

# **Green Technology for Extracting Coffee Oil and Biodiesel Production: An Application of Spent Coffee Ground Management Approach**

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## **Abstract**

Spent Coffee Ground (SCG) was studied as a potential source of oil feedstock for biodiesel production as an alternative waste utilization instead of disposed of as municipal waste. This study using microwave-assisted extraction (MAE). The optimal condition of MAE obtains of n-hexane (4v/v) as extraction solvent, microwave power 460 W, solvent to the material ratio eight mL/g, extraction temperature 72°C, and time 42 min. The oil yield of 24.70 % reaches under optimum conditions. Free fatty acids (FFA) of the extracted oil are converted into fatty acid methyl ester (FAME) by base-catalyzed esterification. The study investigates the effect of temperature, FFA ratio to methanol-CaO solution, and reaction time either. With one step alkali-catalysed transesterification, oil conversion to methyl ester can be enhanced from approx. 42–75% by increasing the reaction time, the reaction temperature, and the Methanol-CaO/FFA ratio. However, a complete oil conversion can only achieve by utilizing a two-steps transesterification process. The results showed that 4.4 L of biodiesel was produced from 1 kg of spent coffee ground. As such, this study expected this study to lead to the paradigm shift in spent coffee ground management with the ultimate target of zero-solid discharge highlights.

## Keywords

Spent coffee ground, microwaved extraction, coffee oil, biodiesel.

## 1. Introduction

Coffee is a drink that is widely consumed by people in the world, for some people drinking coffee is not just a demand for taste but has become part of their lifestyle. Modern society makes coffee shops a place to carry out various social activities such as: meetings, business affairs, discussions, socialization, and rest. Murthy and Naidu (2012) reported that for each tonne of coffee it produces 650 Kg of Spent Coffee Ground (SCG). Mussattu (2011) reports that every year coffee entrepreneurs around the world produce 6 billion tonnes of SCG and an average of 1 kg of coffee produces 2 kg of wet SCG. SCG contain 11-20% oil which is quite high (Phimsen et al, 2016, Brandon et al, 2020, Somnuk et al, 2017). By assuming 16% wt% of coffee oil in SCG, the use as biodiesel feedstock would be able to produce 0.9 million tonnes of biodiesel equivalent to 26 million tonnes or 3.5% of the total fuel supply (Phimsen et al, 2016). Therefore, the large amount of solid waste generated from SCG which can be used for valuable product synthesis requires the development of waste management plan to ensure that it is properly utilized.

Until now, Soxhlet extractor methods were reported to extract coffee oil from SCG with solvent as hexane, heptane, iso propanol, acetone, ethanol (Liang et al, 2018, Al-Hamamre et al, Udin et al, Chokchai et al, 2018). The solvent used to extract coffee oil is a toxic, volatile, harmful (Sara and Jose, 2018). Recently supramolecular solvent has also been proposed. Among the solvent, NADES have been explored for the extraction of natural product, with the objective to reduce toxic waste, to improve selectivity and extraction efficiency (Choi et al, 2019) NADES have excellent properties such low melting point, wide range polarities and high solubility of various compounds, especially poorly water-soluble compounds (Liu et al, 2018).

Conventional extraction such as Soxhlet extraction, maceration, solid liquid extraction, require a long time and high temperature, which in this case does not fit the concept of green chemistry. Currently being developed Microwave Assisted Extraction (MAE), Ultrasound Assisted Extraction (UAE) and Supercritical Fluid Extraction (SFE) which composes to green chemistry criteria (Mena et al, 2019). Yiping et al., (2020) used MAE, UAE and used NADES solvent to extract cumin oil from *Cuminum cyminum L* Seed, and Qiyu et al., (2020) studied was designed to integrate ultrasonic-microwave assisted extraction (UMAE) with ethanol, a green solvent, to extract green coffee oil (GCO) from green coffee beans. This study demonstrated that UMAE with ethanol is a rapid, efficient, and environmentally friendly green technique for the extraction of green coffee oil. In this paper was the development of a simple, economic, and eco-friendly methodology for the extraction of coffee oil from SCG with MAE using NADES solvent. Coffee oil were characterized using GCMS.

