

Modeling the Kinetics of Moisture Desorption from Safou Pulp [*Dacryodes edulis* (G. Don) H.J.Lam] before the Oil Extraction Step

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Abstract

The aim of this work is to model the drying kinetics of Safou pulp with or without endocarp using a phenomenological approach. Oven-drying kinetics at 70°C, 90°C and 105°C were monitored using the curves given by the reduced mass as a function of time, which are modeled according to the Avrami/page, Fick and Peleg models using OringinPro 2018 software. The results showed that parameters k and n of the Avrami/Page model vary very little with fruit size and drying temperature ($0.0018 \pm 0.0002 < k < 0.03328 \pm 0.0079$ and $0.82 \pm 0.05 < n < 1.21 \pm 0.02$). These parameters, which are generally fitting parameters of the model during its numerical resolution, have no physical significance, especially at the macroscopic scale. For all 9 samples studied, the values of a (Fick model) and k (Avrami model/page) were virtually identical, while b (Fick model) and n (Avrami model/page) were virtually identical for the same sample. For the Peleg model, the parameter a , varies from 0.0018 ± 0.0002 to 0.03328 ± 0.0079 , with a ratio of 18.6 for all experimental conditions studied. However, with $0.977 < R^2 < 0.995$ and $0.00079 < \chi^2 < 0.00002$, we have a good fit of the model to the experimental data. The same applies to parameter b , which ranges from 0.82 ± 0.05 to 1.21 ± 0.02 . Thus, drying modeling by these three models can be used to describe and predict the progress of oven-drying of safou pulp.

Keywords

Drying, Modeling, Empirical Models, *Dacryodes edulis*, Congo Basin

1. Introduction

The safflower tree (*Dacryodes edulis*), emblematic of the Central African landscape, is a major economic speculation in the sub-region [1]-[11]. Safou is a very fragile fruit, softening in less than a week and becoming unfit for consumption [12]-[14]. Traditionally, it can be preserved dried or smoked [15]. In recent decades, it has been valorized by extracting the oil from the dried pulp. Drying and oil extraction have been the key operations in process modelling and yield optimization in the production of oil from safou pulp [16]-[21].

Modeling means creating a representation to understand, describe and/or predict 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 oil extraction, the kinetic approach is an essential tool for process modeling and yield optimization.

Theoretical, empirical and semi-empirical mathematical models are often used to describe the behavior of plant products during drying, and of the various metabolites during extraction. The most relevant models are validated by various criteria: “Mean Root Standard Error (MRSE)” as low as possible, coefficient of determination (R^2), close to 1, chi-square (χ^2) tending towards zero. Current studies on drying modeling use numerical resolution of models in their mathematical form without physical significance of the results, which are unfortunately too theoretical for the intended users at this scale. It would therefore make sense to focus on empirical models leading to parameters with physical significance [22] [23]. In addition, to support small-scale producers, it would be preferable to use resources with the fewest mathematical prerequisites and computer inputs, for effective transfer to these users. The diffusion model, fitted, after judicious approximations, by the first-order kinetic model and the Peleg model (pseudo-order 2), gave relevant results for the drying of pulps, notably those of *Dacryodes edulis* fruits [24] and those of raffia sese fruits [25]. These same models also provide a good description of oil extraction. After testing, for reference, the pseudo-first-order diffusional models of Avrami [26] [27] and Fick [28] and the pseudo-second-order desorption model of Peleg [29] in numerical simulation using Originpro18 software. In the context of this work, we propose to model the drying kinetics of Safou pulp with or without endocarp using a phenomenological approach based on [30]. This approach has been widely used in the literature for various extracted metabolites: vegetable oils [31], polyphenols [32], essential oils [33] [34], via correlation lines having a physical meaning for the parameters generated by the models. In this same logic, this work is devoted to modeling the drying kinetics of Safou pulp with or without endocarp using a phenomenological approach.

2. Materials and Methods

2.1. Plant Material

Safflower, native to the Gulf of Guinea, belongs to the genus Burseraceae of the

Dacryodes family and to the species edulis. The characteristics of the fruits studied are listed in **Table 1**.

Table 1. Definition of fruit size [8] [20].

