COMMUNICATIONS IN SCIENCE AND TECHNOLOGY

Homepage: cst.kipmi.or.id



Optimization of ultrasound assisted extraction of sappan (*Caesalpinia sappan* L) wood for preparation of high quality extract

Mohammad Djaeni^{a,*}, Setia Budi Sasongko^a, Devi Yuni Susanti^b, F Widhi Mahatmanti^c, Andri Cahyo Kumoro^a, Laeli Kurniasari^d

^a Department of Chemical Engineering, Universitas Diponegoro, Semarang 50275, Indonesia
^b Department of Agriculture and Biosystem Engineering, Universitas Gadjah Mada, Yogyakarta 55281, Indonesia
^c Department of Chemistry, Universitas Negeri Semarang, Semarang 50229, Indonesia
^d Department of Chemical Engineering, Universitas Wahid Hasyim, Semarang 50222, Indonesia

Article history:

Received: 18 April 2025 / Received in revised form: 24 June 2025 / Accepted: 26 June 2025

Abstract

This study optimized the Ultrasound-Assisted Extraction (UAE) of sappan wood (*Caesalpinia sappan L.*) using Central Composite Design-Response Surface Methodology (CCD-RSM) and investigated its kinetics. Temperature, solvent-to-solid ratio, and extraction time were selected as independent variables with extract yield as the response. Analysis of Variance (ANOVA) showed that the solvent-to-solid ratio significantly affected yield. Optimal extraction conditions were 69.9°C, 29.9 mL/g, and 20.2 min, producing approximately yield of 0,293 mg GAE/g sample. High Performance Liquid Chromatography (HPLC) confirmed the presence of brazilin, while Fourier Transform InfraRed (FTIR) analysis indicated the retention of functional groups. UAE was shown to enhance extraction efficiency and preserve phenolic compounds. Additionally, the extraction process was modeled, resulting in a validated effective diffusivity (De) of 1.8 × 10⁻⁷ cm²/s, The kinetic study was useful in industrial application especially to determine appropriate extraction time.

Keywords: Kinetic study; optimization; sappan wood; ultrasound assisted extraction; response surface methodology (RSM)

1. Introduction

In this modern era, the tendency of human to use natural ingredients is increasing due to their growing awareness of health and the prevention of degenerative diseases. The demand for natural products, particularly those from plants with high antioxidant activity, continues to rise. Among these, phenolic compounds as plant secondary metabolites has been well recognized for their antioxidant activities, mainly in terms of their hydroxyl groups and conjugated double bonds [1,2]. In turn, the extraction of phenolic compounds from plants is seen as a promising alternative to synthetic antioxidants.

Sappan (*Caesalpinia sappan L*.) is a well-known medicinal plant growing widely in Southeast Asia, India, Sri Lanka, Vietnam, and parts of Latin America. Traditionally, the stem of sappan wood is used as a natural remedy and dye. It contains various bioactive compounds—such as flavonoids, steroids, and triterpenoids—with antioxidant, antimicrobial, antifungal, and anti-inflammatory effects [3–5]. Some of key compounds include brazilin, protosappanin, sappanchalcone, and

* Corresponding author Email: moh.djaeni@live.undip.ac.id https://doi.org/10.21924/cst.10.1.2025.1710 hematoxylin. To maximize its potential, a comprehensive research is required on the extraction and optimization of its phenolic content [6].

Common extraction methods such as maceration and Soxhlet are the conventional techniques with some limitations, such as long extraction time, more solvent and energy needed, and potential degradation of active compounds [7–9]. More efficient methods are essential then. Ultrasound-Assisted Extraction (UAE) is a promising alternative of extraction process related to its shorter processing time, lower solvent use, and low-temperature operation [10,11]. These conditions can reduce the energy use and help to preserve compound stability, positioning UAE as a green extraction technique [12].

UAE enhances extraction through cavitation, forming vapor bubbles that collapse and generate microjets, disrupting plant cell structures and facilitating compound release [13–15]. Mechanisms involved consist of fragmentation, erosion, sonocapillarity, detexturation, and sonoporation or combination of these processes [16]. UAE has been successfully applied in extracting phenolics from various sources, such as blackcurrant [17], *Syzygium cumini* leaves [18], cereal bran [19], jujube [20], black jamun [12], dragon fruit peel [2], and beetroot waste [14].



Besides method selection, optimizing extraction parameters and studying kinetics are vital for improving efficiency and scalability. Key variables affecting extraction include solvent concentration, time, temperature, solid-to-solvent ratio, particle size and ultrasound power [21,22]. Since these variables often interact, Response Surface Methodology (RSM) is frequently used to model and optimize the process. RSM has been applied in extracting phenolics from eggplant peel [23], blueberry pomace [22], *Opuntia ficus-indica* (OFI) flowers [24], hog plum pulp [10], turkey berry [21], mulberry leaves [11] and coconut mesocarp [25].

Understanding the extraction profile over time, which is essential for industrial application and process design, can be achieved through kinetic studies [12]. Here, empirical kinetic models such as pseudo-first-order, second-order, power-law and Peleg's are commonly used [18,19,26,27]. Alternatively, mass balance-based models using Fick's Law allow for the determination of effective diffusivity and a better understanding of solute transfer both in solvent and matrix.

