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 pinenes, sabinene, 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 Peleg 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 aethiopia*, Congo-Brazzaville.

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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 aethiopia

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, Habzelia 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 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, $(4^{\circ}0'00' - 4^{\circ}30'00')$ latitudes South and 11°30'00' and 12°0'00' longitudes) 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:

$$Y(\%)db = (m_2/m_1)100$$
 (1)

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 \times 0.25 mm, df: 0, 25 μ m). 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 injection of the sample consisted of 1.0 μL of oil diluted to 10% v/v with acetone.

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 \times 0.25 mm, df: 0,25 μ m). 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 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 C_0 the concentration at the initial time t = 0. (ii) by defining, V_0 , 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 $y = (V_0 - V_0)$ $V)/V_0$, is the degree of extraction, the integration of first order chemical kinetic leads to (1):

$$ln(1/(1-y)) = kt$$
 (2)

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

TABLE I: OVERVIEW OF THE MAIN MODELS USED IN THE STUDY OF METABOLITE EXTRACTION FROM PLANT MATRICES

Authors [ref.]	Models	Term meaning
Ameneghawon et al. [23] Thanh et al. [24]	First order kinetics $dC_t/dt = k (C_0 - C)$ $ln(C/C_0) = -kt$; $t_{1/2} = 0.693/k$ ln[1/(1-y(t)] = kt	C_t , concentration or mass of oil in plant matrix at time t ; C_0 , initial concentration or mass of oil in plant matrix; k , first order kinetic constant (t^{-1}) ; $y = (C_0 - C)/C_0$: extracted EO ratio
Sepidar et al. [25]	Second order kinetics $dC_t/dt = k (C_s - C_t)^2$ $(C_t = C_s^2 kt)/(1+C_skt)$ $(1/C_t) - (1/C_s) = kt$ $t/C_t = 1/(kC_s^2) + t/C_s$	C_t , concentration of extracted oil at time t (g.L ⁻¹); C_s , concentration of extracted oil at saturation (g.L ⁻¹); k, second order kinetic constant (gL ⁻¹ min ⁻¹); $h = kC_s^2$: initial extraction rate (gL ⁻¹ min ⁻¹).
So and Macdonald [26]	$So \ and \ Macdonald \\ Washing: \\ C_L{}^w = C_L{}^{*w} \left(1 - exp(-K_c{}^w.t) \right. \\ Diffusion: \\ C_L{}^d = C_L{}^{*w} \left(1 - exp(-K_c{}^d.t) \right. \\ washing/difusion \\ C_L = C_L{}^{*w} \left(1 - exp(-K_c{}^w.t) \right. \\ + C_L{}^{*w} \left(- exp(-K_c{}^d.t) \right. \\ \end{aligned}$	C_L : concentration of extracted oil; C_L^w : concentration of extracted oil (washing step); C_L^d : concentration of extracted oil (diffusion step) K_c^w : kinetic constant (washing step); K_c^d : kinetic constant(diffusion step)
Milojevic <i>et al.</i> [17], [18] Sovova and Aleksovski [20]	Milojevic $q_t/q_\infty = f \exp(-k_1 t)$ $+ (1-f) \exp(-k_2 t)$	q_p quantity of EO in plant matrix at time t ; q_∞ quantity of EO in plant matrix at time t_∞ ; k_1 et k_2 : kinetic constants (washing and diffusion); f : fraction extracted by washing; 1 - f : fraction extracted by diffusion.
Mejri <i>et al</i> . [22]	$\begin{aligned} & & Monod \\ & & Y_t = Y_{max} \left[1/(K_m + t) \right] \\ & & 1/Y_t = (K_m/Y_{max})(1/t) + (1/Y_{max}) \end{aligned}$	Y_t : yeild of extracted oil at time t (g/100g); Y_{max} yeild at time t_∞ , Y_{max}/K_m : slope of straight line, K_m : Monod equation parameter.
Babu and Singh [27]	$\begin{aligned} & Langmuir \\ & Y_t = Y_{max}t/(b+t) \\ & 1/Y_t = (b/Y_{max}) (1/t) + 1/Y_{max} \end{aligned}$	Y_t : yeild of extracted oil at time t (g/100g); Y_{max} : yeild at time t_∞ , b/Y_{max} = slope of straight line; $1/Y_{max}$, ordinate at the origin.
Bucic-Kojic <i>et al.</i> ,[16] Peleg [21] Shafaei <i>et al</i> . [28]	$\begin{aligned} & Peleg \\ & C_t = C_0 \pm t'(k_1 + K_2 t) \\ & t'C_t = K_1 + K_2 t \\ & k = K_2 / K_1 \end{aligned}$	C_t : mass of extracted EO at time t (m_t); C_∞ : mass of extracted EO at t_∞ (m_∞); $C_0=0$: the mass of extracted EO at $t=0$; K_1 : kinetic extraction constant (second order: $C^{-1}t$); K_2 , extraction capacity constant (C^{-1}); hydrodistillation kinetic constant K_1

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 oxygenated sesquiterpenes. The only sequiterpene 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-cine ole and 11.1% of α -terpine ol [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: βsantalol (14.5 %), α-cadinol (13.0 %), benzyl benzoate and dodecanoic acid (10.0 %) [38].

