

# Optimization of Oil Extraction from Flamboyant (*Delonix regia*) Seeds: Kinetic and Thermodynamic Studies



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**ABSTRACT:** The increasing demand for seed oils globally have prompted the exploration of alternative sources like those of non-edible underexploited agricultural seeds. Flamboyant seeds (FS) are promising options, requiring further research on how to extract and use the oil effectively. This study investigates the optimization of oil extraction from FS through comprehensive kinetics and thermodynamics of the extraction process. The Flamboyant seed oil (FSO) was extracted using Soxhlet extractor and the effect of temperature, time, solvent composition, and solvent type on the oil yield was investigated. The FSO was also characterized for potential uses. The maximum yield of 8.383% was achieved with hexane-to-ethanol ratio of 1:0, at 65°C, and an extraction time of 150 min. Central composite design (CCD) of response surface methodology (RSM) using Design Expert 13.0.5.0 software optimised these conditions, with coefficient of determination values ( $R^2 = 0.9730$ ). Physicochemical analyses revealed the oil's viscosity (19.8 mPa·s), acid value (10.92 mg KOH/g), and saponification value (209.81 mg KOH/g). The hyperbolic model best described the extraction kinetics. Thermodynamic parameters (i.e., change in enthalpy,  $\Delta H = -250.962$  kJ/mol, change in entropy,  $\Delta S = 775.7295$  J/mol·K, and Gibbs free energy,  $\Delta G = -10.0691$  kJ/mol) indicated an exothermic, with a positive entropy change suggesting increased randomness and a negative Gibbs free energy indicating spontaneous process. The study yielded valuable insights into optimization and potential for industrial applications of FSO.

**KEYWORDS:** Extraction; Optimization; Kinetics; flamboyant seed, Thermodynamics;

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## I. INTRODUCTION

The increasing demand for seed oils, driven by their versatile applications in both consumer and industrial sectors, poses significant challenges for the vegetable oil industry (Sarpong, 2024; Zhang *et al.*, 2023). This surge in demand has prompted the exploration of alternative oil sources, including non-edible agricultural seeds, to mitigate the pressure on conventional sources (Malik *et al.*, 2024). Underutilized non-edible agricultural residues hold substantial potential as exploitable resources when appropriately managed. One promising avenue is oil extraction from non-edible agrarian seeds (Paul & Panwar, 2021). Oils extracted from agricultural waste have diverse industrial applications (Putra *et al.*, 2023), including pharmaceuticals (Keskin Çavdar, 2019) and bio-energy generation (Keneni & Marchetti, 2017; Singh *et al.*, 2021).

*Delonix regia*, commonly known as Flamboyant or Royal Poinciana, is a tropical deciduous tree recognized for its fern-like leaves and vibrant red and yellow blooms (Sumitra *et al.*, 2014). The tree produces numerous seed pods that pose waste management challenges due to their littering effect (Abulude *et al.*, 2018; Olugbenga *et al.*, 2020). However, the flamboyant seed (FS) has shown potential in various applications, including bio-energy production (Oyedepi *et al.*, 2017), animal nutrition supplementation (Kaga, 2013; Bake *et al.*, 2016), and pharmaceutical ingredients (Barhate *et al.*, 2014; Modi *et al.*,

2016). Additionally, the oil extracted from FS possesses insecticidal properties, making it a potential alternative to conventional pest management methods (Obembe, 2017; Tura *et al.*, 2015); and has been identified as a safe raw material for moisturizing creams (Abosede *et al.*, 2018); and biodiesel feedstock (Olugbenga *et al.*, 2020).

Various techniques are employed for oil extraction from plant seeds, including mechanical extraction (Bitire *et al.*, 2021), microwave-assisted extraction (Zhang *et al.*, 2018), ultrasonic-assisted extraction (Kumar *et al.*, 2017), supercritical fluid extraction (Brusotti *et al.*, 2014), and solvent extraction using a Soxhlet apparatus (Zhang *et al.*, 2022). Solvent extraction is favored for its economic viability, procedural simplicity, high oil yield, and relatively low operational costs (Martins *et al.*, 2023; Putra *et al.*, 2023).

Extensive studies on solvent extraction of oil from various seeds, such as rapeseed (Li *et al.*, 2013), soursop seeds (Adepoju *et al.*, 2014), *Moringa oleifera* seeds (Bhutada *et al.*, 2016), and rubber seeds (Jisieike & Betiku, 2020), highlight the importance of selecting suitable solvents such as hexane and ethanol to ensure high oil yield and quality. Hexane is preferred for its cost-effectiveness, recyclability, non-polar characteristics, high solvency, low heat of vaporization, and relatively low boiling point (Yusuf, 2018). However, hexane's flammability, non-renewable nature, and associated health

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risks necessitate exploring safer alternatives like ethanol. Ethanol's polarity penetrates cell walls for comprehensive extraction (Ramos *et al.*, 2024). Ethanol is considered renewable, biodegradable, non-toxic, and capable of delivering good yield as an extraction solvent (Meraj *et al.*, 2023; Nath *et al.*, 2024). Despite its advantages, ethanol's polarity limits its solubility with non-polar constituents, resulting in lower oil yields compared to non-polar hexane (Oladipo & Betiku, 2019). Hence, there is a need to investigate the synergy of these solvents in oil extraction from seeds.

In addition to solvent selection, other critical parameters influence oil yield and quality during solvent extraction, including extraction duration, temperature, and solvent composition (Agu *et al.*, 2018; Oladipo & Betiku, 2019). Optimizing these parameters is crucial for maximizing oil yield and quality while minimizing time and energy requirements (Oniya *et al.*, 2017). Central composite design (CCD) of response surface methodology (RSM), which is widely used for experimental design, fits a quadratic surface efficiently, minimizes experimentation, and analyses parameters interactions (Dutta *et al.*, 2015; Olaoye & Busari, 2017). Although CCD has been successfully employed to optimize process parameters for oil extraction from various seeds, to the best of the author's there is no study on the optimization of the process parameters using RSM approach for extraction of oil from FS.

