Kinetic Modeling of Essential Oil Extraction by Hydrodistillation of Xylopia aethiopica (Dunal) A. Rich Fruits from Congo-Brazzaville

Thomas Silou, Jean Bruno Bassiloua, and Rosalie Kama-Niamayoua

ABSTRACT

The extraction kinetics of the essential oil of Xylopia aethiopica (Dunal) A. Rich by hydrodistillation was studied for modeling its process and optimizing its yield. The oils obtained, analyzed by GC/MS, consists mainly of pineses, sabinen, myrenal, terpinene-4-ol, limonene. Experimental data were fitted into first and second order kinetics for a 2-steps extraction, washing and diffusion, of the phenomenological model, according to the hypothesis used. The essential oil which moves inner vegetable cells by diffusion and is extracted at the surface of the particle by washing with an extraction solvent. When the washing step is instantaneous compared to that of diffusion, the mechanism, which is under diffusion control, admits first order. Considering both washing and diffusion steps, kinetic order became 2, in agreement with the Pheleg model. The Monod and Langmuir models also fitted experimental data. All these models validated by the experimental data with determination coefficients R² > 0.96 can be used for optimizing the extraction of the essential oil of Xylopia aethiopica (Dunal) A. Rich.

Keywords: Kinetics, essential oil, Xylopia aethiopica, Congo-Brazzaville.

I. INTRODUCTION

The geographic area of the genus Xylopia extends from Senegal to South Sudan through the Sahel to the East and to Angola, South Africa and Mozambique to the South. It colonizes the lowlands of the tropical rain forest, with a very high concentration in the Congo Basin: Cameroon and Gabon host 27 of 45 African species [1], [2]. There are 160 to 180 species of Xylopia worldwide. The revision of 45 African species of Xylopia due to Johnson and Murray signals 4 new species and brings out new subdivisions.

It is a multipurpose tree. However, its highly "spicy" seeds make it a spice and condiment that is highly prized throughout tropical Africa [3]-[6]. In traditional medicine, Xylopia aethiopica is used in the treatment of scabies [1], [5], [7], [8]. In some countries like Benin, it is threatened with extinction due to anthropogenic pressures that it suffers because of its multiple uses: medicinal uses (more than 60 %), magico-religious uses (22%), firewood, construction wood and food (18%) [9], [10]. To guarantee its sustainability, a program to afforest the coastal savannah of Pointe Noire in Congo Brazzaville was undertaken by planting Xylopia aethiopica for the production of medicinal essential oil in addition to firewood [11]. Extraction optimization has been studied very little and no work on kinetic modeling has been found in the literature. These are two important steps on the way to scale-up the production of this essential oil. We present here first results on the kinetic modeling of the extraction of the essential oil of Xylopia aethiopica fruits from Congo-Brazzaville.

II. MATERIAL AND METHODS

A. Botanical description of Xylopia aethiopica

According to the classification Angiosperm Phylogeny Group (APG) III [12], the species Xylopia aethiopica belongs to the clades Angiosperms / Magnoliidae, in the order of Magnoliales, to the family Annonaceae, to the genus Xylopia, to the species aethiopica. It accepts as synonyms: Unona aethiopica Dunal, Uvaria aethiopica (Dunal) A. Rich, Habezelia aethiopica (Dunal) Kuntze, Xylopia eminii Engler, Xylopia dekeyzeriana De Wild, Xylopia gilletii De Wild. Xylopia aethiopica, commonly known as “African peeper” is a tree 15-30 m high and 60-75 cm in diameter, growing either on rivers or in swamps. It is straight tree. Its leaves are oblong-lanceolate, obtuse, or rounded at the base, acuminate, glabrous, glaucous below, 4 to 10 cm in length, 2 to 4 cm in width. Its scented flowers are white-greenish. Its cylindrical

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linear fruits are arranged in 12 to 20 bacciform capsules of dark green color when ripe. Its seeds (4 to 12 per pod) form a single row. They are ellipsoidal in shape (5.0–6.0 in length; 3.4–3.7 mm in width and 2.5–3.1 mm in thickness), and are dark brown to black [2], [13].