Size	Length (cm)	Width (cm)	Fruit weight (g)
1	<5	<3	<30
2	5 - 8	3 - 4	30 - 70
3	>8	>4	>70

2.2. Drying the Safou Pulp

Fruits were cut lengthwise with a knife to remove seeds. The pulps (exo, meso and endoderm), raw (FH) or blanched for 3 minutes in hot water and freed from endocarp (FB), were oven-dried at 70°C, 90°C and 105°C. Three samples were studied per drying temperature, and for each sample studied, the mt mass was determined for different drying times.

2.3. Modeling of Safou Pulp Drying

2.3.1. Diffusion Models

The term “diffusional model” refers to models whose kinetics are governed by the metabolite diffusion step in the plant matrix prior to extraction. They are derived from Newton’s cooling law and Fick’s diffusion law (**Table 2**). For drying simulation, water content was used in accordance with [35]. Moisture content, also known as humidity, is denoted by $X = m_1/m_2$, with m_1 , the mass of water in the sample, and m_2 , the mass of the sample, on a dry matter basis. The moisture content or reduced moisture content is given by $X_r = (X_t - X_e)/(X_e - X_0)$, with X_t : moisture content at time t , X_0 : moisture content at $t = 0$, X_e : equilibrium moisture content, which in this case is negligible; X_r therefore reduces to: $X_r = X_t/X_0$.

It’s interesting to note that the humidity ratio is similar to the reduced mass that is widely used in numerical model resolution techniques:

$$M_r = (m_t - m_\infty)/(m_0 - m_\infty)$$

With m_0 , m_t , m_∞ the sample masses at times $t = 0$, t and $t = \infty$.

Table 2. Diffusion models used.

Models	Expressions	References
Page (Avrami)	$X_r = \exp(-kt^n)$	[36]
Fick	$M_r = \frac{8}{\pi^2} \sum_{i=1}^{\infty} \frac{1}{2n-1} \exp\left[-\frac{(2n-1)^2 \pi^2 D_e t}{4L^2}\right]$ $\ln M_r = \ln \frac{8}{\pi^2} - \pi^2 \frac{D_e}{4L^2} t = \ln A - kt$ <p>with $k = -\pi^2 \frac{D_e}{4L^2}$</p>	[28]

Drying can also be analyzed as a diffusion of intra-particle moisture to the outside (ambient air) via diffusion pores and under the combined effect of various factors, the most important of which are: 1) the difference in moisture content between ambient air and the moist plant matrix; 2) capillary action in glands and pores due to surface forces; 3) the vaporization/condensation sequence and pressure gradient. The dominant mechanism depends on the nature and water content of the plant sample [37]. Drying kinetics, which can be defined as the variation in the rate of water removal from a moist product, characterizes drying behavior by three characteristic curves: 1) water content vs. time $X = f(t)$; 2) drying rate vs. Time: $-dx/dt = f(t)$ and 3) drying rate vs. water content: $-dx/dt = f(X)$ [38]. A qualitative discussion of these curves already provides sufficiently practical information for process optimization.

2.3.2. Peleg Model

The Peleg model has been proposed to explain the shape of the evolution curves of several natural phenomena, in particular the absorption or desorption of moisture by plant matrices. The phenomenon is assumed to follow a hyperbolic law: $X_t = X_0 \pm t/(k_1 + K_2t)$.

With: \pm : sorption, adsorption (+) and desorption, (-); X_t : water content, or humidity, at time t ; $X_0 = 0$: humidity $t = 0$; k_1 : kinetic constant of order 1, K_2 : extraction capacity, constant linked to equilibrium at the end of the process [39]. Pulp drying can be analyzed by this model respectively as water desorption: $X_t = X_0 - t/(k_1 + K_2t)$, or more generally:

$$X_t = X_0 - t/(a + bt) \quad \text{or} \quad M_r = M_0 - t/(a + bt)$$

with a and b parameters for numerical resolution of the model, minimizing χ^2 or maximizing R^2 . This equation was used, after linearization, to fit the Peleg model, $t/X_t = k_1 + K_2t$ [29]; it gave a straight line, with K^2 the slope, called the Peleg capacity constant (in concentration⁻¹), and the y-intercept, k_1 : called the kinetic constant (in t⁻¹).

Desorption of water from a plant matrix (drying) follows the same pattern, and can therefore be analyzed by Peleg's model.