### 1.1 Research Objectives

As we know, the application of MAE with NADES solvent of coffee oil from SCG has not been reported. Therefore, the main objective of this study was to demonstrate the optimization of coffee oil yields from NADES solvent using three parameters, are : (i) the extraction time, (ii) formulation NADES and (iii) the ratio of SCG-to solvent, (iv) transesterification coffee oil with sulphuric acid catalyst.

## 2. Literature Review

### 2.1 Green Technology extraction

Conventional natural extraction methods generally use organic solvents that have negative impacts such as the presence of toxic residues, chemical changes in extract compounds, and waste that is difficult to degrade. Therefore, the need for a safer and more environmentally friendly extraction method with the use of safe solvents is increasing. Extraction methods that are more environmentally friendly (green extraction) such as Ultrasound Assisted Extraction (UAE) and Microwave Assisted Extraction (MAE) are expected to be a solution (Mena et al., 2019). Green extraction is a separation process that focuses on reducing energy consumption, reducing solvent petroleum by considering the quality and safety of extraction (Chemat et al., 2019).

#### 2.1.1 Microwave Assisted Extraction (MAE)

MAE is an extraction method that utilizes microwaves to extract compounds from natural materials. In a microwave there is an electronic vacuum called a magnetron which produces microwaves. These microwaves are emitted onto a metal wire. If there is a microwave, this stirrer will rotate, so that the microwave will be evenly distributed in the MAE space. The molecules present in the solvent and in the extracted sample will absorb the microwaves, and move and rotate, thus producing heat. The heat produced functions as a heating agent in SCG, so that the cell walls will break and the coffee oil in them can freely come out of the matrix (SCG). The advantages of MAE are the shorter time

required for extraction, less solvent used, higher yields, faster extraction, and lower costs (Sabrina et al, 2019). Pan et al (2020) used MAE with NADES solvent to extract antioxidant component from *O fragrans* flower.

The optimisation of extraction conditions as a microwave power 497.12-Watt, extraction time 59.03 s and solid liquid ratio 31.14 mL/g. Gomez et al (2019) extract of soluble sugars from ripe banana puree with NADES in combination with MAE. Through optimization of reaction conditions, the highest quality of soluble sugar 106.9 g/100 g was obtained with maleic acid: beta alanine: water 1:1:3 molar ratio at 25°C, and extract time 30 min. This result was compared with the extract with ethanol and water, the NADES was clearly more effective than these two traditional extraction solvents. Elena et al (2020) study was the application of NADES to the green extraction of total phenol and anthocyanin from *H. sabdariffa* using a microwave-assisted extraction. Results set the maximum extraction conditions within the experimental domain at liquid-solid ratio of 1:30 mg mL and contain 55% of water. Extractions under these conditions were performed to carry out a comprehensive characterization of the extract. Anthocyanin concentrations were 4.70 and 5.73 mgg<sup>-1</sup> for delphinidin-3-sambubioside and cyaniding-3-sambubioside.

## 2.2 Natural Deep Eutectic Solvent (NADES)

NADES is an environmentally friendly solvent containing a compound that function as an hydrogen bond acceptor (HBA) with electric charge protected through a complex formed by hydrogen bond donator (HBD) with a specific molar ratio (Vanda et al, 2018), The term NADES was first used by Choi et al (2011) to represent a eutectic mixture of two or three natural compounds, namely as HBA (choline chloride, betain, the other amino acid and amines compound), while as HBD (are organic acids, citric acid, maleic acid, acetic acid), carbohydrates (glucose, fructose, sucrose) and water. An obstacle to the extractive efficiency of NADES is their high viscosity, which can be reduced by adding a certain percentage of water or by working at higher temperatures (Mandal et al, 2007). NADES exhibits biodegradability, low toxicity, low cost, non-flammability and environmentally, is used to extract natural bioactive compounds (Gomez et a, 2019). NADES can be classified into five categories to the properties of the compounds used in the synthesis such as: (i) type of ionic liquid, containing acid and bases; (ii) type of ionic liquid, containing acids and bases; (iii) neutral type, formed by sugar or by sugars and polyalcohol's; (iv) neutral acid type, synthesized with sugar or polyalcoholic and organic acids; (v) neutral-basic type, with sugars or polyalcohol's and organic bases and (vii) amino acid types, with amino acids and sugars or organic acids. Table 1 below shows an example of NADES used to extract natural ingredients.

