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Ultrasonic extraction of phenolic compounds from *Ficus Lutea* trunk bark: Impact of solid–liquid ratio

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Abstract

This study examines the ultrasonic recovery of phenolic compounds from *Ficus lutea* trunk bark, with a particular emphasis on how the solid-to-liquid ratio impacts extraction efficiency. Using ultrasound-assisted extraction (UAE), experiments were conducted under controlled conditions at an extraction temperature of 40 °C, ultrasonic power of 240 kW, and frequency of 40 kHz. The experimental setup varied the solid-to-liquid ratios of 1/120, 1/80, and 1/60 g/mL. Phenolic content was measured as milligram gallic acid equivalents (mg GAE) per gram dry weight (g DW). Results showed that optimal ultrasonic cavitation at the specified temperature and power settings led to improved cell disruption and diffusion, thereby enhancing the release of phenolic compounds. The highest recovery was achieved with a solid-liquid ratio of 1/60 g/mL, yielding a polyphenol content of 385±3 mg GAE/g DW. The second-order kinetic model was applied to study the extraction kinetics and was found to be highly suitable for the process, with an $R^2 > 0.99$ and RMSE < 2.14 10^{-3}. In summary, the findings underscore the significance of the solid-liquid ratio in the extraction process, offering a foundation for further refinement and scale-up in industrial applications. The environmental benefits of the ultrasonic method, including reduced solvent usage and energy consumption, also promote sustainability.

Keywords: *Ficus lutea;* Ultrasonic Extraction; Phenolic Compounds; Extraction Kinetics; Second Order Model

1. Introduction

Extraction of phenolic compounds from plant matrices is of significant interest due to the potent antioxidant, antimicrobial, and anti-inflammatory properties of these bioactive molecules [1]. Phenolic compounds contribute to the health-promoting potential of natural products and have wide applications in the food, pharmaceutical, and cosmetic industries [2]. Among the various extraction technologies available, ultrasound-assisted extraction (UAE) has emerged as a promising technique, known for its operational simplicity, reduced extraction time, and enhanced yield compared to conventional methods [3]. UAE capitalizes on acoustic cavitation, where the rapid formation and collapse of microbubbles generate localized high temperatures and pressures, thereby enhancing mass transfer between the plant matrix and solvent [4]. Recent studies have reported that the efficiency of UAE is influenced not only by the ultrasonic parameters including frequency, extraction time, and temperature but also by the physicochemical interactions dictated by the solid-liquid ratio [3]. For instance, investigations by Bin Mokaizh et al., [5] and Ez Zoubi et al., [6] have demonstrated that an optimized solid-liquid ratio can lead to improved solvent penetration and cavitation effects, consequently maximizing the recovery of phenolic compounds. The present study addresses the ultrasonic recovery of

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phenolics from *Ficus lutea* trunk bark, a relatively underexplored botanical source, by focusing on the impact of the solid-liquid ratio.

Despite the advantages of UAE, the optimization of extraction conditions remains challenging due to the multifactorial nature of the process. In particular, the solid-liquid ratio plays a pivotal role in dictating the solvent's access to intracellular contents, affecting both solute solubility and diffusivity [7]. Literature on ultrasonic extraction from various plant materials such as olive leaves [8], grape skins [9], and pomegranate peels [10] illustrates that the favorable range of solid–liquid ratios vary significantly based on matrix composition. However, a comprehensive understanding of how the solid-liquid ratio modulates the UAE efficiency in *Ficus lutea* trunk bark remains important.

The primary objective of this research is to elucidate the effect of the solid-liquid ratio on the ultrasonic extraction efficiency of phenolic compounds from *Ficus lutea* trunk bark. We aim to determine the optimal ratio that maximizes recovery while preserving the integrity of the bioactive compounds. To achieve this, an attempt has been made to study the extraction kinetics by applying the second-order mathematical model in which the extraction parameters such as rate constant (k) and equilibrium concentration (C_0) have been calculated.

2. Material and methods

2.1. Materials

2.1.1. Plant material

F. lutea trunk barks were collected from Man (7° 24′ north, 7° 33′ west) in western Côte d'Ivoire. Thus, they were cleaned to remove impurities. After that, *F. lutea* trunk barks were air-dried in the shade at 33–37 °C for 12 days. The cleaned and dried barks were pulverized using a mortar and the powders were sieved. The fine powders were stored in dark, tightly sealed bottles for further analysis.

