

BUKTI KORENPONDENS

**A Novel Process Production of Calophyllum Inophyllum Biodiesel
with Electromagnetic Induction**

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I. PAPER SUBMISSION

1. Lampiran Manuscript

A Novel Process Production of *Calophyllum Inophyllum* Biodiesel with Electromagnetic Induction

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Abstract: A novel method proposed in the production of *Calophyllum inophyllum* biodiesel has been investigated experimentally. In this study, we report the results of biodiesel processing with electromagnetic induction technology. The method used is to compare the results of *Calophyllum inophyllum* biodiesel processing between conventional, microwave and electromagnetic induction. The degumming, transesterification, and esterification process of the 3 methods are measured by stopwatch to obtain time comparison data. Characteristics of viscosity, density, and *Fatty Acid Metil Ester* (FAME) were obtained from testing of a *Gas Chromatography-mass Spectrometry* (GCMS) at the Integrated Research and Testing Laboratory, Gadjah Mada University, Yogyakarta. The results show that the biodiesel produced by this method satisfies the biodiesel standards and their characteristics are better than the biodiesel produced by conventional and microwave methods. The electromagnetic induction method also offers a fast and easy route to produce biodiesel with the advantage of increasing the reaction rate and improving the separation process compared to other methods. This advanced technology has the potential to significantly increase biodiesel production with considerable potential to reduce production time and costs.

Keywords: renewable energy, microwave, free fatty acid, crude oil

1. Introduction

In the past two decades, the energy crisis has encouraged the development of alternative energy by seeking renewable energy resources. Alternative energy sources such as biodiesel have been developed to replace diesel or fuel oil. Generally, biodiesel is a liquid fuel processed from different sources such as palm oil [1-4], soybean oil [5-10], jatropha [11-18], cottonseed oil [19-25], soursop seed oil [26], recycled cooking oils [27-29], animal fats [30-34], and other potential triacylglycerol-containing feed-stocks [35]. Vegetable oils are also known as triglycerides have a chemical structure composed of 98% triglycerides, and the remainder is diglycerides [36]. Biodiesel is treated with a mixture of fatty acid methyl esters with methanol or ethanol [37, 38].

One of the potential biodiesel plants is *Calophyllum inophyllum* or *Nyamplung* in Indonesia language. The benefits of *Calophyllum inophyllum* as biofuel are seeds that have a higher yield than other crops (jatropha 40-60%, palm kernel 46-54%, and *Calophyllum inophyllum* (40-74%)), and it does not compete with food interests [39]. In addition, the productivity of *Calophyllum inophyllum* seeds of 20 tons/ha/year is higher than jatropha (5 tons/ha/year), and palm (6 ton/ha/year) [40]. *Calophyllum inophyllum* has a very high oil of 75% with an unsaturated fatty acid content of about 71% [41]. It is processed or pressed in the form of yellow-green oil, similar to olive oil, aromatic and tasteless. It usually produces fruit twice a year to 100 kg with the oil content of about 18 kg [42]. *Calophyllum inophyllum* has a higher viscosity, but the capillary ability is lower than kerosene. *Calophyllum inophyllum* oil yields in the process are forged or pressed between 40-70% of the dry seed mass [43], and the degumming process is 62.80 - 65.89% [44].

Generally, biodiesel is processed by thermal or heating system. Biodiesel processing with conventional heating systems is the most widely used method. The conventional biodiesel processes are based on the use of high-power heating and magnetic stirring. Biodiesel processing begins with dry seed pressing into *Crude Calophyllum Oil* (CCO). Through the degumming process obtained *Refined Crude Calophyllum Oil* (RCCO), followed by esterification process. The next transesterification process is producing *Crude Biodiesel* and, finally, biodiesel is obtained through washing and drying.

Due to the existence of several stages in the processing of biodiesel causing high cost of biodiesel production, so that the implementation of the production process is operationally inefficient. Such as, a degumming process is heated at a temperature of 80° C for 30 minutes, until there is sediment. The next step is esterification process for 1-2 hours at temperature 60° C and transesterification process for 1 hour at temperature 30°-65° C. In [45] describes, conventional biodiesel production is generally done at high temperatures with external heat sources. The heat transfer is less effective because it occurs with conduction and convection system. In addition, conventional heating such as hotplate takes a long time and high power. However, although, [46] has improved it by using a homogenizer system for transesterification reactions, they were only able to reduce the reaction time by half from 60 minutes.

To address the challenge of high production cost, many efforts have been made on optimizing the biodiesel production process. One of them is microwave technology as an alternative method with several advantages such as quality issues [47, 48], energy efficient [49], and impact on the environment [50, 51]. The other is a more even warming that comes from the material and it is not transferred from the outside. The heating may also be selective, depending on the dielectric properties of the material. Moreover, microwaves can propagate through the liquid by the process of heating more effectively on the production of biodiesel and the time is shorter [52-55].

Regarding the processing biodiesel, we have proposed a new method of electromagnetic induction heating. Induction heating technology has been widely applied to the manufacture of induction cookers and is able to provide faster heat than the microwave. The induction heater (IH), an alternating electrical current from the power unit flows through a coil made of copper. This current will cause an electromagnetic field of varying magnitude. This field will generate an electric current on the metal material inside it. This electric current is known eddy current generates heat which can be used to heat and melt the metal.

2. Basic Principle

The IH is the process of heating objects that are electrically carried out (normally metal) with electromagnetic induction, where eddy currents are produced in the metal and resistance leads to metal Joule heating. The basic theory of electromagnetic induction, however, is similar to a transformer. The transformer works because of the phenomenon of electromagnetic induction, when there is a current flowing in a closed circuit it will produce various electromagnetic fields. As with transformers, electromagnetic fields (in primary coils) that affect secondary coils produce induced radiation and AC current flows if the secondary coil is a closed circuit. The IH consist of three primary elements: electromagnetic induction, skin effects, and heat transfer [56]. Figure 1 shows the basic principle of induction heating, contain of inductive heating coils and current, to illustrate electromagnetic induction and skin effects. Figure 1(a) indicates the simple structure of a transformer, where the secondary current is in direct proportion to the main current in accordance with the turn ratio.

When the secondary coil is turned on for a moment and is short-circuited, there is a major heat loss that occurs suitable to increase in the secondary current (load current). This is shown in Figure 1 (b). The amount of current in the secondary coil (I_2) is determined from the magnitude of the current on the primary coil (I_1) and the ratio of the windings between the primary and secondary coils (N_1/N_2). In this figure, when the secondary coil we replace with 1 wire ($N_2 = 1$) and made into a closed circuit, we will get a large coherent ratio value of the primary and secondary coils and will cause a large secondary current (I_2).

This will also be followed by a substantial increase in heat due to increase in the load. The concept of induction heaters where energy is supplied from the same source of a combined number of primary and secondary losses is shown in Figure 1 (c). In this figure, the primary inductive windings have many turns, while the secondary windings are only one and are short-circuited. In IH design, it takes the maximum heat energy generated by the secondary windings, where the gap of the inductive heating coil is designed as small as possible and secondary is made with low resistance and high-permeability. Nonferrous metals weaken energy efficiency because of their high resistance and low permeability.