Table 1. NADES solvent for extract natural product

No	HBD	HBA	Water	Molar ratio	Temp	Time (min)
1	Glucose	Choline chloride	Water	2: 5:5	70	60
2	Glucose	Choline chloride	Water	2:4 :4	70	120
3	Glucose	Choline chloride	Water	2:3 :3	70	120
4	Citric acid	Choline chloride	Water	1:1:2	60	60
5	Malic acid	Choline chloride	Water	1:1:2	60	90
6	Malic acid	Glucose	Water	1:1:2	60	60
7	Urea	Glucose	Water	2:2:2	70	60
8	Urea	Fructose	Water	2:1:2	70	60
9	Fructose	Choline chloride	Water	2:5:5	70	60
10	Fructose	Choline chloride	Water	2:3:3	70	60

Source: Gomez e al., (2019)

## 3. Methodology

### 3.1 Preparation NADES solvent

Formula for the NADES solution as shown in the Table 2.

Table 2. Composition of NADES solvent

No	HBA	HBD	Molar ratio	Water (%)
1	Choline clorida	Lactic acid	1:1	10
2	Choline Clorida	Lactic Acid	1:2	10
3	Choline Clorida	Lactic acid	1:3	10
4	Fructose	glucose	1:1	28
5	Fructose	sucrose	2:1	32
6	Citric acid	sucrose	1:2	19

Mix the components of NADES according to the specified mol ratio in a glass bottle with magnetic stirrer, the put in a beaker glass and fill the beaker glass with water. Heat using a hot plate at 70<sup>o</sup> C until a clear solution formed. Then add distilled water according to the formula that has been set, stirring until homogeneous. After the cold, store it in the refrigerator and leave it for 3 days. If the solution remains clear, does not form a precipitate, or crystals, it means that the NADES solution has been formed, The NADES solvent obtained was then put into a bottle.

### 3.2 Extract Coffee Oil

The MAE consists of a distillation flask, a condenser, statif and clamps. The MAE used is the Panasonic NN-ST342M type with a maximum power source of 800-Watt. The outside size of the oven is 485 mm x 287 x 400 mm and the size of the oven chamber 315 mm x 227 x349 mm. Pureed dry SCG using a blender. SCG coffee powder is weighed as much as 20 grams then put it in the distillation flask, add NADES solvent according to the volume that has been determined. Put the distillation flask into the MAE. For extraction, use microwave power 200, 400, 600 and 800 watts, extraction times of 4, 6, 8, and 10 minutes and ratio solvent /SCG 2: 1, 4: 1, 6: 1, and 8: 1 g / g. After extraction, coffee oil and SGC were separated by filtration, then the coffee oil is purified.

### 3.3 Purification Coffee Oil

Coffee oil centrifuge for 20 minutes, left until a complete precipitate is formed. Coffee oil Take 50mL, put in a 150 mL beaker glass and 1 g of bio-flocculant (Tanfloc). Stirrer for 20 minutes, then coffee oil separated by decantation, evaporated with rotary evaporator until the water runs out. Weigh the coffee oil and calculate the yield using the equation.

$$\text{Yield} = \frac{\text{Weight coffee oil(g)}}{\text{Weight SCG(g)}} \times 100 \%$$

Store the coffee oil in a bottle and label it.

### 3.4 Analysis Quality Coffee Oil

Coffee quality analysis includes density measured with a picometer, viscosity measured with viscometer, free fatty acid analysed by titration method and compound distribution in coffee oil analysed by GCMS.