2.4. Statistics

Statistical processing was carried out on Excel 2018 for means, standard deviations, correlation lines and graphs, and on OriginPro 18 for numerical model resolution. The arithmetic mayenne (\bar{x}), the standard deviation (σ), the correlation coefficient (R^2) and the chi-square χ^2 can be represented mathematically as a sequence.

$$R^2 = \frac{(X_{e-exp} - \bar{X}_{e-pre})^2}{\sum (X_{e-exp} - \bar{X}_{e-pre})^2 + (X_{e-exp} - X_{e-pre})^2}; \quad \chi^2 = \sum_{i=1}^N \frac{(X_{e-exp,i} - X_{e-pre})^2}{X_{e-pre,i}}$$

$$\text{with: } \bar{X}_{exp-pre} = \frac{\sum_{i=1}^N X_{e-pre,i}}{N}; \quad X_{e-exp} = \text{Experimental equilibrium water content.}$$

X_{e-pre} = Predicted equilibrium water content, N = Number of experimental points.

$$\bar{x} = \frac{1}{N} \sum_{i=1}^N x_i ; \sigma = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{n-1}}$$

x_i = designates a value from the statistical series; \bar{x} = the arithmetic mean.

3. Results and Discussion

3.1. Characterization of Plant Material

The fruits studied were divided into sizes 1, 2 and 3 according to their morphological data and mass. Their moisture content is shown in **Table 3**.

Table 3. Moisture content of fruit studied.

Parameter	Size	Content (%)
Moisture content	1	54.33 ± 6.11
	2	53.09 ± 7.14
	3	59.38 ± 4.08

These results are in line with those established on much larger samples in the countries of the Congo Basin and Gulf of Guinea: Cameroon [5] [17] [18], Congo-Brazzaville [40] [41], Congo Kinshasa [6] [7], Gabon [10] and Nigeria [3].

3.2. Characteristics of Drying Curves

Figure 1 shows $M_r = f(t)$ and $X_r = f(t)$ and **Figure 2**, $dX/dt = f(t)$, obtained for oven-drying at 105°C of safou pulp (sample size 2).

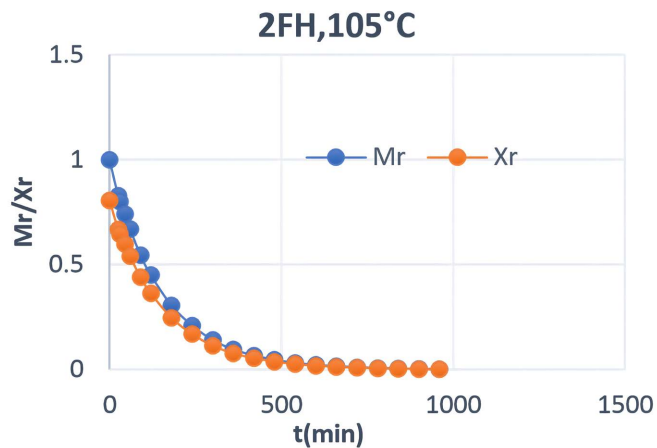


Figure 1. Variation in reduced mass (M_r) and moisture content (X_r) as a function of drying time.

Note the qualitative similarity between $M_r = f(t)$ and $X_r = f(t)$; these 2 curves lead to the same qualitative conclusions about process progress. These curves

pass through three stages [38]: the first stage, called the transition stage corresponding to the temperature rise, is not observable, the constant speed stage has been reduced to a strict minimum, and only the decreasing speed stage has been completely observed. This is confirmed by the $dX/dt = f(t)$ curve, on which the second stage, which is generally a plateau, has been reduced to a peak. Thus, the variation in moisture content passes through a maximum at the first instants of the process to gradually tend towards zero (Figure 2). This observation, for the 9 samples studied at all drying temperatures, is in line with previous data [42].

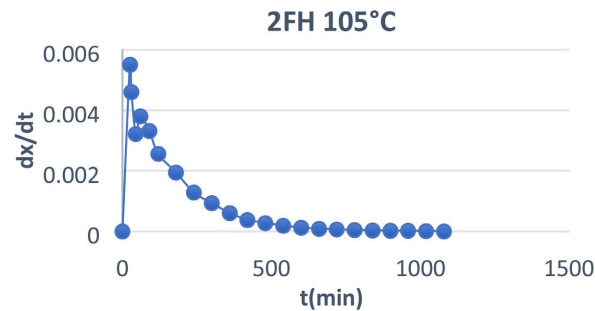


Figure 2. Moisture content as a function of extraction time.

