

Modeling with Simple Graphic Methods of the Extraction of *Corymbia citriodora* Essential Oil on an Artisanal Scale in Congo-Brazzaville

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Abstract

The modelling of pilot-scale extraction of *Corymbia citriodora* essential oil, as a support activity for the development of the national “essential oils” programme, was carried out using steam hydrodistillation on a locally-built distiller. The diffusion and desorption models tested both fitted the experimental data. The diffusional model established that 1) 75% of the essential oil originated from the broken cells and was released during the fast wash step with an extraction rate constant $k_1 = 0.0233 \pm 0.0006 \text{ min}^{-1}$; 2) the slower diffusion step involved the remaining 25% of essential oil with a rate constant of $0.0021 \pm 0.0002 \text{ min}^{-1}$; 3) the rate constant of the overall process is : $k = 0.0305 \text{ min}^{-1}$, assuming first-order kinetics ($t_{1/2} = 22.62 \text{ min.}$). The Peleg desorption model leads to 1) a second-order rate constant $k_1 = 13.3 \pm 3.6 \text{ min}^{-1}$; 2) an extraction capacity constant $K_2 = 0.59\%^{-1} \pm 0.20\%^{-1}$; and finally 3) a first-order rate constant for the overall extraction $k = 0.0474 \pm 0.002 \text{ min}^{-1}$ and a maximum average extraction yield $Y_\infty = 1.8\% \pm 0.5\% \text{ (db)}$. Simple graphical processing with little computer data is well suited to solve this specific problem: the development of the essential oil sector on a small scale and in rural areas as a means of poverty alleviation.

Keywords

Kinetic Modeling, *Corymbia citriodora* Extraction, Pilot Scale, Congo-Brazzaville

1. Introduction

The Australian *Eucalyptus citriodora* was successfully adapted to different condi-

tions throughout the world. In Congo, it is one of some 30 eucalyptus species acclimatized at Pointe-Noire for papermaking. However, *Eucalyptus citriodora*, reclassified *Corymbia citriodora* is better known for the essential oils extracted from its leaves in relatively large amounts. These oils are mostly used in cosmetics and hygiene products. The local populations use infusions of *Eucalyptus citriodora* leaves to treat colds and flu. *Corymbia citriodora* is the species with the highest oil content of the genus *Corymbia* [1]. It was retained as a basic crop for the development of the essential oil sector in Congo Brazzaville. The acclimatization of a dozen of origins from Australia and Madagascar leads to select high biomass production trees with high essential oil content (2% - 7%) and very rich in citronellal (60% - 90%) [2] [3]. These 3 last decades, the Congo launched the production of essential oils initiative in rural areas and on an artisanal scale as a way to fight poverty, with the support of ITTO through a development project [4]. To contribute to the development of this emerging sector, the Higher School of Technology "les Cataractes" supports producers in the cultivation and distillation of aromatic plants by 1) setting up cropping techniques [1] [3] [5] [6]; 2) studying biodiversity, in particular the chemotypes identification [7] [8]. 3) modeling the essential oils extraction [1]-[3] [5] [6]. This important activity highlighted the importance of technology (drying and extraction) in addition to the nature of plant material in the efficiency of the extraction process. The project opted for drying in the shade and in the open air, on the one hand, and hydrodistillation in the open air, on the other hand, and hydrodistillation at atmospheric pressure and over a wood fire. The results on drying were previously published [7]. This work on modelling the extraction of essential oils from *Corymbia citriodora* aims to establish and make available simple methods for controlling the extraction mechanism of essential oils.

2. Material and Methods

2.1. Plant Material

Corymbia citriodora acclimatized in Congo was on an average 4% oil content and belongs to the citronellal chemotype (60% - 80%) [3] [5]. The pilot extraction was carried out at the Center of Valorization of Non-Timber Forest Products (CVPFNL) on raw plant material harvested on the Centre's plantations in Pointe Noire.