Furthermore, FSO obtained from solvent extraction using hexane or ethanol may exhibit variations in yield and properties. Comprehensive characterization analysis is essential to evaluate the oil's quality, composition, and potential applications. Kinetic and thermodynamic studies are conducted to gain insight into the extraction process. The kinetic studies elucidate the extraction rate and mechanism, providing valuable information on reaction rate constants and process comparisons (Tulashie & Kotoka, 2022). While the thermodynamic studies assess factors impacting extraction efficiency, aiding in designing more efficient processes and improving oil yield and quality (Santos *et al.*, 2015). Thus, this study aims to optimize the FS oil extraction process, conduct kinetic and thermodynamic studies, and characterize the oil for potential applications. Addressing these research objectives will contribute to maximizing oil yield, improving efficiency, and finding sustainable solutions for waste management and environmental concerns.

## II. METHODOLOGY

### A. Materials

Freshly harvested Flamboyant (*Delonix regia*) seeds (FS) were used for this study. A Soxhlet apparatus was used for oil extraction, a weighing balance (model: S. Mettler DJ300BS 220 g) for weighing the samples and a 500 mL heating mantle (ZDHW-500 ML) was used as a heat source. All drying process was carried out using DHG-9053A oven with 50-200°C. The extraction reagents comprised n-hexane (99.7% Guangdong Guanghua Sci-Tech Co., Ltd. China) and ethanol (Loba Chemie PVT. Ltd. India), all of which were of analytical grade and required no further purification. Distilled water used were obtained from Central Research Laboratory University of Ilorin, Nigeria.

### B. Flamboyant seed collection and preparation

Mature Flamboyant pods were harvested from trees at the University of Ilorin, Nigeria. The pods were thoroughly washed to remove impurities, and then oven-dried at 80°C to a constant weight before being split open to obtain the seeds. The seeds were soaked in previously-boiled hot water for 30 min (Adejumo *et al.*, 2019) to facilitate the removal of the seed coat, revealing the kernel, and eases oil extraction. The FS were then dehulled, oven-dried at 60°C to a constant weight, and grinded using a Burr mill. The ground seeds were passed through a set of standard screens to remove particle sizes greater than 0.5 mm. The FS sample with 0.5 mm was stored in an airtight container for further analysis.

### C. Oil Extraction

The oil was extracted from the FS sample using solvent extraction with hexane and ethanol. Different combination of process parameters on FSO oil yield was investigated. For each extraction run, 100 mL of the solvent was added to a round bottom flask, and 30 g of the FS sample was placed in a muslin cloth inside the extraction thimble. The extractor was set on a heating mantle at a predetermined temperature for a given time. After each extraction cycle, the solvent was evaporated, leaving the oil behind. The FSO yield was determined gravimetrically as the ratio of the weight of the extracted oil to the weight of the FS sample used (Equation 1). The obtained oil was stored for further processing.

$$\% \text{ oil yield (w/w)} = \frac{\text{weight of extracted oil (g)}}{\text{weight of FPS Sample used (g)}} \times 100 \quad (1)$$

### D. Effect of Extraction Parameters on Flamboyant Seed Oil Yield

The optimum extraction conditions for FSO were determined using a single-factor investigation. Three factors – extraction temperature, extraction time, and solvent composition were varied one at a time while keeping the other parameters constant. The optimal value for each factor was determined and then used to optimize the next. The effect of time on oil yield was investigated by extracting 30 g of FS with 100 mL of n-hexane for 30, 60, 90, 120, and 150 min at the boiling point of hexane. The effect of temperature on oil yield was tested by extracting 30 g of FS with 100 mL of n-hexane for the optimal time at 45, 50, 55, 60, and 65°C using a Soxhlet extractor. The effect of solvent type on oil yield was evaluated by extracting 30 g of FS with 100 mL of hexane and ethanol, respectively, for the optimal time and temperature. The effect of solvent composition on oil yield was determined by extracting 30 g of FS with hexane-to-ethanol ratios of 1:0, 3:1, 1:1, 1:3, and 0:1 for the optimal time and temperature. This procedure was repeated to ensure accuracy and estimate the best conditions for the optimization process.

### E. Statistical Analysis for the Flamboyant Seed Oil Extraction

The response surface methodology (RSM) was used to design the experiments and analyze the data. The central composite design (CCD) under RSM was employed to optimize the FSO extraction process. Appropriate statistical analysis was achieved with the available tools embedded in the Design

Expert (DX, 13.0.5.0; Stat Ease Inc., Minneapolis, USA) Software and a polynomial model (Equation 2) (Kumari & Gupta, 2019) was used to analyse the experimental results generated by CCD.

$$OY = a_0 + \sum_{i=1}^k a_i X_i + \sum_{j=1}^k a_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^k a_{ij} X_i X_j + \dots + \varepsilon \quad (2)$$

Where OY is the predicted FSO yield;  $a_0$  is the constant,  $a_i$ ,  $a_{ii}$  and  $a_{ij}$  are the linear, quadratic and interaction coefficients, respectively; while  $X_i$  and  $X_j$  are the coded value of the process variables; and  $\varepsilon$  is the residual error.

#### F. Optimization

Three-level, three-factor design Central Composite Design (CCD) within the Response Surface Methodology in Design Expert Software was used to optimize the extraction parameters. The selected parameters were extraction time (A, in min), extraction temperature (B, in °C), and solvent composition (C, in %). The CCD comprised a: solvent composition (100:0, 50:50, 0:100) based on n-hexane, temperature (45°C, 55°C, 65°C), and extraction time (30 min, 90 min, 150 min). The coded levels and the actual values of these independent variables are shown in Table 1. Randomization of experiments was employed to minimize unexplained variability.

**Table 1:** Factors and their corresponding levels for Central Composite Design

Factor	Symbol	Coded Factor levels		
		-1	0	+1
Time (min)	A	30	90	150
Temperature (°C)	B	45	55	65
Solvent composition (mL)	C	0:100	50:50	100:0

#### G. Physicochemical Analysis of the Flamboyant Seed Oils

The physicochemical properties of FSO extracted using n-hexane and ethanol were determined by measuring its physical properties, including refractive index, density, viscosity, and specific gravity; and its chemical properties, including iodine value, free fatty acid value, acid value, pH, and saponification value using standard methods adopted by Okwonna *et al.* (2023) and Adejumo *et al.* (2019).

#### H. Kinetic Studies

The kinetics of the oil extraction from FS was investigated using hyperbolic, power law, and parabolic kinetic models. A summary of the models considered, as adopted by Agu and Agulanna (2020), are presented in the next section.

