Chemistry and Materials

The Center for Science Innovation

Research Article

Chem. Mater. 5 (3) 2026, 162-171

Retrospective Recovery Thresholds and Nonlinear Kinetic Modelling of n-Hexane Soxhlet Extraction from *Moringa oleifera* and *Irvingia gabonensis*

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Received
24 August 2026

Received in revised form
01 October 2026

Accepted
15 October 2026

Published online
27 October 2026

DOI
https://doi.org/10.56425/cma.v5i3.148

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Abstract

Selecting a soxhlet extraction time from the last observed maximum can prolong processing after most of the final extract has been collected. This exploratory study compared n-hexane extraction from *Moringa oleifera* and *Irvingia gabonensis* prepared batches. Oven-dried, 1-mm-sieved powders (15 g) were extracted with 250 mL n-hexane; the reported solvent temperature in the flask was 70 °C. Separate extractions were stopped after 1–10 or 1–12 siphon cycles, respectively, with two extractions per endpoint. Mean times and gravimetric yields were fitted directly to Peleg, first-order and Weibull functions. Final observed mean yields were 44.20% and 78.86% on a prepared-material basis. The first means exceeding 95% of these final means occurred at six cycles/89 min and seven cycles/78 min, respectively. These retrospective thresholds do not represent independently determined total extractable oil. With residual variance included in the corrected Akaike criterion, the three time-based models were not clearly separated for *M. oleifera*; first-order was favoured for *I. gabonensis*, with Weibull close. Individual duplicate readings, moisture, extract purity and constant-mass evidence were unavailable to the analysis. The thresholds are batch-specific laboratory candidates for confirmation, not validated stopping rules.

Keywords: solid-liquid extraction, *moringa oleifera*, *irvingia gabonensis*, n-hexane, Weibull model, Peleg model, retrospective thresholds

1. Introduction

Tropical oil-bearing seeds can diversify local lipid resources and provide feedstocks for food, cosmetic and technical applications. *Moringa oleifera* Lam. is especially valued because its seed oil is rich in oleic acid and displays appreciable oxidative stability [1-4]. Reported oil yields nevertheless depend strongly on genotype, whether whole seeds or kernels are used, moisture basis and extraction procedure. The influence of process conditions is well documented. Solvent and aqueous-enzyme extraction can give different recoveries from the same moringa material [5]. Hexane extraction has been optimized against particle size, temperature and residence time [6], while surveys of variable clones have reported wide ranges on kernel and whole-seed bases [7]. Enzymatic, solvent-assisted, supercritical-fluid and hybrid processes further show that oil yield and extract composition are jointly governed by

pretreatment and mass-transfer conditions [8-12]. Consequently, the last gravimetric value obtained in a time series should not automatically be interpreted as either the absolute lipid content or an economically optimal extraction time.

Irvingia gabonensis (Aubry-Lecomte ex O'Rorke) Baill. is an important non-timber forest resource in West and Central Africa. Its kernels, commonly processed as dika or odika, show marked biological and technological variability [13]. Aqueous enzymatic extraction [14], mechanical expression [15] and simultaneous aqueous recovery of lipid and gum fractions [16] demonstrate the diversity of matrices and products obtainable from the kernels. Recent solvent-extraction work reported effects of particle size, temperature and time [17], whereas detailed characterization of Congolese kernels identified a lauric-myristic fat and distinguished total lipid content from mechanical recovery [18].

Soxhlet extraction remains a reference technique for exhaustive laboratory recovery because fresh condensed solvent repeatedly contacts the solid bed [19]. The early part of a curve is commonly interpreted in terms of washing and dissolution of accessible oil, and later recovery in terms of solvent penetration, cell-wall resistance and internal diffusion [20-22]. These are hypotheses here, not mechanisms measured in the investigated materials. Prolonging extraction after this transition adds progressively less material while continuing to consume time, heat and solvent-handling capacity. A fraction of the final observed yield can therefore help describe candidate endpoints, provided it is not interpreted as a validated stopping rule.