The fruits of X. aethiopica studied were collected wild in the Youbi forest, (4°00’00’’ – 4°30’00’’ latitude South and 11°30’00’’and 12°00’00’’longitude) at 100 km north of the town of Pointe-Noire (Congo-Brazzaville).

B. Essential oil extraction by hydrodistillation

The essential oils were obtained by hydrodistillation. Water and plant material (300 g of dried fruits (m1, in dry basis-db-) were placed in a 500 mL round bottomed flask and boiled for 4 h. The organic phase of the condensate was separated from the aqueous phase with diethyl ether and dried over sodium sulphate. The essential oil was recovered after evaporation of the solvent (m2) and extraction yield is given by:

\[ Y(\%)db = \left(\frac{m_2}{m_1}\right)100 \]  

C. Chromatographic Analysis

GC analyses was performed on a Hewlett-Packard 6890 equipped with a split/splitness injector (280 °C, split ratio 1:10), using DB-5 column (30 m ×0.25 mm, df: 0, 25 μm). The temperature program was 50 °C (5 min) rising to 300 °C at rate of 5 °C/min. Injector and detector temperature was 280°C. Helium was used as carrier gas at a flow rate 1 mL/min. The identification was carried out by calculating retention indices and comparing mass spectra with those in data banks [14], [15].

D. Kinetic Modeling

The kinetic modeling of the metabolite extraction from a solid plant matrix is an active field of research [16]-[19]. 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 [17], [20]-[22].

The transposition to the chemical kinetic formalism of the physical extraction of essential oil is done (i) by assimilating the essential oil content to the concentration C, with C0 the concentration at the initial time t = 0, (ii) by defining, V0, as the quantity of essential oil remained in plant material at the initial time (t = 0); V, the amount of essential oil extracted at time t; (V0-V) then becomes the amount of essential oil remaining in plant material at time t [21], [23]. If \[ y = \left(\frac{V_0-V}{V_0}\right) \] is the degree of extraction, the integration of first order chemical kinetic leads to (1):

\[ \ln \left(\frac{1}{1-y}\right) = kt \]  

Table I summarizes the main models used in the study of metabolite extraction from plant matrices.

| Authors [ref.] | Models | Term meaning |
|----------------|--------|--------------|
| Ameneghawon et al. [23] | First order kinetics | \( \frac{dC}{dt} = k (C_0 - C) \) |
| Thanh et al.[24] | ln(C/C0) = -kt; t0 = 0,693/k ln[(1-Y(t))/Y0] = kt |
| Sepidar et al. [25] | Second order kinetics | \( \frac{dC}{dt} = k (C_0 - C)^2 \) |
| | | \( C = \frac{C_0}{k_t} t / (1 + C_0 k_t) \) |
| | | \( (1/C) - (1/C_0) = kt \) |
| | | \( t/C = 1/k (C_0 / k_t)^{1/2} \) |
| So and Macdonald [26] | So and Macdonald Washing : | \( C_t^{w} = C_0^{w} (1 - \exp(-K_w t)) \) |
| | Diffusion : | \( C_t^{d} = C_0^{d} (1 - \exp(-K_d t)) \) |
| | washing/diffusion | \( C_t^{w/d} = C_0^{w/d} (1 - \exp(-K_{wd} t)) + C_0^{w/d} (1 - \exp(-K_{wd} t)) \) |
| Milojevic et al. [17], [18] | Milojevic | \( q_t/q_0 = f/exp(-k_t t) \) |
| Sovova and Aleksovski [20] | Monod | \( f + (1-f) \exp(-k_t t) \) |
| Mejri et al. [22] | Monod | \( Y_t = Y_{max} [1/(K_m + t)] \) |
| | | \( 1/Y_t = [(K_m + t)/Y_{max}] (1/[1 + 1/Y_{max}]) \) |
| Babu and Singh [27] | Langmuir | \( Y_t = Y_{max} / (b + h) \) |
| | | \( 1/Y_t = (b/Y_{max}) (1/h + 1/Y_{max}) \) |
| Bucic-Kojic et al.[16] | Peleg | \( C_t = C_0 + t (k_0 + K_t) \) |
| Peleg [21] | \( t/C = K_t + K_A \) |
| Shafaei et al. [28] | \( k = K_t/K_A \) |

GC/MS was performed on a Hewlett-Packard 5973/6890 system operating in EI mode (70 eV), equipped with a split/splitness injector (280 °C, split ratio 1:20), using DB-5 column (30 m × 0.25 mm, df: 0, 25 μm). The temperature program was 50°C (5 min) rising to 300 °C at rate of 5 °C/min. Injector and detector temperature was 280°C. Helium was used as carrier gas at a flow rate 1 mL/min. The kinetic modeling of the metabolite extraction from a solid plant matrix is an active field of research [16]-[19]. 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 [17], [20]-[22].