#### 1) Hyperbolic model

The hyperbolic model is mathematically given as follows:

$$\underline{q} = \frac{C_1 t}{1 + C_2 t} \quad (3)$$

It is linearly represented as follows:

$$\frac{1}{\underline{q}} = \frac{1}{C_1} \times \frac{1}{t} + \frac{C_2}{C_1} \quad (4)$$

Where  $C_1$  is the initial extraction rate ( $\text{min}^{-1}$ ),  $C_2$  is a constant connected to maximum extraction yield ( $\text{min}^{-1}$ ),  $C_2/C_1$  is the Peleg's constant denoting maximum extraction yield, and  $\underline{q}$  is the oil extraction yield. The plot of  $1/\underline{q}$  against  $1/t$  gives intercept as  $C_2/C_1$  and the slope as  $1/C_1$ .

#### 2) Power-law model

The power-law model is given as follows:

$$\underline{q} = B t^n \quad (5)$$

Where B is the power-law parameter incorporating the characteristics of the extraction system ( $\text{min}^{-1}$ ) and n is the diffusional exponent, indicative of the transport mechanism. Equation 5 is linearized to obtain Equation 6.

$$\ln \underline{q} = \ln B + n \ln t \quad (6)$$

By plotting  $\ln \underline{q}$  against  $\ln t$ , The intercept is obtained as  $\ln B$ , While n is the slope.

#### 3) Parabolic Diffusion Model

The model equation for seed extraction, where chemical reaction is not involved is shown in Equation 7, and it is known as the parabolic diffusion equation.

$$\underline{q} = A_0 + A_1 t^{\frac{1}{2}} \quad (7)$$

Where  $A_1$  is the diffusion rate constant, and  $A_0$  is the washing coefficient and represents the extraction oil yield recovered instantaneously at time  $t = 0$ . An expression for  $A_0$  is given as follows:

$$A_0 = \frac{q_w}{q_0} \quad (8)$$

Where  $q_w$  is the amount of oil washed away instantaneously as the sample material (FSO) is submersed in the solvent and  $q_0$  is the amount of oil in the sample material. A plot of  $\underline{q}$  versus  $t^{\frac{1}{2}}$ , gives  $A_0$  as the intercept and  $A_1$  as the slope.

#### I. Thermodynamic Studies

The thermodynamic parameters such as Gibbs free energy ( $\Delta G$ ), entropy ( $\Delta S$ ), and enthalpy  $\Delta H$  were determined using Equations 9, 10 and 11, respectively (Hernández-Rizo *et al.*, 2023; Silva-Paz *et al.*, 2023).

$$\Delta G = -RT \ln K \quad (9)$$

$$\ln K = -\frac{\Delta G}{RT} = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \quad (10)$$

$$K = \frac{Y_T}{Y_u} = \frac{m_L}{m_s} \quad (11)$$

Where  $K$  is the equilibrium constant,  $Y_T$  is the FSO yield at temperature  $T$ ,  $Y_u$  is the percentage of the unextracted oil.  $m_L$  is the amount of FSO in liquid at equilibrium temperature  $T$  ( $K$ ),  $m_s$  is the amount of FSO in solid at equilibrium temperature  $T$ .  $R$  is gas constant (8.314 J/mol K). The thermodynamic parameters for the extraction of FSO were evaluated by plotting  $\ln K$  against  $1/T$ .

### III. RESULTS AND DISCUSSION

#### A. Yield of Flamboyant Seed Oil

The yield of Flamboyant seed oil (FSO) for the 20 runs of the experiment estimated (using Equation 1) is presented in Table 2. The oil yield varies significantly with changes in the experimental conditions, highlighting the considerable influence of the extraction parameters on FSO yield. The highest oil yield achieved was 8.383%, obtained using an extraction temperature of 65°C, an extraction time of 150 minutes, and a solvent composition of 100 mL hexane. This maximum yield of 8.383% differs significantly from the 29.0% reported by Adejumo *et al.* (2019) in a similar solvent extraction process using n-hexane. This variation in FSO yield is attributable to diverse factors such as geographical and environmental conditions, agro-climatic factors, and variations in seed species (Al-Hamamre *et al.*, 2012). Additionally, extraction parameters in this study were scaled down in contrast to the extraction carried out by Adejumo *et al.* (2019), emphasizing the sensitivity of FSO yield to process parameters.

#### B. Effect of Process Parameters

The effect of extraction parameters such as temperature, time, solvent type and solvent composition on the oil yield obtained from FS are discussed as follows:

##### 1) Effect of Temperature

Figure 1 shows the one-factor plots for the effects of extraction temperature (a), extraction time (b), solvent composition (c), and solvent composition (d) on the yield of FSO. Figure 1(a) showed that the oil yield exhibits an upward trend with increasing extraction temperature, reaching a maximum value at 65°C, slightly below the boiling point of hexane. Beyond this point, the oil yield decreases due to solvent evaporation. Specifically, when using hexane as the solvent, increasing the extraction temperature from 45°C to 55°C leads to a rise in oil yield to 6.476%. At 65°C, the oil yield reaches 8.277%, which is 0.304% higher than at 60°C. However, beyond 60°C, the curve flattens, indicating no significant increase in FSO yield. This suggests that higher temperatures enhance the solubility of oil in the solvent, resulting in a higher extraction rate (Ntalikwa, 2021).

**Table 2: Experimental design with process data and the response for the FSO extraction**

Extraction Process Variables      Oil Yield (wt.%)

Run	Time	Temperature	Solvent Composition	Experimental Yield	Residual
1	30	65	0	1.341	-0.0782
2	30	55	50	0.423	0.6680
3	90	55	50	1.001	-0.6999
4	90	55	50	0.992	-0.7089
5	90	45	50	0.091	0.4480
6	90	55	50	1.851	0.1501
7	30	45	100	0.357	-0.2784
8	150	45	100	0.987	0.0089
9	30	65	100	2.163	-0.1012
10	150	65	100	8.383	0.1410
11	150	65	0	7.597	0.2090
12	90	55	0	2.889	0.0476
13	30	45	0	0.191	-0.2103
14	90	55	50	2.257	0.5561
15	90	55	50	2.039	0.3381
16	150	45	0	0.767	0.0319
17	90	55	50	1.511	-0.1899
18	90	55	100	3.615	0.2296
19	150	55	50	2.52	-0.3908
20	90	65	50	3.613	-0.1708

##### 2) Effect of Time

In Figure 1(b), the oil yield increases with an extended extraction time due to the increased contact time of solvent and oil. However, after 150 min, the curves start to decline, indicating no significant increase in oil yield beyond 180 min. It illustrates that most FSO extraction occurs within the initial 150 min. Extending the extraction time to 180 min only yields a marginal increase of about 0.024%, which does not justify the additional time. Therefore, in this study, 150 min is considered the maximum extraction time, as oil yield beyond this point remains insignificant. Hence, increasing extraction time results in an increase in oil yield.

