Optimized Extraction of Phenolic Compounds from *Ficus exasperata* Using Ultrasound and Maceration: A Comparative Kinetic Study

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ABSTRACT

Ficus exasperata is a medicinal plant used in West Africa for managing inflammation, infections, and oxidative stress. However, its optimized extraction processes remain poorly studied. Conventional maceration (MAC) and ultrasound-assisted extraction (ULT) were compared for phenol and flavonoid recovery from *F. exasperata* leaves at solid-to-liquid ratios of 1/60, 1/80, and 1/120 g/mL. Extraction yields were measured over 35 min and fitted to a second-order kinetic model. ULT markedly increased yields, especially at 1/60 g/mL, achieving 351.5 mg GAE/g DM (phenolic compounds) and 8.88 mg QE/g DM (flavonoids). The model showed excellent fit ($R^2>0.99$) and indicated internal diffusion as the rate-limiting step. Higher kinetic constants under ULT confirmed its faster extraction rate. ULT outperformed MAC, making it a more efficient and sustainable green technology for producing antioxidant-rich extracts while enhancing the mechanistic understanding of plant-based solid–liquid extraction.

Keywords: *Ficus exasperata*, Ultrasound-assisted extraction, Phenolic compounds, Flavonoids, Extraction kinetics, Green technology.

INTRODUCTION

The growing interest in natural bioactive compounds has driven research towards plant sources of antioxidants, particularly polyphenols and flavonoids, due to their health-promoting properties[1,2]. These compounds are widely recognized for their free-radical-scavenging, anti-inflammatory, and antimicrobial activities, making them valuable to the food, cosmetic, and pharmaceutical industries[3,4].

Ficus exasperata, a member of the Moraceae family, is widely distributed across tropical Africa and traditionally used to treat ailments such as ulcers, inflammation, diarrhea, asthma, gastric pain, and hypertension[5]. Its leaves are rich in phenolic compounds, which are responsible for its therapeutic effects[6]. Despite this importance,

systematic optimization of extraction conditions for these bioactive compounds remains limited.

Extraction efficiency depends on several parameters, including the extraction medium, temperature, processing time, and solid-to-liquid (S/L) ratio[7]. Recently, eco-friendly techniques such as ultrasound-assisted extraction (ULT) have attracted attention as alternatives to conventional maceration, offering faster processing, reduced solvent use, and enhanced yields[8,9].

However, no comparative kinetic modeling studies have yet been performed for phenolic extraction from *F. exasperata* using different S/L ratios in maceration (MAC) and ultrasound-assisted extraction (ULT). It is expected that ULT will yield higher extraction rates and equilibrium concentrations than MAC under identical conditions. This study was designed to:

- Compare the extraction yields of total phenolic compounds, including flavonoids, from *F. exasperata* leaves using MAC and ULT.
- Evaluate the influence of three S/L ratios (1/60, 1/80, and 1/120 g/mL) on extraction efficiency.
- Apply a second-order kinetic model to interpret the extraction dynamics and identify rate-limiting steps.

MATERIAL AND METHODS

Plant Material

Fresh leaves of *Ficus exasperata* were collected in Kassiapleu, Tonkpi region (7°' 24 N, 7° 33' W), in December 2024. Botanical identification was confirmed by the National Floristic Center in Abidjan. The leaves were cleaned, air-dried in the shade under ambient conditions for seven days, and milled using a laboratory grinder. The resulting powder was stored in airtight containers at room temperature until extraction.

Chemicals

All reagents used in this study were of analytical grade. Gallic acid and quercetin standards (Sigma-Aldrich, Germany) were used for the quantification of total phenolic and flavonoid contents, respectively. The Folin–Ciocalteu reagent and aluminum chloride were employed for the colorimetric assays.

Extraction Procedures

Two extraction methods were evaluated: conventional maceration (MAC) and ultrasound-assisted extraction (ULT). Each extraction was performed using three solid-to-liquid (S/L) ratios: 1/60, 1/80, and 1/120 g/mL. For both techniques, a quantity of plant powder was mixed with the appropriate volume of 60% acetone solution.

