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Research Paper

Application of the Box-Behnken Experimental Design to Optimize Coffee Ground Oil Extraction Using the Soxhlet Method

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ABSTRACT*: Coffee is a widely traded and consumed commodity worldwide. In 2023, Indonesia's coffee consumption reached 312,000 tons, placing it as the second-largest consumer in Asia. This high consumption generates approximately 48% of coffee grounds as waste, which still contains valuable compounds such as carbohydrates, proteins, oils, minerals, and polyphenols, thus potentially being utilized in various applications. This study was conducted to optimize the Soxhlet extraction of oil from spent coffee grounds (SCGs), with the objective of maximizing yield while preserving oil quality. The investigated independent variables comprised the ethanol concentration in n-hexane (0, 50, and 100% v/v), extraction time (15, 30, and 45 min), and the solvent-to-solid (L/S) ratios (15:1, 20:1, and 25:1 mL/g), all systematically arranged using a Box–Behnken design (BBD). The study found that using ethanol – n-hexane mixtures at certain compositions significantly improved oil yield while reducing saponification value, acid value, and FFA content. Longer extraction times and higher L/S ratios also contributed to increasing yield and saponification value, while decreasing acid value and FFA up to an optimum point. The optimum condition was obtained at 17.89% v/v ethanol in n-hexane, 45 minutes of extraction, and a L/S ratio of 22.47 mL/g, resulting in a 25.34% yield with oil characteristics of 179.12 mg KOH/g saponification value, 4.30 mg KOH/g acid value, and 0.73% FFA. These findings demonstrate that Soxhlet extraction can produce SCG oil with favorable characteristics, although the saponification value was slightly lower (-0.49%) than the minimum limit set by SNI 7431:2015. Overall, SCG oil shows great potential as an alternative vegetable oil for use in food, pharmaceutical, cosmetic, and renewable energy applications.*

Keywords: *BBD; extraction; SCG oil; optimization; Soxhlet*

1. INTRODUCTION

Coffee was recognized as one of the most extensively traded and consumed commodities globally, primarily due to its distinctive aroma and flavor profile [1]. The caffeine content in coffee exerted stimulant effects that enhanced concentration, reduced fatigue, and accelerated body metabolism [2]. According to the International Coffee Organization [3], coffee consumption in Indonesia reached 312,000 tons in 2023, positioning the country as the second-largest coffee consumer in Asia. This high level of consumption generated a substantial amount of spent coffee grounds (SCGs), approximately 48% of the total coffee powder [4]. SCGs contained carbohydrates, proteins, oils, minerals, and polyphenols, which render them suitable as raw material for value-added products in the food, cosmetic, pharmaceutical, and renewable energy industries [5], [6]. Moreover, SCGs also contained chlorogenic acid [7], which acted as an antioxidant [8], anti-inflammatory agent [9], and antimicrobial compound [10]. Unfortunately, SCGs were still generally regarded as waste and discarded, even though their disposal contributed to the release of CH_4 and CO_2 gases, which exacerbated global warming [2].

Today, SCGs containing 20–30% oil [4], [11], comparable to jatropha seed [12] and papaya seeds [13], had attracted substantial research interest due to its high content of linoleic acid (34.99–42.45%), palmitic acid (32.98–41.83%), and nutraceutical compounds [14], [15], all of which provided multiple health benefits [16],

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[17]. Despite this great potential, oil extraction from SCGs required an appropriate method to achieve optimal yield without compromising their nutraceutical properties, as exposure to elevated temperatures during extraction can lead to thermal degradation of bioactive compounds [18].

Oil extraction from SCGs could be carried out using various techniques such as solvent extraction, mechanical pressing, extrusion–expeller, and sublimation. Conventional solvent-based extraction methods, including maceration, percolation, and reflux extraction (Soxhlet), were also commonly applied in different contexts. However, these methods typically suffer from limitations such as prolonged extraction times, high solvent usage, and comparatively low extraction efficiency [19]. In addition, the use of highly pure solvents was essential to prevent extract contamination, which was often difficult to obtain. Despite these limitations, conventional solvent extraction methods offered advantages in terms of simplicity, low equipment costs, ease of operation, and the ability to process multiple samples simultaneously [20].

The Soxhlet extraction method was selected because it has been demonstrated to be effective in numerous studies for extracting oil from a variety of raw materials. Its efficiency is significantly enhanced when parameters such as solvent type, particle size, extraction time, and solvent-to-solid (L/S) ratio are optimized. Andriana and Broto [21] reported that papaya seed oil extraction using petroleum ether (20 mesh, 180 minutes, 1:9 ratio) yielded 57.03%. In addition, watermelon seeds achieved a yield of 87.5% with a 1:4 ratio [22]. Recent studies also demonstrated that Soxhlet extraction produced higher yields compared to cold pressing and enzymatic extraction in pumpkin seeds [23], making it a superior choice for oil extraction from solid matrices.

This study investigated the extraction process and characterization of oil obtained from SCGs, employing a statistical approach to optimize extraction conditions. The optimization aimed to obtain the highest oil yield while simultaneously evaluating its chemical properties, including saponification value, acid value, and FFA. The independent variables investigated included the concentration of mixed ethanol and n-hexane solvents, extraction time, and L/S ratio. These factors were analyzed to evaluate their influence on both yield and oil characteristics and were incorporated into response surface modeling to identify optimal extraction parameters. The experiments were designed, and the data were analyzed using a Box-Behnken design (BBD). Oil extracted under these optimized conditions exhibited high quality, indicating promising applications in food, cosmetics, pharmaceuticals, and renewable energy sectors, thereby enhancing the value and utilization of coffee waste.