##### 3) Effect of Solvent Type

The solvents used in this study include both polar (ethanol) and non-polar (n-hexane) solvents. The polarity of the solvent affects the extraction yield, with less polar solvents leading to higher oil yields, as shown in Figure 1(c). Ethanol exhibits a slightly lower oil yield compared to hexane. This difference may be due to ethanol's ability to extract non-fatty components such as antioxidants and pigments, which can interfere with oil extraction as a polar solvent (Oladipo & Betiku, 2019). Non-polar solvents, like hexane, extract both non-fatty and fatty constituents efficiently. The lower polarity and lower boiling points of n-hexane contribute to its higher yield as it traverses the pulverized kernels more rapidly (Yang *et al.*, 2014). Additionally, n-hexane lacks the OH group, which can hinder oil extraction from certain seeds (Oladipo & Betiku, 2019).

##### 4) Effect of Solvent Composition

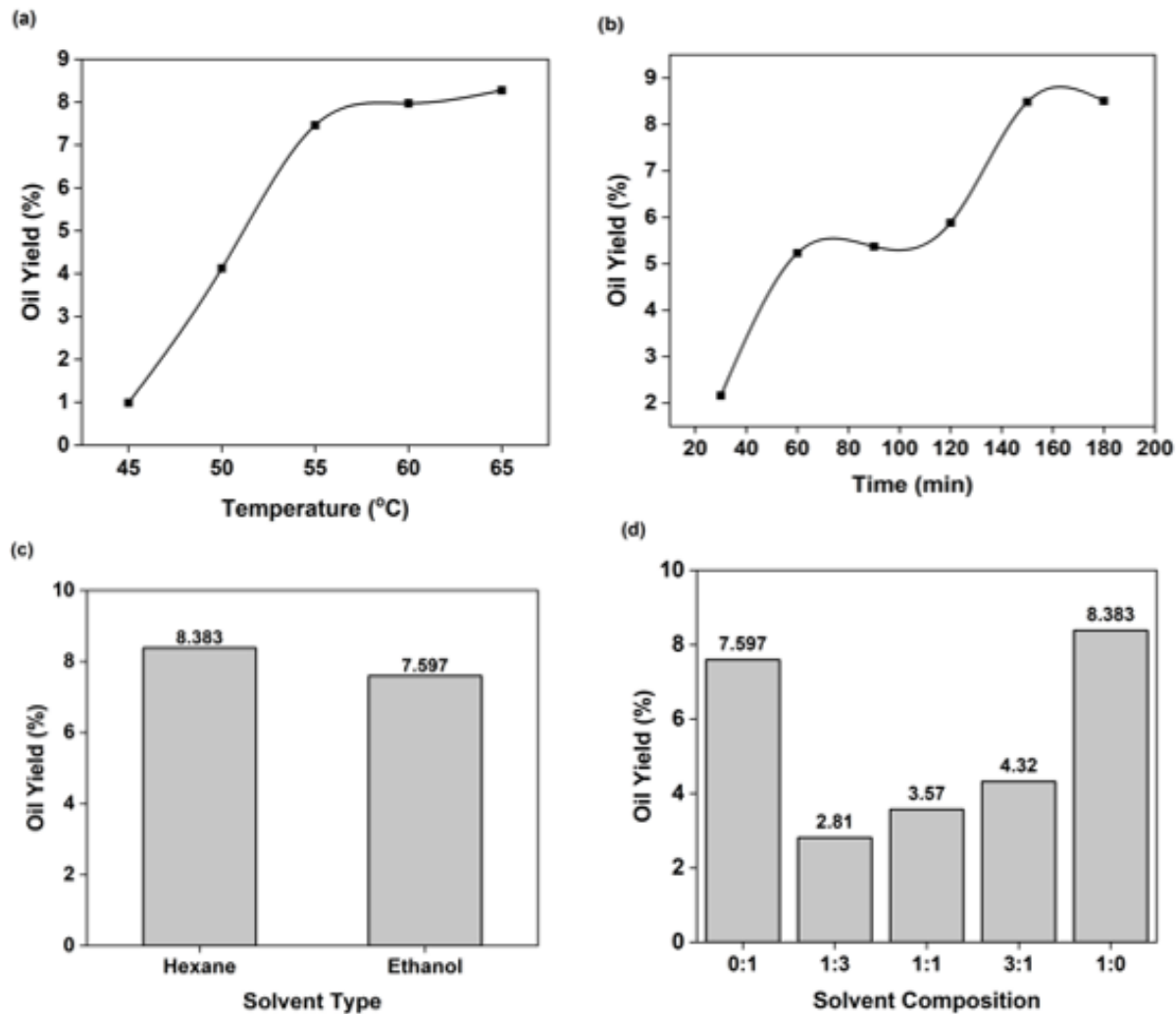
In extracting FSO, a solvent mixture of hexane and ethanol in varied ratios was utilized. The results in Figure 1(d) indicate that hexane to ethanol ratio significantly impacts the oil yield. Increasing the non-polar solvent from 1:1 to 3:1 and 1:0 leads to higher FSO yields. However, increasing the polar solvent in ratios 1:3 and 0:1 reduces oil yields, which is attributable to the impaired solubility of ethanol caused by non-polar constituents in the sample matrix (Li *et al.*, 2013). Additionally, polar

compounds extraction from the seeds may lead to complex interactions with fatty acids and monoglyceride components, inhibiting oil extraction and reducing overall yield (Al-Hamamre *et al.*, 2012). These findings align with previous research on oil extraction from plant seeds using hexane and ethanol as co-solvents (Bhutada *et al.*, 2016; Ntalikwa, 2021).