### 3.5 Synthesis Biodiesel

A 250 mL of 3-necked round bottomed flask was employed to carry out trans-esterification reaction. The centered neck was utilized to insert a mechanical stirrer, another side neck was fitted with a water-cooled condenser, and the third one neck was fitted with a temperature indicator. A mechanical stirrer was employed to stir the reaction mixture. 50.0 g of coffee oil, 150,0 g methanol and CaO (1% of weight of oil) are refluxed for 3 hours at 65<sup>o</sup>C while continuing stirrer. After chilling, the biodiesel is purified with column chromatography. The resulting mixture obtained was added into chromatographic column packed with 10 g silica gel and 60 g alumina as eluent 50 mL a mixture of hexane: ethyl acetate (19:1). To these biodiesel -rich phase were added 200 mL hexane and left at rest for additional 15 min, the upper phase was separated and added to previously obtained fraction. Biodiesel was washed with 50 mL distilled water (to remove residues of CaO and methanol) several times until the water pH was 7, then heated with an oven at ± 80<sup>o</sup>C for 24 hours. The sample was centrifugated at 5000 rpm for 1 minute to separate the remaining impurities. Fraction methyl ester analyse with Gas Chromatography–Mass Spectroscopy (GC-MS) Shimadzu QP 5000 using column type Agilent 19091S-105: 3409.48779 HP-5MS,split mode and heater are set at 280<sup>o</sup>C. Sample injected at GC and initial temperature for 5 min at 50<sup>o</sup>C with a rate of 10<sup>o</sup>C /min and split ratio 100:1 and run time for 30 min.

#### 4. Data Collection

Data obtained through experiment in a chemistry laboratory Universitas Negeri Jakarta. The data observed were Stability NADES solvent, Effect time extraction on yield coffee oil, Effect microwave power on yield coffee oil, Effect of NADES/SCG ratio on yield coffee oil, Quality of coffee oil : density, viscosity, FFA, Compound distribution in coffee oil and compound distribution in biodiesel

#### 5. Results and Discussion

##### 5.1 Stability NADES Solvent

The stability test results, show that the NADES solvent remains a transparent liquid, no sediment was formed, nor formed crystals and wastable until the 7<sup>th</sup> day. The effect of temperature on the stability of NADES shows that the higher the %Wt. of water in NADES, the higher the decomposition temperature of NADES. Table 3 below shows the decomposition of NADES.

Table 3. Decomposition NADES

No	HBA	HBD	Molar ratio	%water	physical	Decomposition (t <sup>o</sup> C)
1	Fructose	glucose	1:1	28	liquid	128
2	Fructose	sucrose	2:1	32	liquid	131
3	Citric	sucrose	1:1	19	liquid	121
4	Choline Chloride	Lactic acid	1 :3	15	liquid	100

In this study, the coffee oil was extracted using NADES Choline Chloride: lactic acid: water with molar ratio 1:3:10

##### 5.2. Effect of microwave power

The results of the power effect on coffee yield are shown in Figure 1 below:

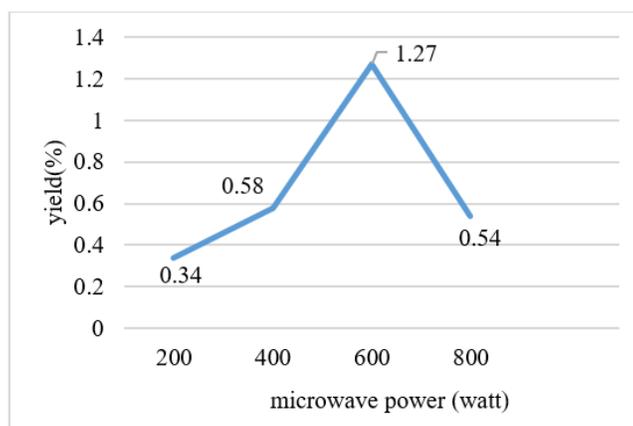


Figure 1. Effect of microwave power on the yield coffee oil

From Figure 1, we can identify that the greater the power used from 200 to 560 watt, the coffee oil yield up to 1.27%, but with increase radiation power of 800 Watt, the yield decreases. This indicates that the increase in power is proportional to mass transfer until it reached a certain point, namely the optimum yield is obtained. The microwave breaking down of plant cell tissue and will control the amount of energy that will be received by the material to be converted into heat energy. It is this, heats energy that helps the diffusion process or the extraction of the oil extract from the material into the solvent (Mandal et al.). When the microwave hits polar molecules', such as water, the waves will be absorbed, and the polar molecules will vibrate strongly, producing heat energy, detected by increase temperature, so the great of power, the greater the energy produced.