2. RESEARCH METHOD

2.1. Materials

The equipment utilized in this research includes a 250 mL Soxhlet extractor, heating mantles, analytical balances, ovens, thermometers, beaker glass, cellulose thimbles, and a UV-Vis spectrophotometer (Optizen 2120 UV+Mecusys). The research materials were 1 kg of robusta coffee bean dregs obtained from a coffee shop after one brewing, as well as ethanol (96%) and n-hexane (99%) solvents purchased from a chemical store. For analytical purposes, KOH (99.8%), HCl (36%), and PP indicator (1%) were used, obtained from the marketplace, and 10 mL of standard coffee bean oil was obtained from a chemical store.

2.2 Drying and Grinding of SCGs

Robusta SCGs obtained from a local coffee shop after a single brewing cycle were separated from the residual liquid using filter paper. Subsequently, 100 g of the SCGs were dried under sunlight in open air for three days until a moisture content of less than 14% (wet basis) was achieved [24]. The dried SCGs were then ground using a mortar and pestle and passed through a 30-mesh sieve to ensure uniform particle size.

2.3 Extraction of Oil from SCGs

Soxhlet extraction of SCGs was carried out following the procedure reported by Thilakarathna *et al.* [25], with minor modifications. Fifteen grams of dried, ground, and sieved SCGs were placed in a cellulose thimble for solvent extraction. The extraction employed ethanol–n-hexane mixtures at concentrations of 0, 50, and

100% v/v, while variations in the L/S ratio (15:1, 20:1, and 25:1 mL/g) and extraction time (15, 30, and 45 min) were also examined. The process was conducted at the boiling temperature of the solvent–oil mixture (~79 °C). Upon completion, the extract–solvent mixture was analyzed using a UV–Vis spectrophotometer to measure absorbance values. To calculate the oil concentration and yield, a mixture of solvent and commercial coffee oil was used as a standard. The oil yield was determined according to Equation (1):

$$Y\ (\%) = \frac{C_{oil} \times V \times \rho_{oil}}{m_{SCG}} x100\% \quad (1)$$

where Y is the extraction yield (%), C_{oil} represents the oil concentration in the extract (mL/L), V is the extract volume (L), ρ_{oil} is the oil density (g/mL), and m_{SCG} refers to the mass of SCGs used (g).

2.4 Determination of Saponification Value

About 1 g of SCG oil was placed in a 250 mL Erlenmeyer flask and mixed with 25 mL of 1 N KOH. The mixture was refluxed for 30 min with occasional stirring. After cooling slightly, 1 mL of phenolphthalein was added, and the hot solution was titrated with 0.5 M HCl until the pink color disappeared, giving the titrant volume a mL. A blank test using the same procedure but without the oil provided volume b mL. The saponification value was then computed using Equation (2) [26].

$$SV = \frac{(b-a) \times N \times 56.1}{m_{oil}} \quad (2)$$

where SV is the saponification value (mg KOH/g), a and b represent the titrant volumes for the sample and blank (mL), N is the normality of HCl, and m_{oil} is the mass of SCG oil (g).

2.5 Determination of Acid Value and FFA

The acid value was determined by weighing approximately 1 g of SCG oil into a volumetric flask. To this, 25 mL of 95% ethanol and two drops of phenolphthalein indicator were added. The mixture was then titrated with 0.1 M KOH until a light pink endpoint was observed [26]. The acid value was subsequently calculated using Equation (3):

$$AV = \frac{V_{titrant} \times N \times 56.1}{m_{oil}} \quad (3)$$

where AV is the acid value (mg KOH/g), V is the volume of KOH used for titration (mL), and N is the normality of KOH.

The FFA content, expressed as oleic acid equivalent, was then derived from the acid value using Eq. (4):

$$FFA\ (\%) = \frac{AV}{1.99} \quad (4)$$

where 1.99 is the conversion factor corresponding to the molecular weight of oleic acid (282 g/mol).

2.6 Experimental Design and Process Optimization

The Box–Behnken design (BBD) was utilized to optimize the extraction parameters, namely ethanol concentration in n-hexane, L/S ratio, and extraction time (Table 1). Statistical analyses were conducted at a significance level of $p < 0.05$, with optimization based on the highest desirability value [27].

Table 1. The BBD for the extraction of SCG oil

Parameters	Unit	Range and constraints		
		-1	0	1
Ethanol concentration in n-hexane	%v/v	0	50	100
Solvent-to-solid (L/S) ratio	mL/g	15:1	20:1	25:1
Extraction time	min.	15	30	45

3. RESULTS AND DISCUSSION

3.1. Oil Yield of Extracted from SCGs

Based on Soxhlet extraction, the oil yield from Robusta SCGs exhibited considerable variation, ranging from 17.39% to 26.73%, with an average yield of 20.08% (Table 2). This variation reflected the influence of process conditions, namely the ethanol concentration in n-hexane, extraction time, and L/S ratio. The highest oil yield (26.73%) was achieved under the conditions of 50% v/v ethanol in n-hexane, 45 minutes of extraction, and a L/S of 25:1. These findings indicated that extended extraction time combined with a higher solvent ratio enhanced solvent contact and penetration into the solid matrix, thereby facilitating more efficient oil recovery [28].