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\[ \ln \left(\frac{1}{1-y}\right) = kt \]  

Notes:
- The model was tested using the data presented in [21], [26], [28].
The models most found in the literature are based on the phenomenological approach assumptions [26], [29], summarized in following seven points (Table I) : (i) Plant particles are considered to have properties such as shape, size and contain the initial essential oil [18]; (ii) The concentration gradients in the fluid phase develop at scales greater than the size of the particles; (iii) The solvent flow rate is uniformly distributed in each section of the extractor [30]; (iv) Part of the essential oil is located on the outer surfaces of the plant particles, f, and the rest is evenly distributed inside the plant particles (1-f); (v) The essential oil is considered to be a single component; (vi) The effective diffusion coefficient through plant particles is constant, and (vii) There is no resistance to essential oil mass transfer from the outer surfaces of plant particles [18].

One simple order kinetic (pseudo first order kinetics, Table I) and 3 empirical models (Sovova-Milojevic, Peleg, Monod models, Table I) were tested here for the essential oil extraction of Xylopia aethiopica fruits.

### III. RESULTS AND DISCUSSION

#### A. Content and Composition of the Essential Oil of Fruits

Only the fruits have a significant essential oil content in Xylopia aethiopica (2-8 %), the leaves come in second position with contents of 0.05-0.30 % and the bark, in third position (0.01-0.07 %) [11].

Table II shows chemical composition of the studied sample from Congo Brazzaville.

It is a hydrocarbon monoterpenic oil consisting mainly of α and β pinenes, sabinene, myrtenal, terpinene-4-ol, limonene. These six major constituents, which represent more than 75 % of the total oil, are followed by around fifty minor constituents (0.10-0.50 %) giving to the chromatogram a high qualitative complexity. The 55 constituents identified by their retention indices and their mass spectra are distributed in 19 hydrocarbon monoterpenes, 27 oxygenated monoterpenes, 2 hydrocarbon sesquiterpenes and 6 oxygenated sesquiterpenes. The only sesquiterpene present at more than 1 % is isospathulenol (1.35%). The studied sample from Congo-Brazzaville belongs to the pinene/sabinene chemotype. Many works, carried out on qualitative and quantitative analysis across Tropical Africa indicate that the essential oils of Xylopia aethiopica are very largely constituted of hydrocarbon monoterpenes represented by β-pinene (37.0-40.5% [31]; 12-42% [32]; 18.3% [33]); sabinene: 36.0 % [34]. Germacrene D is the most abundant sesquiterpene and the oxygenated compounds identified are mainly 1,8-cineole and terpinen-4-ol. One essential oil sample from Egypt studied by Karawya et al. [35] consists mainly of oxygenated compounds: 23.4% of terpinen-4-ol, 16.3% of 1,8-cineole and 11.1% of α-terpineol [36], [37]. The Xylopia aethiopica fruits from Nigeria allowed the isolation and characterization for the first time of a fairly rare sesquiterpene ketone : Zerumbone (4.0%), in addition to the constituents more commonly found in essential oils: β-santalol (14.5 %), α-cadinol (13.0 %), benzyl benzoate and dodecanoic acid (10.0 %) [38].

#### B. Kinetic Modeling of the fruit Essential Oil Extraction of Xylopia aethiopica

Table III gathers the data on the variation of the yield versus the extraction time as well as the data necessary to test the models studied.