2.1.2. Chemicals and Reagents

Folin–Ciocalteau reagent (Sigma Aldrich, USA), gallic acid (Sigma Aldrich, USA), calcium carbonate (Na_2CO_3) (Fischer Chemical, France) and acetone (Carlo Erba, France) were used in this study.

2.2. Methods

2.2.1. Ultrasonic-assisted extraction (UAE)

Different exposures to solid-liquid ratios were examined using an ultrasonic bath with a rated output power of 240 kw and a frequency of 40 kHz (Model 1: PS-40, Mumbai, India). Specifically, three different solid-liquid ratios were used: 1/120, 1/80, and 1/60 g/mL in the acetone/water system (60/40, v/v). All treatments were maintained at 40 °C. The mixtures were transferred after 0, 5, 10, 15, 20, 25, 30, 35, and 40 min to a centrifuge and centrifuged at 3000 rpm for 10 min. Aliquots of the supernatants were collected for the evaluation of total phenolic content.

2.2.2. Determination of Total Phenolic Content (TPC)

The total phenolic contents of the supernatants of the hydroacetone extract at different solid/liquid ratios (120, 1/80 and 1/60 g/mL) of the trunk bark of *F. lutea* were estimated using Folin-Ciocalteu reagent, as previously described by Singleton and Rossi [11]. The calibration curve was plotted from the gallic acid standard solutions at 31.25, 50, 62.5, 100, 125, 200, 250, 400 and 500 µg/mL, added to 500 µL of Folin-Ciocalteu reagent diluted 10 times and 500 µL of a saturated Na_2CO_3 solution. The samples were incubated for 40 min and the absorbance was read after at 765 nm using a UV-Vis spectrophotometer (ONDA spectrophotometer, UV-30SCAN, China). For the supernatants of the hydroacetone extract, 100 µL was added to the same reagents as previously performed for the calibration curve. TPC was expressed as milligrams of gallic acid equivalent per gram of dry weight (mg GAE/g DW).

2.2.3. Second-order kinetic model

The extraction kinetics of phenolic compounds by sonication was studied by fitting the obtained experimental data to the equation of the Second-order kinetic model.

The model was used in the study of the extraction kinetics of polyphenols and anthocyanins from saffron floral bio-residues [12], phenolic compounds from oak chips into model wine [13].

The equation for Second-order kinetic model is:

$$C_t = \frac{C_0^2 kt}{1 + C_0 kt}$$

where C_t is the phenolic content at time t during extraction, C_0 is the phenolic content at saturation, and k is a constant related to the rate constant of the second-order model.

2.2.4. Statistical Analysis

The adequacy of the fitting of the predicted data to the experimental data was analyzed were calculated by the statistical software R Studio (version 2024.12.0) using the correlation coefficient (R^2) and the root mean square error (RMSE), defined by the following expressions:

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

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

where N is the number of experimental data points. q_{cal} and q_{exp} are the estimated and experimental values, respectively. A higher value of R^2 and a lower value of RMSE indicates a better quality of fit between the experimental and predicted data.

3. Results and discussion

3.1. Effect of S/L ratio on TPC

In **Figure 1**, three S/L ratios (1/120, 1/80, and 1/60 g/mL) were examined while the extraction temperature was set at 40 °C, the extraction solvent was acetone/water (60/40, v/v), the sampling times were every 5 min up to 40 min, and other parameters such as ultrasonic power (240 kW) and frequency (40 kHz) were kept constant.

For all ratios, it was observed that the TPCs increased rapidly at the beginning of the extraction process (between 5 and 10 min) and then tended to constant values as they approached equilibrium. The phenomenon can be clarified by Fick's second law of diffusion, uncovering that last equilibrium will be achieved between the solution concentrations within the solid matrix and solvent after a specific duration [14].

Furthermore, the TPCs showed an increasing trend as the S/L ratios increased, showing that the S/L ratios significantly influenced the TPCs. At equilibrium, the highest TPC (385±3 mg/g DW) was obtained with the S/L ratio of 1/60 g/mL, while the S/L ratio of 1/120 g/mL produced 251±7 mg/DW as the lowest TPC. The increase in the S/L ratio corresponds to a higher quantity of the extraction solvent which results in an easier and faster diffusion of bioactive compounds, including phenolic compounds [15]. In other words, the solvent penetrates more easily inside the cell and facilitates the extraction of polyphenols [16]. Many authors have previously reported a strong positive correlation between S/L ratio and TPC.