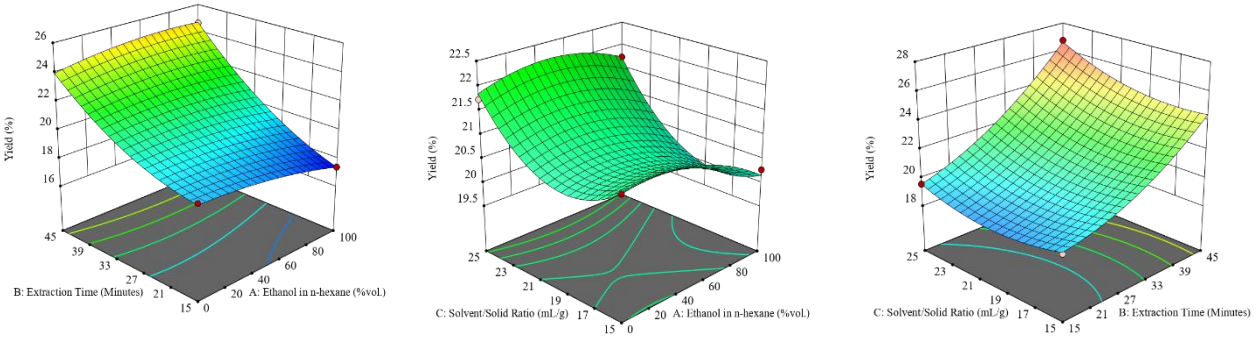
Conversely, the lowest oil yield of 17.39% was obtained under the conditions of 100% ethanol as a single solvent, with an extraction time of 15 minutes and a L/S ratio of 20:1. This outcome highlighted that the use of mixed solvents, extended extraction time, and a higher solvent ratio enhanced the efficiency of the extraction process. These findings emphasize the critical role of optimizing extraction parameters to maximize oil yield from Robusta SCGs.

Table 2. Oil yield of Robusta SCGs obtained by Soxhlet extraction

Ethanol concentration in n-hexane (%)	Extraction time (minutes)	Solvent-to-solid ratio (mL/g)	Yield (%)
100	45	20:1	24.63
100	15	20:1	17.39
0	30	15:1	21.01
0	15	20:1	19.20
100	30	25:1	21.73
50	45	25:1	26.73
100	30	15:1	20.28
50	15	15:1	19.20
0	30	25:1	21.73
50	15	25:1	19.56
0	45	15:1	23.91
100	45	20:1	24.63

Based on Figure 1, the effects of extraction time, ethanol concentration in n-hexane, and L/S ratio on the oil yield from SCGs are illustrated through 3D surface plots. The color gradient from blue to red represents increasing oil yield, where blue signifies low yield, green–yellow represents medium yield, and red indicates high yield. All three plots (a–c) demonstrate that oil yield improves with longer extraction times and higher L/S ratios, whereas higher ethanol concentrations in the n-hexane mixture tended to reduce the yield.

Figure 1(a) highlights the combined effects of extraction time and ethanol concentration in n-hexane on oil yield at a L/S ratio of 20:1. Prolonged extraction enhanced oil recovery across different ethanol concentrations due to the extended contact between solvent and matrix, allowing more oil to be solubilized. However, the rate of increase diminished at longer times as the system approached equilibrium. A similar trend was observed for the interaction between extraction time and L/S ratio at 50% ethanol in n-hexane, as shown in Figure 1(c). These results concur with Al-Hamamre *et al.* [29], who found that increasing extraction time across various solvents enhanced coffee oil recovery. Likewise, Hanif *et al.* [30] observed that the extraction of coffee oil with n-hexane improved from 18.35% to 22.71% as extraction time increased from 60 to 300 minutes, after which the yield plateaued due to equilibrium.



(a) **(b)** **(c)**

Figure 1. 3D surface plot depicting the effect of extraction variables on oil yield from SCG Robusta: (a) interaction between extraction time and ethanol concentration in n-hexane at a L/S ratio of 20:1 (mL/g); (b) interaction between ethanol concentration in n-hexane and L/S ratio at a extraction time of 30 minutes; and (c) interaction between L/S ratio and extraction time at an ethanol concentration of 50% v/v in n-hexane.

Meanwhile, variations in ethanol concentration within the n-hexane mixture significantly influenced the overall polarity of the solvent system. The addition of ethanol enhanced the solvent's ability to extract more polar components, thereby increasing the oil yield, as illustrated in Figure 1(b), which displayed a curved profile with a peak at 50% v/v ethanol in n-hexane. However, when the ethanol fraction became excessively high, the capacity of n-hexane to dissolve nonpolar oil constituents decreased, resulting in reduced oil yield (Figure 1a). These results are consistent with those reported by Efthymiopoulos *et al.* [31], who found that ethanol, being more polar than pentane, produced higher yields of coffee oil, although still lower than those obtained with n-hexane. In addition, the residual moisture content of the sample also appeared to affect extraction efficiency across solvents of varying polarity. Kondamudi *et al.* [32] noted that dichloromethane (polar) yielded slightly higher oil recovery than hexane (nonpolar). This effect was likely attributed to incomplete moisture removal during drying at 50 °C, similar to the present study, where sun-drying was employed. Consequently, higher solvent polarity was presumed to facilitate oil extraction from samples containing residual water [33].

