

Conversion of Crude *Hevea brasiliensis* Oil to Biodiesel Utilizing *Perna viridis* Waste Shells as Solid Catalysts

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ABSTRACT

The over-reliance and environmental damage caused by the use of fossil fuels have created the need to explore an alternative source of renewable and clean energy. Free Fatty Acid (FFA) from *Hevea brasiliensis* oil or commonly known as rubber seed oil (RSO) was utilized as feedstock with the aid of waste mollusk shells (*Perna viridis*) using domestic household batch microwave irradiation technique. The mollusk shells were sieved to particles sizes ranging from 1 mm to 2 mm and were calcined at 900°C for 4 hours at 5°C/min heating rate. SEM-EDS, XRF, and XRD were performed to characterize the surface morphologies and composition of the catalyst. The two-step esterification-transesterification process reduced the total acid number of RSO from 104.70 mg KOH/g to 0.463 mg KOH/g. The optimum yield of fatty acid methyl ester (FAME) reached up to 96.6% under optimal conditions of 12:1 methanol molar ratio, catalyst loading of 12 weight percent (wt%) oil, 7 minutes of reaction time, and 400-Watt microwave power input. The produced methyl ester fatty acids composition was analyzed via GC-MS and the physicochemical properties such as kinematic viscosity, acid value, flash point, density, water content, and calorific value were analyzed according to the American Society for Testing and Materials D6751 (ASTM D6751) and European Committee for Standardization (EN 14214) and were found to be within the requirements.

Keywords: Biodiesel, heterogeneous catalyst, microwave, transesterification.

1. INTRODUCTION

The decline of fossil fuels and the environmental harm caused by their use has stimulated research in renewable energy, energy conservation, and the development of energy policies worldwide. *Hevea brasiliensis* or rubber seed oil (RSO) which is considered the 2nd generation of biodiesel grows abundantly in tropical regions like India, Brazil, and across Southeast Asia [1]. In 2019, the Malaysian Rubber Board in their annual report recorded that more than 1 million hectares of land had been converted to rubber plantations which contributed to an average production of 1000 kg of rubber seeds per ha/year [2]. The government and industries are more focused on natural rubber, rubberwood products, and other rubber-based products, thus these rubber seeds are normally perceived as low in value and not used. Until recently,

rubber seed oil was underutilized, and it was still disregarded as a waste by-product [3].

Conversely, diversifying the applications of the rubber tree possibly will economically benefit the country while also reducing the production of waste. Oleic acid (C18:0) is abundant in rubber seed oil, can withstand high oxidation levels, thus suitable for biodiesel and bio-lubricants application that require strong oxidative stability at high temperatures. Recently, rubber seed oil is gaining more attention and becoming the preferred source of feedstock among biodiesel producers because of its special features such as easy propagation, high viscous oil yield, fast growth, and the ability to adapt to various climate conditions.

Homogeneous catalysts such as commercial potassium hydroxide and sodium hydroxide are widely used in biodiesel development due to their ease of use

[4]. However, this method is tedious and the cost to separate and recover this homogeneous catalyst particularly in a large-scale production has made the industry shift focus on heterogeneous biodiesel production [5].

Calcium oxide (CaO) catalyst is a promising solid catalyst that can withstand high temperatures, easy to recycle, and can produce biodiesel up to par with homogeneous catalysts. The reusability of heterogeneous catalysts which can be reused up to ten times reduces the amount of water used in the processing stage and the length of purification. However, shells of the mollusk are frequently found in the sea, on land, and in freshwater. These shells are often useless and left to accumulate without any use. *Perna viridis*, the Asian green mussel or locally known as *Kupang* is a natural waste shell. It can be easily found in coastal waters in the Asia Pacific region. Among the investigated waste mollusk shell-derived catalysts [6], *Perna viridis* derived CaO catalyst produced higher yields.