##### 5.3 Effect of Liquid/Solid Ratios

The effect of the ratio of the material to the solvent to produce the yield of the extract, as seen in Figure 2 below:

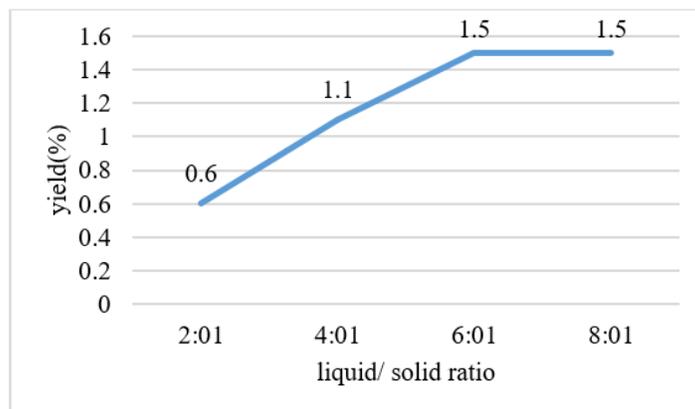


Figure 2. Effect of liquid/solid ratio on the yield coffee oil

As the liquid amount NADES with 10% water grew, the coffee oil yield increased rapidly. The experiment showed that the more amount of NADES were added, the more celluloses were dissolved. And more substances in the plant were released. Moreover, the yield of coffee oil was not significantly improved after the material ratio (g/g) was 6:1. This phenomenon may be due to that the NADESS have come into full contact with the SCG, dissolving almost all the cell walls. Thus, when the liquid-solid ratio was too large, the increase of yield was small. Considering the cost and extraction efficiency, 6:1 of liquid/solid ratio (g/g) was selected for the subsequent experiments.

#### 5.4 Effect of Extraction Time

The extraction time shows the length of contact time between solvent and SCG. The longer the contact time, the greater the yield produced, because the longer the extraction time, the greater the microwave radiation energy applied to the SCG, causing the water temperature distributed inside the SCG to increase.

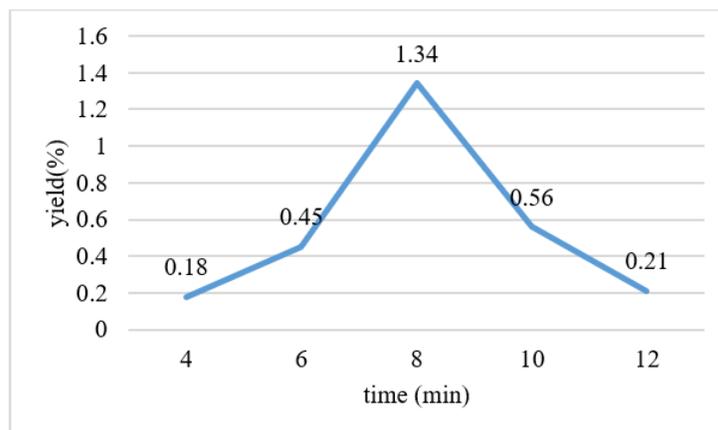


Figure 3. Effect of extract on the yield coffee oil

Figure 3 illustrates the yield increase lasts quite a long time for 4 to 8 min. After 8 min there was a decrease in yield due to the heating of the material has lasted so long that it degrades the thermolabile oil component. the cell wall and release the dye in the solvent.

#### 5.5. Quality Coffee Oil

The quality of coffee oil is indicated by density, viscosity and FFA. The values for the three parameters, as seen in Table 4 below:

Table 4. Quality coffee oil

No	Parameter	Value
1	Density(g/mL)	0.9149
2	Viscosity(cSt)	20,65
3	FFA (mg KOH/g)	24,68

The density and viscosity of the coffee oil produced have a high enough value. This shows that phenol content in coffee oil is quite a lot. The FFA content which is also quite high indicates the amount of long chain FFA in coffee oil. Figure 4 shows the GCMS spectrum of coffee oil. Based on this spectrum, coffee oil contains phenolics compound, fatty acid, and organic acid. The high level of these compounds in coffee oil makes coffee oil very potential to be used biodiesel through esterification and transesterification reaction.