Furthermore, the L/S ratio also significantly influenced oil yield from SCGs. As illustrated in Figures 1(b) and 1(c), increasing the solvent volume across various extraction times and ethanol concentrations consistently led to higher oil yields. A higher solvent volume generally facilitated greater solubilization of oil, resulting in higher recovery. Efthymiopoulos *et al.* [34] reported a similar trend, with yields increasing from 17.6 ± 0.96% to 24.6 ± 1.33% when the solvent ratio was raised from 4:1 to 9:1. Le *et al.* [35] likewise demonstrated that increasing the L/S ratio up to an optimal point improved oil extraction across different techniques. However, excessive solvent use was considered inefficient, as it increased both extraction costs and solvent waste generation.

3.2. Saponification Value of Oil Extracted from SCGs

The saponification value serves as an essential indicator of SCG oil quality, representing the mass of alkali (mg KOH/g oil) needed to completely saponify a given weight of oil or fat. This parameter was measured by treating the sample with an excess of KOH solution, followed by titration of the remaining unreacted alkali with a standard acid, using phenolphthalein as the endpoint indicator. The quantity of alkali consumed corresponded to the number of fatty acyl groups present in 1 g of oil, meaning that the saponification value was closely linked to the average chain length of the triacylglycerols. Oils dominated by long-chain fatty acids typically displayed lower saponification values, whereas higher values were associated with triacylglycerols containing shorter acyl chains. Consequently, the saponification value provides a valuable estimation of the fatty acid chain-length distribution in SCG oil [36].

According to the Indonesian National Standard (SNI 7431:2015), the saponification value of vegetable oils, including SCG oil, should range from 180 to 265 mg KOH/g [37]. As presented in Table 3, the saponification values obtained in this study varied from 167.73 to 181.83 mg KOH/g, with an average of 173.76 mg KOH/g. The relatively narrow range indicated that the extraction process yielded oil with a fairly uniform fatty acid composition, although slight variations occurred due to differences in extraction conditions. Nevertheless, most of the obtained values did not fully comply with the SNI 7431:2015 standard. The highest value, 181.83 mg KOH/g, was obtained under extraction conditions of 50% ethanol in n-hexane, an extraction time of 45 minutes, and a L/S ratio of 25:1. The relatively low saponification values suggested that the oil from SCGs was largely composed of long-chain triglycerides [38].

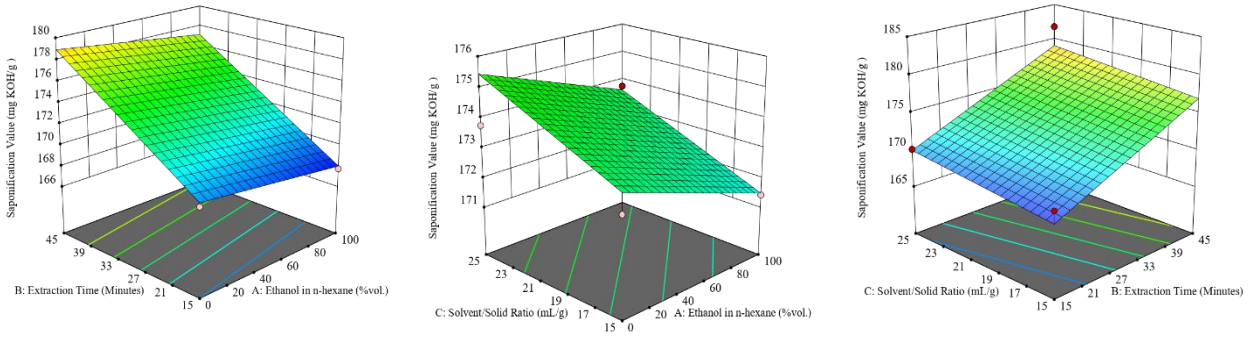
Table 3. Saponification Values of SCG Oil Extracted by Soxhlet Method

Ethanol concentration in n-hexane (%)	Extraction time (minutes)	Solvent-to-solid ratio (mL/g)	Saponification values (mg KOH/g)
100	45	20:1	176.08
100	15	20:1	167.73
0	30	15:1	172.63
0	15	20:1	169.44
100	30	25:1	173.76
50	45	25:1	181.83
100	30	15:1	171.42
50	15	15:1	169.44
0	30	25:1	173.76
50	15	25:1	170.12
0	45	15:1	178.29
100	45	20:1	176.08

Based on Figure 2, the saponification value of SCG oil extract was influenced by extraction time, ethanol concentration in n-hexane, and the L/S ratio. The 3D response surfaces indicated that the saponification value generally increased with longer extraction times and larger L/S ratios, whereas higher ethanol concentrations tended to reduce the value. As illustrated in Figures 2(a) and 2(c), the shift in color from blue (lower values) to yellow (higher values) reflected the rise in saponification value with extended extraction time.

Although longer extraction durations can enhance oil yield, they may also promote partial thermal degradation of triglycerides into FFA due to prolonged exposure to elevated temperatures, which can contribute to higher saponification values [39]. However, saponification value is fundamentally governed by the average fatty acid chain length and molecular weight of triglycerides, where shorter-chain lipids produce higher values. Therefore, the direct impact of extraction time on the saponification value was less significant compared to factors such as temperature and alkali concentration during the saponification analysis.