Three raw fruits of each size were dried at 105°C, and the drying curves obtained are shown in Figure 3. They have the same shape for fruits of the same size, on the one hand, and show a very slight decrease in the slope at the origin, from small to large fruits, on the other. The same behavior was observed at 70°C and 90°C.

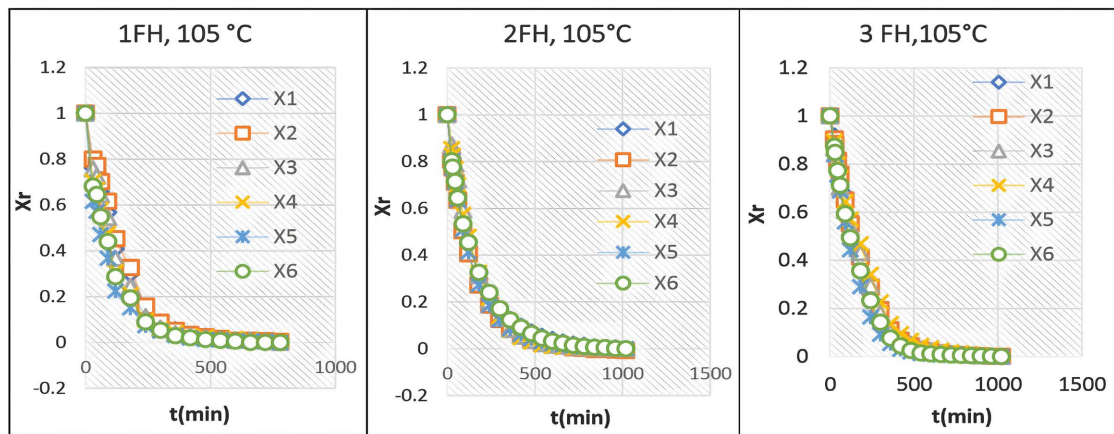


Figure 3. Drying curves for raw half-fruits at 105°C, Grades: 1 (1FH), 2 (2FH) and 3 (3FH). Fruit1 (X1, X2), Fruit 2 (X3, X4), Fruit 3 (X5, X6).

3.3. Influence of Fruit Size and Drying Temperature on the Drying Process

The curves in Figure 4 confirm that small fruits dry more quickly, with slopes at initial times higher than those of larger fruits. The difference between drying rates decreases as the drying temperature increases. There is therefore a com-

promise to be found between speed and drying time for large fruits. The temperature of 105°C would be more suitable for drying this type of fruit.

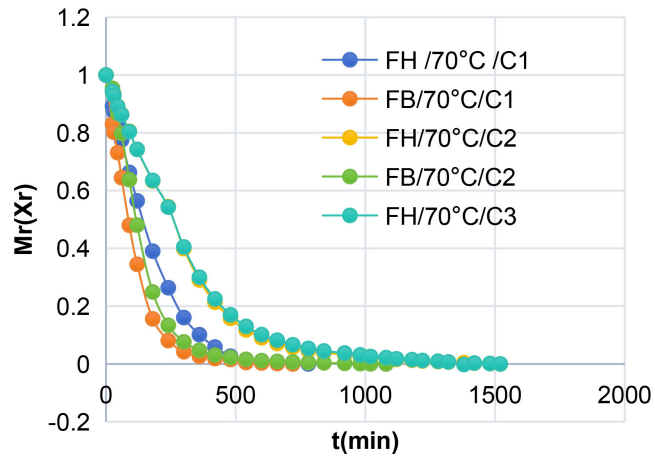


Figure 4. Variation in reduced mass versus fruit size during drying at 70°C.

Drying at 105°C is faster than drying at 90°C and 70°C (Figure 5). The corresponding curves have slightly steeper slopes at the beginning of the process (0 - 500 min). But the differences are small because the differences between the drying temperatures are too small.