Empirical asymptotic functions provide compact descriptions of these trajectories. The Peleg equation was originally proposed as a two-parameter non-exponential approach to equilibrium [23], whereas the Weibull cumulative function supplies independent scale and shape terms [24]. Both have been adapted to solid-liquid extraction, alongside first-order models [20,25]. Direct nonlinear fitting is preferable to regression of transformed variables because transformation changes the error structure and point weighting [26]. Model comparison should also penalize unnecessary flexibility [27]; the small-sample correction to Akaike's information criterion is particularly relevant for short kinetic series [28]. A high R^2 alone is not a sufficient selection criterion for nonlinear models [29].

This study reanalyses the recorded Soxhlet endpoint means obtained in Brazzaville for *M. oleifera* seed and *I. gabonensis* kernel materials. For these two prepared batches, its objectives were to: (i) compare recovery profiles across siphon cycles; (ii) fit Peleg, first-order and Weibull functions directly to the observed yields; and (iii) identify retrospective empirical 90%, 95% and 99% thresholds relative to the last observed yield. The work deliberately remains within the recorded dataset: no missing replicate, composition measurement or unreported response was reconstructed.

2. Materials and methods

2.1. Raw materials and preparation

Moringa oleifera seeds were collected directly from plants in the Diata district of Makélékélé, Brazzaville, Republic of the Congo. After opening the pods, seeds were manually decorticated and protected from light. Decorticated *I. gabonensis* kernels originating from Ouesso, Sangha Department, were purchased at the Texaco market in Brazzaville. Visibly deteriorated kernels were discarded, and retained lots were protected from moisture before processing.

Both materials were oven-dried at 70 °C, mechanically ground and passed through a 1-mm sieve. The duration of predrying and residual moisture were not recorded in the available extraction dataset; yields are therefore reported on the prepared-material basis rather than asserted as dry-matter lipid contents. All portions of each material came from the same homogenized batch of dried, ground powder. Collection/purchase dates, harvest season, botanical authentication or voucher records, numbers of source plants or commercial lots, storage duration and temperature, and detailed mixing procedures were not included in the available documentation.

2.2. Soxhlet extraction and gravimetric yield

For each terminal siphon-cycle count, fresh 15-g portions of ground material were placed on Whatman No. 1 filter paper in Soxhlet thimbles. Each flask initially contained 250 mL n-hexane, and no solvent was added during a run. The solvent-to-solid ratio of 16.67 mL g^{-1} is therefore the initial charge ratio, not the solvent volume maintained in the thimble. Separate extractions were stopped after 1–10 cycles for *M. oleifera* and 1–12 for *I. gabonensis*, with two extractions at every endpoint. A terminated run was not resumed to obtain the next point. The reported 70 °C refers to solvent in the boiling flask, not the heater setting or thimble temperature. After termination, the miscella was concentrated in a rotary evaporator. The extract was oven-dried at 103 °C for 1 h, cooled in a desiccator and weighed. Gravimetric extract yield was calculated as:

$$Y\ (\%) = \left(\frac{m_{ext}}{m_0}\right) \times 100 \quad (1)$$

where m_{ext} is the flask-plus-extract mass minus the empty-flask mass, and m_o is the initial prepared sample mass (15 g). The fixed 1-h drying treatment is not evidence of

constant mass: repeated drying/weighing results and a constant-mass criterion were not available. Residual solvent and triacylglycerol (TAG) purity were not independently quantified, so the response is termed gravimetric extract yield, not verified oil content.

Rotary-evaporator settings, cooling duration, pressure, temperature-monitoring details, equipment manufacturers/models and precision, and n-hexane grade, supplier and stated purity were not available in the documentation used for revision. Thus, neither solvent-free constant mass nor thermally stable cycling can be asserted.

2.3. Data structure and kinetic coordinates

The analysis comprised 10 endpoint means for *M. oleifera* and 12 for *I. gabonensis.* Both time and yield are means of two separate extractions at each endpoint. Extract mass is cumulative within each run, but the curve combines different terminal runs. The time origin was the onset of solvent boiling, and elapsed time ended at the assigned terminal siphon. Mean elapsed time was the primary kinetic coordinate; terminal siphon-cycle count was a secondary coordinate.

The 89- and 91-min means for *M. oleifera* belong to different pairs of extractions stopped after six and seven cycles; their difference is not a measured 2-min seventh cycle. Individual duplicate times and yields were unavailable to the analysis, so original timing entries could not be independently checked and all means were retained unchanged. No dispersion estimates, error bars or between-material hypothesis tests were reconstructed. Duplicates within one prepared batch do not establish between-batch reproducibility.