Vo et al., [17] also investigated the impact of S/L ratio on the efficiency of ultrasound-assisted extraction of phenolic compounds under fixed conditions: water content (20%), ultrasound power (300 W) and temperature (30 °C) for 5 minutes as exposure time. The results illustrated an improvement in the extraction yield of phenolic compounds when the S/L ratio was increased from 10 to 80 g/mL.

Another author demonstrated that solid/liquid ratio, temperature and water content in deep natural eutectic solvents greatly influenced the TPC and antioxidant activity from olive pomace [18].

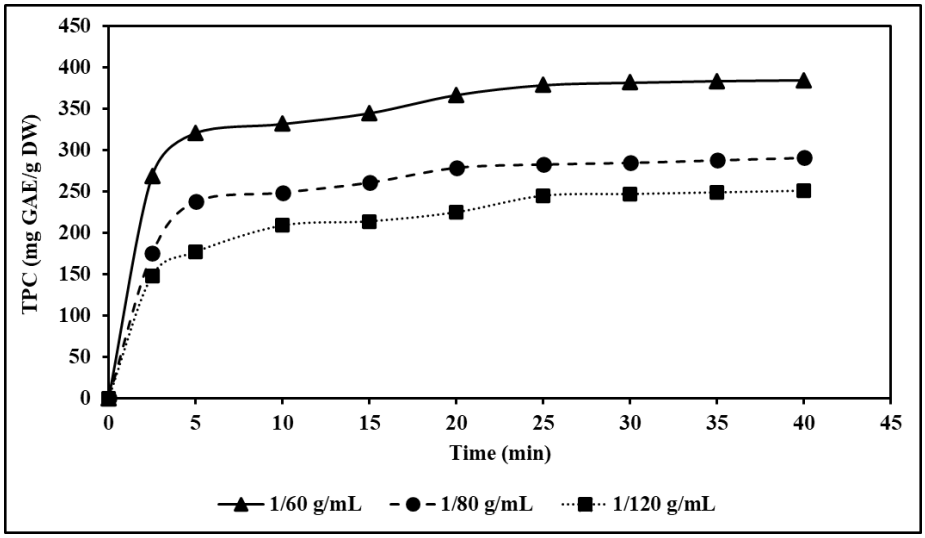

Figure 1 Effect of solid/liquid ratio on TPC by ultrasound

3.2. Second-order kinetic model

Experimental TPCs obtained by ultrasound were analyzed using a second-order kinetic model for a better understanding of the diffusion and mass transfer phenomena of phenolic compounds from the trunk bark of *F. lutea*.

Figure 2 shows the TPCs obtained by ultrasound as a function of time using the S/L ratios 1/120, 1/80 and 1/60 g/mL with the linearized form of the second-order model.

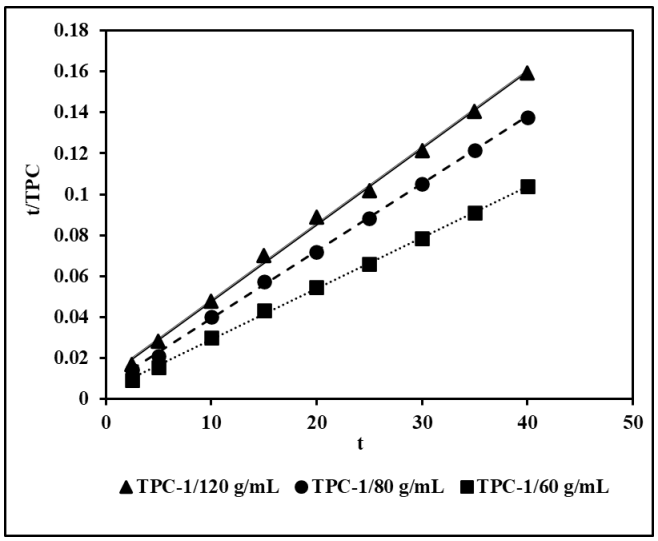

Figure 2 Second-order linear model for TPC using different solid/liquid ratios

The plot of t/C_t versus t provides the values of the rate constant k and the saturation concentration C_0. A lower value of k and a higher value of C_0 suggest a faster extraction and a higher equilibrium concentration, respectively. The rate constant k, experimental and theoretical equilibrium concentrations, and statistical parameters R^2 and RMSE are presented in **Table 1**.

R^2 and RMSE were calculated to assess the goodness of fit. A good fit is indicated by a high R^2 value and a low RMSE value. The R^2 values presented were greater than 0.99, and the RMSE values were less than 2.13×10^{-3}, demonstrating a better fit to the second-order model.