- For maceration (MAC), the mixtures were stirred continuously at 40°C.
- Ultrasound-assisted extraction (ULT) was performed using a GT SONIC ultrasonic cleaner, model PS-40 (240 W, AC 110/220 V) at 40°C.

Samples were collected every 5 min for 35 minutes. The extracts were subjected to centrifugation at 3000 rpm for 10 min, and the resulting supernatant was retrieved for the determination of phenolic and flavonoid contents. All extractions were performed in triplicate.

Determination of Total Phenolic Content

Total phenolic content (TPC) was determined using the Folin–Ciocalteu colorimetric method, following the protocol described by Singleton *et al.,*[10], with minor modifications. Briefly, 100 µL of crude extract were mixed with 500 µL of 10% (w/v) Folin–Ciocalteu reagent. After 10 min of incubation at room temperature, 500 µL of saturated sodium carbonate (Na_2CO_3) solution were added, and the mixture was incubated for an additional 40 min in the dark. Absorbance was measured at 765 nm using a UV–Visible spectrophotometer (ONDA UV-30SCAN, China), with a reagent blank as reference. A standard calibration curve was prepared using gallic acid, and results were expressed as milligrams of gallic acid equivalents per gram of dry matter (mg GAE/g DM).

Determination of Total Flavonoid Content

Total flavonoid content (TFC) was determined using the aluminum chloride colorimetric method described by Galgano *et al.,*[11], with minor modifications. Briefly, 2 mL of crude extract or quercetin standard solution (3.125–50 µg/mL) were mixed with 1 mL of 10% (w/v) aluminum chloride ($AlCl_3$) prepared in methanol. After 30 min of incubation at room temperature, absorbance was measured at 434 nm using a UV–Visible

spectrophotometer (ONDA UV-30SCAN, China), with a blank as reference. A standard calibration curve was prepared using quercetin, and results were expressed as milligrams of quercetin equivalents per gram of dry matter (mg QE/g DM).

Kinetic Modeling of Total Phenol and Flavonoid Extraction Using a Second-Order Equation

To better understand the kinetic mechanisms governing the release of total phenols, including total flavonoids, from the leaves of *Ficus exasperata*, the extraction profiles were modeled using a second-order kinetic equation. This approach is consistent with previous studies demonstrating the relevance of second-order models for describing solid–liquid extraction dynamics (Lazar *et al.*, 2016 ; Peng *et al.*, 2023 ; Rodríguez-Fernández *et al.*, 2023). The second-order kinetic model provided the best fit to the experimental data and offered a robust explanation of the mass transfer phenomena involved. The differential form of the model is expressed as Equation (1):

$$\frac{dC_t}{dt} = k(C_S - C_t)^2 \tag{1}$$

Where Ct is the concentration (yield) at time t, CS is the equilibrium concentration, and k is the rate constant.

The model parameters CS and k were estimated by fitting the experimental data to the integrated form of the Equation (2):

$$C_t = \frac{C_S^2 kt}{1 + C_S kt} \tag{2}$$

Nonlinear regression was performed in RStudio (version 2024.12.1+563), minimizing the sum of squared residuals between observed and predicted values. The goodness of fit between experimental and predicted data was evaluated using the coefficient of determination (R²) and the root mean square error (RMSE), calculated according to Equations (3) and (4).

$$R^2 = 1 - \frac{\sum_{j=1}^{N}(q_{exp} - q_{cal})^2}{\sum_{j=1}^{N}(q_{exp} - \bar{q}_{cal})^2} \tag{3}$$

$$RMSE = \sqrt{\frac{1}{N}\sum_{t=1}^{N}(\bar{q}_{exp} - \bar{q}_{cal})^2} \tag{4}$$

Statistical Analysis

All statistical analyses were performed using RStudio software (version 2024.12.1+563). Data are expressed as mean±standard deviation from three independent replicates. A 95% confidence level (p<0.05) was applied for all statistical tests. Differences among means were assessed using one-way analysis of variance (ANOVA), and significant differences were determined using Tukey's post-hoc test. Scatter plots with regression curves were generated to visualize the kinetic modeling fits, while boxplots were used to compare extraction yields between treatments. Boxplots display medians, interquartile ranges, whiskers, and potential outliers unless otherwise specified.