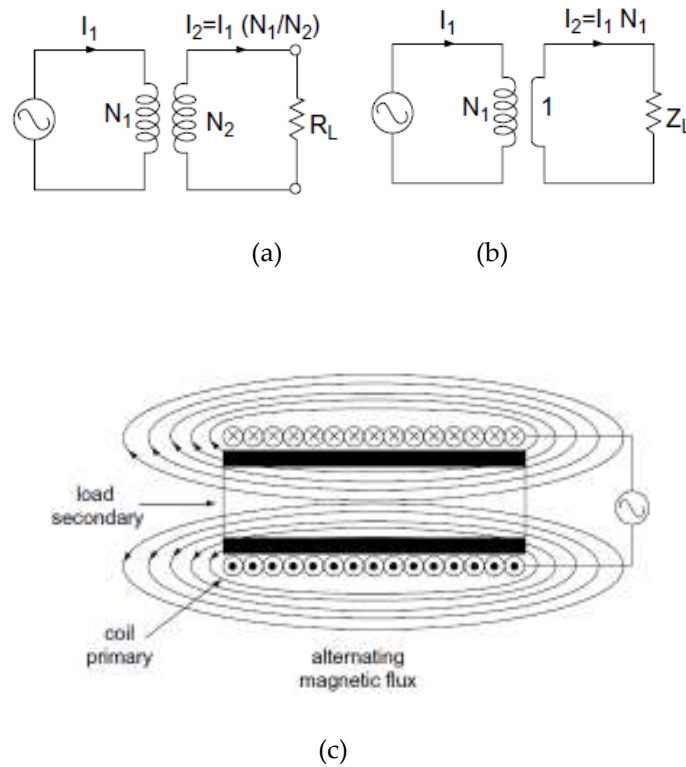


Figure 1. Basic induction: (a) equivalent circuit of transformer; (b) secondary of a short circuit; (c) concept of induction heating.

2.1 Electromagnetic Induction

The output of IH flow alternating current (AC) to supply an electric coil (induction coil). The induction coil is a source of heat that induces an electric current to a work-piece or the metal section to be heated. In addition, the induction coil as a heat source has no contact with the work-piece, and heat only occurs in the local area, especially the zone inside the coil. This occurs because the presence of AC in the induction coil has an invisible electromagnetic force field (flux) around it. When the work-piece is placed inside the induction coil, the force lines concentrate in the air gap between the coil and the work-piece. The induction coil functions as the major transformer with the work-piece to be heated into a secondary transformer. As a result, the force field in the induction coil causes the same and opposite electric current in the work-piece. After that, the work-piece heats up due to the current flow resistance of the electric current induced. The level of heating of the work-piece is affected by the

induction current frequency, induction current intensity, material specific heat, material magnetic permeability, and material resistance to current flow.

Based on Figure 1, when the AC current enters the coil, the magnetic field is formed around the coil, according to Ampere Law can be calculated as in equation (1):

$$\int H di = Ni = f \quad (1)$$

$$\phi = \mu HA$$

This formula state, when object that are inserted into a magnetic field will cause changes in the velocity of magnetic motion.

The density of the magnetic field decreases when the object reaches close to the center of the surface. In Faraday's Law states, the electric current produced on the surface of a conductive object has an inverse relationship with the current in an inductive circuit as in Equation (2).

$$E \frac{d\lambda}{dt} = \frac{d\phi}{dt} \quad (2)$$

Then, Equation (3) describes electrical energy caused by induced currents and eddy currents changed into heat energy.

$$P = \frac{E^2}{R} = i^2 R \quad (3)$$

Where,

- R = Resistance is determined by resistivity (ρ) and permeability (μ) of conductive objects
- i = Current is determined by the magnetic of field intensity.
- E = Induction voltage
- P = Power

2.2 Eddy Current

Induction heating arises when an electric current or eddy current is induced into the work-piece under conditions of poor electrical conductors. To produce the induction heating to be efficient and practical, the specific relationship between the frequency of an electromagnetic field that produces an eddy current, and the properties of a work-piece, must be satisfied. The basis of induction heating is eddy currents are produced outside the work-piece or commonly called "skin effect" heating. The level of induction heating depends on the high frequency of electric current, electrical resistance, and relative magnetic permeability of the work-piece.

Eddy Current is the induction of alternating electric current in a conductive material by an alternating magnetic field (generated by alternating electric current). The induced current inside the modified material causes a change in the value of the induced current through the material. The eddy current principle is based on Faraday's law which states that when a conductor is cut out the force lines of the magnetic field or electromotive force (EMF) will be induced into the conductor. The amount of EMF depends: (1) size, strength, and magnetic field density; (2) the speed at which the magnetic force lines are cut; (3) quality of conductors. Because eddy current is the current of electric current in the conductor, it will produce magnetic field too. Lenz's law states that the magnetic field of the induced current has a direction opposite to the cause of the induced current.

The magnetic field of eddy current is in opposite direction to the magnetic field of the coil as shown in Figure 2.

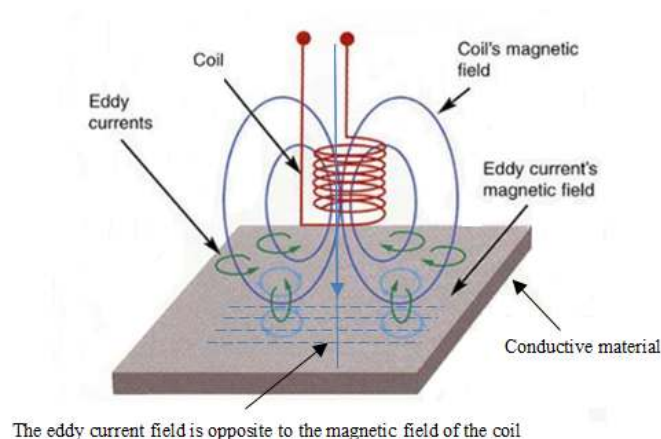


Figure 2. Direction of magnetic field eddy current opposing to the direction of magnetic field of the coil.

3. Topology of IH

3.1 Power System of IH

Nowadays, IH is heating technology has been developed in industrial, domestic and medical applications. The advantages are efficiency factor, fast heating, safety, cleanliness and accurate control [57]. Since the discovery of semiconductor technology, the second major revolution of IH technology is developed. The use of high-power semiconductor devices and high frequency power supply application has made great progress. Among them are silicon-controlled rectifiers (SCR), and gate turn-off thyristor which have been widely used to implement very reliable power converters. Then, the expansion of high frequency power electronics for IH needs such as metal-oxide-semiconductor field effects (MOSFETs) and bipolar junction transistors (BJT), allows higher efficiency power converter designs. Further progress in semiconductor technology is the discovery of an isolated-gate bipolar transistor (IGBT) that can improve the performance of IH design. IGBT has a low resistance and requires a very small power supply with a frequency of up to 100 kHz, but a frequency of round 400 kHz is difficult to achieve. On the contrary, SIT has defects such as high conduction loss compared to IGBT, complicated fabrication processes, high costs and limited application. Therefore, switching with very high frequencies can be achieved using MOSFETs.

With regard to power systems, a new soft-switched converter circuit has been developed that integrates the advantages of conventional PWM converters and resonance converters. This soft-switched converter has a switching waveform that is identical to a conventional PWM converter, except that the edges rise and fall from the waveform 'smoothed' without a temporary surge. In contrast to resonant converters, new soft-switched converters usually use resonance in a controlled manner. In this case, efficient energy conversion at high-frequency switching is absolutely necessary by manipulating the voltage or current when switching to zero. The concept is to combine the resonant tank in the converter to create an oscillation voltage (generally sinusoidal) and/or the current waveform so that the zero voltage switching (ZVS) or zero current switching (ZCS) conditions can be made for the power switch. The ZVS principle refers to the elimination the turn-on switching loss having the voltage of the switching circuit set to zero just before the circuit is turned "on". On the contrary, ZCS avoids turn-off switching loss by allowing no current to pass through the circuit right before turning it "off". The voltage or current given to the switching circuit can be made zero using resonance generated by the L-C resonance circuit. This is the topology "resonant converter". Resonance is allowed to occur just before and during the turn-on process and turn-off so as to produce ZVS and ZCS conditions. In addition, they seem to behave like conventional PWM converters.