These results confirm that the second-order model accurately predicts the optimal conditions required to achieve high extraction yields of phenolic compounds based on the solid-to-liquid ratios used for extracts from the trunk bark of *F. lutea* (**Figures 3-5**).

Table 1 Kinetic parameters of the second-order model and equilibrium and predicted contents at different solid/liquid ratios

Parameters	**k (g DW/mg GAE.min)**	**Experimental TPC (mg GAE/g DW)**	**Predicted TPC (mg GAE/g DW)**	**R^2**	**RMSE**
S/L ratios (g/mL)					
1/120	$1.356\ 10^{-3}$	251 ± 7	267.379	0.998	$2.136\ 10^{-3}$
1/80	$1.742\ 10^{-3}$	291 ± 6	303.593	0.995	$9.308\ 10^{-4}$
1/60	$1.519\ 10^{-3}$	385 ± 3	400.605	0991	$9.307\ 10^{-4}$

Furthermore, it can be seen from **Figure 6** that the TPC increases from 251±7 to 385±3 mg GAE/g DW when increasing from an S/L ratio of 1/120 to 1/60 g/mL.

These observations align well with those of Casazza et al., [19], who reported a 31% increase in the extraction yield of polyphenols from grape seeds when the solid-to-liquid (S/L) ratio was varied from 0.30 to 0.20 g dw/mL, and a 12% increase when the ratio was further reduced from 0.20 to 0.10 g DW/mL.

Additionally, Nhut Pham et al., [20] demonstrated that the anthocyanin content extracted from Vietnam Hibiscus sabdariffa L increased from 87.47 mg/L to 158.56 mg/L when the liquid-to-solid ratio increased from 2:1 to 8:1, but decreased significantly when the ratio was increased further from 8:1 to 10:1.

Rajha et al., [21] studied the influence of the S/L ratio and other parameters on the optimization of polyphenol extraction yield from grape by-products. In contrast, their study found that TPC values decreased when the solid/liquid ratio increased from 0.16 to 0.43 g/mL.

Indeed, the ratio between the mass of the solid raw material (usually the plant matrix) and that of the liquid solvent is a critical parameter that directly influences the kinetics and thermodynamics of the extraction process [16,22].

Furthermore, the rate constant increased from 1.356×10^{-3} to 1.742×10^{-3} g DW/mg GAE.min before decreasing to 1.519×10^{-3} g DW/mg GAE.min (**Figure 7**).

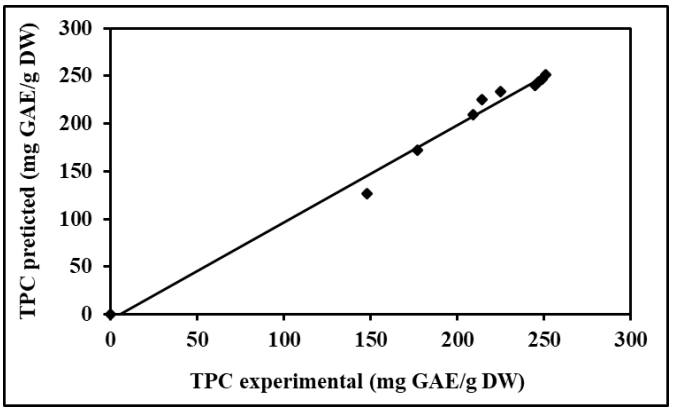

Figure 3 Correlation between experimental and predicted TPC values using the second-order model for the solid/liquid ratio 1/120

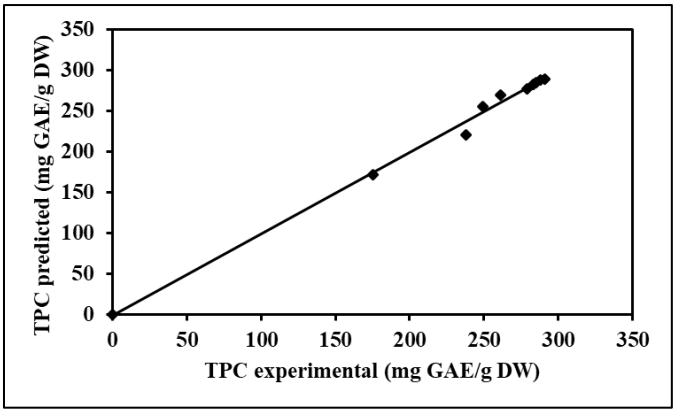

Figure 4 Correlation between experimental and predicted TPC values using the second-order model for the solid/liquid ratio 1/80