2.4. Kinetic models

Three positive-parameter asymptotic functions were fitted directly to mean yield by unweighted nonlinear least squares. The Peleg function was written as:

$$Y(t) = \frac{t}{(K_1 + K_2 t)}, Y\infty = \frac{1}{K_2} \quad (2)$$

where $\frac{1}{K_1}$ describes the initial slope and $\frac{1}{K_2}$ is the modelled asymptote. The first-order function was:

$$Y(t) = Y\infty[1 - exp(-kt)] \quad (3)$$

where *k* is an apparent extraction rate constant, not an intrinsic chemical-reaction constant. The Weibull function was:

$$Y(t) = Y\infty\left\{1 - exp\left[-\left(\frac{t}{\alpha}\right)^{\beta}\right]\right\} \quad (4)$$

where α is the time scale and β is the dimensionless shape parameter; β = 1 reduces to the first-order form. Times to a fraction f of the fitted model asymptote were calculated as $t(f) = \left[\frac{f}{(1-f)}\right]\frac{K_1}{K_2}$ for Peleg, $t(f) = -\ln\left(\frac{1-f}{k}\right)$ for first-order and $t(f) = \alpha[-\ln(1-f)]^{\frac{1}{\beta}}$ for Weibull. Thus, t_{90} and t_{95} refer to $Y\infty$, not the final observed mean. Numerical fitting settings, starting values, bounds and convergence handling are specified in Supplementary File 1, Section S1, and the executable code in Supplementary File 2.

2.5. Fit comparison and retrospective thresholds

Fit quality was summarized by residual sum of squares (RSS), root mean squared error (RMSE), mean absolute error (MAE) and descriptive R^2. For information-criterion comparison [27,28], endpoint-mean errors were provisionally assumed independent, additive Gaussian with a common unknown variance within each material series; the meantime coordinate was treated as fixed. The small-sample corrected Akaike information criterion (AICc) was calculated as:

$$AICc = n\left[\ln(2\pi) + 1 + \ln\left(\frac{RSS}{n}\right)\right] + 2K + 2K\left(\frac{K+1}{n-K-1}\right) \quad (5)$$

Here *n* is the number of endpoint means (10 or 12), and *K* = *p* + 1 includes the estimated residual variance $\frac{RSS}{n}$: *p* = 2 for Peleg and first-order, *p* = 3 for Weibull, hence *K* = 3 or 4. Within each material and coordinate, *ΔAICc* was calculated from the minimum *AICc* and weights as $\frac{exp\left(\frac{-\Delta AICc}{2}\right)}{\sum exp\left(\frac{-\Delta AICc}{2}\right)}$. *ΔAICc* < 2 was interpreted as no clear separation. Equation (5) includes the Gaussian constant; its conventional correction is used conditionally for these nonlinear fits, not as validation of the error assumptions.

Residuals (observed mean minus fitted yield) were inspected against mean time and fitted yield (Supplementary Figs. S1–S4). Conditional regression intervals are possible, but were not reported because mean-time uncertainty and the assumed mean-response error model could not be assessed. Without individual duplicate values, replicate dispersion and pure error cannot be separated from lack of fit.

Independently of model extrapolation, the empirical 90%, 95% and 99% thresholds were defined as the first sampled endpoint mean meeting the selected fraction of the final observed mean yield. This denominator is not independently determined total extractable oil. The 95% level was selected post hoc as an illustrative central criterion, with 90% and 99% as sensitivity cases. Duration differences were calculated against the final mean elapsed time, not total processing time or energy. Calculations used Python 3, NumPy, SciPy, pandas and Matplotlib; the complete mean dataset and revision code are supplied as Supplementary File 2.

3. Results and Discussion

3.1. Observed recovery and retrospective thresholds

Both investigated batches showed rapid initial recovery followed by a progressively flatter trajectory (Table 1 and Fig. 1). *M. oleifera* increased from 16.80% at the first cycle to 32.46% at the third, equivalent to 73.4% of its final observed mean yield. It reached 41.33% at the fifth cycle and 44.20% at the final two observations. The last yield corresponds to 6.63 g of gravimetric extract from 15 g of prepared material.