Despite the fact that sappan wood is known for its phenolic content, studies on UAE optimization and kinetic modeling using mass balance, so far, are limited still. Previous works have focused on the optimization of enzyme-assisted extraction using RSM Box-Behnken [28] and empirical kinetic models [29–31], but little research applied a mass transfer approach. This study, in turn, aims to optimize the UAE process for sappan wood using RSM and to model the extraction kinetics using mass balance principles and Fick's Law.

2. Materials and Methods

2.1. Materials

Sappan wood used in this study was sourced from community-grown plants in Ungaran, Central Java, Indonesia. Ethanol was obtained from PT Bratachem Indonesia, and distilled water (aquadest) was supplied by UD. Mitra Jaya Indonesia. Analytical-grade reagents used for analysis included brazilin standard, Folin-Ciocalteu reagent, gallic acid (Sigma Aldrich), and sodium carbonate and methanol (Merck).

2.2. Extraction process for optimization

Fig 1 illustrates the schematic process of sappan wood extraction. The heartwood of sappan was first ground into fine particles that passed through a 20-mesh sieve. Subsequently, a total of 20 g of the powdered wood was added with a 60% ethanol solution and extracted by means of ultrasonic extractor (probe type of Ultrasonic Cell Disruptor TUE-500, operating at 22 kHz with a 12 mm probe diameter). The selection of 60% ethanol as the solvent was based on the findings from previous study [31]. The extraction process examined three independent variables: extraction temperature, extraction time, and solventto-solid ratio, all of which were configured in accordance to the experimental design as shown in Table 1. Following extraction, the solvent was removed using a rotary evaporator (Heidolph, Hei-VAP Advantage) to obtain extract, which was then weighed. The resulting extract was stored in a sealed container at 4°C until further analysis.

2.3. Analysis

2.3.1. Extraction yield

Extraction yield was counted by comparing the mass of extract with sample used and counted using Eq. (1).

$$yield = \frac{mass\ of\ extract\ gained}{mass\ of\ raw\ material\ used}\ x100\% \tag{1}$$

2.3.2. Total phenolic content (TPC) of extract

The analysis of Total Phenolic Content (TPC) was carried out by following the Folin-Ciocalteu method [32] with gallic acid as the standard. A total of 0.1 g of extract was dissolved in methanol to a final volume of 10mL. From this solution, 0.2mL was taken and mixed with distilled water and 1mL of Folin-Ciocalteu reagent. After shaking, 3 mL of 20% Na₂CO₃ solution was added. The mixture was then incubated at room temperature for 2 hr. Absorbance was measured at 765 nm using a UV-Vis spectrophotometer (Genesis 10S). The TPC values used for the optimization study were expressed in mg GAE/g of sample, calculated based on the TPC result multiplied by the extract yield and divided by the mass of the dry sample.

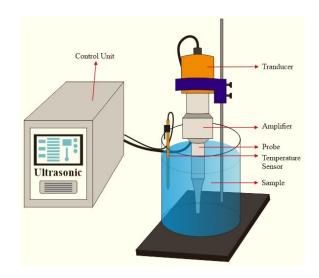


Fig. 1. Schematic diagram of ultrasound-assisted extraction

2.3.3. FTIR and HPLC analysis

To investigate the functional groups present in sappan wood samples and the extract, Fourier Transform InfraRed (FTIR) spectroscopy was employed. The analysis was conducted using a Perkin Elmer FTIR spectrometer within the wavelength range of 400–4000 nm. High Performance Liquid Chromatography (HPLC) was performed to validate the determination of brazilin content in the extract following the method as described by [33].

2.4. Design of response surface methodology (RSM)

The optimization of extraction process was carried out using Central Composite Design (CCD)-based RSM. Three

independent variables here were chosen: extraction temperature; solvent-to-solid ratio; and extraction time with response of the TPC of extract (mg GAE/g sample) (Table 1). There were twenty run of experiment with centered point (55°C, 20 mL/g, 30 min) repeated six times. Statistical analysis and 3D graph of the model were obtained using Design Expert V13 software.

Table 1. Experimental design of CCD

Factors	Low Level	Center	High Level
A: extraction temperature (°C)	40	55	70
B: solvent-to-solid ratio (mL/g)	10	20	30
C: extraction time (min)	20	30	40

2.5. Kinetic modeling

The extraction of sappan wood using ethanol was considered as thin slab's solid-liquid extraction. In this process, there were two important steps. The first step was diffusion from solid to the solid's interface and the second one was mass transfer from the interface of solid to the liquid [34]. The diffusion of solute in solid followed Fick Law as presented in Eq. (2).

$$N_A = -D_e \cdot \frac{\partial C_A}{\partial x} \tag{2}$$

where N_A is solute diffusion in solid (g solute/sec/cm²), C_A is the amount of solute in solid (g solute/cm³ solid), x is the position (cm) and De is the effective diffusion of solute in solid (cm²/sec). Mass balance of solute in volume is written according to Eq. (3).

