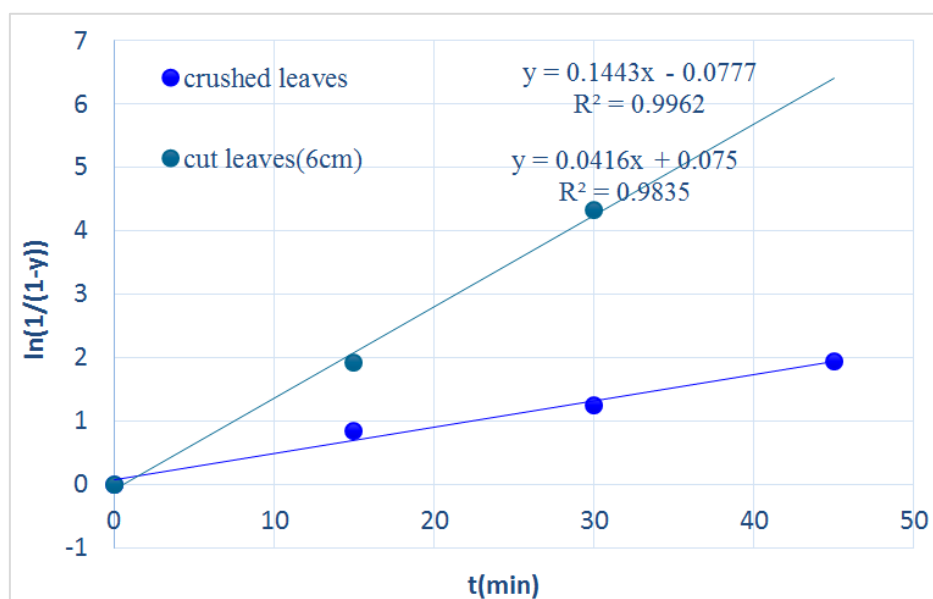


Figure 10. Sovova-Milojevic model validated straight lines for *C. citratus* leaves.

Drying could therefore be described by the empirical methods widely used in the literature to model the extraction of various metabolites from plant matrices.

Abbreviations

ITTO	International Tropical Timber Organization
CVPFNL	Non Timber Forest Products Valorization Center
UMNG	Marien Ngouabi University
MRSE	Mean Root Standard Error
EO	Essential Oil

Acknowledgments

Authors thank the Higher School of Technology “Les Cataractes” (EPrES) and the Faculty of Sciences and Techniques, Marien Ngouabi University (UMNG), Non timber forest products Valorization Center (CVPFNL) for the scientific and logistic support.

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Data Availability Statement

The data is available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare no conflicts of interest.

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