I. gabonensis increased from 23.93% at the first cycle to 60.53% at the third, or 76.8% of its final observed yield. Recovery then rose to 77.53% at the seventh cycle and only to 78.86% at the final observation, corresponding to 11.83 g extract from 15 g material. The final gravimetric yield ratio between *I. gabonensis* and *M. oleifera* was 1.78. This descriptive ratio uses prepared-material masses, not dry masses, and cannot isolate an effect of species from differences in moisture, origin, composition or handling.

The 99% criterion was met after eight cycles and 98 min for *M. oleifera* (44.13%) and after nine cycles and 108 min for *I. gabonensis* (78.73%). Neither criterion defines an exhaustive extraction or a universal optimum. Both are retrospective descriptions relative to the last observed mean and require independent confirmation before prospective use.

3.2. Nonlinear model performance

All three functions represented the approach to a plateau, but their extrapolated asymptotes and recovery times differed markedly (Fig. 2 and Table 3). For *M. oleifera*, Weibull produced the lowest RMSE (0.541 percentage point), an estimated asymptote of 45.98% and β = 0.8743. The first-order asymptote (44.62%) remained close to the final observation. After correcting the AICc parameter count, Peleg had the minimum *AICc*, with Weibull and first-order at *ΔAICc* = 0.472 and 1.313; their weights were 0.433, 0.342 and 0.225, respectively. Thus, the three candidates were not clearly separated. Peleg's higher asymptote (54.61%) and t_{95} of 484.5 min still limit the plausibility of its long-range projection.

Table 1. Mean cumulative n-hexane extract yields and elapsed times from separate terminal extractions (two extractions per endpoint; prepared-material basis).

Cycle	Mean time (min)	*M. oleifera* mean yield (%)	Cycle	Mean time (min)	*I. gabonensis* mean yield (%)
1	12	16.80	1	13	23.93
2	26	28.00	2	27	43.26
3	40	32.46	3	34	60.53
4	52	37.26	4	46	61.66
5	72	41.33	5	55	65.93
6	89	42.40	6	67	71.12
7	91	42.86	7	78	77.53
8	98	44.13	8	84	77.80
9	115	44.20	9	108	78.73
10	125	44.20	10	118	78.80
-	-	-	11	128	78.80
-	-	-	12	139	78.86

Times run from boiling onset to the terminal siphon of each separate extraction. Adjacent mean times are not successive cycle durations from one run. Individual duplicate values were unavailable.

The 90% threshold was first met after 72 min for *M. oleifera* and 67 min for *I. gabonensis* (Table 2). At the illustrative 95% criterion, *M. oleifera* produced 42.40% after six cycles and 89 min, while *I. gabonensis* produced 77.53% after seven cycles and 78 min. These means actually reached 95.93% and 98.31% of the final means; discrete sampling does not locate the exact 95% crossing. Their recorded durations were 36 min (28.8%) and 61 min (43.9%) shorter than the final mean durations. Yield differences were 1.80 and 1.33 percentage points, or 4.1% and 1.7% of the final yields, equivalent to 0.270 and 0.200 g per 15-g charge. These are comparisons between distinct terminal runs, not increments from a resumed extraction.

For *I. gabonensis,* first-order kinetics had the minimum *AICc*, with Y_∞ = 81.34% and k = 0.031885 min^{-1}. Weibull returned a lower RMSE (2.466 versus 2.768 percentage points) and Y_∞ = 79.47%, but its extra shape parameter was penalized; *ΔAICc* = 1.944 and the corresponding weights (0.715 and 0.271) indicate relative preference, not decisive separation. Peleg was weaker (*ΔAICc* = 7.831; weight 0.014) and projected Y_∞ = 100.66% and t_{95} = 565.4 min despite near-stability of the observations by 108–139 min. Peleg can be written as $Y = \frac{Y_\infty t}{(\tau + t)}$, where $\tau = \frac{K_1}{K_2}$. Its