![Table II: Chemical Composition of the Studied Sample from Congo Brazzaville](image)

| Constituents          | Content (%) |
|-----------------------|-------------|
| alpha thujene         | 0.40        |
| alpa pinene           | 8.24        |
| camphene              | 0.24        |
| sabinene              | 35.84       |
| bêta pinene           | 28.65       |
| myrcene               | 0.32        |
| menth-1(7),8-diene     | 0.09        |
| alpha terpiene        | 0.16        |
| p-cymene              | 1.29        |
| limonene              | 1.13        |
| beta phellandrene     | 0.51        |
| 1,8-cineole           | 0.69        |
| (E)-beta-ocimene      | 0.12        |
| gamma terpinene       | 0.46        |
| cis sabinene hydrate  | 0.14        |
| terpinolène           | 0.09        |
| linalol               | 0.48        |
| trans sabinene hydrate| 0.15        |
| para-Menth-2-en-1-ol   | 0.12        |
| alpha campholenal     | 0.09        |
| sabinacetone          | 0.50        |
| pinocarvone           | 2.97        |
| verbenol              | 0.69        |
| sabinol               | 0.30        |
| terpinene-4-ol        | 3.16        |
| thuj-3-én-10-al       | 0.17        |
| cryptone              | 0.55        |
| myrtanol              | 0.42        |
| myrtanol              | 4.37        |
| verbenol              | 0.32        |
| verbenoil             | 0.22        |
| cuminal               | 0.36        |
| myrtényle formiate    | 0.19        |
| phellandral           | 0.19        |
| bornyle acetate       | 0.11        |
| para-cymen-7-ol       | 0.12        |
| perilic alcohol       | 0.12        |
| 4-hydroxy-creptenone  | 0.13        |
| alpha copaene         | 0.31        |
| beta cubebene + beta eleme  | 0.21 |
| gamma eleme           | 0.09        |
| germacrene-D          | 0.27        |
| epitoreilenol         | 0.29        |
| germacrene-B          | 0.11        |
| caryophyllene oxide   | 0.52        |
| salvial-(4) en-1-one  | 0.14        |
| tortilenol            | 0.24        |
| isospathulenol        | 1.35        |
| amophora-4,9-dien-2-ol| 0.13        |
| isopiramadiene        | 0.51        |
| manoyle oxide         | 0.33        |
| kaurene               | 0.10        |

na: not available.
TABLE III: DATA FOR TESTING MODELS USED IN ESSENTIAL OIL EXTRACTION OF XYLOPIA AETHIOPICA FRUIT FROM CONGO-BRAZZAVILLE

| t (min) | 0   | 15  | 30  | 45  | 60  | 75  | 90  | 105 | 150 | 165 |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 1A      | -   |     |     |     |     |     |     | 3.01| 3.01|     |
| Y       | 1.85| 2.31| 2.57| 2.77| 2.92| 2.97| 2.99|     |     |     |
| 1/Y     | 0.54| 0.43| 0.39| 0.36| 0.34| 0.34| 0.33| 0.33| 0.33| 0.33|
| ln[1/(1-Y)] | 0.00| 0.61| 0.77| 0.86| 0.92| 0.97| 0.99| 1.00| 1.00| 1.00|
| t/Y     | 8.11| 13  | 17.51| 21.66| 25.68| 30.3| 35.12| 49.83| 54.82|     |
| u/Y     | 24.69| 38.96| 52.33| 62.22| 77.32| 90.91| 105.00| 150.00| 165.00|     |

1. Test of pseudo first order model

According to Sovova-Milojevic model, the two-step extraction process leads to:

\[
\frac{q}{q_e} = f \exp (-k_1 t) + (1-f) \exp (-k_2 t)
\]

(3)

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

\[
\frac{q}{q_e} = 1 - \exp (-k_2 t)
\]

(4)

dealing to following linear form:

\[
\ln(1/(1-y)) = k_2 t
\]

with \( y = Y/Y_e \)

The deviation at the origin of the validation line reflects the existence of a very rapid washing step prior to that of diffusion.

The slope of the straight line (\( R^2 = 0.9908 \)) is a pseudo first order constant: \( k = 0.0476 \) min

Fig. 1. Variations of extraction yield versus extraction time with Xylopia fruit essential oil.