3.2 Half-Bridge Resonance Inverter

In terms of the number of switching devices, the inverter topology normally used in IH is a single switch [58], full bridge circuit [59], and half bridge circuit [60], [61] resonance inverter. In this paper, power system with a half bridge resonance series inverter circuit has been applied. In [60], explaining the advantages of half bridge resonance series inverters is stable switching, low cost, and a streamlined design. Figure 3 shows the power operating system a half-bridge-resonant inverter. This system consists of AC power supply, main electrical circuit, control unit, input current detection circuit, resonance current detection circuit, and gate operation circuit. All procedures are needed to design and test the system shown in the block diagram. The system for IH, however, does not need large capacitors to produce DC flatter, because the major goal of this system is to generate heat energy. In contrast, a rough DC form helps increase the system's power factor.

AC power (220V/60Hz) passes through the rectifier diode to be transmitted to the capacitor (C). The capacitor in the available power system is too small in capacity to act leveling work, which leads to the action of an increased current at 120 Hz, which is not the right level for DC operation. The system for IH, however, does not need large capacitors to produce DC flatter, because the major purpose of this system is to generate heat energy. In contrast, a rough DC form helps increase the system's power factor. Based on this circuit, the leveling capacitor functions as a filter that prevents high-frequency currents from flowing into the inverter and entering the input part. Later, the input current becomes the average current of the inverter and the flow of ripple to the leveling capacitor.

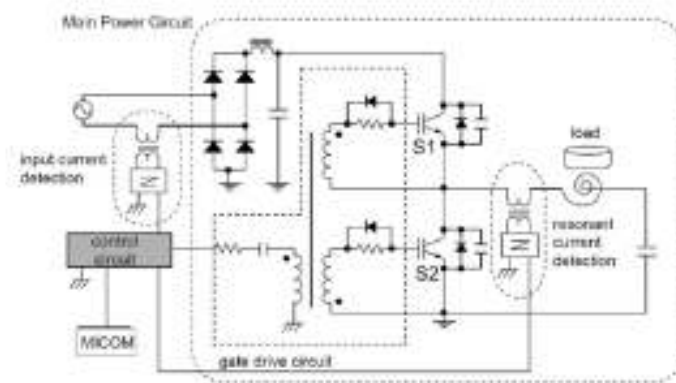


Figure 3. Half-bridge series resonant inverter.

4. Experimental Set-up

4.1 Testing of *Calophyllum Inophyllum* Oil

Calophyllum inophyllum oil used in this study was obtained from Kebumen, East Java Province (Indonesia). This type of crude oil is an inedible oil, which belongs to the *Guttiferae* family, usually called to as “Nyamplung” oil in Indonesia. *Nyamplung* trees usually grow along coastal area and adjacent lowlands forests, having no incubation in yielding, and seeds can be obtained throughout the year. Figure 4 shows the crude oil of *Calophyllum inophyllum*. Table 1 shows the characteristics of *Calophyllum inophyllum* crude-oil based on the Gas Chromatography-mass Spectrometry, (GCMS) testing at the Integrated Research and Testing Laboratory, Gadjah Mada University, Yogyakarta.



Figure 4. *Calophyllum inophyllum* crude oil.

Table 1. Composition of *Calophyllum inophyllum* crude oil

Fatty Acid	Mass content (%)
Palmitic acid	13.92
Stearic acid	13.12
Oleic acid	69.11
Linoleic acid	3.85

Based on the Table 1, it can be seen that there are many variations in the content of palmitic, oleic, linoleic, and stearic acid among the oils studied. This crude oil shows the content of saturated fatty acids including stearic acid (13.12%) and palmitic (13.92%). In contrast, high unsaturated fatty acids, representing 72.96% mainly consisted of oleic acid (69.11%) and linoleic (3.85%).

4.2 Biodiesel Processing

A schematic of the process for producing biodiesel is shown in Figure 5. First of all, biodiesel processing starts from the degumming process using 3 heating methods at 80°C. The goal is to separate crude oil from gum. The hotplates (conventional), microwaves, and electromagnetic induction heating are used in processing as a comparison. A total of 200 ml of crude oil was mixed with H₂SO₄ catalyst with a ratio of 1:20. The duration of the degumming process depends on the heating method until the color changes to light brown.

The next step, the esterification process is carried out by heating to a temperature of 65°C. A catalyst mixture of H₂SO₄ catalyst with 2% mass of oil dissolved into methanol until it is completely mixed. According to [62], the molar ratio and dosage of catalyst are one of the most important factors in the yield of biodiesel production, whereas the humidity is one of the lowest factors. In this process, the ratio of oil molar methanol used is 1:6 assuming that crude oil consists of all triglycerides with a density of 0.944 grams/ml and a molecular weight of 704.2 grams/mol. After that, 5 grams of oil samples were tested to determine free fatty acid content below 2%. If the FFA is still high or above 2%, the esterification process is repeated 2 times until the FFA actually becomes low below 2%.

After that, the transesterification process is a chemical reaction involving triglycerides and alcohol in the presence of a catalyst to form esters and glycerol. The transesterification process of *Nyamplung* oil was obtained at a ratio of 6:1, KOH 1% catalyst with a temperature of 65°C. The time period of the transesterification process depends on the heating method used. It usually can be seen in oil color changes and the glycerol is formed. Finally, glycerol is formed must be separated from biodiesel, then washed and dried.