2.2. Extraction Kinetics

50 kg of leaves and 100 litres of water were introduced into a 300-litre distiller fitted with a grid to separate the water from the plant matter. This first trial was repeated 2 times. The third trial involved 16 kg of leaves and 30 litres of water. The distillate was collected separately in different flasks at 15, 30, 45, 60, 90, 120, 180 minutes. Let m_1 be the mass of plant material on dried matter basis (db) and m_2 the quantity of essential oil collected at time t , the extraction yield is given by:

$$Y_t(\%) = (m_2/m_1)100 \quad (1)$$

In all cases, the extractions were carried out in a locally built distiller, at atmos-

pheric pressure with water in the steam and liquid states (steam-hydrodistillation).

Different models were used in the literature to simulate essential oil extraction by hydrodistillation; two of these, used in this study were previously presented [10]-[12].

The first-order diffusional model [13]-[15] and the Peleg model [16] through their simple graphical resolution respond well to the context of the study, namely extraction in rural areas at small scale using atmospheric pressure hydrodistillation. **Table 1** summarizes the mathematical equations and the meaning of the terms used in this study.

Table 1. Diffusional model and Peleg model: mathematical equations and term meanings.

| Mathematical equations [references] | Term meanings |
|---|--|
| DIFFUSIONNEL MODEL | |
| Two steps mechanism (washing and diffusion) | |
| $q_t/q_\infty = f \exp(-k_1 t) + (1 - f) \exp(-k_2 t)$ <p style="text-align: center;">[13] if $f = 0$:</p> $q_t/q_\infty = 1 - \exp(-kt)$ $\ln(1/(1 - y)) = kt$ <p style="text-align: center;">[14] [15]</p> | q_t, q_∞ : quantity of the essential oil extracted at time t and t_∞ f : oil fraction extracted in the washing step. k_1 : kinetic constant of washing step k_2 : kinetic constant of diffusion step $y = q_t/q_\infty$ k : kinetic constant of global extraction (order 1) |
| $Y_t/Y_\infty = k_1 t/Y_\infty + (1 - \exp(-kt))$ <p style="text-align: center;">[17]</p> | Y_t, Y_∞ : yield à t and t_∞ k_1 : kinetic constant of linear step k : kinetic constant of curvilinear step |
| $q_t/q_\infty = b + kt$ <p style="text-align: center;">[18]</p> | q_t, q_∞ : quantity of the essential oil extracted at time t and t_∞ k : kinetic constant of linear step b : characteristic parameter of the curvilinear step |
| PELEG MODEL | |
| $q_t = q_\infty + t/(k_1 + K_2 t)$ $t/q_t = k_1 + K_2 t$ $k = K_2/k_1,$ $q_\infty = 1/K_2$ <p style="text-align: center;">[16]</p> | q_t : quantity of the essential oil extracted at time t (q_t); (q_∞ : quantity of the solute extracted at t_∞ ; $q_0 = 0$: the quantity of the metabolite extracted at $t = 0$; k_1 : kinetic extraction constant (order 2), K_2 constant extraction capacity linked to end equilibrium of the process; k kinetic constant of global extraction (ordre 1) |

2.3. Statistical Processing

Statistics and graphs were carried out on Excel 2019 software.

3. Results and Discussion

3.1. Highlighting the Two-Stage Process and Assessing the Importance of Each Stage

Table 2 reports the extraction data using a rural distiller. Experiments 1 (run 1) and 2 (run 2) were carried out with 50 kg of plant raw material and experiment 3 (run 3) with 16 kg. The first two experiments relating to the extraction of 50 kg of leaves lead to different yields (1.08% et 1.78%); thus confirming the heterogeneity of used raw material. Experiment 3 with a filling rate 3 times lower (16 kg) leads to the highest yield of 3 extractions (1.91%). On the other hand, examination of the curves $Y_t/Y_\infty = f(t)$ (**Figure 1**) which represents the fraction of oil extracted as a function of time leads to three superimposable curves and corresponding to an extraction in two steps with different durations. They are identical to those obtained previously in the laboratory scale [10].