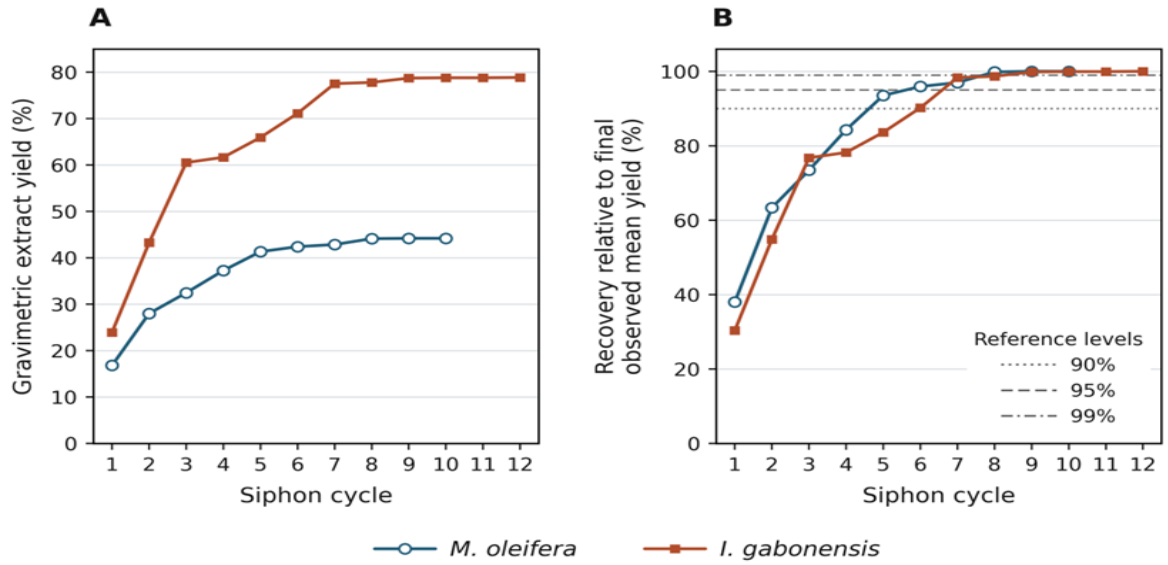


Figure 1. Observed extraction profiles. (A) Gravimetric extract yield and (B) yield relative to the final observed mean as functions of terminal siphon-cycle count. Symbols are means of two separate extractions per endpoint; connecting lines are guides to the eye. Horizontal lines mark 90%, 95% and 99% of the last observed mean, not independently determined total extractable oil. No error bars are shown because individual duplicate readings were unavailable.

remaining fraction decays algebraically as $Y - Y\infty = \frac{Y\infty t}{(\tau+t)}$, so a short series can yield a plausible local fit but an excessive long-range plateau. Positive-parameter fitting does not constrain $Y\infty$ below 100%. The 100.66% asymptote is therefore physically implausible, not an observed yield. This behaviour cannot be attributed to linearization because the reported fits were directly nonlinear [26,29]. Residual plots show patterned Peleg departures for *I. gabonensis* and a positive residual near 6.7 percentage points at its 34-min/60.53% observation for all three time-based models (Supplementary Figs. S1–S4). That point was retained. The limited means do not establish constant variance or Gaussian errors, and fit metrics should not conceal these departures.

and Weibull RMSE values were 0.617 and 0.606 percentage point; corrected weights were 0.941 and 0.056, with Weibull at $\Delta AICc$ = 5.631. For *I. gabonensis*, their RMSE values were 2.101 and 2.086, and corrected weights were 0.888 and 0.092. Coordinate choice therefore affects model ranking, but not the retrospective threshold calculated from the endpoint means. Neither coordinate establishes a unique mechanism.

3.3. Matrix-dependent yield and chemical interpretation

The 44.20% *M. oleifera* yield lies near the upper end of ranges reported for solvent-extracted kernels and variable clones [3,4,7], and exceeds some optimized hexane yields [6]. This is chemically plausible for a decorticated, finely ground kernel material, but direct numerical comparison

Table 2. First endpoint means meeting retrospective thresholds relative to the final observed mean yield, not independently determined total extractable oil.

Material	Target	Mean time (min)	Cycle	Mean yield (%)	Recorded-duration difference (%)	Unrecovered fraction relative to the final observed yield (%)
M. oleifera	90%	72	5	41.33	42.4	6.5
M. oleifera	95%	89	6	42.40	28.8	4.1
M. oleifera	99%	98	8	44.13	21.6	0.2
I. gabonensis	90%	67	6	71.12	51.8	9.8
I. gabonensis	95%	78	7	77.53	43.9	1.7
I. gabonensis	99%	108	9	78.73	22.3	0.2

Final observed mean yield: 44.20% at 125 min/10 cycles for *M. oleifera* and 78.86% at 139 min/12 cycles for *I. gabonensis*. unrecovered fraction $= 100(1 - \frac{Y_{selected}}{Y_{final}})$, not a percentage-point difference; duration difference $= 100\ \frac{(t_{final} - t_{selected})}{t_{final}}$, not an energy or cost saving. The 95% target is post hoc.