### C. Statistical Analysis of Data for Flamboyant Seed Oil Extraction

The coded variables, experimental and predicted values obtained from the experiments are presented in Table 2. The results obtained from the significance tests for the regression

analysis of the model is presented in Table 3. The F-value of the model is 39.99, indicating the statistical significance of the RSM quadratic model. Moreover, the p-value  $< 0.0001$  suggests only a 0.01% chance of the F-value causing interference. In terms of relevance, the operating parameters follow this order: solvent composition (167.37)  $>$  temperature (97.21)  $>$  extraction time (39.99). The ANOVA results indicate that all terms of the regression model are significant ( $p < 0.05$ ) except one. As shown in Table 3, factors A, B, AB, and  $C^2$  all have p-values  $< 0.05$ , signifying their significant influence on essential oil extraction when these factors vary. The coefficient of determination ( $R^2$ ) of 0.9730 indicates that 97.30% of the experimental data align with the model's predictions.



**Figure 1:** One-factor plots for the effects of (a) extraction temperature, (b) extraction time, (c) solvent composition and (d) solvent composition on the yield of FSO.

**Table 3: Analysis of Variances (ANOVA) for model regression**

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
<b>Model</b>	92.18	9	10.24	39.99	< 0.0001	Significant
<b>A – Time</b>	24.90	1	24.90	97.21	< 0.0001	
<b>B – Temperature</b>	42.87	1	42.87	167.37	< 0.0001	
<b>C - Solvent Composition</b>	0.74	1	0.74	2.89	0.1200	
<b>AB</b>	15.88	1	15.88	61.99	< 0.0001	
<b>AC</b>	0.00	1	0.00	0.00	0.9902	
<b>BC</b>	0.19	1	0.19	0.73	0.4132	
<b>A<sup>2</sup></b>	0.37	1	0.37	1.45	0.2556	
<b>B<sup>2</sup></b>	0.00	1	0.00	0.00	0.9681	
<b>C<sup>2</sup></b>	5.49	1	5.49	21.42	0.0009	
<b>Residual</b>	2.56	10	0.26			
<b>Lack of Fit</b>	1.14	5	0.23	0.80	0.5940	not significant
<b>Pure Error</b>	1.42	5	0.28			
<b>Cor Total</b>	94.74	19				
					<b>R<sup>2</sup></b>	0.9730
					<b>Adjusted R<sup>2</sup></b>	0.9486
					<b>Predicted R<sup>2</sup></b>	0.8940
					<b>Adeq Precision</b>	24.0296

*D. Optimization of FSO Extraction*

The relationship between oil yield (OY) and independent variables A, B, and C in coded terms for the quadratic model is expressed as follows:

$$OY = 1.7009 + 1.5779A + 2.0704B + 0.272C + 1.40875AB + 0.00225AC + 0.15275BC - 0.368A^2 + 0.0125B^2 + 1.4125C^2 \tag{12}$$

Where A is time (min), B is the temperature (°C), and C is the solvent composition (mL). The positively correlated factors with FSO yield include time (A), temperature (B), solvent composition (C), time-temperature (AB), time-solvent composition (AC), temperature-solvent composition (BC), temperature squared (B<sup>2</sup>), and solvent composition squared (C<sup>2</sup>). Increasing the values of these factors leads to higher FSO yields. Contrarily, FSO yield tends to decrease with an increase in time squared (A<sup>2</sup>) due to negative quadratic effect.

The graphical representations of the regression equation for optimizing FSO extraction are depicted as contour and 3-D dimensional surface plots in Figure 2-4, illustrating the relationships between independent and dependent variables. Figure 2 suggests that seed oil yield increases with a longer extraction time and higher temperature, with an optimal temperature and extraction time for maximum oil yield. Temperature appears to have more impact on FSO yield than extraction time. This observation aligns with previous research, highlighting the importance of temperature and extraction time in seed oil extraction. Ntalikwa (2021) found that *Jatropha* oil yield increased with higher temperatures and longer extraction times, although oil yields decreased at temperatures exceeding the solvent's boiling point of 68°C. Figure 3 and Figure 4 illustrate that the FSO yield experiences an increase due to the interaction between extraction time and solvent composition, as well as the interaction between temperature and solvent composition. However, solvent composition appears to have a more pronounced impact on FSO yield, as depicted in Figure 4.

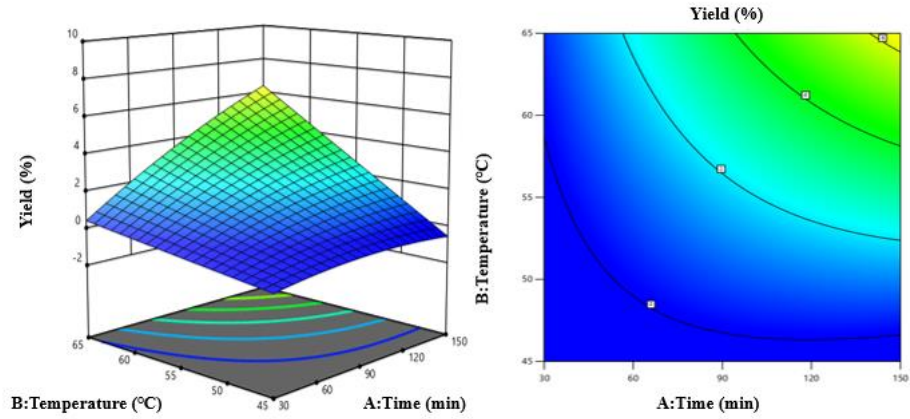


Figure 2: The response surface and contour plots of FSO yield as the function of extraction time and temperature.

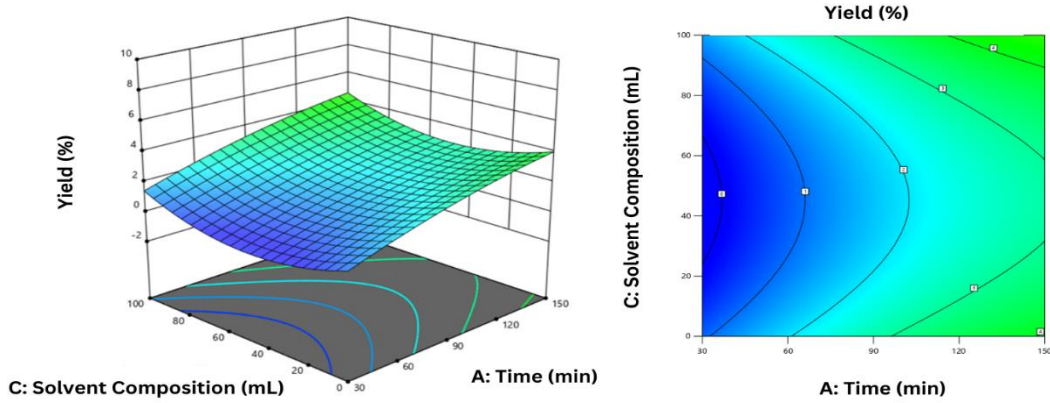


Figure 3: The response surface and contour plots of FSO yield as the function of extraction time and solvent composition