TABLE II: CHEMICAL COMPOSITION OF THE STUDIED SAMPLE FROM CONGO BRAZZAVILLE

		Congo	Brazzaville		
RT	RT RI _{calc}		Constituents	Content (%)	
5.714	919	924	alpha thujene	0.40	
5.863	926	932	alpa pinene	8.24	
6.182	943	946	camphene	0.24	
6.669	967	969	sabinene	35.84	
6.758	972	974	béta pinene	28.65	
6.953	982	988	myrcene	0.32	
7.214	995	1003	mentha-1(7),8-diene	0.09	
7.455	1008	1014	alpha terpiene	0.16	
7.599	1016	1029	p-cymene	1.29	
7.679	1021	1024	limonene	1.13	
7.708	1022	1025	beta phellandrene	0.51	
7.742	1024	1026	1,8-cineole	0.69	
7.983	1038	1044	(E)-beta-ocimene	0.12	
8.190	1050	1054	gamma terpinene	0.46	
8.411	1062	1065	cis sabinene hydrate	0.14	
8.649	1076	1086	terpinolène	0.09	
8.898	1090	1095	linalol	0.48	
8.930	1092	1098	trans sabinene hydrate	0.15	
9.313	1117	1118	cis para-Menth-2-en-1-ol		
9.348	1119	1122	alpha campholenal	0.09	
9.554	1134	1135	nopinone	0.49	
9.586	1136	1135	trans pinocarveol	2.97	
9.651	1141	1140	trans verbenone	0.69	
9.844	1155	1154	sabinacetone	0.50	
9.917	1160	1160	pinocarvone	0.12	
10.017	1167	na	sabinol	0.30	
10.201	1180	1174	terpinene-4-ol	3.16	
10.234	1182	1182	thuj-3-èn-10-al	0.17	
10.299	1187	1183	cryptone	0.55	
10.360	1191	1194	myrtenol	0.42	
10.432	1197	1195	myrtenal	4.37	
10.608	1209	1204	verbenone	0.32	
10.755	1219	na	verbenol	0.22	
11.112	1244	1238	cuminal	0.36	
11.230	1253	na	myrtényle formiate	0.19	
11.620	1280	na	phellandral	0.19	
11.698	1285	1287	bornyle acetate	0.11	
11.792	1292	1292	para-cymen-7-ol	0.41	
11.901	1300	1294	perillic alcohol	0.12	
12.074	1313	1314	4-hydroxy-cryptenone	0.12	
12.932	1379	1374	alpha copaene	0.31	
			beta cubebene + beta		
13.089	1391	1387	elemene	0.21	
13.603	1431	1434	gamma elemene	0.09	
14.272	1485	1484	germacrene-D	0.27	
15.147	1559	na	epitorilenol	0.29	
15.207	1564	1559	germacrene-B	0.11	
15.484	1588	1582	caryophyllene oxyde	0.52	
15.602	1598	na	salvial-4(14)-en-1-one	0.14	
15.850	1620	na	torilenol	0.24	
15.982	1632	na	isospathulenol	1.35	
16.683	1694	1700	amorpha-4,9-dien-2-ol	0.13	
19.207	1940	1905	isopiramadiene	0.51	
19.967	2019	1997	manoyle oxyde	0.33	
20.274	2052	2034	kaurene	0.10	
			Total	99.60	

na: not available.

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 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
1/t	-	0.067	0.033	0.022	0.017	0.013	0.011	0.01	0.007	0.006
\mathbf{Y}_{t}	0	1.85	2.31	2.57	2.77	2.92	2.97	2.99	3.01	3.01
$1/Y_t$		0.54	0.43	0.39	0.36	0.34	0.34	0.33	0.33	0.33
$y_t \!\!=\!\! Y_t \!/ Y_{\infty}$	0.00	0.61	0.77	0.86	0.92	0.97	0.99	1.00	1.00	1.00
$ln[1/(1-y_t)]$	0.00	0.94	1.47	1.97	2.81	3.51	4.51	-	-	-
t/Y_t	-	8.11	13	17.51	21.66	25.68	30.3	35.12	49.83	54.82
t/y _t	-	24.69	38.96	52.33	62.22	77.32	90.91	105.00	150.00	165.00

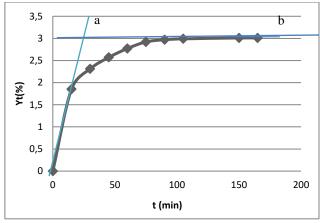


Fig. 1. Variations of extraction yield versus extraction time with Xylopia fruit essential oil.