The secondary siphon-cycle analysis retained first-order as the lowest-*AICc* model for both materials (Supplementary Tables S1–S2). For *M. oleifera*, first-order

remains sensitive to genotype, particle size, moisture basis, solvent ratio and desolventization. Studies using enzymatic, supercritical-fluid or assisted extraction

confirm that recovery and extract composition can shift together [5,8-12].

The 78.86% *I. gabonensis* value is high relative to the 63.8% fat content and 47.9% hot-press recovery reported for one Congolese material [18] and to the 68.80% maximum of a solvent study [17]. Biological variability and differences in kernel preparation may contribute [13], but the safest interpretation remains a gravimetric n-hexane extract on the prepared-sample basis. It should not be equated with verified TAG content. Incomplete solvent removal or coextracted constituents other than TAG, such as waxes, resinous material and low-polarity pigments, could contribute to the mass; none was quantified here. Fixed-time drying at 103 °C does not establish constant mass. Moisture in the starting solid affects the denominator: at equal extract mass, dry-basis yield would be $\frac{Y_{prepared}}{(1-w)}$, where *w* is its water mass fraction. It does not itself inflate the weighed extract; retained water or solvent in that extract would affect the numerator. No moisture correction is possible from the available data.

The normalized trajectories nevertheless carry useful descriptive information: the first three cycles recovered 73.4% and 76.8% of the final observed means. Washing and dissolution of accessible material, followed by restricted access, are plausible hypotheses, not measured mechanisms. Both materials were decorticated and passed through a 1-mm sieve; this does not establish equal particle-size distributions, fines contents or agglomeration. Different intact-cell fractions, cell-wall resistance and bed structure could change access and diffusion paths, but were not measured [20–22]. Literature describes oleic-rich *M. oleifera* oils [1–4] and lauric-myristic *I. gabonensis* fat [18]; their fluid properties could affect dissolution and flow, but lipid and miscella viscosities were not measured here. Native gums/mucilages could affect matrix structure; aqueous lipid–gum recovery [14,16] is not evidence that gums were extracted by n-hexane. The yield difference therefore cannot be assigned to any one structural or chemical factor.