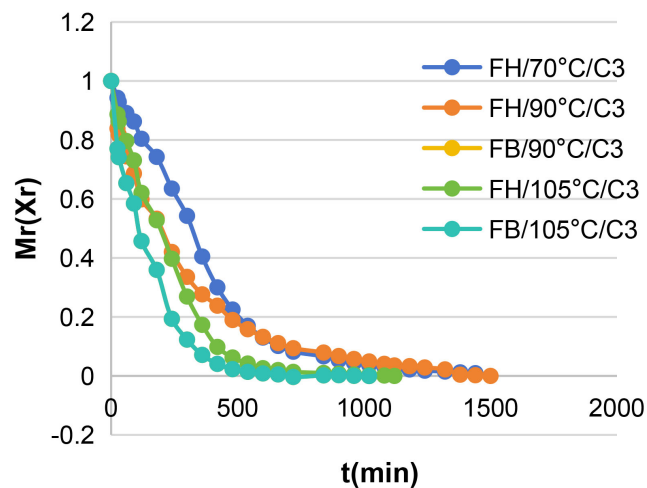


Figure 5. Variation in reduced mass as a function of drying temperature.

Ultimately, the moisture content or reduced water content at temperatures ranging from 70°C to 105°C is insensitive to fruit size and pulp condition (raw or cooked).

3.4. Model Testing

3.4.1. Numerical Resolution of the 3 Models for Oven-Dried Safou Pulp

The diffusional models (Avrami/Page and Fick) and Peleg's desorption model were tested. The various model parameters were determined by automatic cal-

culuation maximizing R^2 and minimizing chi-square (χ^2) on OringinPro 2018 software. Data obtained under different experimental conditions (raw or cooked fruit, sizes 1, 2 or 3; drying at 70°C, 90°C and 105°C) and for the Avrami, Fick or Peleg models are collated in **Tables 4-6**. For more specific, targeted studies (effective diffusivity coefficients, activation energies, etc.), the scale of variation of model parameters can be evaluated.

Table 4. Parameters of the Fick diffusion model for safou pulp drying.

Samples	$T(^{\circ}\text{C})$	Model constants		R^2	χ^2
		A	k		
1FH*	70	0.0034 ± 0.0004	1.20 ± 0.02	0.99911	$1.2445 \text{E}-4$
1FB*		0.0018 ± 0.0002	1.21 ± 0.02	0.99913	$1.33097\text{E}-4$
1FH*	90	0.0091 ± 0.0009	0.84 ± 0.02	0.99769	$2.24432\text{E}-4$
2FB*		0.0333 ± 0.0079	0.82 ± 0.05	0.99204	$7.86626\text{E}-4$
2FH*	105	0.0083 ± 0.0005	1.12 ± 0.02	0.99963	$4.47554\text{E}-5$
3FB*		0.0079 ± 0.0002	0.966 ± 0.006	0.99986	$1.67645\text{E}-5$

*1FH: Raw fruit, size 1; 1FB: Fruit without endocarp, size 1; 2FH: Raw fruit, size 2; 2FB: Fruit without endocarp, size 2; 3FB: Fruit without endocarp, size 3.

Table 5. Parameters of the diffusion model (Avrami/Page) for safou pulp drying.

Samples	$T(^{\circ}\text{C})$	Model constants		Validation criteria	
		k	n	R^2	χ^2
1FH	70°C	0.0034 ± 0.0004	1.20 ± 0.02	0.99911	$1.2445 \text{E}-4$
1FB		0.0018 ± 0.0002	1.21 ± 0.02	0.99913	$1.33097\text{E}-4$
1FH	90°C	0.0091 ± 0.0009	0.84 ± 0.02	0.99000	$2.24432\text{E}-4$
2FB		0.03328 ± 0.0079	0.82 ± 0.05	0.99000	$7.86626\text{E}-4$
2FH	105°C	0.0083 ± 0.0006	1.12 ± 0.02	0.99963	$4.47554\text{E}-5$
3FB		0.0079 ± 0.0002	0.97 ± 0.01	0.99986	$1.67645\text{E}-5$

Table 6. Peleg model parameters for safou pulp drying.

Samples	$T(^{\circ}\text{C})$	Model constants			Validation criteria	
		a^*	b^*	$k = b/a^*$	R^2	χ^2
1FH	70°C	95.14 ± 9.10	0.81 ± 0.03	0.0085	0.97723	0.00317
1FB		172.06 ± 12.83	0.71 ± 0.03	0.0041	0.98692	0.00199
1FH	90°C	42.73 ± 3.18	0.90 ± 0.02	0.0210	0.98761	0.00122
2FB		42.73 ± 3.18	0.90 ± 0.02	0.0210	0.98761	0.00122
2FH	105°C	113.42 ± 5.22	0.84 ± 0.02	0.0074	0.99360	0.00077
3FB		87.48 ± 3.50	0.86 ± 0.01	0.0098	0.99523	0.00055

*By identifying the terms with Peleg's empirical model $a = k_1$ and $K_2 = b$, we deduce the kinetic constant for drying (order 1): $k = K_2/k_1 = b/a$.