RESULTS AND DISCUSSION

Kinetics and Comparative Analysis of Phenolic Compound Extraction

The evolution of phenolic compound yields over time (Fig. 1) showed a rapid increase during the first 20 min, followed by a slower phase and stabilization, indicating saturation. This pattern was well described by a pseudo-second-order kinetic model, confirming that internal diffusion within the plant matrix is the rate-limiting step, as previously reported[12,15].

Model parameters (Table 1) displayed excellent agreement between experimental and theoretical values (R²>0.99; RMSE<10), validating the model's suitability. Ultrasound-assisted extraction (ULT) consistently achieved higher equilibrium yields (C_S) and rate constants (k) than maceration (MAC). At a 1/60 g/mL solid/liquid ratio, ULT reached 351.5 mg GAE/g DM versus 303.5 mg GAE/g DM for MAC, with k values of 0.0024 and 0.0021 g DM·mg GAE⁻¹·min⁻¹, respectively. These gains resulted from cavitation-induced cell wall disruption, which enhanced solvent penetration and solute desorption[16].

Boxplots (Fig. 2) confirmed these trends, with higher medians and narrower interquartile ranges (IQR) for ULT, reflecting greater reproducibility, especially at 1/80 and 1/120 g/mL. A few outliers in MAC suggest lower process control due to slower mass transfer and possible degradation. Yield declined with dilution from 1/60 to 1/120 g/mL, consistent with earlier reports[17] that higher solvent volumes reduce solute–solvent interactions and increase losses through oxidation or adsorption. ULT mitigated this effect through improved mixing and penetration[18].
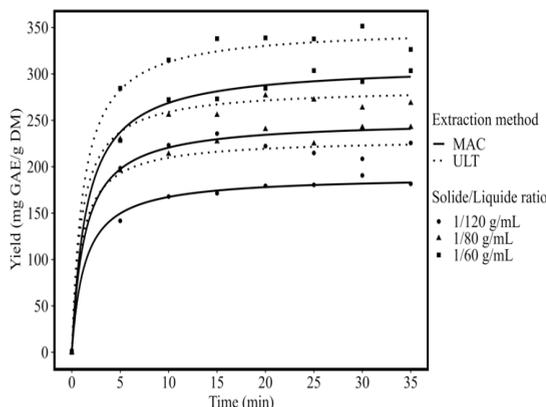
Fig. 1. Comparison of experimental and modeled phenolic compound extraction kinetics from *F. exasperata* at different solid-to-liquid (S/L) ratios using maceration (MAC) and ultrasound-assisted extraction (ULT). Yields are expressed as milligrams of gallic acid equivalents per gram of dry matter (mg GAE/g DM)
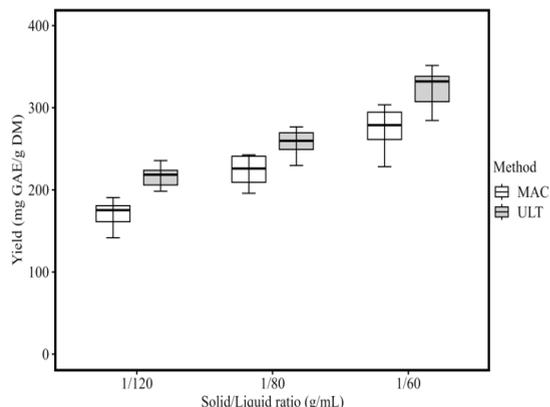