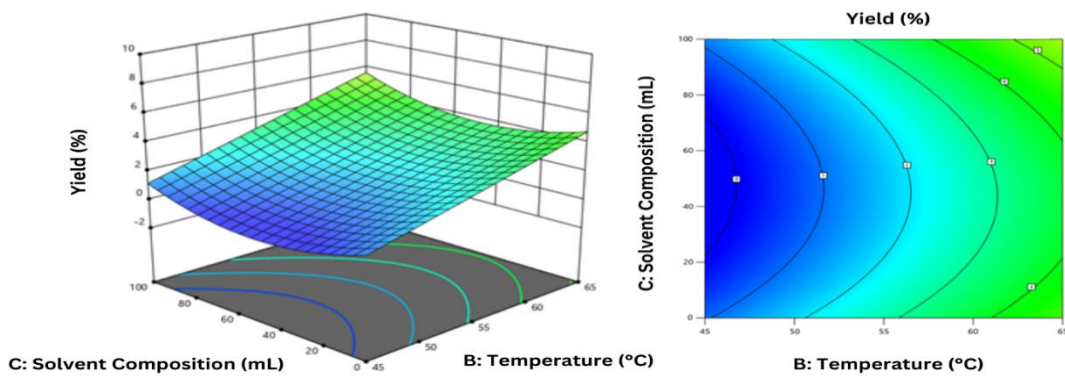


Figure 4: The response surface and contour plots of FSO yield as the function of extraction temperature and solvent composition.

E. Validation of Processing Parameters for Maximum Oil Yield

The optimum yield was determined with numerical optimization using Design Expert software v 13. The maximum oil recovery was achieved with solvent composition, extraction temperature, and extraction time set of 100 mL, 65°C, and 150 min, respectively, deviating slightly from the literature. Figure 5 presents a surface plot illustrating the optimum values. Under these conditions, the predicted oil recovery was 8.242% with a desirability of 0.983 at a 95% Prediction Interval (PI) High. These optimal conditions closely matched the experimental value of 8.383%, above the projected value of 8.242%. Two confirmatory experiments were conducted under the optimized conditions to validate the model's accuracy, resulting in an average FS oil yield of 8.319%. This result falls within the range predicted by the model and is presented in Table 4. Thus, the experiment confirmed the optimal conditions for solvent extraction of oil from FS using response surface methodology for optimization.

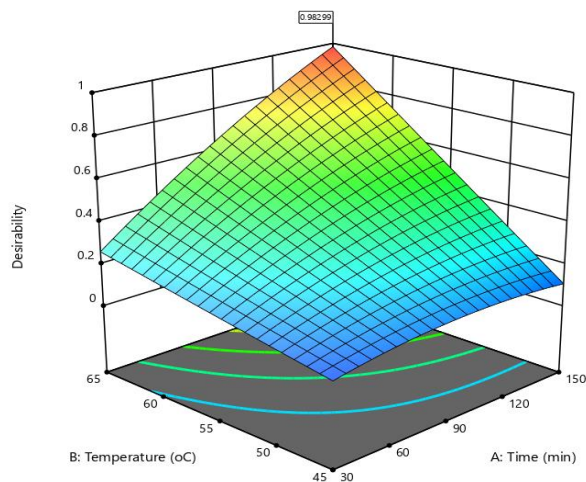


Figure 5: 3-D Response surface plot showing the effect of extraction time and temperature on the oil yield

Table 4: FSO CCD matrix of independent variables and their corresponding experimental, predicted, and residual values

Optimum Conditions	Coded Levels	Actual Levels
Time (min)	1	150
Temperature (°C)	1	65
Solvent Composition (ml)	1	100
Response	Predicted Value	Experimental Value
Yield	8.242	8.277
	-	8.362

F. FSO Extraction Kinetics

Three models were used to analyse FSO extraction kinetics: parabolic diffusion, power law, and hyperbolic models using linear regression to fit experimental data. The determination coefficient ( $R^2$ ) was employed to evaluate and compare the fit quality for each model. Figure 6(a-c) shows the results of fitting experimental kinetic data for FSO extraction with hexane. The experimental data fit well with all three models, as indicated by their  $R^2$  values: 0.9142 for the parabolic diffusion model, 0.9153 for the power law model, and 0.9524 for the hyperbolic model. The hyperbolic model was chosen as the most suitable for characterizing the kinetics of oil extraction from flamboyant seeds, given its highest  $R^2$  compared to the other models. Additionally, Figure 6(c) presents the fit of the hyperbolic model to the experimental data and kinetic parameters.

The kinetic parameters obtained from Figures 6a-c. The slope obtained for the parabolic model ( $A_1$ ) is 12.05 represents the parabolic diffusion parameters, and the intercept ( $A_0$ ) is 0.0435 denotes the initial extraction rate. The slope obtained for the power law model, which represents  $n$ , is 0.7236. The intercept is -1.5614, representing  $\ln B$  relating to the extraction rate constant characteristics. The slope of the hyperbolic model represents the diffusion coefficient, indicating the moderate diffusion rate. The intercept represents the initial extraction rate. The slope of the parabolic model represents  $1/C1$  of the parabolic diffusion model, which was obtained to be 0.7705, which indicates that the increase in the extraction is moderate, meaning the system is dominated by diffusion. The negative slope of -1.679, representing  $C2/C1$ , reflects an offset that arises in the early stage of the extraction process. It can be deduced that the extraction process is rapid at first followed by a gradual decrease in extraction rate as the maximum oil extraction yield is approached.