$$\left[-D_e A \frac{\partial C_A}{\partial x} | x \right] - \left[-D_e A \frac{\partial C_A}{\partial x} | x + \Delta x \right] = A. \Delta x. \frac{\partial C_A}{\partial t}$$
 (3)

where A is slab's area (cm²) and t is time (sec). Taking limit Δx closes to 0, we then could get Eq. (4).

$$\frac{\partial^2 C_A}{\partial x^2} = \frac{1}{D_e} \frac{\partial C_A}{\partial t} \tag{4}$$

The boundary conditions follow Eq. (5)-(7).

$$C_A(x,0) = C_{A0} \tag{5}$$

$$C_A(0,t) = C_f/H (6)$$

$$C_A(L,t) = C_f/H \tag{7}$$

Where C_{A0} was initial solute in solid (g solute initial/cm³ solid), C_f was solute amount in solvent (g solute/g solvent) and H was Henry's constant. Material balance of solute in solid and liquid can be written using Eq. (8).

$$W. C_{f0} + n. A. L. C_{A0} = W. C_f + n. A \int_0^L C_A. dx$$
 (8)

Where W was the amount of solvent (g), n was amount of slab and L was slab's width (cm). The C_f then can be calculated using Eq. (9).

$$C_f = C_{fo} + \frac{n.A}{W} \left(L. C_{A0} - \int_0^L C_A. dx \right)$$
 (9)

The value of diffusivity and Henry's constant was calculated using Microsoft Excel through trial and error method in order that minimum Sum of Square Error (SSE) as in Eq. (10) can be reached. The mathematical model was solved using Finite Different Approximation that convert partial differential equation into algebraic equation [35].

$$SSE = \left(A_{extracted\ data} - A_{extracted\ calc}\right)^2 \tag{10}$$

Twenty grams of sappan wood was extracted using 300 ml ethanol 60% for 5, 10, 15, 20, 25, 30, 40 and 50 and 60 min. Temperature was kept at 50°C and after extraction, solvent was separated using rotary evaporator and amount of extract was weighed.

3. Results and Discussion

3.1. Optimization of extraction process

The results of extraction process along with variable data are presented in Table 2. The extraction result data were phenolic content (TPC) of extract, expressed in mg GAE/g sample. From Table 2 it can be seen that the range of TPC obtained was between 0.035 mg GAE/g sample to 0.292 mg GAE/g sample. The regression equation suggested was stated in Eq. (11).

Coefficient with positive sign in the model showed that variable could increase the response, while minus sign showed that variable could lower the response [10]. From Eq. (11), it can be concluded that both extraction temperature and solventto-solid ratio in linear form could increase the response of TPC, while time reduced the response. Moreover, solvent-to-solid ratio affected the response of TPC more since the coefficient was higher than extraction temperature. A similar result was also found in the ultrasound-assisted extraction of phenolic from Empetrum nigrum where solvent-to-solid ratio had the most significant effect [15]. While the extraction of turkey berry fruits found that extraction temperature, solvent-to-solid ratio and ethanol concentration gave significant effect on the TPC of extract, time was insignificant [21]. The interaction of extraction temperature with solvent-to-solid ratio and temperature with time increased the TPC response, while solvent-to-solid ratio and time's interaction tended to give a negative impact.

Table 3 presents the tabulation of the ANOVA results. Based on the data, the proposed model was statistically significant at a 95% confidence level (p-value = 0.0007), indicating only a 0.07% chance that the F-value occurred due to random noise. The lack of fit had a p-value of 0.6654 (>0.05),

showing it insignificant compared to the pure error, suggesting that the regression model closely matched the experimental data [11]. A significant model combined with an insignificant lack of fit confirmed the model's reliability. The high coefficient of determination ($R^2 = 0.9779$) indicated the strong predictive accuracy, while the adjusted R2 confirmed consistency between predicted and actual values. An adjusted R² above 0.9 further supported model adequacy, and the small gap between predicted and adjusted R² (<0.2) highlighted its reliability [10]. The model also achieved an adequate precision of 19.8571 above the minimum desirable value of 4 [22], confirming a strong signal-to-noise ratio. Meanwhile, significant factors identified were the solvent-to-solid ratio (B), its interaction with time (BC), and its quadratic term (B2), all with p-values below 0.05. The ranked positive effects of variables and their interactions (from Equation 11) were: solvent-to-solid ratio (B) > temperature × solvent-to-solid ratio × solvent-to-solid ratio (AB²) > temperature (A) > temperature² (A^2) > temperature × solvent-to-solid ratio (AB) > temperature \times time (AC).