Fig. 2. Comparison of phenolic compound extraction yields obtained by maceration (MAC) and ultrasound-assisted extraction (ULT) at three solid-to-liquid (S/L) ratios (1/60, 1/80, and 1/120 g/mL). Results are expressed as milligrams of gallic acid equivalents per gram of dry matter (mg GAE/g DM)

Table 1: Kinetic parameters and equilibrium concentrations for phenolic compound extraction from *F. exasperata* leaves using maceration (MAC) and ultrasound-assisted extraction (ULT) at different solid-to-liquid (S/L) ratios. Yields are expressed as milligrams of gallic acid equivalents per gram of dry matter (mg GAE/g DM)

Method	S/L ratio(g/mL)	Theoretical C_s (mg GAE/g DM)	k (g DM.mg GAE^{-1}.min^{-1})	Experimental C_s (mg GAE/g DM)	RMSE	R^2
MAC	1/60	316.6	1.656 10^{-3}	303.6	5.685	0.996
ULT	1/60	355.0	2.373 10^{-3}	351.5	7.870	0.995
MAC	1/80	249.8	2.762 10^{-3}	242.6	8.389	0.995
ULT	1/80	279.8	3.400 10^{-3}	276.7	5.347	0.996
MAC	1/120	195.7	2.741 10^{-3}	190.6	3.102	0.997
ULT	1/120	226.5	8.583 10^{-3}	235.7	9.030	0.984

Kinetics and Comparative Analysis of Flavonoid Extraction

Flavonoid extraction kinetics (Fig. 3) exhibited a biphasic profile: a rapid uptake in the first 20 min, then a plateau at 30–35 min, characteristic of internal diffusion-controlled processes[19,20]. The pseudo-second-order model fitted the data well ($R^2>0.99$; low RMSE; Table 2), confirming that internal mass transfer limits the extraction, in agreement with previous observations[21,22].

At 1/60 g/mL, ULT achieved C_s = 9.18 mg QE/g DM versus 6.61 mg QE/g DM for MAC, with k = 0.08276 and 0.07158 g DM·mg QE^{-1}·min^{-1}, respectively. The performance boost is attributed to ultrasound cavitation, which disrupts cell structures, increases solvent penetration, and accelerates solute release[23,24].

Dilution from 1/60 to 1/120 g/mL decreased both C_s and k, reflecting lower biomass concentration, weaker solute gradients, and increased solvent viscosity-all factors that slow diffusion[25,26]. Similar dilution effects were observed during flavonoid recovery from *Flos sophorae* with infrared-assisted extraction[27].

Boxplots (Fig. 4) further illustrated ULT's advantage: at 1/60 g/mL, median yield was 8.88 mg QE/g DM for ULT and 6.12 mg QE/g DM for MAC, with narrower IQRs under ULT, indicating higher reproducibility. Minimal outliers under ULT contrast with MAC at 1/120 g/mL, where variability increased. Comparable patterns were reported for ultrasound-assisted deep eutectic solvent extraction from *Flos sophorae* Immaturus[28]. Slightly higher dispersion in some ULT conditions may reflect sensitivity to acoustic intensity or sample positioning[29].

Overall, this study confirms that kinetic modeling not only quantifies performance differences

between ULT and MAC but also substantiates that internal diffusion limits the extraction of phenolic compounds and flavonoids from *Ficus exasperata*.

ULT consistently overcomes these limitations through enhanced mass transfer, even under less favorable solid/liquid ratios.
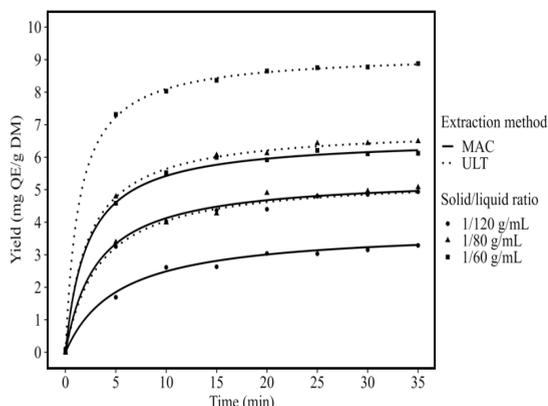