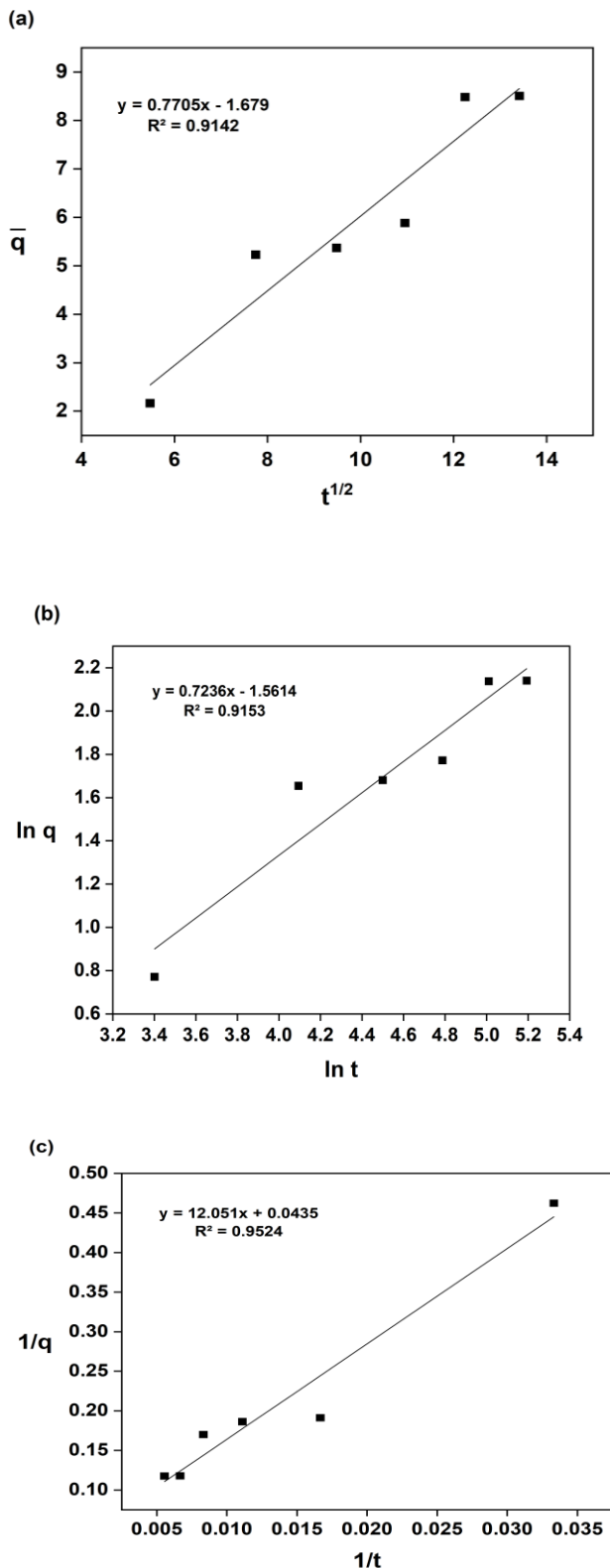


Figure 6: Fit of experimental data for kinetics of FSO extraction to (a) Parabolic diffusion, (b) Power law, and (c) Hyperbolic model.

G. Thermodynamic Parameters

Thermodynamic parameters of the oil extraction process were determined using FSO yield data. The oil yield at temperature T is represented by  $Y_T$  as shown in Table 5, percentage of unextracted oil represented by  $Y_u$  and the thermodynamic constant, K, derived from Equation 8. The Van't Hoff equation (Equation 9) was successfully fitted to the data with a high coefficient of determination ( $R^2=0.9596$ ), as depicted in Figure 7, enabling the calculation of enthalpy and entropy changes during FSO extraction. A negative  $\Delta G$  value of  $-10.0691$  kJ/mol indicates that the process is thermodynamically favorable and spontaneous under the given conditions. The negative Gibbs free energy change drives the solute extraction process from the mixture without external energy input. The negative enthalpy change,  $\Delta H$  of  $-250.962$  kJ/mol, signifies that the extraction process releases heat energy. This heat release can be advantageous in maintaining the necessary temperature conditions for the solvent's vaporization and condensation cycles in a Soxhlet extraction process. Furthermore, a positive entropy change,  $\Delta S$  of  $775.7295$  J/mol·K, suggests an increase in disorder or randomness during FSO extraction due to solvent and solute mixing during extraction.

In summary, the determined thermodynamic parameters indicate that the extraction process is thermodynamically favorable (negative  $\Delta G$ ) and exothermic (negative  $\Delta H$ ) and results in an increase in disorder (positive  $\Delta S$ ). These findings align with the expectations for a favorable extraction process, such as Soxhlet extraction, that employs heat to facilitate the separation of components from a solid sample. The findings in this study are similar to what was obtained for neem leave by Sunday et al. (2021 ) and *Treculia africana* by Okwonna et al. (2023).

**Table 5:** Equilibrium Constant Data (K) at Different Extraction Temperatures.

T	$Y_T$	$Y_u$	K	ln K
318	0.987	7.52	0.13125	-2.03065
323	4.125	4.382	0.94135	-0.06044
328	7.463	1.044	7.14847	1.966898
333	7.973	0.534	14.9307	2.70342
338	8.277	0.23	35.98696	3.583157

T is temperature in kelvin,  $Y_T$  is the oil yield at temperature T,  $Y_u$  is the percentage of unextracted oil represented and K is the thermodynamic constant

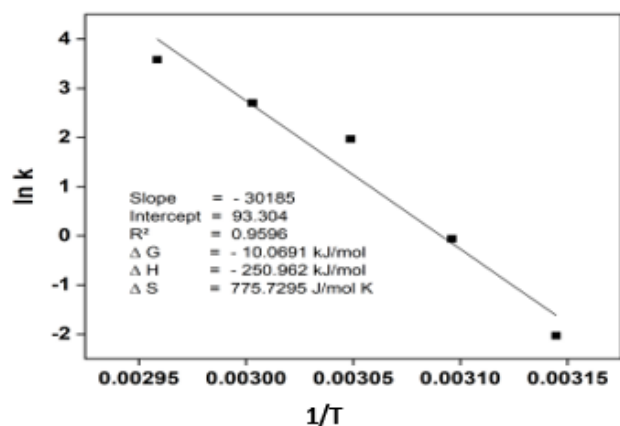


Figure 7: The Van't Hoff equation fit experimental thermodynamic data and obtained thermodynamic parameters.