Table 2. Result of experiment

Run	Temperature (°C)	Solvent- to-solid ratio (mL/g)	Time (min)	Response (mg GAE/g sample)	
				measured	predicted
1	70	30	40	0.219	0.221
2	55	20	46.8	0.162	0.160
3	70	30	20	0.292	0.294
4	55	36.8	30	0.244	0.239
5	55	20	30	0.168	0.166
6	55	20	30	0.188	0.166
7	70	10	40	0.149	0.151
8	55	20	30	0.160	0.166
9	40	10	40	0.085	0.087
10	70	10	20	0.113	0.115
11	55	20	30	0.181	0.166
12	40	30	40	0.138	0.140
13	29.8	20	30	0.166	0.163
14	55	20	30	0.159	0.166
15	40	10	20	0.075	0.077
16	55	3.2	30	0.035	0.041
17	55	20	30	0.141	0.166
18	55	20	13.2	0.171	0.169
19	40	30	20	0.218	0.220
20	80.2	20	30	0.215	0.209

 $Y = 0.1663+0.0146A+0.0621B-\\ 0.0027C+0.0066AB+0.0041AC-\\ 0.0249BC+0.0077A^2-0.0103B^2-0.0008C^2-\\ 0.0024ABC-0.0065A^2B-0.0107A^2C+0.0176AB^2$

(11)

Table 3. ANOVA analysis

Source	SS	df	Mean Square	F-value	p-value	
Model	0.0657	13	0.0051	20.44	0.0007	significant
A-	0.0012	1	0.0012	4.86	0.0697	
temperature						
B-solvent- to-solid ratio	0.0218	1	0.0218	88.37	<0.0001	
C-time	0.0000	1	0.0000	0.1639	0.6997	
AB	0.0004	1	0.0004	1.42	0.2783	
AC	0.0001	1	0.0001	0.5508	0.4860	
BC	0.0050	1	0.0050	20.03	0.0042	
\mathbf{A}^2	0.0009	1	0.0009	3.45	0.1128	
\mathbf{B}^2	0.0015	1	0.0015	6.24	0.0467	
\mathbb{C}^2	9.15E-	1	9.15E-	0.0370	0.8538	
	06		06			
ABC	0.0000	1	0.0000	0.1826	0.6841	
A^2B	0.0001	1	0.0001	0.5684	0.4794	
A^2C	0.0004	1	0.0004	1.53	0.2616	
AB^2	0.0010	1	0.0010	4.13	0.0883	
Residual	0.0015	6	0.0002			
Lack of Fit	0.0001	1	0.0001	0.2109	0.6654	not
						significant
Pure error	0.0014	5	0.0003			
Cor Total	0.0671	19				
Std Dev	0.0157			\mathbb{R}^2	0.9779	
Mean	0.1639			Adjusted R ²	0.9301	
C.V.%	9.59			Predicted R ²	0.7725	
				Adeq precision	19.8571	

Fig. 2 shows the 3D surface of the result and the interaction effects of the variables in extracting TPC from sappan wood. The contour plot in each picture used two variables, while another variable kept constant at center point. Fig. 2(a) shows the effect of extraction temperature and solvent-to-solid ratio on the TPC of extract at the constant time of 30 min. The increase of temperature and solvent-to-solid ratio increased the TPC of extract. Temperature is considered crucial in extracting phenolic compounds and several researches showed the increase of TPC along with the increasing extraction temperature [10]. Increasing temperature results in softening cell tissues, weakening interaction between polyphenol and polysaccharide, increasing solute solubility, decreasing surface tension and solvent viscosity and over increasing diffusion coefficient, rate of diffusion and mass

transfer increasing the quantity of phenolic extracted [10,11,36]. But elevated temperature may also harm the extraction process, since increasing temperature in UAE can enhance the vapor pressure of solvent, thus reducing cavitation force and lower the yield. The extraction of phenolic compound from OFI flower provided the highest TPC on temperature of 70°C. It is mentioned that temperature above this level can cause the degradation of extract and reduce the extraction rate [24], while optimum extraction temperature of phenolic compounds from hog plum using UAE was 52.03°C [10].

Fig. 2(b) presents the effect of temperature and time on the TPC extract at the constant solvent-to-solid ratio of 20 mL/g. The increase of time tended to decrease the yield and it showed that increasing both time and temperature decreased TPC slightly. Long extraction time causes degradation of phenolic since it can expose with temperature, air/oxygen and also light in longer time. Degradation also can occur because radiation generated by ultrasound or the phenolic can react with other components and the degradation decreases the TPC yield [10,24]. The extraction of OFI flower showed that, although TPC increased when extraction time was prolonged up to 40 min, but, in fact, there was no different statistical result from 20 to 50 min [24].

Fig. 2(c) showed the interaction effect between solvent-to-solid ratio and extraction time on the TPC of extract at the constant extraction temperature of 55°C. In general, an increase in the solvent-to-solid ratio at a fixed extraction time can enhance the TPC yield, particularly at shorter extraction durations. However, extending the extraction time at a high

solvent-to-solid ratio tends to reduce the TPC. A higher solvent-to-solid ratio is generally beneficial for extraction as it enhances the concentration gradient that acts as the driving force in mass transfer process. A greater concentration gradient improves the diffusion rate, thereby increasing the extraction efficiency [6]. Nevertheless, at very high solvent-to-solid ratios, the solubility of phenolic compounds reaches a saturation point, after which no further increase in yield is observed as equilibrium is achieved [21].