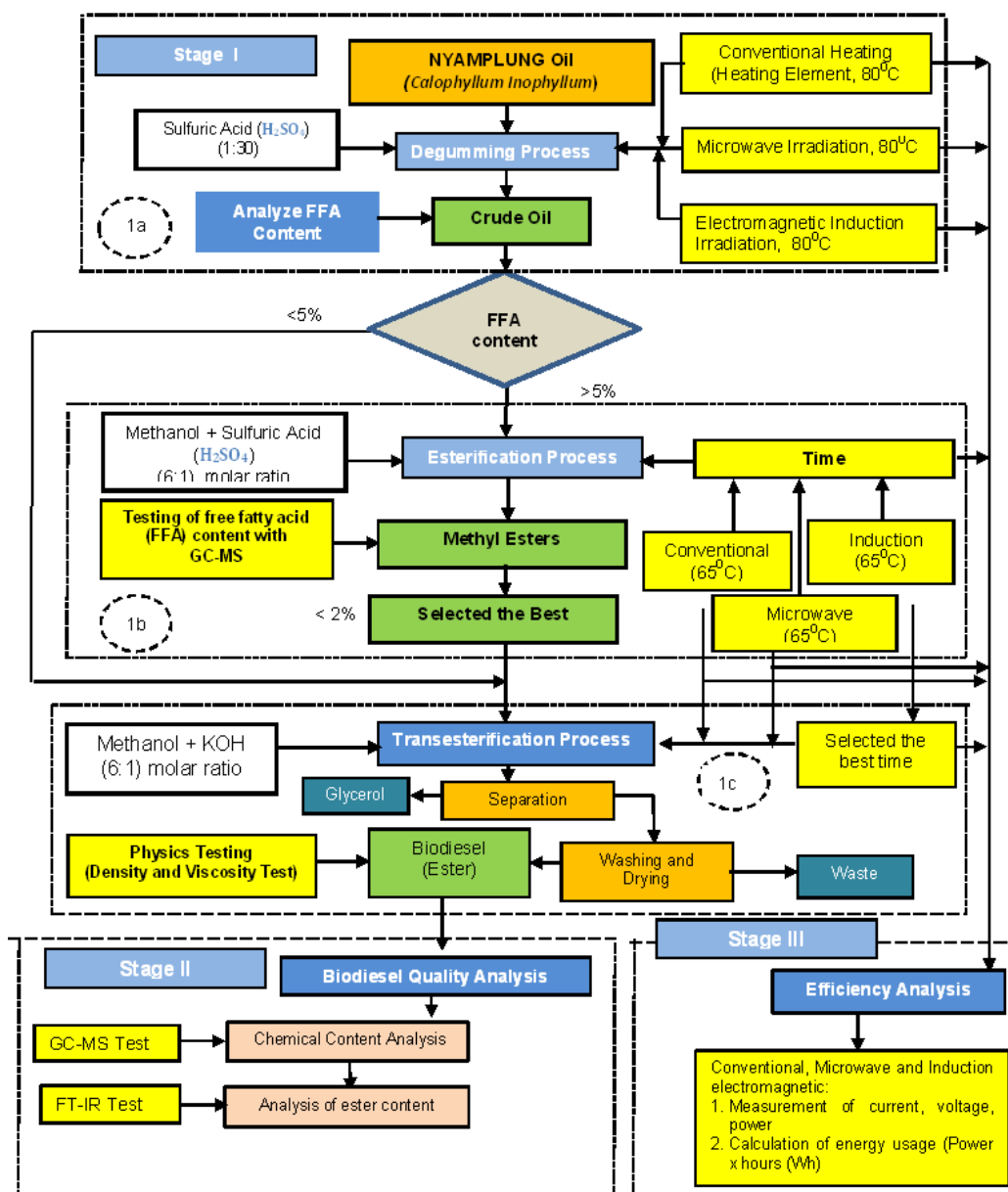


Figure. 5 Schematic of biodiesel process.

4.3 Induction Heating Irradiation

In this study, an electromagnetic induction heater has been used in the processing of *Calophyllum inophyllum* biodiesel. The equipment specifications have been detailed in Table 2. The schematic diagram of the experimental set up is given in Figure 6. The electromagnetic induction heat radiation induced by the metal mounted inside the coil is absorbed by the sample oil, which may lead to the appearance of warming in the sample. Heating with induced magnetic field radiation is faster and evenly distributed along the metal mounted inside the coil, it does not transfer heat from the outside. The duration of induction given to metals affects the rise in temperature/heat of the metal which affects the passage of the transesterification reaction. The greater the temperature given, the transesterification reaction runs faster and will result in more biodiesel conversion.

Table 2. Equipment specification

No.	Name	Specification
1.	Dc Voltage	30 Volts

2.	Power Input	200 watts
3.	Current Output	1.2 Amps
4.	Temperature	0-1000 ^o C (adjustable)

Vibrations in molecules induced by induction heat radiation will produce a uniform heat on the molecule, where the resulting induction heat penetrates the molecule and excites the molecules evenly, not just the surface. Induced heating radiation can speed up the reaction by vibrating the reactant molecule quickly. The longer the radiation time is given to the transesterification reaction the heat generated by the reactant molecule's vibration will be greater until reaching its optimum state. The transesterification time at 60^o C has been controlled automatically by a microcontroller. Output data such as temperature, current, power, and voltage are displayed and stored on a laptop.

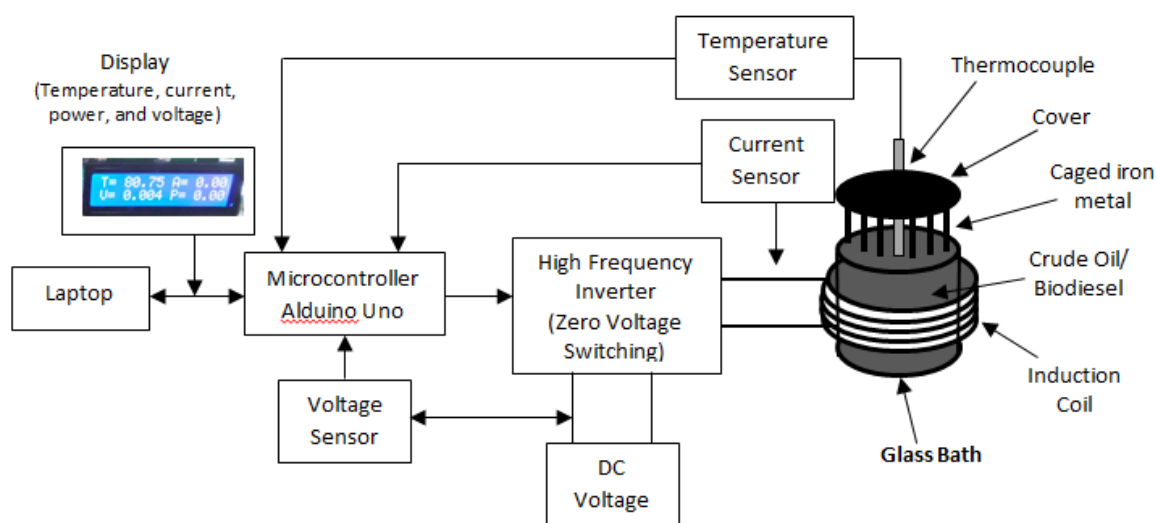


Figure 6. Schematic diagram of electromagnetic induction irradiation.

Eddy currents have the most dominant role in induction heating process. The heat generated on the material depends on the amount of eddy current induced by the inductor winding. When the winding is fed by alternating current, a magnetic field will occur around the conductor wire. The magnetic field varies according to the current flowing in the coil. According to [63], if there is conductive material around the changing magnetic field, then the conductive material will flow a current called eddy current. The Eddy Current principle is based on Faraday's law which states that when a conductor is cut out the force lines of the magnetic field or electromotive force (EMF) will be induced into the conductor. The amount of EMF depends on (1) size, strength, and magnetic field density; (2) the speed at which the magnetic force lines are cut; (3) quality of conductors.