H. Physicochemical Analysis of Extracted FSO

The results of the physicochemical analysis conducted to evaluate the quality of oils as presented in Table 6. At room temperature, all samples exhibited a liquid state. The color of the oil varied; it appeared amber for the n-hexane extract and dark brown for the ethanol extract, as illustrated in Figure 8. The ethanol-extracted FSO's dark color is attributable to its ability to dissolve other components that affect the oil's appearance. Previous studies have shown that the coloring matter content of oilseeds gradually increases with an elevated

Table 6: Physicochemical analysis of hexane-extracted FSO extracted in comparison to similar studies

Parameters	This study	(Adejumo <i>et al.</i> , 2019)	(A. L. Adepoju, 2019)	(Oyediji <i>et al.</i> , 2017)	(Tura <i>et al.</i> , 2015)	(Adewuyi <i>et al.</i> , 2010)	(Abosede <i>et al.</i> , 2018)
<b>Yield</b>	8.383%	29.0%	29.50%	17.16%	30.8%	7.0%	-
<b>Density (g/cm<sup>3</sup>)</b>	0.97	0.942	0.99	0.97	0.621	-	-
<b>Specific gravity</b>	-	-	0.960	-	0.743	0.951	0.99
<b>Refractive index @ 25°C</b>	1.432	-	1.46	1.47	-	1.442	1.43
<b>Iodine value (g/100 g)</b>	23.42	127.92	10.28	121.03	23.48	127.7	37.56
<b>Saponification value (mg KOH/g)</b>	209.81	213.48	37.48	203.40	22.44	195.4	30.00
<b>Acid value (mgKOH/g)</b>	10.92	1.97	-	-	1.683	-	22.40
<b>Free fatty acid (mg KOH/g)</b>	5.61	0.985	1.60	-	-	10.4	-
<b>Viscosity (mPa.s)</b>	19.8	-	22	-	-	-	47.60
<b>Color</b>	Amber	-	-	Amber	Deep green	Brown	Light green

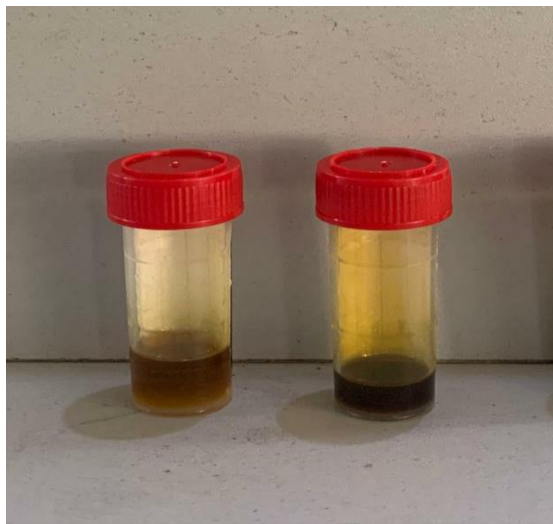


Figure 8: FSO extracted with n-hexane and ethanol respectively

dielectric constant (Oladipo & Betiku, 2019), aligning with these observations.

The density of FSO obtained is  $0.97 \text{ g/cm}^3$ , which aligns with Adepoju's (2019) reported density of  $0.99 \text{ g/cm}^3$ . This similarity implies that *Delonix regia* seed oil is suitable for use as biofuel, as a higher mass of oil provides more energy output per unit volume. The oil exhibits a refractive index of 1.47, which aligns with the range reported in the literature (Table 8) and is consistent with values of 1.47 found for both *Citrullus lanatus* and *Sesamum indicum* seed oils (Adepoju, 2019). The iodine value of the oil was determined to be  $23.42 \text{ g/100 g oil}$ , indicating a low degree of unsaturation and categorizing it as a non-drying oil.

The saponification value for FSO was found to be  $209.81 \text{ mg KOH/g}$ , closely aligning with values reported by Adejumo *et al.* (2019) and Oyedeji *et al.* (2017) as  $213.48 \text{ mg KOH/g}$  and  $203.40 \text{ mg KOH/g}$ , respectively. This value provides insights into the oil's average molecular weight and suggests its suitability for soap and shampoo production (Oyedeji *et al.*, 2017). The acid value was determined to be  $10.92 \text{ mg KOH/g}$ , lower than the  $22.40 \text{ mg KOH/g}$  reported by Abosede *et al.* (2018). Additionally, the viscosity, which measures the oil's resistance to shear, was found to be  $19.8 \text{ mPa}\cdot\text{s}$ .

Notably, the quality parameters investigated in Table 8 fall within the ranges reported in the literature for FSO, indicating that the oil produced in this study exhibits similar quality characteristics. Variations in quality parameters among studies can be attributed to differences in species, extraction techniques, and operating conditions. Nevertheless, this study demonstrates the potential of the

extracted oil for biodiesel production despite its poor yield and the manufacturing of soaps and shampoos.

#### IV. CONCLUSION

The kinetics and thermodynamics of FSO extraction were investigated under varying extraction times, temperatures, and solvent compositions. The study revealed that increasing extraction temperature and time enhanced oil yield, with the maximum yield obtained using n-hexane at  $65^\circ\text{C}$  and an extraction time of 150 min. The nonpolar solvent, n-hexane, outperformed ethanol, highlighting the critical role of solvent selection. Optimization using Response Surface Methodology and Central Composite Design confirmed these conditions, yielding a high coefficient of determination value ( $R^2 = 0.9730$ ). Kinetic modelling showed that the hyperbolic model best described the extraction process, with a high coefficient of determination ( $R^2 = 0.9524$ ), making it a reliable predictor of extraction behavior. Thermodynamic analysis indicated that the process was exothermic ( $\Delta H = -250.962 \text{ kJ/mol}$ ) and thermodynamically favorable, as evidenced by the negative Gibbs free energy change ( $\Delta G = -10.0691 \text{ kJ/mol}$ ). The positive entropy change ( $\Delta S = 775.7295 \text{ J/mol}\cdot\text{K}$ ) suggested increased disorder, consistent with solvent-solute interactions. The physicochemical analysis confirmed that the extracted FSO met quality standards, with key parameters such as viscosity ( $19.8 \text{ mPa}\cdot\text{s}$ ), acid value ( $10.92 \text{ mg KOH/g}$ ), and saponification value ( $209.81 \text{ mg KOH/g}$ ), indicating its suitability for industrial applications, including biodiesel production, soap, and shampoo manufacturing. These findings underscore the importance of optimizing extraction conditions and solvent selection to maximize yield and quality. For commercial viability, future studies should explore scaling up FSO extraction to an industrial level while maintaining product integrity and efficiency.

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