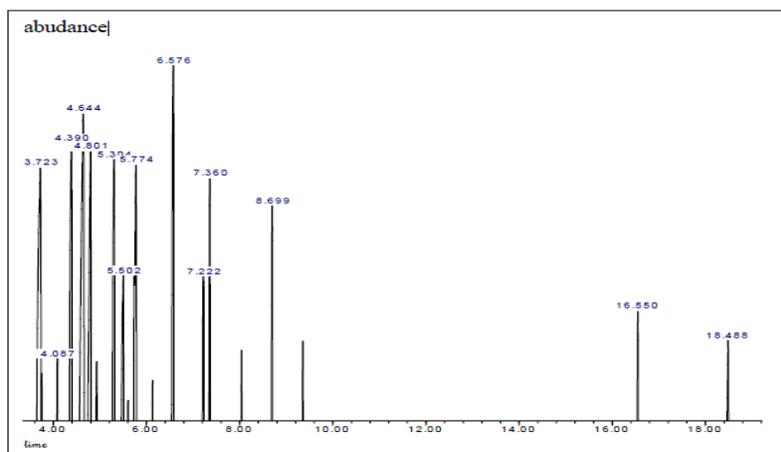


Figure 4. Spectra GCMS coffee oil

## 5.6 Biodiesel

Biodiesel was produced from coffee oil via esterification and transesterification reaction has a density as much as 865.65 mL/g, viscosity 1,23cSt and FFA 3.15 mgKOH/g. The results of GC-MS analysis, as seen in Figure 5 below:

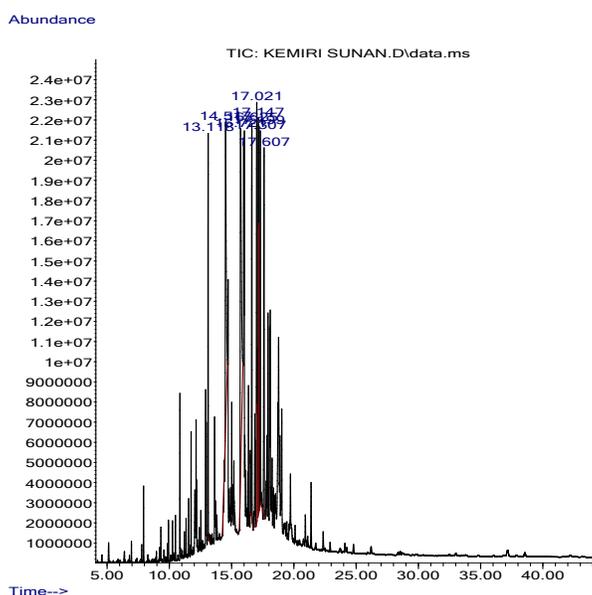


Figure 5. Chromatogram GC-MS coffee oil

Figure 5 shows the presence of four peaks with the retention times of 13.119, 14.515, 15.722 and 17.415 min, respectively. showing methyl myristate, methyl palmitate, methyl oleate, methyl cis-9,10, and methyl acrylate compounds. The existence of heating causes the oleic acid double bond to be oxidized to become an epoxy methyl cis-9,10 compound. This compound causes the high acid value of biodiesel, because this compound can be re-oxidized to form short chain fatty acids which can increase the value of the acid number.

## 6. Conclusion

In this study, extraction of coffee oil from a spent coffee ground powder was successfully extracted by using microwave-assisted extraction method. To obtain maximum results, the impact of several parameters was investigated such as microwave power, the ratio of material to the solvent and the duration of extraction. The results showed that the best condition required to achieve maximum yield 1.52% was the power of 600 W with material to solvent ratio 6:1, time of 8 min. Therefore, it can be concluded that the extraction of coffee oil from SCG with MAE is an efficient and efficient extraction method. The GCMS analysis showed that. Coffee oil contains high triglycerides and free fatty acid.

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