Table 4 shows the values of the Fick diffusion model parameters under different experimental conditions (size: 1, 2, 3; temperature: 70°C - 105°C).

The parameter a , ranges from 0.0018 ± 0.0002 to 0.03328 ± 0.0079 , with a ratio of 18.6 for all experimental conditions studied. However, with $0.977 < R^2 < 0.995$ and $0.00079 < \chi^2 < 0.00002$, we have a good fit of the model to the experimental data. The same applies to parameter b , which ranges from 0.82 ± 0.05 to 1.21 ± 0.02 .

In **Table 5**, k and n , the parameters of the Avrami/Page model, vary very little with fruit size and drying temperature ($0.0018 \pm 0.0002 < k < 0.03328 \pm 0.0079$ and $0.82 \pm 0.05 < n < 1.21 \pm 0.02$). These parameters, which are generally fitting parameters of the model during its numerical resolution, have no physical significance, especially at the macroscopic scale. The Avrami model is a special case of the Page model, with $n = 1$ (**Table 1**). For all 9 samples studied, the values of a (Fick model) and k (Avrami/page model) were virtually identical, while b (Fick model) and n (Avrami/page model) were virtually identical for the same sample. **Table 6** shows the parameters of the Peleg model: a , which can be identified with the Peleg kinetic constant, varies from 42.73 ± 3.18 to 172.06 ± 12.83 , and b , the Peleg capacity constant, varies from 0.71 ± 0.03 to 0.90 ± 0.02 . From these two constants, we deduce the drying kinetic constant $k = b/a$, which varies from 0.0041 to 0.0210 min^{-1} , and the equilibrium moisture content at the end of drying $X_c = 1.11 - 1.41 \text{ g/g}$. These values will be compared with those obtained by graphical resolution.

Figure 6 illustrates the level of validation of the 3 models for the entire study (size, drying temperature, equilibrium humidity).

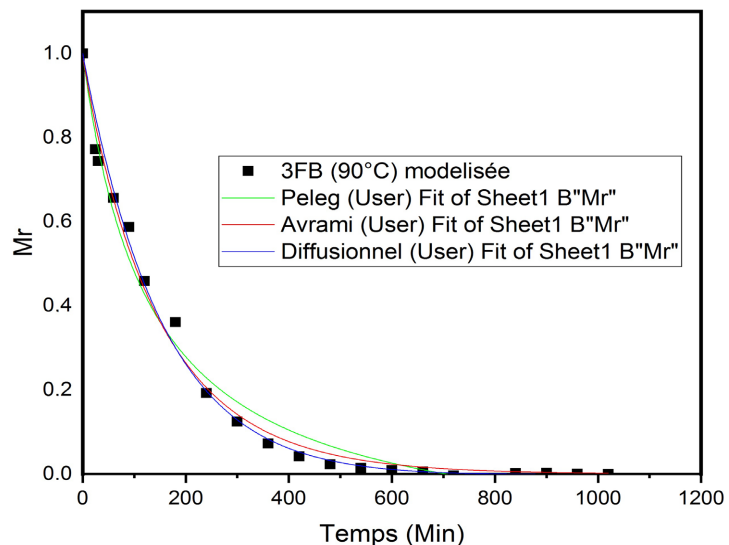


Figure 6. Validation of Peleg, Avrami and Fick models for drying safou pulp without endocarp (size 3, $T = 90^\circ\text{C}$).

3.4.2. Graphical Resolution of Models Used for Oven-Drying Safou Pulp

1) Diffusion models

The Avrami/Page diffusion model based on Newton's law on the one hand

and the model based on Fick's pseudo-first-order law on the other were fitted by $\ln(1/(1-y))$. **Figure 7** shows the pseudo-first-order kinetics validation line with a very good coefficient of determination ($R^2 = 0.9989$) and a pseudo-first-order kinetic constant $k = 0.006 \text{ min}^{-1}$. This value is to be compared with those obtained for other metabolites studied in the literature: essential oils, with $k = 0.007 - 0.115 \text{ min}^{-1}$ [43] and $k = 0.004 - 0.504 \text{ min}^{-1}$ [31]. Furthermore, the kinetic constant of the diffusional model is of the same order of magnitude as those obtained with the Peleg model ($0.0041 - 0.0210 \text{ min}^{-1}$, **Table 6**).