Table 2. Pilot extraction of essential oil from *Corymbia citriodora* leaves.

| | | | | | | | | | |
|-------|----------------|------|------|------|------|------|------|------|------|
| Run 1 | $Y_t(\%)$ | 0.00 | 0.40 | 0.78 | 0.88 | 0.94 | 0.99 | 1.03 | 1.08 |
| | Y_t/Y_∞ | 0.00 | 0.37 | 0.72 | 0.81 | 0.87 | 0.92 | 0.95 | 1.00 |
| Run 2 | $Y_t(\%)$ | 0.00 | 0.60 | 1.19 | 1.36 | 1.49 | 1.60 | 1.67 | 1.73 |
| | Y_t/Y_∞ | 0.00 | 0.35 | 0.69 | 0.79 | 0.86 | 0.92 | 0.97 | 1.00 |
| Run 3 | $Y_t(\%)$ | 0.00 | 0.67 | 1.31 | 1.56 | 1.69 | 1.81 | 1.86 | 1.91 |
| | Y_t/Y_∞ | 0.00 | 0.35 | 0.69 | 0.82 | 0.88 | 0.95 | 0.97 | 1.00 |

In first approximation, Y_∞ was substantially equal to Y_{180} , taking into account the asymptotic ending of the extraction.

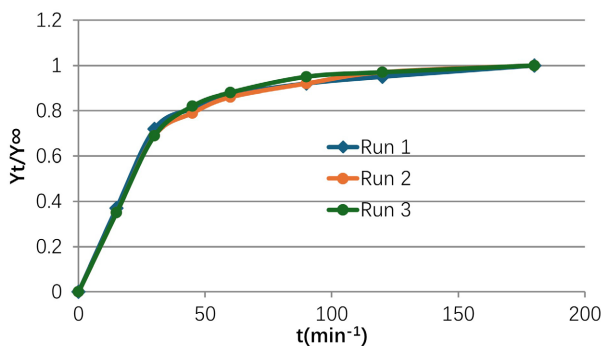


Figure 1. Pilot scale extraction curves of the essential oil from the leaves of *Corymbia citriodora* ($Y_t/Y_\infty = f(t)$).

[17] hypothesis considers the first step of the process as linear and the second, curvilinear:

$$Y_t/Y_\infty = (k_1/Y_\infty)t + (1 - \exp(-kt)) \tag{2}$$

In **Figure 2**, the break is around 45 min of the distillation time. The linear part was noted:

$$Y_t/Y_\infty = (k_1/Y_\infty)t \tag{3}$$

with a slope (k_1/Y_∞) leading to k_1 : the rate constant of the fast washing step.

The initial ($0 < t < 45$ min) straight line equation: $Y_t/Y_\infty = 0.024x + 0.0033$ ($R^2 = 0.9998$) leads to following value of k_1 (Figure 2)

$$k_1/Y_\infty = 0.024$$

$$k_1 = 0.024Y_\infty = 0.024 \times 1.08 = 0.026 \text{ min}^{-1}$$

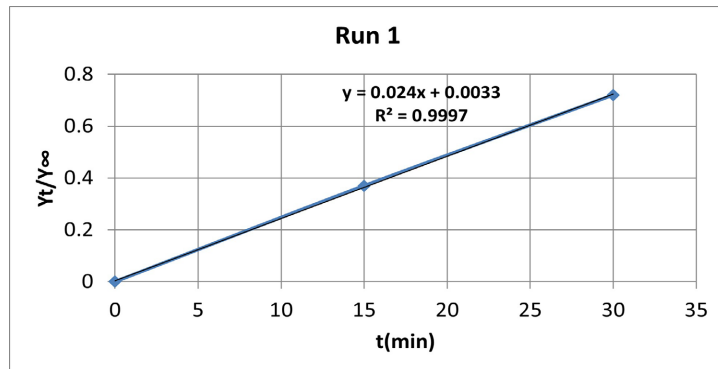


Figure 2. Determination of the kinetic rate constant (order 1) of the washing step in accordance with the hypothesis [17].