The saponification value was also strongly affected by the polarity of the solvent system employed for SCG oil extraction. As shown in Figures 2(a) and 2(b), increasing the ethanol content in n-hexane resulted in a decline in the saponification value. Generally, higher solvent polarity is associated with greater extraction of short-chain fatty acids, including FFAs, which tends to increase the saponification value [33]. Chatepa dan Masamba [40] similarly found that pumpkin seed oil extracted with polar solvents (acetone, ethanol) showed higher saponification values than oil extracted with nonpolar solvents (hexane, cyclohexane, petroleum ether). However, this effect can vary depending on the oil type. A high proportion of polar solvent may also promote the co-extraction of non-lipid polar components, which can lower the measured saponification value. This observation is consistent with Thao *et al.* [41], who found that Vietnamese tea seed oil exhibited its highest saponification value when extracted with a less polar ethanol - ethyl acetate mixture (1:3 v/v), whereas pure ethanol (a more polar solvent) produced the lowest value.



(a) **(b)** **(c)**

Figure 2. 3D surface plot depicting the effect of extraction variables on the saponification value of SCG oil: (a) interaction between extraction time and ethanol concentration in n-hexane at a L/S ratio of 20:1 (mL/g); (b) interaction between ethanol concentration in n-hexane and L/S ratio at a extraction time of 30 minutes; and (c) interaction between L/S ratio and extraction time at an ethanol concentration of 50% v/v in n-hexane.

In addition, the L/S ratio also had a marked impact on the saponification value of SCG oil. Increasing this ratio provided a greater volume of solvent, which improved the extraction efficiency for oil constituents, particularly FFA and short-chain fatty acids, that contributed to the saponification value. Consequently, the effect of the L/S ratio was indirect, facilitating greater extract recovery. This phenomenon accounted for the observed increase in saponification value at higher ratios (Figures 2(b) and 2(c)). In contrast, lower ratios restricted solvent availability, thereby reducing the amount of extracted compounds and resulting in lower saponification values. Figures 2(b) and 2(c) also demonstrated that these changes were relatively linear, indicating a consistent effect of L/S ratio on saponification value across increasing and decreasing trends.

3.3. Acid Value of Oil Extracted from SCGs

The acid value served as a key indicator for assessing the quality of oils and fats. This parameter represented the amount of FFAs formed as a result of triglyceride hydrolysis or degradation during extraction and storage [42]. Beyond FFAs, the acid value could also rise due to the presence of other acidic compounds, including acetic, propionic, vanillic, hydroxybenzoic, and syringic acids, as well as hydroxy-methyl-furfural and various phenolic compounds [43]. The analysis results showed that the acid value of SCG oil ranged from 4.30 to 9.21 mg KOH/g (Table 4). According to the Indonesian National Standard (SNI) 7431:2015, vegetable oils were considered acceptable when their acid value was < 4 mg KOH/g [37]. The acid values of the SCG oil extracted in this study did not meet the required standards. Therefore, further processing, such as purification or refining, is necessary to enhance the oil quality and ensure it complies with established specifications.

Based on Table 4, the acid value of SCG oil obtained through the Soxhlet method ranged from 4.30 to 9.21 mg KOH/g, with an average of 7.82 mg KOH/g. The lowest acid value of 4.30 mg KOH/g was observed when pure ethanol was used for 15 minutes at a L/S ratio of 20:1. In contrast, the highest value of 9.21 mg KOH/g was observed with pure ethanol extraction for 30 minutes at a L/S ratio of 15:1. These findings highlight that ethanol concentration, extraction duration, and L/S ratio significantly affected the acid value of SCG oil.

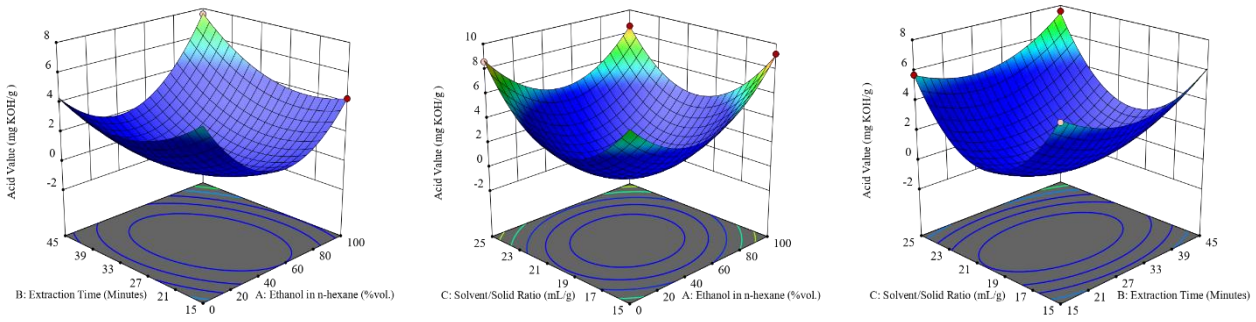
Based on Figure 3, the acid value of SCG oil was affected by the combination of extraction time, ethanol concentration in n-hexane, and L/S ratio. Figures 3(a)–(c) show similar patterns for these variables, forming a valley-shaped curve with the lowest acid value observed at an extraction time of 30 minutes, 50% ethanol concentration in n-hexane, and a L/S ratio of 20:1. This trend suggested that in the early stages of extraction, short-chain fatty acids such as FFAs were extracted relative to triglycerides, which mainly contribute to acid value [34]. As the extraction time progressed, a larger proportion of triglycerides was extracted, causing a

decline in acid value, depicted as the valley base in Figures 3(a) and 3(c). Further prolongation of the extraction time (up to 45 minutes) potentially caused the hydrolysis of triglycerides, leading to an increase in the acid value. Similar behavior was reported by Agustina *et al.* [44], who observed rising acid values with longer extraction times for oil recovered from cooling pond wastewater.