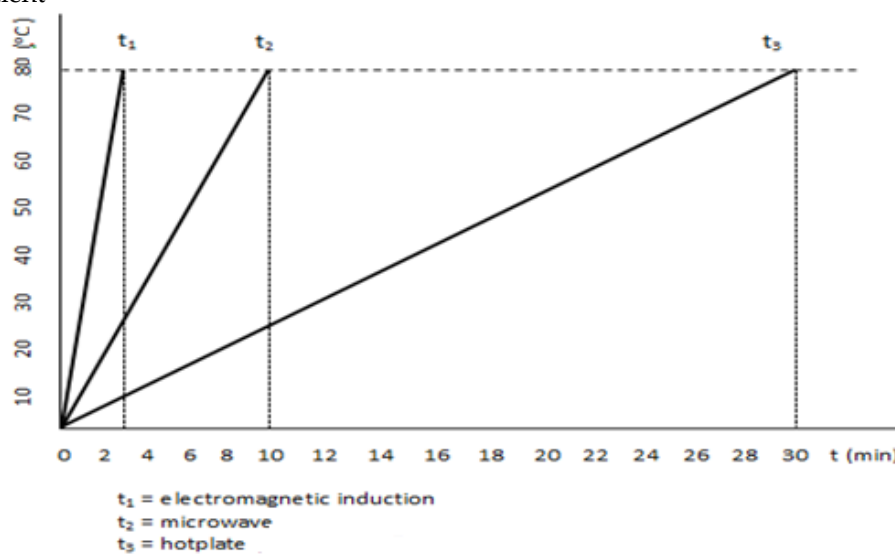
5. Result

5.1 Characteristics of IH Irradiation

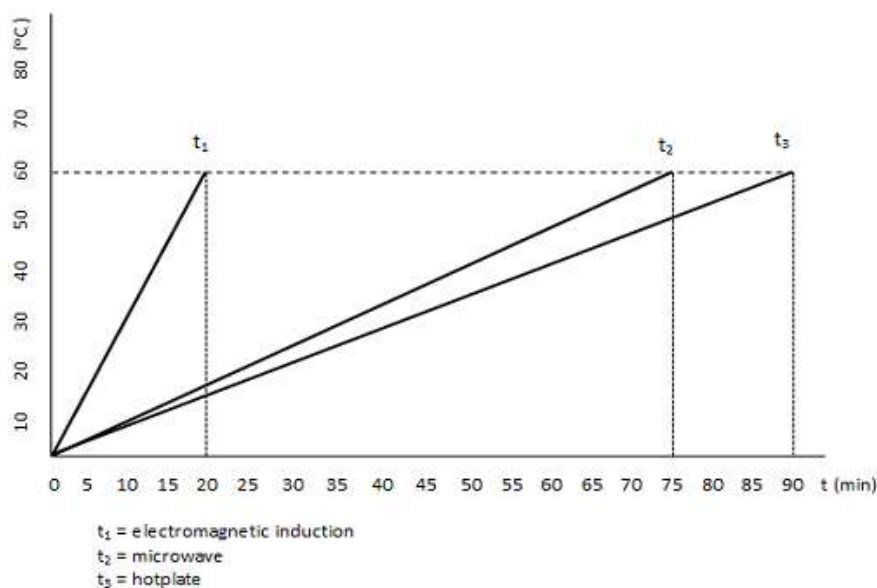
One method of heating to allow the reaction to run faster is to perform a transesterification reaction using electromagnetic induction heat radiation. The electromagnetic IH radiation induced by the metal mounted inside the coil is absorbed by the sample, which may lead to the appearance of warming in the sample. Heating with induced magnetic field radiation is faster and evenly distributed along the metal mounted inside the coil, rather than transferring heat from the outside. The duration of induction given to metals affects the rise in temperature / heat of the metal which affects the passage of the transesterification reaction. The greater the temperature given, the transesterification reaction runs faster and will result in more biodiesel conversion.

Vibrations in molecules induced by IH radiation will produce a uniform heat on the molecule, where the resulting induction heat penetrates the molecule and excites the molecules evenly, not just the surface. Induced heating radiation can speed up the reaction by vibrating the reactant molecule quickly. The longer the radiation time is given to the transesterification reaction; the heat generated by the reactant molecule's vibration will be greater, so that at any given time the transesterification reaction will reach its optimum state. Eddy current has the most dominant role in the induction heating process. The heat generated in the material depends on the amount of eddy current induced by the inductor winding. When the windings are energized by alternating current, a magnetic field will occur around the conductor wire. Magnetic fields vary according to the current flowing in the coil.

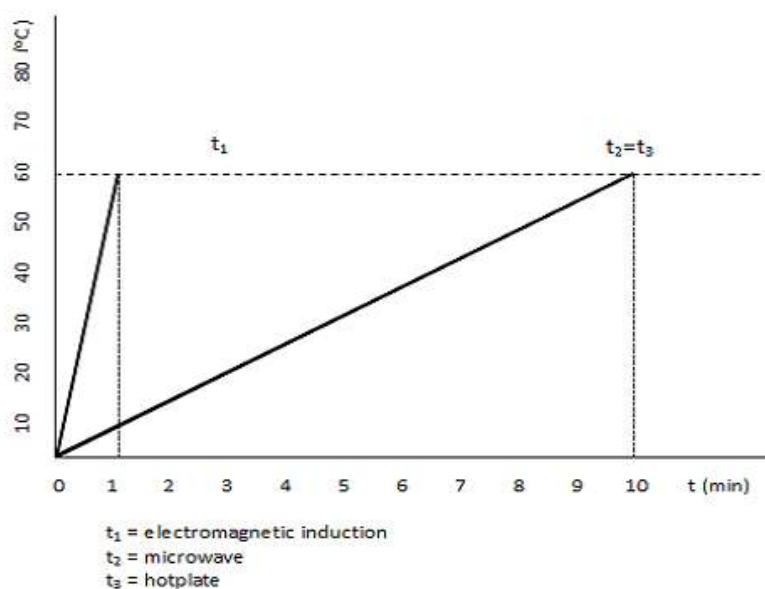
Technically, the induction heater has the following characteristics: able to release heat in a relatively short time. This is because the energy density is high. With induction, it is possible to reach very high temperatures. Heating can be done at a specific location and the system can be made to work automatically. Induction heating, in general, has high energy efficiency, but it depends on the characteristics of the heated material. Heating losses can be minimized. According to [64], several factors determine the number of eddy currents in the metal, including: (1) the magnetic field that induces metal; (2) metal materials used to generate heat. The smaller the resistance of the metal type, the better it is to be the object of heat of the metal; (3) metal surface area, the more surface area of the metal the more eddy currents will be on the metal surface; and (4) the greater the frequency, the greater the frequency the more the magnetic field is generated. In addition, there are several advantages of using induction heater, including: heat is generated directly inside the barrel wall; heat can be applied uniformly across the barrel; cold element operation, so it has no time limit; fast start up time; and energy-efficient



(a)



(b)



(c)

Figure 7. A Comparison of the time of biodiesel production between conventional, microwave, and induction electromagnetic (a) degumming; (b) esterification; (c) transesterification.

Figure 7 shows a comparison of the results of testing of biodiesel manufacturing processes between conventional, microwave and electromagnetic induction. It can be seen that electromagnetic induction technology is a great opportunity in the production process in the future. Compared to other methods, the proposed new method has a very short time at every stage of the production process such as degumming (3 minute), esterification (20 minute, and transesterification (1.2 minute).

5.2 Analysis Free Fatty Acid (FFA)

5.2.1 Esterification Process

Esterification is the process of reacting free fatty acids into esters with short chain alcohols (methanol or ethanol) to produce metal fatty acid esters (FAME) and water. In this study sulfuric acid catalyst (H_2SO_4) was used for esterification reactions. The esterification process with an acid catalyst is needed if vegetable oil contains FFA above 2%. If oil with a high FFA level ($> 2\%$) is directly transesterified with an alkaline catalyst, then the FFA will react with the catalyst to form soap. The formation of large amounts of soap can inhibit the separation of glycerol from methyl esters and result in the formation of emulsions during the washing process. Esterification is used as a preliminary process to convert FFA to methyl ester so that it reduces the levels of FFA in vegetable oils and then is transesterified with a base catalyst to convert triglycerides to methyl esters.

Esterification is the conversion stage from FFA to esters. Ester reacts fatty oils with alcohol. To encourage the reaction to proceed to perfect conversion at low temperatures (for example, at most 120°C), the methanol reactant must be added in very excessive amounts (usually greater than 10 times the stoichiometric ratio) and the product water following the reaction must be removed from the reaction phase, namely the oil phase. The 1:20 molar ratios between oil and methanol was used in this study. Heat treatment is carried out at an average temperature of 65°C with a period of time depending on the characteristics of the heating method used. The esterification process is carried out twice, because in the first esterification process obtained FFA content which is still high above 2%. Table 3 shows the comparison of FFA values from the esterification results for the three heating methods used.

Table 3. Comparison of FFA values from esterification results between the three heating methods with a molar ratio of 1:20 (2% b/b) and sulfuric acid catalyst

Method	Temperature (Degree)	Time (Minute)	Esterification	
			FFA (I)	FFA (II)
			(%)	(%)
Hot Plate	60	90	10,6	2.58
Microwave	60	75	2.468	1.267
Induction Electromagnetic	60	15	5,8	0.7

5.2.2 Transesterification Process

This stage is the main stage in this study, where triglycerides which are the main components of oil are converted into biodiesel and glycerol. This process was carried out using a 1% b/b KOH catalyst with a 1:6 molar ratio of oil-methanol. By using the same method as in the esterification process, the heating is carried out at a temperature of 65°C . According to [65] the optimal temperature in the transesterification process uses a KOH catalyst which is 65°C . Comparative data from the results of the transesterification of the three heating methods are shown in Table 4. Figure 8 and 9 shows the transesterification process using electromagnetic induction, respectively. Here, clearly biodiesel production using electromagnetic induction has a higher FAME value than conventional and microwave

Table 4. A comparison of transesterification process of nyamplung oil with KOH catalyst 2% (b/b), with molar ratio 1:6

Method	Temperature	Time	Power	FFA	FAME
	(Degree)	(Minute)	(Watts)	(%)	(%)
Hot Plate	61	10	600	0.56	35,1

Microwave	60	10	40	0.58	40.51
Induction Electromagnetic	60	0.43	130	0.5	65.96



Figure 8. The esterification process using electromagnetic induction



Figure 9. FAME: (a). esterification (b) biodiesel.