[18] adopts the symmetric hypothesis and considers the washing step as curvilinear and the diffusion step as linear (Figure 3; $45 \text{ min} < t < 180 \text{ min}$)

$$Y_t/Y_\infty = b + kt \tag{4}$$

with b as the characteristic parameter of the first stage and corresponding to the fraction of the oil extracted during this stage; this is the parameter f of [13].

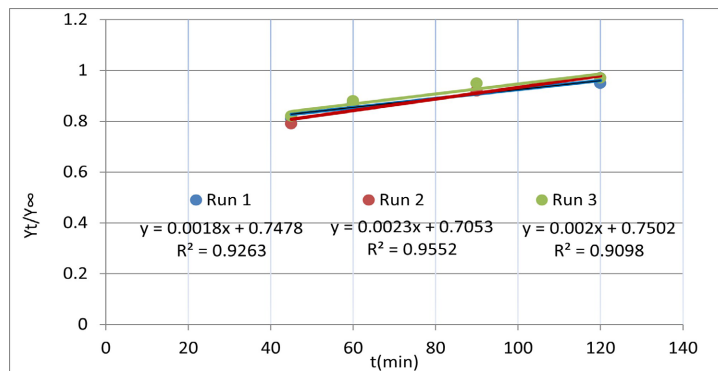


Figure 3. Determination of the kinetic constant (order 1) of the diffusion step and of the fraction of essential oil extracted during the washing step.

Table 3 summarizes the information for the two steps of the pilot-scale extraction of essential oil from the leaves of *Corymbia citriodora*.

Table 3 shows that: 1) the kinetic rate constant of the washing step is on average $0.0233 \pm 0.0006 \text{ min}^{-1}$ and that of the diffusion step, $0.0021 \pm 0.0002 \text{ min}^{-1}$, leading to an average rate ratio of 11 in favor of the washing step; 2) the fraction of extracted essential oil *via* the washing step is 74% on average.

Table 3. Summary of the parameters relating to the two stages extraction of *Corymbia citriodora* essential oil.

| | *k _{washing} (R ²) min ⁻¹ | **k _{diffusion} (R ²) min ⁻¹ | ***R | **% extracted EO |
|-----------|---|--|------|------------------|
| Run 1 | 0.0240 (0.9997) | 0.0018 (0.9263) | 13 | 75 |
| Run 2 | 0.0230 (0.9999) | 0.0023 (0.9552) | 10 | 71 |
| Run 3 | 0.0230 (0.9999) | 0.0020 (0.9098) | 11 | 75 |
| Mean (SD) | 0.0233 (0.0006) | 0.0020 (0.0002) | 11 | 74 |

*[17]; **[18], ***R: k_{washing}/k_{diffusion}; EO: Essential oil.

Finally, despite the heterogeneity of raw material and filling ratio of the distiller, the extraction takes place according to the same mechanism for the 3 experiments.

3.2. Testing the Different Models

3.2.1. Diffusional Model (Phenomenological Approach)

The two-steps (two-sites) diffusional model (broken and intact cells) of [13] postulates that the extraction yield can be described by the expression:

$$q_t/q_\infty = f \exp(-k_1 t) + (1 - f) \exp(-k_2 t) \tag{5}$$

with *f*, the oil fraction extracted in the washing step.

When the extraction is done without a washing step, *i.e.* *f* = 0 [15], this expression is reduced to:

$$q_t/q_\infty = 1 - \exp(-kt) \tag{6}$$

leads to a linear form:

$$\ln(1/(1 - y)) = kt \tag{7}$$

with

$$y = q_t/q_\infty = Y_t/Y_\infty \tag{8}$$

The validation of the model can therefore be done graphically by this last equation.

Table 4 gathers the data necessary for the validation of the two models tested.