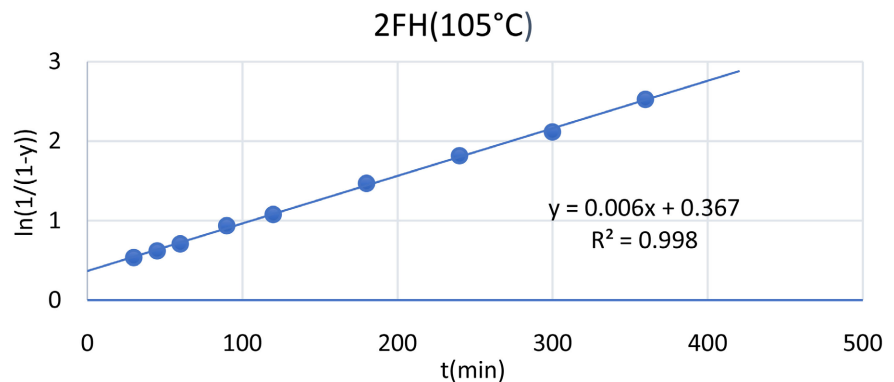


Figure 7. Validation line for the pseudo-first-order diffusional model for safou pulp drying.

2) Peleg model

Figure 8 shows the variation in water content as a function of drying time at 105°C . The curve obtained shows the characteristic shape of the Peleg model [29], as observed by [32], when extracting grape polyphenols; [33], when extracting essential oils and [30], when extracting vegetable oils.

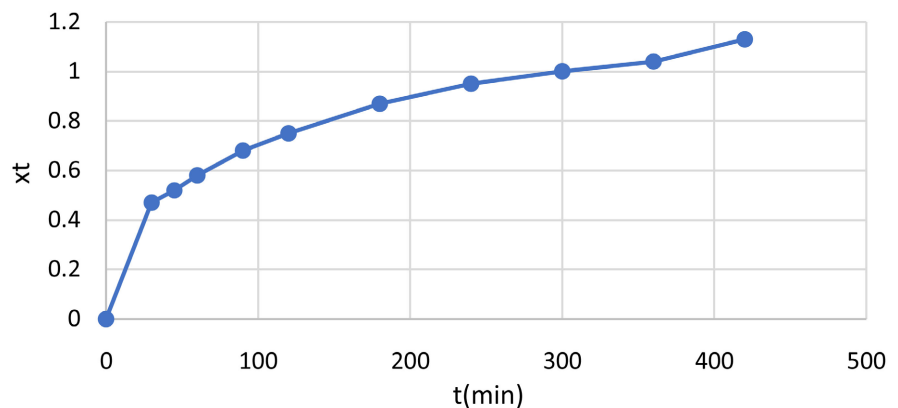


Figure 8. Variation in extracted moisture as a function of drying time at 105°C .

The graphical validation of the diffusion models (Avrami/Page, Fick) and the Peleg model uses the linearized forms of the mathematical expressions for the different models: $\ln X_r = \ln A + kt$, first-order pseudokinetics for the diffusion models, and $t/X_r = k_1 + K_2 t$, second-order pseudokinetics for the Peleg model.

Table 7 brings together the data necessary for the graphical validation of the models studied.

Table 7. Graphical validation data of the models studied (2FH at 105°C).

t (min)	0	30	45	60	90	120	180	240	300	360	420	t_{∞}
$1/t$		0.033	0.022	0.017	0.011	0.008	0.006	0.004	0.003	0.003	0.002	-
M_t	23.61	18.36	17.82	17.18	16.07	15.21	13.91	13.05	12.43	12.02	11.76	11.15
$m_{eau}(ext)$	0	5.25	5.79	6.43	7.54	8.40	9.70	10.56	11.18	11.59	12.45	-
$X_t =$	0	0.47	0.52	0.58	0.68	0.75	0.87	0.95	1.00	1.04	1.13	
t/X_t	0	63.83	86.54	103.45	132.35	160.00	206.90	252.63	300.00	346.15	371.68	
$1/X_t$		2.13	1.92	1.72	1.47	1.33	1.15	1.05	1.00	0.96	0.88	
$y = X_t/X_{\infty}$		0.42	0.46	0.51	0.61	0.66	0.77	0.84	0.88	0.92	1.00	
$1 - y$		0.58	0.54	0.49	0.39	0.34	0.23	0.16	0.12	0.08	0.00	
$1/(1 - y)$		1.72	1.85	2.04	2.56	2.94	4.35	6.25	8.33	12.50	-	
$\ln(1/(1 - y))$		0.54	0.62	0.71	0.94	1.08	1.47	1.82	2.12	2.53		