2. Test of Peleg-Bucic model

The curve \( Y_t = f(t) \) (figure 1) is similar to that of moisture sorption by flour studied by Paleg [21]. This similarity has led a number of authors to apply Peleg's model to desorption metabolites of plant matrices under kinetic control. For the extraction of essential oil from the fruits of *XYLOPIA AETHIOPICA*, the validation curve of the Peleg model is a straight line noted \( t/Y_t = 0.309 t + 3.214 \) with a coefficient of determination \( R^2 = 0.9989 \). It leads to the two parameters of the model: the ordinate intercept \( K_0 = 3.214 \) min

We deduce the kinetic constant at the initial time \( B_0 = 1/K_0 = 0.3111 \) min

Fig. 2: Validation of the first order test for Xylopia aethiopica essential oil extraction (Moljevic Model).

with content at equilibrium (end of the extraction): \( C_e = 1/0.309 = 3.24 \) % and hydrodistillation kinetic constant \( k = K_0/K_2 = 0.0961 \) min

Fig. 3: Peleg model test line for extraction essential oil from the fruits of *Xylopia aethiopica*.
3. Test of Monod and Langmuir models

The two models are validated by the same linearized equation of \(1/Y_t = f (1/t)\). Monod's model, inspired by Michaelis enzymatic kinetics leads to the mathematical expression: \(Y_t = Y_{\text{max}} \frac{t}{(t + K_m)}\) and to its linearized inverse: \(1/Y_t = (K_m/Y_{\text{max}}) (1/t) + 1/Y_{\text{max}}\). We obtain a line allowing to calculate \(Y_{\text{max}}\) and \(K_m\) knowing that \(Y_{\text{max}}\) were the yield at \(t = \infty\) (end of the extraction process) and \(K_m\) and \(Y_{\text{max}}\): slope of the line and that the yield \(Y_t\) is expressed in g of essential oil extracted/100 g of sample [22].

Langmuir's gas adsorption isotherm model is mathematically expressed by the equation: \(Y_t = Y_{\text{max}}.t/(b + t)\) with \(Y_t\) = yield at time \(t\); \(Y_{\text{max}}\) the yield at \(t = \infty\) and \(b/Y_{\text{max}}\); the slope of the curve at the initial instants. The opposite form: \(1/Y_t = (1/Y_{\text{max}}) (1/t) + 1/Y_{\text{max}}\) gives a straight line with a slope = \(b/Y_{\text{max}}\) and an ordinate at the origin \(1/Y_{\text{max}}\) [27].

The linear form of Monod's model equation (figure 4, \(R^2 = 0.9904\)) makes it possible to graphically determine the parameters of the model \(Y_{\text{max}} = 1.0/0.3021 = 3.31\) % and \(K_m/Y_{\text{max}} = 3.6083, K_m = 11.9440\) min.-%. With \(Y_{\text{max}} = 3.31\) % (\(t = \infty\)), the value of 3.01 assumed for \(Y_{\text{max}}\) corresponds to \((3.01/3.31) 100 = 91\) % for the extraction rate at 150 minutes. The Langmuir model leads to the same value of \(Y_{\text{max}} = 3.31\) % and the same value of \(b = 11.9440\) min.-%; and identifiable at \(K_m\).

IV. CONCLUSION

The extraction of essential oil from *Xylopia aethiopica* fruits can be described by the phenomenological model in two steps according Sovova-Milojevic: a rapid washing of the oil located in the broken cells and at the solid-liquid interface of intact cells followed by a much slower step controlled by the oil diffusion from the inside to the outside of the intact cells of the plant matrix. Assuming the absence of the washing step, the process run to a pseudo-first-order mechanism characterized by a kinetic constant \(k=0.0476\) min\(^{-1}\) (Milojevic model). Assuming an instantaneous washing step, the process run to a second-order mechanism (Peleg model) characterized by a Peleg kinetic constant \(K_i=3.214\) min\(^{-1}\) and a Peleg extraction capacity constant \(K_e=0.309\) %\(^{-1}\) from which the hydrodistillation kinetic constant \(k=K/K_i=0.0961\) min\(^{-1}\) can be deduced. The models of Monod and Langmuir are also validated by kinetics of order 2 are also of possible reading grid of the experimental data relating to the extraction of essential oil from the fruits of *Xylopia aethiopica*.

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