Table 4. Acid Value of SCG Oil Extracted by Soxhlet Method

Ethanol concentration in n-hexane (%)	Extraction time (minutes)	Solvent-to-solid ratio (mL/g)	Acid values (mg KOH/g)
100	45	20:1	7.59
100	15	20:1	4.30
0	30	15:1	7.11
0	15	20:1	5.84
100	30	25:1	8.60
50	45	25:1	7.59
100	30	15:1	9.21
50	15	15:1	5.84
0	30	25:1	8.60
50	15	25:1	5.73
0	45	15:1	7.82
100	45	20:1	7.59

On the other hand, the acid value also increased with higher concentrations of ethanol or n-hexane in the solvent mixture. As depicted in Figures 3(a) and 3(b), the lowest acid values were observed with a balanced ethanol – n-hexane mixture (50:50%). An increase in the ethanol fraction had a more pronounced effect on raising the acid value compared to increasing n-hexane, as indicated by the contour color shift from green to red in Figure 3(b). This finding was consistent with Al-Hamamre *et al.* [29], who reported that SCG oil extracted with polar solvents like ethanol and isopropanol tends to have higher acid values compared to those obtained with nonpolar solvents such as pentane and hexane. Furthermore, the acid values measured in this study were relatively lower than those published by Caetano *et al.* [45] and Hanif *et al.* [30] despite using the same solvent, n-hexane, indicating an improvement in the quality of SCG oil and approaching the standards set by SNI 7431:2015.



(a) **(b)** **(c)**

Figure 3. 3D surface plot depicting the effect of extraction variables on the acid value of SCG oil: (a) interaction between extraction time and ethanol concentration in n-hexane at a L/S ratio of 20:1 (mL/g); (b) interaction between ethanol concentration in n-hexane and L/S ratio at a extraction time of 30 minutes; and (c) interaction between L/S ratio and extraction time at an ethanol concentration of 50% v/v in n-hexane.

Additionally, Figures 3(b) and 3(c) showed that the acid value of the extracted SCG oil initially decreased as the L/S ratio increased from 15:1 to 20:1. However, the acid value rose again when the L/S ratio increased further from 20:1 to 25:1, creating a valley-shaped response with a minimum at a ratio of 20:1. The reduction in acid value at this ratio is likely due to a higher extraction of triglycerides, which lowers the relative amount of FFA in the oil. Conversely, under conditions of excessive solvent, the solubility of FFAs increased, leading to a subsequent rise in the acid value.

3.4. FFA Content of Oil Extracted from SCGs

FFAs represent the fraction of fatty acids that are not bound in the form of triglycerides. The FFA content in oil needed to be maintained as low as possible, as higher levels could accelerate oxidation and reduce oil stability [29], [46]. According to the Indonesian National Standard (SNI) 7431:2015, vegetable oils are considered acceptable when the acid value is below 4 mg KOH/g, corresponding to an FFA content of less than 2% [37]. Additionally, FFA content influences the oil's physical properties, such as viscosity. Al-Hamamre *et al.* [29] reported that increases in FFA were linearly correlated with higher kinematic viscosity of the oil. Therefore, controlling FFA levels is vital for assessing the quality of extracted oils.

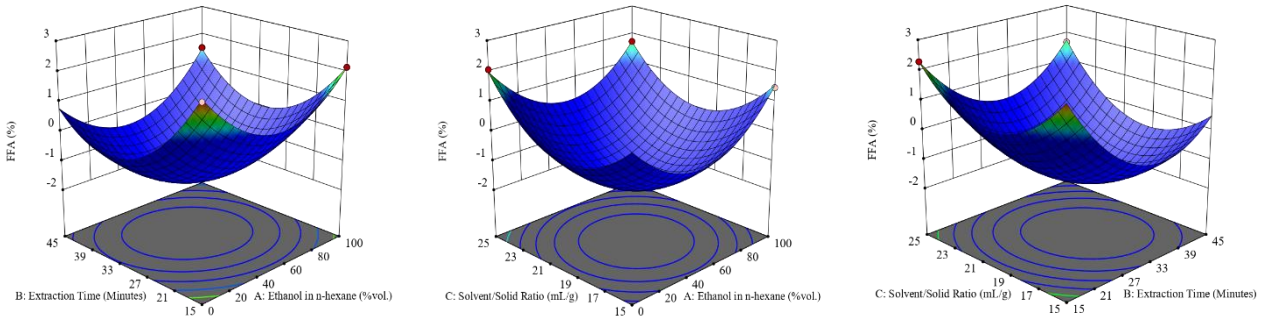
Based on Table 5, the FFA content of SCG oil extracted through the Soxhlet method ranged from 1.06% to 2.53%, with an average value of 2.05%. The lowest FFA value of 1.06% was achieved using pure n-hexane (0% ethanol) for 30 minutes at a L/S of 15:1. Conversely, the highest FFA value of 2.53% was recorded under extraction conditions involving either a 50:50 ethanol – n-hexane mixture or pure n-hexane, with L/S ratios of 15:1 and 20:1, and an extraction time of 15 minutes. Despite these variations, most samples met the quality standards for SCG oil as defined by SNI 7431:2015, suggesting that the oils extracted were of acceptable quality.