$$X_t = (M_0 - M_t)/M_{\infty} = [m_{eau}(ext)]/M_{\infty}$$

The experimental data were fitted by the Peleg model (**Figure 9**); $t/X_t = f(t)$ gives a straight line with a correlation coefficient $R^2 = 0.9923$, leading to a kinetic constant of order 2: $k_1 = 56.146 \text{ min (g/g)}^{-1}$ and an extraction capacity constant: $K_2 = 0.7924 \text{ (g/g)}^{-1}$. This gives the maximum extraction content (t_{∞} : equilibrium at the end of extraction): $X_e = 1/K_2 = 1/0.7924 = 1.26 \text{ g/g}$ and the drying kinetic constant $k = 0.7924/56.146 = 0.0141 \text{ min}^{-1}$.

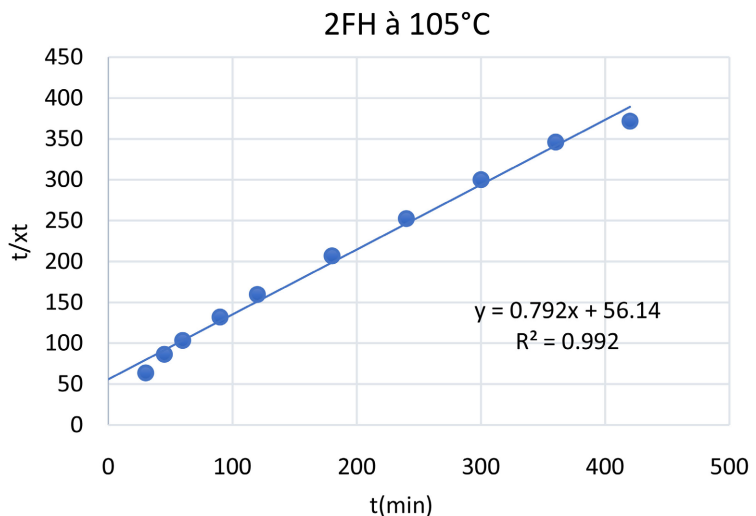


Figure 9. Peleg model validation line for safou pulp drying at 105°C.

The numerical and graphical methods lead to concordant results (**Table 8**). This validates the approximations used for the graphical method and, above all, the direction chosen for the model parameters.

Table 8. Peleg model parameters from numerical and graphical resolutions.

Parameters	$k_1(a)$ min (g/g) ⁻¹	$K_2(b)$ (g/g) ⁻¹	k (min ⁻¹)	X_e (g/g)
Digital resolution	42 - 172	0.71 - 0.90	0.0041 - 0.0210	1.11 - 1.41
Graphics solution	56	0.7924	0.0141	1.26

4. Conclusion

The drying of safou pulp can be considered as the desorption of a “particular metabolite”, water. It can be studied by empirical graphical resolution of the diffusion and Peleg models used in the literature. Its comparative treatment using numerical resolution and approximate graphical resolution of the diffusional and Peleg models leads to concordant results. This finding validates the approximations underlying formal first- and second-order kinetic graphical methods. We successively considered drying as a second-order desorption of the Peleg type or a first-order pseudo-diffusion of the Avrami/Page, Fick type, and used correlation lines to meet approximate solutions of the models, taking into account the physical context of the process. These methods, widely used in the literature for various metabolites, have been validated on drying by mechanisms showing a high degree of similarity with other metabolite extractions, probably due to the same limiting step: intra-particle diffusion in the plant matrix. However, all these hypotheses on a promising preliminary work will be deepened by a systematic study on Congo Basin oilseeds.

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Conflicts of Interest

No conflict of interest for this article.

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