Table 4. Data used to test the two studied models.

| | Run 1 | | | | | | | |
|------------------------------------|-------|-------|-------|-------|-------|-------|--------|--------|
| t (min ⁻¹) | 0 | 15 | 30 | 45 | 60 | 90 | 120 | 180 |
| 1/t | | 0.07 | 0.03 | 0.02 | 0.02 | 0.01 | 0.01 | 0.01 |
| Y _t (%) | 0 | 0.4 | 0.78 | 0.88 | 0.94 | 0.99 | 1.03 | 1.08 |
| y = Y _t /Y _∞ | 0 | 0.37 | 0.72 | 0.81 | 0.87 | 0.92 | 0.95 | 1.00 |
| t/Y _t | | 37.50 | 38.46 | 51.14 | 63.83 | 90.91 | 116.50 | 166.67 |
| 1 - y | 1 | 0.63 | 0.28 | 0.19 | 0.13 | 0.08 | 0.05 | 0.00 |
| 1/(1 - y) | 1 | 1.59 | 3.60 | 5.40 | 7.71 | 12.00 | 21.60 | |
| ln[1/(1 - y)] | 0 | 0.46 | 1.28 | 1.69 | 2.04 | 2.48 | 3.07 | |

Continued

| Run 2 | | | | | | | | |
|--------------------|---|------|-------|-------|-------|-------|-------|--------|
| Y_t (%) | 0 | 0.6 | 1.19 | 1.36 | 1.49 | 1.6 | 1.67 | 1.73 |
| $y = Y_t/Y_\infty$ | 0 | 0.35 | 0.69 | 0.79 | 0.86 | 0.92 | 0.97 | 1.00 |
| t/Y_t | | 25 | 25.21 | 33.09 | 40.27 | 56.25 | 71.86 | 104.05 |
| $1 - y$ | 1 | 0.65 | 0.31 | 0.21 | 0.14 | 0.08 | 0.03 | 0.00 |
| $1/(1 - y)$ | 1 | 1.53 | 3.20 | 4.68 | 7.21 | 13.31 | 28.83 | |
| $\ln[1/(1 - y)]$ | 0 | 0.43 | 1.16 | 1.54 | 1.98 | 2.59 | 3.36 | |

| Run 3 | | | | | | | | |
|--------------------|---|-------|-------|-------|-------|-------|-------|-------|
| Y_t (%) | 0 | 0.67 | 1.31 | 1.56 | 1.69 | 1.81 | 1.86 | 1.91 |
| $y = Y_t/Y_\infty$ | 0 | 0.35 | 0.69 | 0.82 | 0.88 | 0.95 | 0.97 | 1.00 |
| t/Y_t | | 22.39 | 22.90 | 28.85 | 35.50 | 49.72 | 64.52 | 94.24 |
| $1 - y$ | 1 | 0.65 | 0.31 | 0.18 | 0.12 | 0.05 | 0.03 | 0.00 |
| $1/(1 - y)$ | 1 | 1.54 | 3.18 | 5.46 | 8.68 | 19.10 | 38.20 | |
| $\ln[1/(1 - y)]$ | 0 | 0.43 | 1.16 | 1.70 | 2.16 | 2.95 | 3.64 | |

The data in **Table 4** have enabled the first-order diffusional model to be validated by the line $\ln(1/(1 - y)) = kt$ (**Figure 4**).