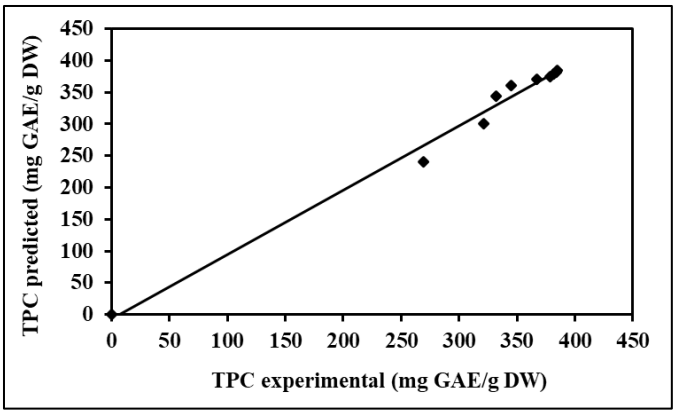

Figure 5 Correlation between experimental and predicted TPC values using the second-order model for the solid/liquid ratio 1/60

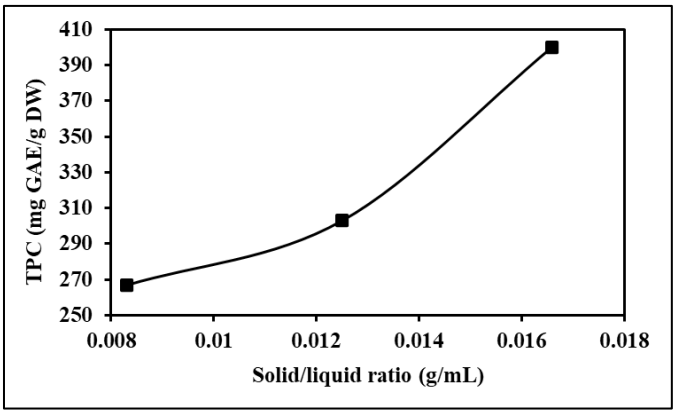

Figure 6 Extractable phenols (TPC) as a function of solid/liquid ratio

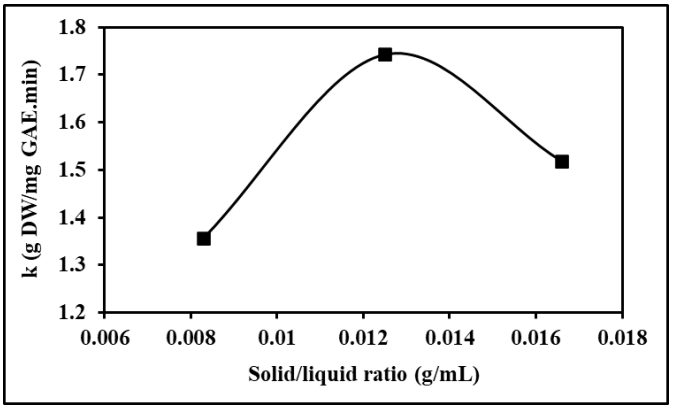

Figure 7 Rate constant k as a function of solid/liquid ratio

4. Conclusion

The ultrasonic extraction of phenolic compounds from *Ficus lutea* trunk bark highlights the importance of process optimization in achieving high extraction yields. By systematically varying experimental conditions, particularly the solid-to-liquid ratio, it was observed that the phenolic compound yield is significantly influenced by the liquid medium available for mass transfer. Optimal conditions, including a solid-to-liquid ratio of 1/60 g/mL, resulted in a yield of 385 ± 3 mg GAE/g dry weight. This outcome compares favorably with previously published optimization studies on ultrasonic-assisted extraction processes, validating the methodology's value in refining parameters. The influence of the solid-to-liquid ratio was especially pronounced: lower ratios did not provide sufficient solvent penetration, leading to suboptimal cavitation and limited phenolic release. Additionally, the study emphasizes the environmental benefits of ultrasonic extraction. By reducing the need for harsh solvents and minimizing energy consumption, this method offers a more sustainable and environmentally friendly alternative to conventional extraction techniques. Future research could focus on scaling up the process and applying similar optimization protocols to other plant matrices, thereby expanding the application potential of ultrasonic-assisted extraction in the field of natural product recovery.

Compliance with ethical standards

Acknowledgements

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Disclosure of Conflict of interest

No conflicts of interest to be disclosed.

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