1. Test of pseudo first order model

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

$$q_t/q_\infty = f \exp(-k_1 t) + (1-f) \exp(-k_2 t)$$
 (3)

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

$$q_t/q_\infty = 1 - \exp(-k_2 t) \tag{4}$$

dealing to following linear form:

$$ln(1/(1-y) = k_2t$$

with $y = Y_t/Y_{\infty}$

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⁻¹ (Fig. 2). This constant should be compared with those relating to the extraction of Ocimum basilicum $k = 0.0328 - 0.0741 \text{ min}^{-1}$, $R^2 = 0.9359 - 0.9670$ [39]; $k = 0.118 \text{ min}^{-1}$, $R^2 = 0.99 [19]$; the extraction of Cymbopogon winteranius: k=0.047 min-1, R²=0.99 [19]; the extraction of Cymbopogon citratus: $k=0.074-0.090 \text{ min}^{-1}$ [18]; the extraction of Cymbopogon flexuosus, $k = 0.025 \text{ min}^{-1}$, $R^2=0.99$ [40]; Curcuma mangga k=0.962 h^{-1} , $R^2=0.969$ [41]. Its value is of the same magnitude (0.010-0.090 min⁻¹) as those of cymbopogons and Ocimum basilicum found in the literature with a very good coefficient of determination $(R^2=0.97-0.99).$

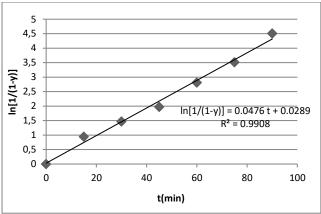


Fig. 2: Validation of the first order test for Xylopia aethipica essential oil extraction (Moljevic Model).

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 $K_1=3,214$ min.%⁻¹, the kinetic constant of Peleg and the slope of the line $K_2 = 0.309$ %⁻¹, the capacity constant of extraction of Peleg.

We deduce the kinetic constant at the initial time $B_0 =$ 1/K₁=0.3111 % min⁻¹, content at equilibrium (end of the extraction): C_e=1/0.309=3.24 % and hydrodistillation kinetic constant $k = K_1/K_2 = 0.0961 \text{ min}^{-1}$. The value of these two constants is of the same order of magnitude as for those of the extraction of essential oil from the leaves of Ocimum basilicum: $K_1 = 1.8398-2.7007 \text{ min.}\%^{-1}$ and $K_2 = 0.3406$ - $0.4990 \% ^{-1}$ with $R^2 = 0.99 - 1.00 [39]$ and Cymbopogon winteranius: $K_1 = 19.09 \text{ min. } (g/g^{-1}) \text{ and } K_2 = 0.0301 \%^{-1}$ [42].

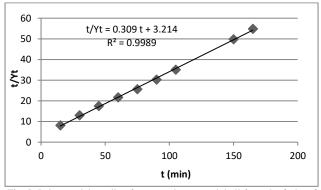


Fig. 3. Peleg model test line for extraction essential oil from the fruits of Xylopia aethiopica.

3. Test of Monod and Langmuir models

These 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: $Yt = Y_{max} [t/(K_m + t)]$ and to its linearized inverse: $1/Y_t = (K_m/Y_{max}) (1/t) + 1/Y_{max}$. We obtain a line allowing to calculate Y_{max} and $K_{\text{m}},$ knowing that Y_{max} were the yield at t $=\infty$ (end of the extraction process) and K_m/Y_{max} : slope of the line and that the yield Y_t is expressed in g of essential oil extracted/100 g of sample [22].

adsorption isotherm Langmuir's gas model mathematically expressed by the equation: $Y_t = Y_{max} \cdot t/(b+t)$ with Y_t = yield at time t; Y_{max} , the yield at $t = \infty$ and b/Y_{max} , the slope of the curve at the initial instants. The opposite form: $1/Y_t = (/Y_{max}) (1/t) + 1/Y_{max}$ gives a straight line with a slope = b/Y_{max} and an ordinate at the origin $1/Y_{max}$ [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 $Y_{max} = 1/0.3021 = 3.31$ % and K_m $/Y_{max} = 3.6083$, $K_m = 11.9440$ min.% ⁻¹. With $Y_{max} = 3.31$ % $(t = \infty)$, the value of 3.01 assumed for Y_{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_{max} : 3.31% and the same value of $b = 11.9440 \text{ min.}\%^{-1}$ and identifiable at K_m

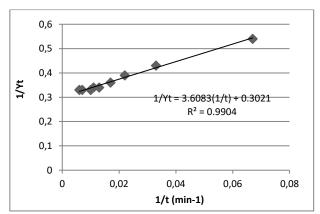


Fig. 4. Test straight line for Monod and Langmuir models for extraction essential oil from the fruits of Xylopia aethiopica.

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 K₁=3,214 min %⁻¹ and a Peleg extraction capacity constant K₂=0,309 %⁻¹ from which the hydrodistilation kinetic constant $k=K_2/K_1=0.0961 \text{ min}^{-1}$ 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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