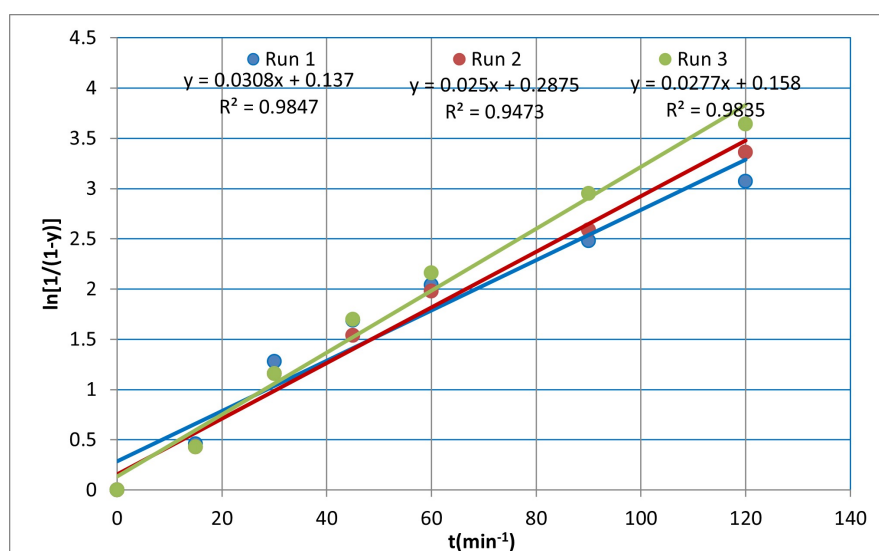


Figure 4. Validation of the diffusional model of order 1 by the line $\ln(1/(1 - y)) = kt$.

The curve $\ln[1/(1 - y)]$, a straight line with a slope = 0.0308, leads to a kinetic rate constant of essential oil extraction by steam-hydrodistillation: $k = 0.0308 \text{ min}^{-1}$ and $t_{1/2} = 0.69/k = 22.62 \text{ min}$ (**Figure 4**). The fraction of essential oil released by washing step is 74%, accounts for the low duration ratio of the two extraction steps ($R = 11$).

The pseudo first order kinetic constant obtained is within the same variation range as that observed by [19] for *Cymbopogon citratus* ($0.0286 - 0.0411 \text{ min}^{-1}$)

using the line $\log(q_0 - q_t) = f(t)$, with q the extraction yield.

3.2.2. Peleg's Model

Figure 5 represents the validation lines of the Peleg model. The ordinate at the origin lead to the kinetic rate constant of Peleg (order 2), the slope and the Peleg constant of extraction capacity.

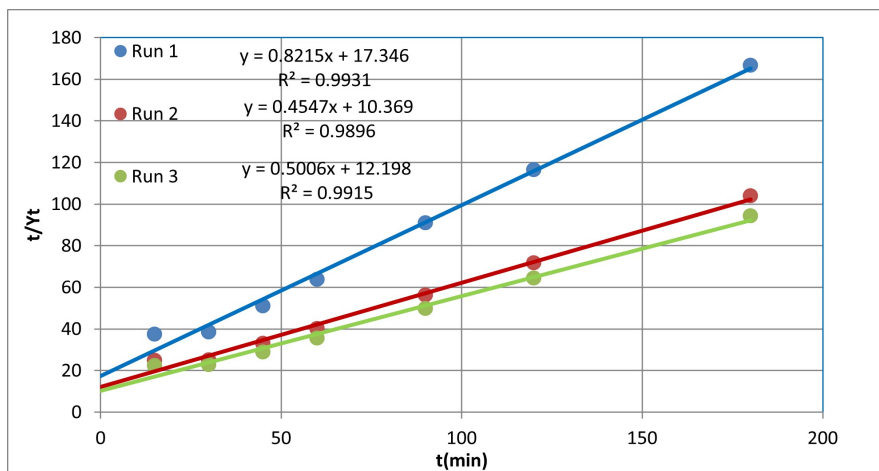


Figure 5. Validation of the Peleg model from the straight line $t/Y_t = k_1 + K_2t$ (second order kinetics).

The Peleg model which considers the extraction as a desorption of a metabolite from a plant matrix postulates a hyperbolic evolution of the process given by following equation:

$$q_t = q_0 + t/(k_1 + K_2t) \tag{9}$$

which leads to the following linear form:

$$t/q_t = k_1 + K_2t \tag{10}$$

Here, it takes following form:

$$t/Y_t = k_1 + K_2t \tag{11}$$

The results obtained on Run 1; for example, lead to the following equation for the overall duration of the experiment (Figure 5):

$$t/Y_t = 0.8215t + 17.346 \quad (R^2 = 0.9931)$$

From this equation we deduce the extraction capacity:

$$K_2 = 0.8215\%^{-1}$$

We can finally access the kinetic rate constant of extraction (order 1):

$$k = K_2/k_1 = 0.8215/17.346 = 0.04736 \text{ min}^{-1}$$

This value is of the same order of magnitude as that obtained above with the diffusional model which is 0.0308 min^{-1} .