Based on the analysis, the optimal extraction conditions were identified at a temperature of 69.9°C, a solvent-to-solid ratio of 29.9 mL/g, and an extraction time of 20.2 min, which theoretically produced a TPC yield of 0.293 mg GAE/g sample. The actual TPC yield obtained from validation experiments under these conditions was 0.278 mg GAE/g sample. The optimum extraction temperature in this study was similar to that of reported by [24] but higher than that of observed by [10]. The optimal extraction temperature for phenolic compounds from hog plum was 52.03°C, but it required a longer extraction time of 30 min, compared to only 20.2 min in this study. In contrast, the extraction of phenolics from turkey berry fruits showed an optimum temperature of 80°C with an extraction time of 17.3 min and a solvent-to-solid ratio of 49.7 mL/g [21]. These conditions involved a higher temperature and solvent-tosolid ratio, while reducing the extraction time. This indicated the interactions among the variables, and optimizing one parameter (e.g. reducing time) may require compensatory adjustments in others (e.g. increasing temperature or solvent ratio).

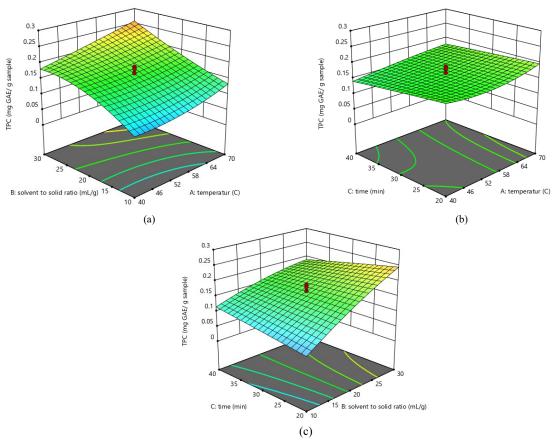


Fig. 2. 3D contour of (a) interaction of temperature and solvent-to-solid ratio, (b) interaction of temperature and time, and (c) interaction of solvent-to-solute ratio and time

3.2. HPLC and FTIR analysis

Fig. 3 shows the chromatogram of extract and brazilin standard. From this figure, brazilin was identified at retention time (Rt) of 2.063 min; this showed that the method used for the analysis was selective to detect the target. The result also showed that brazilin was successfully extracted using UAE and ethanol as solvent. The chromatogram found unknown components besides brazilin and this result is in accordance with [28, 33, 37]. Another peak shown in the chromatogram could be another component found in the extract since the extract was crude extract.

FTIR analysis was performed to identify the functional groups present in the material, with the spectra of both the raw sample and the extract as displayed in Fig. 4. The FTIR spectrum of sappan wood sample showed a distinct peak at 1040 cm⁻¹, indicating the presence of phenolic rings, suggesting that sappan wood is a rich source of phenolic compounds. Another peak was observed around 3200 cm⁻¹, corresponding to the hydroxyl (-OH) functional group of alcohols although this peak appeared less intense compared to the one at 1040 cm⁻¹. Several other peaks also existed in the sample, but their intensity were very low. After extraction process, the peaks could be identified more obvious. The emergence of these peaks was probably due to the extraction process that took specific component based on the method and solvent used, and left the unwanted compounds in the residue. The extract still had peaks at 1040 cm⁻¹ and 3200 cm⁻¹, while peaks at 1600 cm⁻¹ had higher intensity than that of in the sample and it referred to C=C bond [38]. Peak arounds 1400 cm⁻¹ refers to C=O group, indicating brazilin component as a result of brazilin's oxidation [5]. The transmittance of sample and extract were also different where extract transmittance was higher than sample one. In other words, the availability of extract component was higher than the sample. Then, it can be concluded that extraction process can concentrate the active compounds while keeping its molecular structure.

3.3. Kinetic model

Fig.5 presents the result of the kinetic study using the mass balance proposed. Fig. 5(a) shows the solute concentration profile in the slab at different time (t=0, 30 and 60 min), while Fig. 5(b) presents the solute concentration profile at various slab thicknesses over time. The extraction profile in Fig. 5(a) indicated a decrease in extract concentration over time with the central part of the slab having a higher concentration. The center of the slab naturally is the most difficult part to reach, and the extract diffusion occurs unidirectionally along the xaxis from the central part of the slab to the edges. Phenolic compounds, as secondary metabolite compounds, are located in the heartwood [39] - the innermost part of the wood and it is difficult for the solvent to reach. Furthermore, Fig. 5(b) shows a decrease in solute concentration along the thickness of the material. The farther distance from the center of the slab gave the greater decrease in concentration. This was because solvent penetration was still relatively high at the slab's edges, allowing for greater solute extraction. This then resulted in a higher decrease of solute concentration at the solid's edges. Meanwhile, the central part of the slab, being the innermost region, is more difficult for the solvent to access, leading to a relatively smaller decrease in solute concentration (as observed in the less significant concentration drop, and taking a longer time).

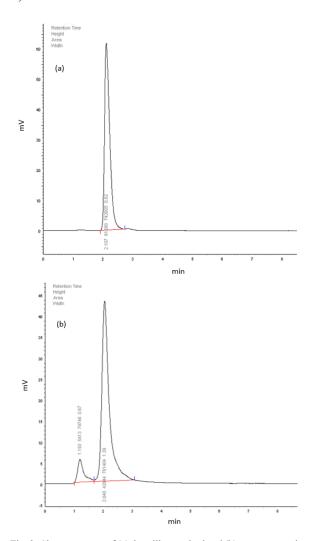


Fig. 3. Chromatogram of (a). brazilin standard and (b). sappan wood extract

Theoretically, extending the extraction time will continue to decrease the solute concentration in the solid until equilibrium concentration is reached. Initially, equilibrium concentration achieved at the solid's edges and then gradually at the center of the slab. However, excessively prolonged extraction times may potentially degrade the extracted phenolic compounds [10]. This kinetic study gave the diffusivity of 1,8x10⁻⁷ cm²/sec and Henry's constant of 0,5.