5.3 Analysis of Energy Use

Based on the measurement results, the amount of power used by all heating methods is the multiplication of voltage and output current. Energy consumption is calculated between the time multiplier and the output power. Based on Table 5, it can be seen that electromagnetic induction method using energy more efficient compared to conventional and microwave method, that is, 0.056 kWh. The amount of energy obtained is based on the multiplication of the overall time of the degumming, esterification and transesterification steps by the amount of heating equipment power.

Table 5. Comparison of energy consumption for *Calophyllum inophyllum* oil (200 ml)

Methods	Energy consumption (kWh)			Total Energy (kWh)
	Degumming	Esterification	Transesterification	
Hotplate	0.3	0.9	0.1	1.3
Microwave	0.02	0.15	0.02	0.19
Electromagnetic Induction	0.007	0.046	0.003	0.056

5.4 Testing of Viscosity and Density

The viscosity of biodiesel has an important role in the fuel injection process. According to [66], very small viscosity can cause leaks in the fuel injection pump. Conversely, too high viscosity will produce large droplets of fuel and have high momentum and collide with the cylinder wall, but the injection pump cannot fog properly. Density is related to the value of heat and energy produced by a diesel engine. Low density will produce high calorific value and vice versa. Density is one parameter that shows the success of the transesterification reaction. Fulfillment of density parameters indicates that the biodiesel purification process is successful. The lack of biodiesel due to impurity can produce high density values. Table 6 shows the comparison of laboratory testing densities, and viscosity values for each test sample. The test results showed that the biodiesel viscosity obtained was lower than the others, namely 5.54 Cst. This value meets the requirements of the Indonesian National Standard (SNI) in the fuel injection process between 2.3-6.0 Cst.

Table 4. Comparison of testing of viscosity and density values

Properties	<i>Calophyllum inophyllum</i> Oil			Biodiesel				SNI
	Hotplate	Microwave	Electromagnetic Induction	ASTM D6751 (USA)	ASTM PS 121	EN 14214	C1 Biodiesel	
Density (20°C) (g/ml)	0.883	0.885	0.882	0.87-0.9	0.7328	No specific	0.877	0,850 - 0,890
Kinematic viscosity, 40°C (mm ² /s)	6.353	5.9847	5.54	1.9-6.0	1.9-6.0	3.5-5.0	5.6872	2.3-6.0

4.5 Analysis of FTIR (Fourier Transform Infrared)

The purpose of FTIR testing is to prove that what is produced in the transesterification process is methyl ester by knowing the functional groups formed. Biodiesel produced from this study was analyzed using FTIR instruments to prove and ensure that the results of this study have a functional group of methyl ester which is the main functional group of biodiesels. The presence of esters, can be seen from the typical absorption of C = O and C - O groups. Figure 10 shows the testing of FTIR of nyamplung oil.

Based on the spectrum data produced, the reaction has a typical group of methyl esters. Typical absorption bands of long chain fatty acids include stretch C-O vibrations in the frequency region around 1170.09; 1195.87, and 1244.09 (No. 3-5); the strain absorption band C-H alkane at the frequency 2854.65 (No. 11); and methyl groups on the frequency of 2924.09 (No.12). The C = O stretch absorption band is detected at the frequency 1743.65 (No.10) which is very clearly seen as a strong band in the mid-

spectrum area. The stretch vibration absorption band $C = C$ at wavelength 1641.42 shows that the biodiesel component contains a double bond. Thus, the functional groups contained in nyamplung seed oil biodiesel analyzed by FTIR include methyl groups, ester groups and carbonyl groups.

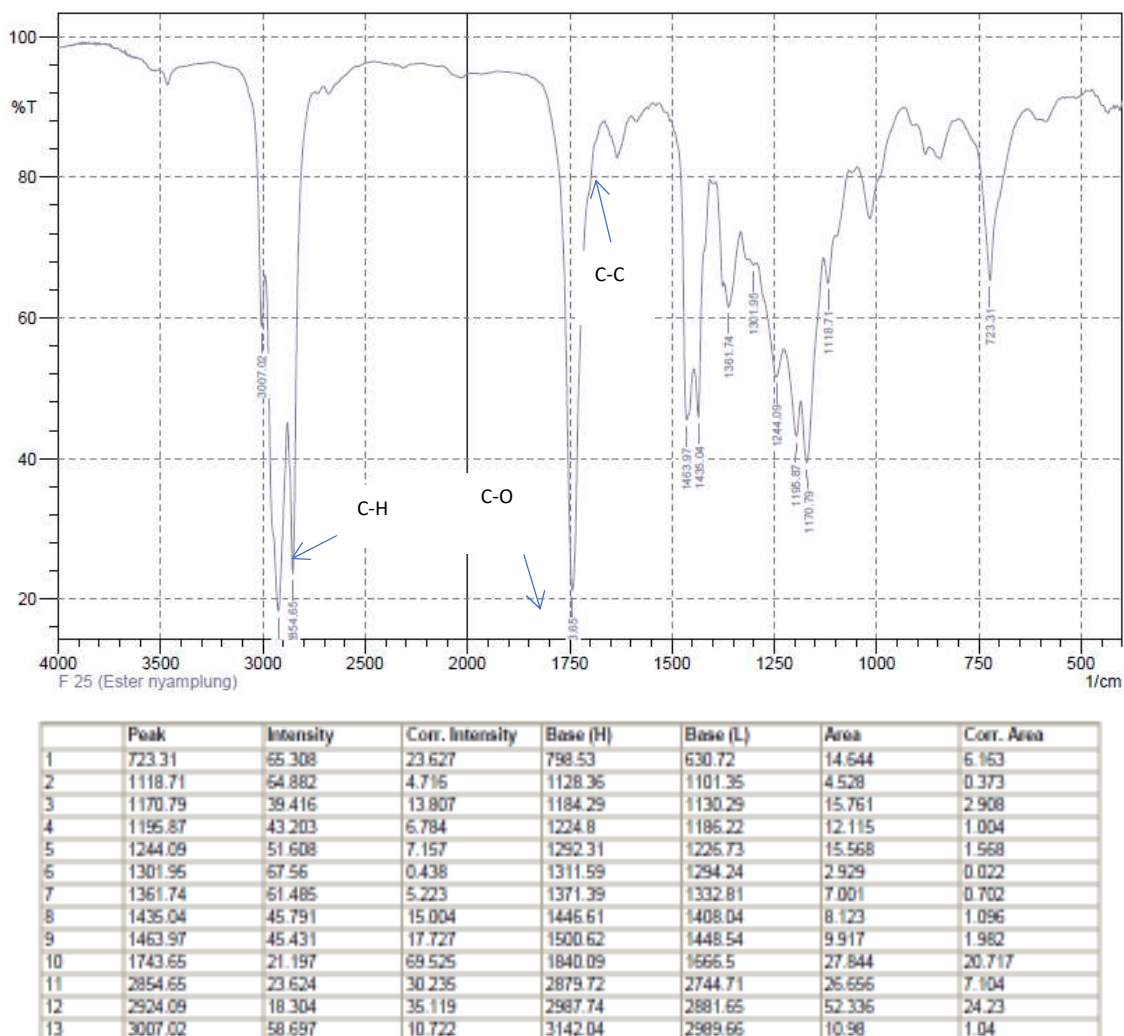


Figure 10. FTIR charts of nyamplung methyl esters

5.2 Discussion

Generally, the biodiesel production process is used by a heating device such as a hot plate or heating coil. With the development of high technology, the microwave radiation method in making biodiesel is a smart choice compared to conventional systems. An alternative heating system "microwave irradiation" has been used in transesterification reactions in recent years. Microwaves are electromagnetic radiation that represents non-ionizing radiation that affects molecular motions such as ion migration or dipole rotation but does not change the molecular structure [67, 68].