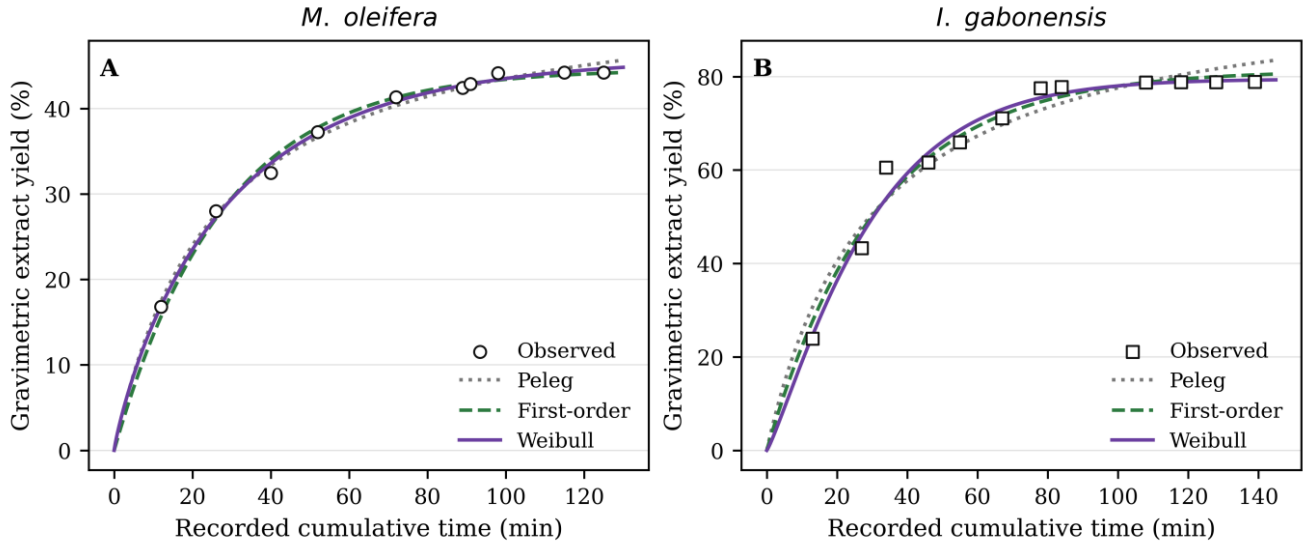


Figure 2. Recorded gravimetric yields and direct nonlinear fits for (A) Moringa oleifera and (B) Irvingia gabonensis. Symbols are mean time–yield pairs from two separate terminal extractions; lines are model fits (Table 3A). Time is elapsed from boiling onset. Curves extend to zero without adding an observation or experimental uncertainty band.

For Weibull, β = 0.8743 gives a decreasing fractional approach rate, $\frac{\left[\frac{dY}{dt}\right]}{(Y\infty - Y)} = \left(\frac{\beta}{\alpha}\right)\left(\frac{t}{\alpha}\right)^{\beta-1}$, compatible with declining accessibility. First-order assumes a constant fractional approach rate *k* and can summarize the *I. gabonensis* profile with fewer curve parameters. Neither description proves washing, cell-wall control or intraparticle diffusion, especially given the corrected weak model separation.

3.4. Laboratory implications and solvent perspective

The 95% thresholds identify laboratory-scale candidates for confirmatory trials, not validated operating optima. Prospective testing would fix six and seven cycles before new independent experiments and compare them with a separately justified exhaustive reference; the present final observations are not such validated references. Heating, condensation, evaporation, drying, cooling and solvent recovery were not quantified, so duration differences do not measure proportional energy or cost savings. The 103 °C/1-h drying step is outside the reported boiling-to-siphon time. Comparable kinetic studies with ethanol show dependence on temperature, particle size and moisture [22,30]. A subsequent process assessment would need an objective function including extract value, time, energy, solvent recovery and cake quality.

Periodic solvent renewal is intrinsic to Soxhlet operation [19], but the initial 16.67 mL g^{-1} ratio does not determine local miscella concentration. Chamber fill volume, reflux rate, per-cycle concentration and solvent dependence or extraction time. Their capital and energy requirements differ substantially from Soxhlet operation. A meaningful follow-up should therefore compare processes on the same seed lot and report extract

Table 3. Direct nonlinear fits to endpoint mean yield–time data. (A) Parameter estimates and times relative to each fitted asymptote. (B) Complete information-theoretic and error statistics.

(A) Parameter estimates and model-asymptote times

Material	Model	Parameter estimates	Y∞ (%)	t_{90} (min)	t_{95} (min)
M. oleifera	Peleg	K_1 = 0.46696; K_2 = 0.018312	54.61	229.5	484.5
M. oleifera	First-order	k = 0.036025 min^{-1}	44.62	63.9	83.2
M. oleifera	Weibull	α = 29.324 min; β = 0.8743	45.98	76.1	102.9
I. gabonensis	Peleg	K_1 = 0.29562; K_2 = 0.0099347	100.66	267.8	565.4
I. gabonensis	First-order	k = 0.031885 min^{-1}	81.34	72.2	94.0
I. gabonensis	Weibull	α = 30.530 min; β = 1.1671	79.47	62.4	78.2

(B) Fit statistics

Material	Model	RSS	RMSE	MAE	R^2
M. oleifera	Peleg	5.084	0.713	0.632	0.9932
M. oleifera	First-order	5.798	0.761	0.583	0.9922
M. oleifera	Weibull	2.925	0.541	0.413	0.9961
I. gabonensis	Peleg	176.598	3.836	3.129	0.9463
I. gabonensis	First-order	91.954	2.768	2.107	0.9720
I. gabonensis	Weibull	73.000	2.466	1.744	0.9778

Y∞ is a fitted asymptote, not verified total extractable oil; t_{90}/t_{95} refer to Y∞. Units: K_1 is min per percentage point; K_2 is inverse percentage point; k (min^{-1}); α (min); β (dimensionless). RMSE/MAE are in percentage points and RSS in squared percentage points. Full AICc includes residual variance (K = 3 or 4; n = 10 or 12 means) and the Gaussian constant. Weights apply within each material's time-based candidate set. No confidence intervals are asserted.