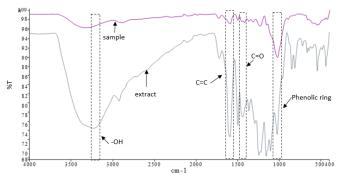


Fig. 4. FTIR spectra of (a). sappan wood sample, (b) sappan wood extract

The diffusivity value obtained in this research was lower than the De value reported for the extraction of *Moringa oleifera* leaves using microwave assisted extraction [40], as well as the extraction of myrcene from *Cymbopogon citratus* using microwave-assisted hydrotropic distillation [34]. The lower De value in this study was likely related to differences in the structure of the raw materials used. Commonly, leaves have a thin layer and a more fragile structure, which facilitates easier and faster solvent penetration. In contrast, sappan wood has a denser matrix with a well-organized and rigid structure [41]. However, the De value in this study was higher than that reported for the extraction of paclitaxel from *Taxus chinensis*

leaves [42]. This difference was likely attributed to the extraction method used; while paclitaxel was extracted using conventional methods, this study employed ultrasound-assisted extraction, which was able to accelerate the extraction process. Overall, the study of extraction kinetics can be used to predict concentration profiles in both solid and liquid phases, evaluate extraction efficiency, and determine the optimal time to terminate the process [35]. These parameters are highly valuable in industrial applications, particularly for designing extraction vessel volumes and determining appropriate extraction time.

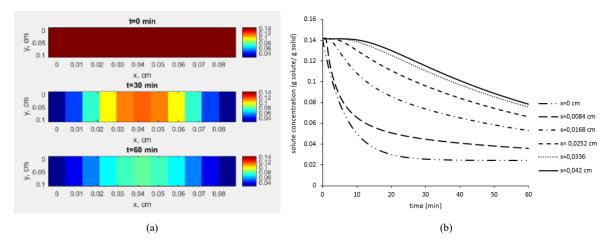


Fig. 5. (a) solute concentration profile in the slab at different time (t=0, 30 and 60 min), (b). solute concentration profile at various slab thickness over time

4. Conclusion

The optimization of UAE on sappan wood was carried out using RSM. Three independent variables were examined: extraction temperature, solvent-to-solid ratio and extraction time. Significant variables for this research included solventto-solid ratio, interaction solvent-to-solid ratio with time and solvent-to-solid ratio quadratic with p-value <0.05. The optimum extraction condition was temperature of 69.9°C, solvent-to-solid ratio of 29,9 mL/g and time 20.2 min with expected yield of 0,293 mg GAE/g sample. HPLC analysis showed the peak of brazilin as main phenolic compound in sappan wood, while FTIR spectra of extract showed several peaks corresponding to specific bonds in phenolic component. Kinetic study was arranged using mass balance concept and obtained the effective diffusion (De) of 1,8 x 10⁻⁷ cm²/sec with Henry constant of 0.5. The result showed that UAE could speed up extraction process and parameter of kinetic study is useful for predicting the extract profile over time, especially to determine appropriate extraction time.

Acknowledgements

Authors acknowledge to the Ministry of Education, Culture, Research and Technology Republic of Indonesia and Universitas Diponegoro for its financial funding through UNDIP's research program 2024

References

- A. V. González de Peredo, M. Vázquez-Espinosa, E. Espada-Bellido, M. Ferreiro-González, A. Amores-Arrocha, M. Palma, G.F. Barbero, A. Jiménez-Cantizano, Alternative Ultrasound-Assisted Method for the Extraction of the Bioactive Compounds Present in Myrtle (Myrtus communis L.), Molecules 24 (2019).
- G.V.S. Bhagya Raj, K.K. Dash, Ultrasound-assisted extraction of phytocompounds from dragon fruit peel: Optimization, kinetics and thermodynamic studies, Ultrason. Sonochem. 68 (2020) 105180.
- R. Srinivasan, G.G. selvam, S. Karthik, K. Mathivanan, R. Baskaran, M. Karthikeyan, M. Gopi, C. Govindasamy, *In vitro antimicrobial activity of Caesalpinia sappan L.*, Asian Pac. J. Trop. Biomed. 2 (2012) 136–139.
- 4. N.P. Nirmal, M.S. Rajput, R.G.S.V. Prasad, M. Ahmad, *Brazilin from Caesalpinia sappan heartwood and its pharmacological activities: A review*, Asian Pac. J. Trop. Med. 8 (2015) 421–430.
- L. Ngamwonglumlert, S. Devahastin, N. Chiewchan, G.S.V. Raghavan, Color and molecular structure alterations of brazilein extracted from Caesalpinia sappan L. under different pH and heating conditions, Sci. Rep. 10 (2020) 1–10.
- B.Y. Wong, C.P. Tan, C.W. Ho, Effect of solid-to-solvent ratio on phenolic content and antioxidant capacities of "Dukung Anak" (Phyllanthus niruri), Int. Food Res. J. 20 (2013) 325–330.
- J. Liao, Z. Guo, G. Yu, Process intensification and kinetic studies of ultrasound-assisted extraction of flavonoids from peanut shells, Ultrason. Sonochem. 76 (2021) 105661.
- I.M. Yusoff, Z. Mat Taher, Z. Rahmat, L.S. Chua, A review of ultrasoundassisted extraction for plant bioactive compounds: Phenolics, flavonoids, thymols, saponins and proteins, Food Res. Int. 157 (2022) 111268.