According to [49], the advantage of using a microwave in biodiesel processing is that transesterification is accelerated efficiently in a short reaction time. In addition, a drastic reduction in the number of byproducts is obtained, a short separation time is obtained and a high yield of a very pure product is achieved in a short time [69]. Compared to conventional systems, production costs are also reduced and fewer by-products occur. Therefore, microwave heating is very favorably compared to conventional methods, where heating time very slow and inefficient because the transfer of energy to the sample depends on the convection current and the thermal conductivity of the reaction mixture. However, a shorter heating reaction has been achieved through electromagnetic induction than microwave technology.

Experimentally, we have reported a comparison of 3 methods in the production process of *Calophyllum inophyllum* biodiesel. In the transesterification process, electromagnetic induction needs only 1.15 minutes, compared with both microwave and conventional methods for 10 minutes. Likewise, other stages such as degumming and esterification, such as Figure 7a, 7b, and 7c show a significant time difference between the three methods. Some characteristics of the electromagnetic induction method show better progress than others, such as the time is shorter, energy saving, the quality of FAME, viscosity, and yield is higher. Therefore, the findings of this method have promising expectations for biodiesel production.

6. Conclusions

This study has used a new method to process biodiesel at each stage. The esterification reaction is only used for 20 minutes, even for the transesterification reaction to be shorter. Compared to heating hotplates and microwaves, the reaction time is significantly reduced. As a result, the overall energy use from the biodiesel production stage is also more economical and efficient. The optimal condition for this experiment were an oil-to-methanol molar ratio of 1:6, 2% (b/b) KOH catalyst, a reaction temperature of 65°C, reaction time of only 1.2 minutes, and FAME of 65.96%. Electromagnetic induction can shorten reaction time effectively. The proposed method is very competitive compared to the two commonly used methods. Faster heating has been enhanced by electromagnetic induction compared to the microwave is considered the preferred method due to several advantages such as low energy consumption, a substantial reduction in reaction time and solvent requirements, enhanced selectivity, and better conversion with fewer byproducts.

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2. Major Revisions Tgl 26 Desember 2018



Sudirman S <sudirman_s@staf.undana.ac.id>

[Energies] Manuscript ID: energies-413947 - Major Revisions

2 pesan

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26 Desember 2018 15.34

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Dear Dr. Kurniati,

Thank you for submitting the following manuscript to Energies:

Manuscript ID: energies-413947

Type of manuscript: Article

Title: A Novel Process Production of Calophyllum Inophyllum Biodiesel with Electromagnetic Induction

Authors: Sri Kurniati, Sudjito Soeparman, Sudarminto Setyo Yuwono, Lukman Hakim, Sudirman Syam *

Received: 9 December 2018

E-mails: sri_kurniati@staf.undana.ac.id, sudjitospn@yahoo.com, ssyuwono2004@yahoo.com, lukman.chemist@ub.ac.id, sudirman_s@staf.undana.ac.id

Submitted to section: Sustainable Energy,

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Biomass Processing for Biofuels, Bioenergy and Chemicals

http://www.mdpi.com/journal/energies/special_issues/biomass_processing

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If the reviewers have suggested that your manuscript should undergo extensive English editing, please address this during revision. We suggest that you have your manuscript checked by a native English speaking colleague or use a professional English editing service. Alternatively, MDPI provides an English editing service checking grammar, spelling, punctuation and some improvement of style where necessary for an additional charge (extensive re-writing is not included), see details at <http://www.mdpi.com/authors/english>.

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1/10/2021

Email Universitas Nusa Cendana (Undana) - [Energies] Manuscript ID: energies-413947 - Major Revisions

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Thank you for your information.
In the next few days, I will revise my manuscript in accordance with the reviewer's comments.

Best regards,

Sri Kurniati

Pada tanggal Rab, 26 Des 2018 pukul 16.34 Ana Djakonov
<djakonov@mdpi.com> menulis:
[Kutipan teks disembunyikan]

3. Manuscript Revision

a. Response to Reviewer 1 Comments

Point 1: The conclusions should be amplified and improved to clarify the results obtained in this work.

Response 1: Conclusions have been revised

Point 2: The key results should be stated in a more quantitative way.

Response 2: Some test results data have been revised and added in table form

Point 3: In Figures 4, 8 and 9, the authors of the manuscript must improve the quality of images.

Response 3: This image has been upgraded

Point 4: It would be very interesting that the authors of the manuscript cite some recent papers in the introduction like the year 2016, 2017 and 2018.

Response 4: : Some references have been added

Point 5: References. Standards and regulatory specifications used in the present study should appear in the references.

Response 5: Yes, it has been checked and corrected in accordance with this journal format

Point 6: The English style requires improvements. The authors must be sure that there are no grammatical errors and that the manuscript is perfectly written in English.

Response 6: Several sentences and paraphrases have been revised

Point 7: The authors must be sure that the bibliographical references, figures and tables are in accordance with the format of the journal.

Response 8: Yes, it has been checked and corrected in accordance with this journal format

Point 8: It would be very interesting that the authors of the manuscript include more bibliographical references and, specially, references from the journal itself.

Response 8: Some references from Energies have been added [ref. 26, 46, 67 and 70]

b. Response to Reviewer 1 Comments

The authors of the current manuscript describes a novel process production of Calophyllum 3 inophyllum biodiesel with electromagnetic induction This paper presents a work that is of interest to the reading audience of Energies, also this work could be a significant step forward for the field of research. The paper may be recommended for publication as it meets most of requirements to such type of articles; however, the conclusions have to be extended, and the authors of the manuscript must improve the quality of Figures 11, 12, 13 and 14

Response :

Conclusions and Figures 11, 12, 13, and 14 have been revised

c. Response to Reviewer 3 Comments

Point 1: The description of the basic principles and topology of induction heating (Section 2) must be included as Supporting Information.

Response 1: I have added chapter 2.3 (Operation Fundamental of IH) to support information about the description of basic principles and topology of induction heating (Section 2)

Point 2: Figure 5 shows a schematic process for producing biodiesel consisting in three steps: degumming, esterification and transesterification. However, there is not information about the pre-treatment of feedstocks before each process. For instance, details about the degumming process, like the oil:H₂SO₄ ratio (molar or weight?), are not clear, since in the text appears 1:20 (oil:H₂SO₄?) and 30:1 in the schematic diagram. Is H₂SO₄ neutralized after the degumming and esterification processes? Is water formed in the esterification process removed before transesterification? Moreover, in the text (p. 12, l. 346), it is affirmed that an oil: methanol molar ratio of 1:20 is used in the present study, but on page 7 (l. 247) a molar ratio of 1:6 is indicated

Response 2:

- Figure 5 (becomes Figure 6) has been revised, the degumming process uses H_2PO_4 (5% v/v) instead of H_2SO_4
- This paraphrase is wrong (~~A total of 200 ml crude oil was mixed with H_2SO_4 catalyst with a ratio of 1:20.~~) and this paraphrase was replaced by: A degumming process carried out using phosphoric acid (H_2PO_4) as much as 5% (v/v) of 400 ml *Calophyllum-inophyllum* oil, then it was heated to 80°C .
- Added Table 1 which provides information about the pre-treatment of feedstocks before each process
- This statement was omitted: ~~In this process, the ratio of oil molar methanol used is with ratio of 1:6 assuming that crude oil consists of all triglycerides with a density of 0.944 grams/ml and a molecular weight of 704.2 grams/mol. Afterwards, a 5 gram oil sample is tested to determine free fatty acid content (below 2%).~~
- **Additional explanation about the esterification process:**
Briefly, the first catalyst is dissolved in methanol and added 5 grams of *Calophyllum-inophyllum* oil is put in a 100 ml beaker. The mixture of methanol and catalyst H_2SO_4 was put into Erlenmeyer and stirred with a magnetic stirrer for 5 minutes. The beaker glass is filled with a mixture of methanol, *Calophyllum-inophyllum* oil, H_2SO_4 , and it is heated using 3 heating methods by keeping the temperature up to 60°C . After esterification, testing is carried out to determine the content of FFA. If the FFA is still high or in above 2%, the esterification process is repeated for 2 times until the FFA becomes precisely low (below 2%). After that methanol, and the product is separated using a 100 mL separatory funnel for the purification stage. The product is washed with hot distilled water ($> 80^\circ\text{C}$) to remove the remaining acid in the product, continuing until the washing water shows $\text{pH} \sim 7$.