In addition, we can get from K_2 , the quantity of essential oil extracted at equilibrium, Y_∞ (end of extraction):

$$Y_\infty = 1/K_2 = 1/0.8215 = 1.22\%$$

This maximum value of the essential oil extraction yield during experiment 1 is consistent with the value of 1.08% accepted as a first approximation and which corresponded to 180 min of extraction time, taking into account the asymptotic end of the process. This validates *a posteriori* the approximate method for calculating the fraction of recovered essential oil as a function of time Y_t/Y_∞ .

Table 5 groups the parameters of the Peleg model obtained during these 3 experiments.

Table 5. Peleg model parameters for extraction by distillation of *Corymbia citriodora* oil.

| | $k_1 \text{ min}^{-1}$ | $K_2 (\%)^{-1}$ | R^2 | $k = K_2/k_1$ (min^{-1}) | $Y_\infty = 1/K_2$ (%) |
|-------|------------------------|-----------------|--------|--|---------------------------|
| Run 1 | 17.346 | 0.8215 | 0.9931 | 0.0474 | 1.22 |
| Run 2 | 10.369 | 0.4547 | 0.9896 | 0.0482 | 2.20 |
| Run 3 | 12.198 | 0.5006 | 0.9915 | 0.0450 | 2.00 |
| Mean | 13.304 | 0.592 | 0.991 | 0.047 | 1.807 |
| SD | 3.618 | 0.200 | 0.001 | 0.002 | 0.518 |

This table shows that 1) the second-order kinetic rate constant k_1 varies from 10.369 to 17.346 min^{-1} with an average of $13.3 \pm 3.6 \text{ min}^{-1}$ and the extraction capacity constant K_2 varies from 0.4547% at 0.8215% with an average of $0.6\% \pm 0.2\%$; which leads to a first-order extraction kinetic constant $k = 0.0474 \pm 0.002 \text{ min}^{-1}$ and a maximum extraction yield $Y_\infty = 1.8\% \pm 0.5\%$, on average.

4. Conclusions

Three rotations of the artisanal extraction of the essential oil of *Corymbia citriodora* acclimatized in Congo-Brazzaville were monitored for the purpose of modeling the process. The two models tested, the diffusional model of Sovova and Aleksovski and the desorption model of Peleg, fitted experimental data, thus suggesting that each model makes it possible to capture part of the progress of the complex process that is steam hydrodistillation.

It emerges from this study that:

- ✓ According to the diffusional model that, 1) the fraction of essential oil extracted by the washing step is 75%; 2) the washing and diffusion steps take place respectively with extraction kinetic constants of $0.0233 \pm 0.0006 \text{ min}^{-1}$ and $0.0021 \pm 0.0002 \text{ min}^{-1}$, *i.e.* a rate ratio of 11 in favor of the washing step; 3) the kinetic constant, with the first order approximation, varies from 0.0250 to 0.0308 min^{-1} with an average: $0.028 \pm 0.002 \text{ min}^{-1}$.
- ✓ According to the Peleg desorption model, 1) the maximum extraction yield varies from 1% to 2% with an average of $1.2\% \pm 0.5\%$ (db); 2) the kinetic rate constants k_1 and extraction capacity K_2 are respectively, on average $13.3 \pm 3.6 \text{ min}^{-1}$ and $0.6\%^{-1} \pm 0.2\%^{-1}$ and lead to an average of the first order extraction rate constant $k = 0.047 \pm 0.002 \text{ min}^{-1}$.

The results obtained by the two models, using judicious approximations for

their simplification, were consistent with each other and with those of the literature. The simplified graphical processing of the diffusional [13]-[15] and the desorption [16] models lead to a validation with high validation criteria values ($R^2 > 0.9$) of the models tested by of the three experiments. This processing with low computer inputs is largely sufficient to model the processes implemented in artisanal essential oil units for small family businesses in Congo-Brazzaville.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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