- R. Arbianti, Angelina, B. Suryapranata, L.P. Latifah, N.F. Putri, T.S. Utami, Y. Muharam, Slamet, Combined enzymatic and ultrasound-assisted aqueous two-phase extraction of antidiabetic flavonoid compounds from Strobilanthes crispus leaves, Commun. Sci. Technol. 8 (2023) 113–123.
- T. Ahmed, M.R. Rana, M.R. Maisha, A.S.M. Sayem, M. Rahman, R. Ara, Optimization of ultrasound-assisted extraction of phenolic content & antioxidant activity of hog plum (Spondias pinnata L. f. kurz) pulp by response surface methodology, Heliyon 8 (2022).
- 11. S. Insang, I. Kijpatanasilp, S. Jafari, K. Assatarakul, Ultrasound-assisted extraction of functional compound from mulberry (Morus alba L.) leaf using response surface methodology and effect of microencapsulation by spray drying on quality of optimized extract, Ultrason. Sonochem. 82 (2022) 105806.
- M. Sharma, K.K. Dash, Microwave and ultrasound assisted extraction of phytocompounds from black jamun pulp: Kinetic and thermodynamics characteristics, Innov. Food Sci. Emerg. Technol. 75 (2022) 102913.
- 13. I. Lavilla, C. Bendicho, Fundamentals of Ultrasound-Assisted Extraction, Elsevier Inc., 2017.
- G.S.N. Fernando, K. Wood, E.H. Papaioannou, L.J. Marshall, N.N. Sergeeva, C. Boesch, Application of an Ultrasound-Assisted Extraction Method to Recover Betalains and Polyphenols from Red Beetroot Waste, ACS Sustain. Chem. Eng. 9 (2021) 8736–8747.
- Y. Gao, S. Wang, S. Dang, S. Han, C. Yun, W. Wang, H. Wang, Optimized ultrasound-assisted extraction of total polyphenols from Empetrum nigrum and its bioactivities, J. Chromatogr. B 1173 (2021) 122699.
- F. Chemat, N. Rombaut, A.G. Sicaire, A. Meullemiestre, A.S. Fabiano-Tixier, M. Abert-Vian, *Ultrasound assisted extraction of food and natural* products. *Mechanisms, techniques, combinations, protocols and* applications. A review, Ultrason. Sonochem. 34 (2017) 540–560.
- M.J.A. González, C. Carrera, G.F. Barbero, M. Palma, A comparison study between ultrasound–assisted and enzyme–assisted extraction of anthocyanins from blackcurrant (Ribes nigrum L.), Food Chem. X 13 (2022).
- K. V. Mahindrakar, V.K. Rathod, Ultrasound-assisted intensified aqueous extraction of phenolics from waste Syzygium cumini leaves: Kinetic studies and evaluation of antioxidant, antidiabetic and anticancer potential, Food Biosci. 46 (2022) 101547.
- N. Milićević, P. Kojić, M. Sakač, A. Mišan, J. Kojić, C. Perussello, V. Banjac, M. Pojić, B. Tiwari, Kinetic modelling of ultrasound-assisted extraction of phenolics from cereal brans, Ultrason. Sonochem. 79 (2021).
- F. Berkani, M.L. Serralheiro, F. Dahmoune, A. Ressaissi, N. Kadri, H. Remini, Ultrasound assisted extraction of phenolic compounds from a jujube by-product with valuable bioactivities, Processes 8 (2020) 1–17.
- A. Weremfo, F. Adulley, K. Dabie, S. Abassah-Oppong, E. Peprah-Yamoah, Optimization of ultrasound-assisted extraction of phenolic antioxidants from turkey berry (Solanum torvum Sw) fruits using response surface methodology, J. Appl. Res. Med. Aromat. Plants 30 (2022) 100387.
- X. Zhang, S. Wang, Q. Wu, M. Battino, F. Giampieri, W. Bai, L. Tian, Recovering high value-added anthocyanins from blueberry pomace with ultrasound-assisted extraction, Food Chem. X 16 (2022) 100476.
- K. Philippi, N. Tsamandouras, S. Grigorakis, D.P. Makris, Ultrasound-Assisted Green Extraction of Eggplant Peel (Solanum melongena) Polyphenols Using Aqueous Mixtures of Glycerol and Ethanol: Optimisation and Kinetics, Environ. Process. 3 (2016) 369–386.
- 24. F. Brahmi, F. Blando, R. Sellami, S. Mehdi, L. De Bellis, C. Negro, H. Haddadi-Guemghar, K. Madani, L. Makhlouf-Boulekbache, Optimization of the conditions for ultrasound-assisted extraction of phenolic compounds from Opuntia ficus-indica [L.] Mill. flowers and comparison with conventional procedures, Ind. Crops Prod. 184 (2022) 114977.
- J. Yang, N. Li, C. Wang, T. Chang, H. Jiang, Ultrasound-homogenizationassisted extraction of polyphenols from coconut mesocarp: Optimization