Point 3: Data displayed in Figure 7 compare the three heating processes (hotplate, microwave and electromagnetic induction), using time units in the y-axis, but the targets to be attained in the degumming, esterification and transesterification processes must be included.

Response 3: The target to be achieved in the degumming, esterification and transesterification process has been described in section 5.2

Point: 4. The esterification process is repeated twice to reach FFA values lower than 2% (mg KOH/g as acid value?), but it is not clear whether the heating process is carried out twice or new reactants (H_2SO_4 and methanol) are added. Please clarify.

Response 4: The esterification process is carried out twice. On esterification I the FFA value is above 2%. Then the second esterification process is carried out to reduce the FFA value below 2%. Before esterification II was carried out, the old methanol was first discarded or separated from the oil produced by esterification I. Furthermore, mixing the new molar ratio (20: 1) was made like the esterification process I and then reheated to 60°C . The treatment of time used in the esterification process of the three heating methods has been summarized in Table 3.

Point :5. Is a fraction of methanol removed before the transesterification process? A different oil:methanol molar ratio is used for transesterification (1:6 versus 1:20).

Response 5:

- Yes, before all the transesterification process is carried out all the methanol content is eliminated. The following is the explanation: After the esterification process is complete, the oil color changes will appear to be clear yellow with reduced FFA content in *Calophyllum-inophyllum* oil. Then the remaining oil and methanol are separated gravitatively, and washed with 70°C warm water, then separated by oil and water using a separating funnel as shown in the figure below. Then the oil is heated on an electric stove with a temperature of 100°C which aims to remove the remaining water contained in the oil. The results of the esterification process will show colour differences from reddish yellow to unclear yellow.

- The esterification process uses a ratio of 20: 1
- The transesterification process uses a ratio of 6: 1



Point :6. The analysis of energy use only takes into account the time of application of the three different heating systems to calculate the amount of power utilized, by nothing is said about the cost of instruments and equipment, and their corresponding amortization.

Response 6: In this study, I only calculate the energy usage of the three-equipment used without calculating the price of the tool used. However, a brief explanation of equipment costs is explained in section 5.2. In this case, the current, voltage, and time are measured at each stage (degumming, esterification and transesterification) and summing the overall power used by the formula: $W = V \times I \times t$ (in watt-hours, wh). The aim is to compare the use of electrical energy among the three-heating method equipment in an effort to reduce the production costs of biodiesel processing.

Point :7. The evolution of the FAME yield with the time must be included in order to show the kinetic of the transesterification process by using electromagnetic induction. It is expected that biodiesel properties given in Table 6 correspond to a biodiesel sample obtained with a FAME content higher than 96.5% (EN 14103).

Response 7: Already described in Table 4

Point :8. FTIR spectroscopy is not conclusive about the effectiveness of the transesterification process for biodiesel production from vegetable oils, since the FTIR spectra of triglycerides (esters of fatty acids with glycerol) present in this feedstock also exhibit the absorption bands of C=O and C-O groups, and methyl groups are also observed as terminal groups in fatty acids chains. HPLC or GC must be used, among other techniques, for the characterization of FAMEs present in biodiesel.

Response 8: Section 5.6 about FTIR analysis was replaced by GC-MS analysis

Point 9. Table 1 can be removed because all the information shown in this Table is cited in the text. Otherwise, data concerning saponification value, iodine number, water content, acid value, density, among others, could be included.

Response 9:

- Yes, I agree that table 1 can be deleted because all information displayed in this table is quoted in the text.
- **This statement has been revised:** Table 1 shows the characteristics of *Calophyllum inophyllum* crude oil based on the Gas Chromatography mass Spectrometry (GCMS) testing at the Integrated Research and Testing Laboratory, Gadjah Mada University, Yogyakarta, (The characteristics of *Calophyllum inophyllum* crude-oil have been tested using Gas Chromatography-mass Spectrometry (GCMS) at the Integrated Research and Testing Laboratory, Gadjah Mada University, Yogyakarta)

- **This statement has been revised :** ~~Table 1 presents many variations in the oil content of palmitic, oleic, linoleic, and stearic acid among the studied oils.~~ (Based on this test the contents of palmitic, oleic, linoleic, and stearic acid were obtained from the oil studied.)

Point 10. English must be revised in depth because there are several typos and wrong sentences. For instance: (Title) A Novel Production Process instead of A Novel Process Production; (p. 1, l. 40) vegetable oils are mainly composed of triglycerides (98%) and a small proportion of diglycerides, since vegetable oils are not known as triglycerides; (p. 1, l. 41-42) Biodiesel is not treated with a mixture of fatty acid methyl esters with methanol and ethanol, but that biodiesel is a mixture of fatty acid alkyl esters obtained by treating triglycerides with methanol or ethanol; (p.11, l. 334) fatty acid methyl esters instead of metal fatty acid esters.

Response 10: Ok, all of these sentences have been revised

4. Manuscript Re-Submitt Tgl 5 Januari 2019

1/10/2021 Email Universitas Nusa Cendana (Undana) - [Energies] Manuscript ID: energies-413947 - Manuscript Resubmitted

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[Energies] Manuscript ID: energies-413947 - Manuscript Resubmitted
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Dear Dr. Kurniati,

Thank you very much for resubmitting the modified version of the following manuscript:

Manuscript ID: energies-413947
Type of manuscript: Article
Title: A Novel Process Production of Calophyllum Inophyllum Biodiesel with Electromagnetic Induction
Authors: Sri Kurniati, Sudjito Soepaman, Sudarminto Setyo Yuwono, Lukman Hakim, Sudirman Syam *
Received: 9 December 2018
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Submitted to section: Sustainable Energy,
http://www.mdpi.com/journal/energies/sections/sustainable_energy
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A member of the editorial office will be in touch with you soon regarding progress of the manuscript.

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ssyuwono2004@yahoo.com, lukman.chemist@ub.ac.id, sudirman_s@staf.undana.ac.id

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6. Minor Revisions, Tgl 14 Januari 2019

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Biomass Processing for Biofuels, Bioenergy and Chemicals

http://www.mdpi.com/journal/energies/special_issues/biomass_processing

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7. Accepted for Publication, 17 Januari 2019

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Type of manuscript: Article

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8. Manuscript Resubmitted, 17 Januari 2019

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ssyuwono2004@yahoo.com, lukman.chemist@ub.ac.id, sudirman_s@staf.undana.ac.id

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Authors: Sri Kurniati, Sudjito Soeparman, Sudarminto Setyo Yuwono, Lukman

Hakim, Sudirman Syam *

Received: 9 December 2018

E-mails: sri_kurniati@staf.undana.ac.id, sudjitospn@yahoo.com, ssyuwono2004@yahoo.com, lukman.chemist@ub.ac.id, sudirman_s@staf.undana.ac.id

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Hakim, Sudirman Syam *

Received: 9 December 2018

E-mails: sri_kurniati@staf.undana.ac.id, sudjitospn@yahoo.com,

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