Table 5. FFA Content of SCG Oil Extracted by Soxhlet Method

Ethanol concentration in n-hexane (%)	Extraction time (minutes)	Solvent-to-solid ratio (mL/g)	FFA (%)
100	45	20:1	1.51
100	15	20:1	2.15
0	30	15:1	1.06
0	15	20:1	2.53
100	30	25:1	1.72
50	45	25:1	1.67
100	30	15:1	1.47
50	15	15:1	2.53
0	30	25:1	2.06
50	15	25:1	2.29
0	45	15:1	1.56
100	45	20:1	1.51

Based on Figure 4, the FFA content of SCG oil extracts was influenced by the interaction among ethanol concentration in n-hexane, extraction time, and L/S ratio. As shown in Figures 4(a) and 4(c), extending the extraction time from 15 to 30 minutes led to a reduction in FFA content, whereas further prolonging the extraction to 45 minutes caused an increase. This trend was likely caused by enhanced hydrolysis of the oil, which released additional FFA [47], [48]. These findings align with those of Zamanhuri *et al.* [49], who identified increased FFA content in crude palm oil after extraction times exceeding 30 minutes. The FFA content reflects the degree of oil hydrolysis, potentially facilitated by water vapor during extended heating as well as lipolysis of triglycerides, where water molecules in the SCG matrix cleave ester bonds in triglycerides to yield glycerol and FFAs [50].

The FFA content in SCG oil extracts was also influenced by the ethanol concentration in n-hexane, as shown in Figures 4(a) and 4(b). Extraction with either pure n-hexane (0% ethanol) or pure ethanol (100%) resulted in relatively high FFA levels, whereas a balanced ethanol–n-hexane mixture (50:50% v/v) tended to produce the lowest FFA content. This finding aligns with the study by Amin and Abdallah [51], who reported that rice bran oil extraction generated high FFA content when using either nonpolar solvents (petroleum ether and n-hexane) or polar solvents (methanol and ethanol), while the lowest FFA was obtained with isopropanol, which exhibits intermediate polarity. Similarly, Gebreegziabher *et al.* [52] reported that dichloromethane, a solvent with moderate polarity, yielded the lowest FFA content during *Argemone mexicana* seed oil extraction compared to nonpolar solvents such as n-hexane and petroleum ether.



(a) **(b)** **(c)**

Figure 4. 3D surface plot depicting the effect of extraction variables on the FFA content of SCG oil: (a) interaction between extraction time and ethanol concentration in n-hexane at a L/S ratio of 20:1 (mL/g); (b) interaction between ethanol concentration in n-hexane and L/S ratio at a extraction time of 30 minutes; and (c) interaction between L/S ratio and extraction time at an ethanol concentration of 50% v/v in n-hexane.

Furthermore, the L/S ratio also affected the FFA content in SCG oil extracts. As shown in Figures 4(b) and 4(c), FFA content decreased as the ratio increased from 15:1 to 20:1, then increased again when the ratio was raised from 20:1 to 25:1, forming a surface response with a minimum at a 20:1 ratio. Under this condition, the extracted SCG oil exhibited FFA content within the vegetable oil quality standard set by SNI 7431:2015 [37]. Additionally, the FFA levels observed in this study were lower compared to previous research using similar solvent systems like ethanol and n-hexane , indicating improved extraction outcomes [29], [53].

3.5 SCG Oil Extraction Optimization

The SCG oil extraction experiments conducted using the Soxhlet method were designed according to a BBD with three independent variables: ethanol concentration in n-hexane (A), extraction time (B), and L/S ratio (C). The experimental design matrix and the corresponding responses, including oil yield, saponification value, acid number, and FFA content, are presented in Tables (2) – (5). To optimize the extraction process, the levels of variables A, B, and C were systematically adjusted with the objectives of maximizing oil yield and saponification value while minimizing acid value and FFA content. Both the main effects and interactions of these factors were considered. Relationships between independent variables and response outcomes were modeled using linear and polynomial regression analyses to estimate empirical coefficients, enabling the prediction and optimization of extraction conditions. The empirical equations obtained for oil yield (Y), saponification value (S), acid number (AN), and FFA content are presented in Eqs. (5) – (8):

$$Y = 12{,}9539 - 0{,}0060A + 0{,}2071B + 0{,}1412C \quad (5)$$

$$S = 160{,}9506 - 0{,}0181A + 0{,}3038B + 0{,}2149C \quad (6)$$

$$AN = 86{,}3750 - 0{,}1497A - 0{,}6270B - 7{,}4634C + 0{,}0016AB - 0{,}0021AC + 0{,}0051BC + 0{,}0015A^2 + 0{,}0080B^2 + 0{,}1867C^2 \quad (7)$$

$$FFA = 35{,}2275 - 0{,}05918A - 0{,}6259B - 2{,}5917C + 0{,}0004AB - 0{,}0008AC + 0{,}0047BC + 0{,}0006A^2 + 0{,}0078B^2 + 0{,}0636C^2 \quad (8)$$