temperature fluctuations were not measured or documented. Local saturation or thermal contributions to the apparent rate constants therefore cannot be excluded or quantified from these observations.

n-Hexane is effective for nonpolar lipid recovery and its volatility facilitates separation, but flammability, occupational exposure and fossil origin motivate solvent substitution. Experimental studies have compared alcohols and terpenes with hexane in rapeseed systems [31], evaluated bio-based 2-methyloxolane for oil extraction [32,34], and directly compared ethanol and hexane on press cake [35]. Broader assessments emphasize that extraction selectivity, solvent recycling, energy use and product quality must be evaluated together [33,36]. Any such comparison would require a consistent, independently justified recovery reference rather than assuming that the present final yields represent exhaustive extraction.

For *M. oleifera* specifically, supercritical CO_2 with ethanol pretreatment [37], ultrasound and microwave intensification [38], pilot-scale supercritical CO_2 [39] and response-surface optimization of supercritical extraction [40] demonstrate feasible routes for reducing petroleum-yield, triacylglycerol purity, fatty-acid profile, solvent residue, specific energy demand and residual-meal quality.

3.5. Scope, uncertainty and validation priorities

This study provides exploratory descriptions of endpoint means from two prepared batches. Duplicate extractions were performed, but their individual readings and dispersion were unavailable to the analysis. Residuals describe departures from fitted means, not independently estimated experimental variability. The Akaike weights are conditional on the working likelihood and the recorded means; they do not quantify between-batch reproducibility. Uncertainty in mean time cannot be propagated from the table, and preparation, origin and moisture differences prevent isolation of a causal species effect. Further confirmation would require moisture determination before extraction, extract drying to demonstrated constant mass with residual-solvent verification, and an independent total-lipid or chromatographic mass balance. Recording each run's terminal time, yield and variability, preferably across independent raw-material batches, would support uncertainty estimation and prospective assessment of the candidate thresholds. These are future validation

priorities; no new experiments were performed for this revision.

4. Conclusion

Under the reported laboratory conditions of 15 g prepared material, passage through a 1-mm sieve, an initial 250 mL n-hexane charge and 70 °C solvent in the flask, final observed mean gravimetric yields were 44.20% for *M. oleifera* and 78.86% for *I. gabonensis*. These are prepared-material, batch-specific observations, not verified dry-basis oil contents. The first endpoint means exceeding 95% of the final means occurred at six cycles/89 min and seven cycles/78 min. Recorded durations differed from the final means by 28.8% and 43.9%, with yield gaps of 4.1% and 1.7% of the final observed yields. These comparisons do not establish energy savings or validated stopping rules.

Corrected time-based AICc values did not clearly separate the three models for *M. oleifera*. First-order was favoured for *I. gabonensis*, with Weibull remaining close; cycle-based comparisons favoured first-order for both. Peleg extrapolated higher plateaus and recovery times, particularly for *I. gabonensis*. The mean-data analysis is exploratory because individual duplicate readings, moisture, extract purity and constant-mass evidence were unavailable. The retrospective thresholds are candidates for laboratory confirmation against independently justified exhaustive extraction, not scale-up prescriptions.

Author contributions

Anicet Frédéric Binaki: Investigation, methodology, data curation, writing - original draft. **Eliane Thérèse Biassala**: Investigation, writing—review, manuscript revision. **Bob Wilfrid Loumouamou**: Conceptualization, formal analysis, visualization, supervision, writing - review and editing. **Hubert Makomo**: Writing - review and editing. **Lobin Paniche Damba-Kabi**: Investigation, resources, writing - review and editing. **Jean Mathurin Nzikou**: Supervision, writing - review and editing. **Thomas Silou**: Conceptualization, supervision, writing - review and editing. All authors reviewed and approved the submitted version. Experimental validation of the proposed thresholds is not claimed.

Conflicts of interest

There are no conflicts to declare.

Acknowledgement

The authors thank the technical staff of the Laboratory of Food Chemistry and Technology and the Process Engineering Laboratory of Marien Ngouabi University for the institutional and laboratory support provided during this work.

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