- study, Ultrason. Sonochem. 78 (2021) 105739.
- N.A.A.R. Zahari, G.H. Chong, L.C. Abdullah, B.L. Chua, Ultrasonicassisted extraction (UAE) process on thymol concentration from Plectranthus amboinicus leaves: Kinetic modeling and optimization, Processes 8 (2020).
- H.S. Kusuma, P.D. Amelia, C. Admiralia, M. Mahfud, Kinetics study of oil extraction from Citrus auranticum L. By solvent-free microwave extraction, Commun. Sci. Technol. 1 (2016) 15–18.
- D. Septiani, H. Suryadi, A. Mun'im, Improving Enzyme-Assisted Extraction of Brazilin from Sappanwood (Caesalpinia Sappan L.) Extract by Fungal Cellulase, Pharmacogn. J. 14 (2022) 21–28.
- M. Djaeni, A.C. Kumoro, F.D. Utari, I.E. Septiani, Enhancement of The Sappanwood Extract Yield by Aqueous Ultrasound-Assisted Extraction Using Water Solvent, Int. J. Adv. Sci. Eng. Inf. Technol. 11 (2021) 1514– 1520.
- 30. L. Kurniasari, A.C. Kumoro, M. Djaeni, *Ultrasound-Assisted Extraction of Sappan Wood-Kinetic Modeling*, AIP Conf. Proc. 3250 (2025).
- L. Kurniasari, M. Djaeni, A.C. Kumoro, Ultrasound-Assisted Extraction (UAE) of sappan wood (Caesalpinia sappan L.): Effect of solvent concentration and kinetic studies, Brazilian J. Food Technol. 26 (2023) 1– 11.
- 32. I.S. Arsiningtyas, Antioxidant *Profile of Heartwood and Sapwood of Caesalpinia sappan L. Tree's Part Grown in Imogiri Nature Preserve, Yogyakarta*, IOP Conf. Ser. Earth Environ. Sci. 810 (2021).
- A.S. Sakti, F.C. Saputri, A. Mun'im, Optimization of choline chlorideglycerol based natural deep eutectic solvent for extraction bioactive substances from Cinnamomum burmannii barks and Caesalpinia sappan heartwoods, Heliyon 5 (2019) e02915.
- W.B. Sediawan, I. Hartati, H. Sulistyo, M.M. Azis, U. Al Rahma, Microwave Assisted Hydrotropic Distillation of Myrcene-Rich Essential Oil of Cymbopogon Citratus, Int. J. Technol. 14 (2023) 310–319.
- 35. D.Y. Susanti, W.B. Sediawan, M. Fahrurrozi, M. Hidayat, *A mechanistic model of mass transfer in the extraction of bioactive compounds from intact sorghum pericarp*, Processes 7 (2019).
- 36. B. Dulo, T. De Somer, M. Moyo, E. Nakyese, J. Githaiga, K. Raes, S. De Meester, Kinetic modeling of phenolic compounds extraction from nutshells: influence of particle size, temperature and solvent ratio, Biomass Convers. Biorefinery (2023) 23565–23579.
- S. Warinhomhaun, B. Sritularak, D. Charnvanich, A simple highperformance liquid chromatographic method for quantitative analysis of brazilin in caesalpinia sappan L. extracts, Thai J. Pharm. Sci. 42 (2018) 208-213
- A. Hasrudin, Z. Abidin, N. Hiedayati, V. Prajaputra, Hydrothermal Synthesis of Multi-Color Fluorescent Carbon Dots from Sappan Wood (Caesalpinia sappan) as an Acid-Base Indicator, Rev. Chim. 71 (2020) 106–112.
- R. Arisandi, S. Nugroho Marsoem, G. Lukmandaru, T. Ashitani, K. Takahashi, The contents of phenolics and cell wall component of eucalyptus pellita f. muell stemwood and bark, Wood Res. 64 (2019) 411–422.
- R. Albarri, İ. Toprakçı, E. Kurtulbaş, S. Şahin, Estimation of diffusion and mass transfer coefficients for the microwave-assisted extraction of bioactive substances from Moringa oleifera leaves, Biomass Convers. Biorefinery 13 (2023) 5125–5132.
- 41. T. Vij, P.P. Anil, R. Shams, K.K. Dash, R. Kalsi, V.K. Pandey, E. Harsányi, B. Kovács, A.M. Shaikh, *A Comprehensive Review on Bioactive Compounds Found in Caesalpinia sappan*, Molecules 28 (2023).
- Y.J. Jo, J.H. Kim, Effective Diffusivity and Mass Transfer Coefficient during the Extraction of Paclitaxel from Taxus chinensis Using Methanol, Biotechnol. Bioprocess Eng. 24 (2019) 818–823.