Fig. 3. Comparison of experimental and modeled flavonoid extraction kinetics from *F. exasperata* at different solid-to-liquid (S/L) ratios using maceration (MAC) and ultrasound-assisted extraction (ULT). Flavonoid yields are expressed as milligrams of quercetin equivalents per gram of dry matter (mg QE/g DM)
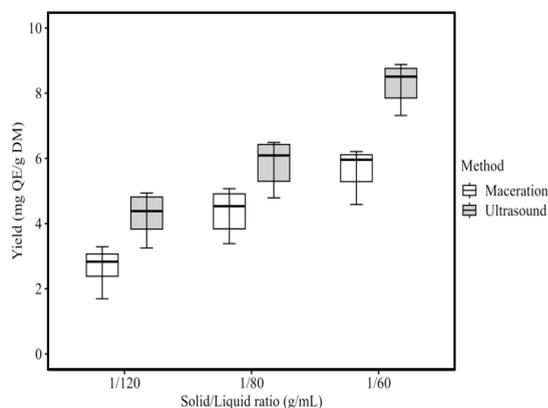

Fig. 4. Comparison of flavonoid extraction yields obtained by maceration (MAC) and ultrasound-assisted extraction (ULT) at three solid-to-liquid (S/L) ratios (1/60, 1/80, and 1/120 g/mL). Results are expressed as milligrams of quercetin equivalents per gram of dry matter (mg QE/g DM)

Table 2: Kinetic parameters and equilibrium concentrations for flavonoid extraction from *F. exasperata* leaves using maceration (MAC) and ultrasound-assisted extraction (ULT) at different solid-to-liquid (S/L) ratios. Yields are expressed as milligrams of quercetin equivalents per gram of dry matter (mg QE/g DM)

Method	S/L Ratio (g/mL)	Theoretical C_s (mg QE/g DM)	k (g DM·mg QE^{-1}·min^{-1})	Experimental C_s (mg QE/g DM)	RMSE	R^2
MAC	1/60	6.61	7.300 10^{-2}	6.21	0.104	0.997
ULT	1/60	9.18	8.276 10^{-2}	8.88	0.048	0.999
MAC	1/80	5.51	5.288 10^{-2}	5.07	0.120	0.994
ULT	1/80	6.93	6.188 10^{-2}	6.49	0.076	0.998
MAC	1/120	3.78	4.743 10^{-2}	3.29	0.094	0.991
ULT	1/120	5.35	5.624 10^{-2}	4.94	0.086	0.996

CONCLUSION

This study confirmed the superiority of ultrasound-assisted extraction (ULT) over conventional maceration (MAC) for recovering phenolic compounds, including flavonoids, from *Ficus exasperata* leaves. Both extraction rate (k) and maximum yield (C_s) were enhanced under ULT, with the highest values (351.5 mg GAE/g DM for phenolic compounds and 8.88 mg QE/g DM for flavonoids) obtained at a 1/60 g/mL solid-to-liquid ratio.

Beyond these results, the findings highlight ULT as a green, energy-efficient, and cost-effective technique, with potential to reduce solvent consumption and processing time, thereby lowering environmental impact while improving industrial productivity. The valorization of *F. exasperata*,

a plant long used in African ethnomedicine, offers promising opportunities for applications in nutraceuticals, functional foods, and pharmaceutical formulations.

Future work should focus on optimizing ultrasound parameters such as frequency, power, and extraction time; extending the approach to other medicinal or aromatic plant matrices; and evaluating the antioxidant and therapeutic bioactivity of the obtained extracts to confirm their functional potential in real-world applications.

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Conflict of Interest

The authors confirm that no conflicts of interest are associated with the publication of this article.

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