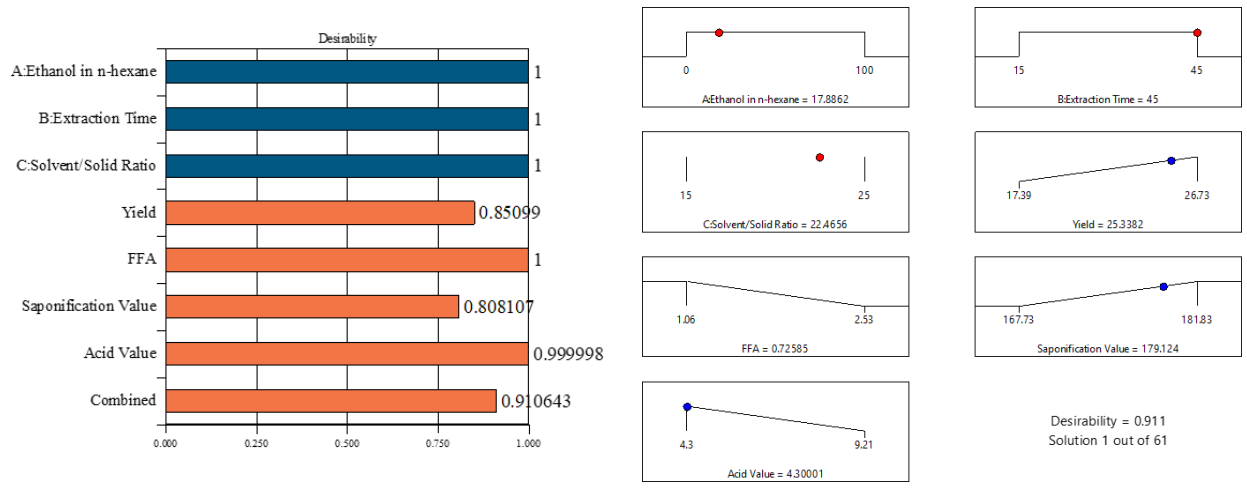
where Y represents the SCG oil yield (%), S denotes the saponification value (mg KOH/g), AN is the acid number (mg KOH/g), and FFA corresponds to the free fatty acid content (%); meanwhile, A, B, and C represent the ethanol concentration in n-hexane (% v/v), extraction time (minutes), and L/S ratio (mL/g), respectively.

The results showed that SCG oil yield (Y) and saponification value (S) increased with longer extraction times and higher L/S ratios. In contrast, raising the ethanol concentration in n-hexane, a more polar solvent, generally resulted in lower values for both responses. These results are consistent with previous research on ultrasonic-assisted SCG extraction, which identified extraction time and L/S ratio as key factors significantly affecting oil yield [54], [55].

For the acid number (AN) and free fatty acid (FFA) content, the independent variables influenced the responses through linear, interaction, and quadratic effects. In particular, the interactions between ethanol concentration and extraction time, as well as between extraction time and the L/S ratio, played a significant role in increasing both AN and FFA contents. Quadratic terms (A^2, B^2, C^2) also revealed non-linear relationships that tended to elevate AN and FFA. However, such effects were undesirable, as the acid number must meet the SNI quality standard of < 4 mg KOH/g or FFA < 2% [37]. Therefore, the determination of optimal extraction conditions required careful consideration of these quality limits, ensuring that the extraction process produced not only a high oil yield but also oil that met established quality standards.

To determine the optimal conditions, each optimization criterion, encompassing four response variables, was assigned equal importance. The analysis was performed within the range of the three independent variables outlined in Table 1. The objective was to maximize both the oil yield and saponification value while minimizing the acid number and FFA content. Optimization was evaluated using the desirability function, calculated individually for each response and collectively for the overall objectives.

Desirability values ranged from 0 to 1, increasing as the independent variables and responses approached the optimization targets and decreasing as they deviated from them. The optimization results are shown in Figure 5. Figure 5(a) presents the individual desirability scores for independent variables, responses, and overall desirability. Each independent variable scored 1, indicating that the optimization was conducted within the ranges specified in Table 1. For the responses, the desirability scores were 0.8510 for oil yield, 0.8081 for saponification value, and 1 for both acid number and FFA. Overall, these scores indicate a favorable optimization outcome.



(a) **(b)**

Figure 5. Optimal conditions for Soxhlet extraction of SCG oil: (a) Pareto desirability plot, (b) Ramp plot of optimal points for independent variables

Figure 5(b) presents the optimum conditions in a ramp plot, where all independent variables and responses were optimized simultaneously, achieving the highest overall desirability of 0.911. The optimum parameters were an ethanol concentration of 17.89% v/v in n-hexane, an extraction time of 45 minutes, and a L/S ratio of 22.47 mL/g. At these conditions, the SCG oil yield reached 25.34%, the saponification value 179.12 mg KOH/g, the acid number 4.30 mg KOH/g, and the FFA 0.73%. Overall, the SCG oil characteristics at these optimum conditions complied with the SNI 7431:2015, except the saponification value, which was marginally below the minimum standard (~0.49%) [37].

4. CONCLUSION

The experimental results demonstrated that the optimal conditions for SCG oil extraction by the Soxhlet method were achieved at an ethanol concentration of 17.89% v/v in n-hexane, an extraction time of 45 minutes, and a L/S ratio of 22.47 mL/g. Under these parameters, the oil yield was 25.34%, with a saponification value of 179.12 mg KOH/g, an acid number of 4.30 mg KOH/g, and an FFA content of 0.73%. Utilizing a solvent mixture at these optimized proportions enhanced the oil yield and reduced the acid number and FFA compared to using exclusively polar or nonpolar solvents. Both extraction time and L/S ratio positively influenced oil yield and saponification value, while optimizing acid number and FFA to acceptable levels. Despite a slightly lower saponification value compared to the SNI 7431:2015 standard (~0.49%), the optimized SCG oil shows strong potential as an alternative vegetable oil source for food, pharmaceutical, cosmetic, and renewable energy applications.

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