The use of microwave irradiation techniques to produce biodiesel is still limited only to batch production since it is not easy to upscale biodiesel synthesis from laboratory to industrial production. There is limited research done on microwave chemistry and technology [7] and until today the report on utilizing bulk size of solid catalyst on the high FFA conversion of feedstock is still limited. Research must be carried out to increase the safety of this technique and address the microwave radiation's poor penetration depth into absorbing materials based on their dielectric properties. This study aims to investigate the performance of high FFA *Hevea brasiliensis* oil as green biodiesel with the aid of *Perna viridis* as a solid catalyst, done via batch microwave method. This study aims to produce biodiesel that is more environmentally friendly and economically productive.

2. MATERIALS & METHODS

2.1. Materials

Crude *Hevea brasiliensis* feedstocks were purchased from a local company, Kinetics Chemical Sdn. Bhd in Kuala Lumpur, Malaysia. Waste *Perna viridis* shells were obtained from seafood restaurants in Melaka, Malaysia. All chemicals that are used in this research such as 99.9% methanol, (95 – 98%) sulfuric acid, Sigma Aldrich (phenolphthalein), isopropyl alcohol, and KOH pellets were purchased from Polyscientific Chemicals Sdn Bhd.

2.2. Catalyst preparation and characterization

Existing methodologies were used to produce a heterogeneous CaO catalyst from waste *Perna viridis*

[8]. The surface morphologies and elemental composition of the calcined catalyst were analyzed using scanning electron microscopy (SEM) combined with the Energy Dispersive X-ray spectroscopy (EDS) model SUI510 (HITACHI). To obtain high reflectivity throughout the scanning procedure, the samples were coated with gold using a sputter coater (JEOL: JFC-1600) for five minutes. The element composition was analyzed by using an EDS mounted on the microscope. An X-ray fluorescence spectroscopy (XRF-Oxford, ED-2000) was employed to obtain the elemental chemical compositions of the calcined sample for more specific measurement.

The chemical composition of the catalyst was performed on an X-ray diffractometer (XRD) (Shimadzu model XRD-6000) using *Cu-K α* as a radiation source operating at 25 mA and 30 kV over a temperature ranging from 20° to 90° with a step size of 0.04° (2 θ) and a scan step time of 3° per minute. Diffraction patterns observed in XRD were confirmed by comparing their diffraction lines and intensities against solid material samples from the Joint Committee on Powder Diffraction Standards (JCPDS) files. The catalytic activity of the heterogeneous catalysts was determined using the XRD approach.

2.3. Pre-treatment and acid esterification

The physical characteristics and the fatty acid composition of crude *Hevea brasiliensis* oil were examined by conducting physical and chemical testing on the feedstock before biodiesel production. The physical characteristics of all the non-edible feedstock such as acid number, free fatty acid, kinematic viscosity, water content, and density were investigated to ensure the feasibility of using the feedstock to produce biodiesel. Several testing such as viscosity, TAN number, flash point, and density were carried out in Oil Analysis Laboratory at Universiti Teknikal Malaysia (UTeM), Melaka. The test results were tabulated in Table 1. All tests were carried out according to ASTM D6751, EN 14214, and American Oil Chemists' Society (AOCS) methods.

Pre-treatment of the raw feedstock must be carried out to remove any undesired trace components before the acid esterification process. One kg of raw *Hevea brasiliensis* was filled into the reactor and vigorously stirred at a speed of 350 rpm using a magnetic stirrer for an hour. The moisture and water content in the raw feedstock was removed by ensuring that the reaction temperature in the reactor was in the range of 90°C to 100°C using a Type-K thermocouple.

The acid esterification process was a mandatory step carried out to reduce the high FFA content in the feedstock. Approximately 100 grams of unrefined high FFA non-edible *Hevea brasiliensis* were placed into a

beaker and heated to 60°C while being stirred under atmospheric conditions to homogenize the crude oil. The process was carried out using 0.225 to 1 wt% sulfuric acid (H₂SO₄). The molar ratio of methanol used to refine the feedstock also varied from 6:1 to 12:1 in 3:1 ratio interval. The solution in the beaker was placed into a water bath before adding the heated crude oil to avoid burning the methanol and was stirred at a speed of 400 to 600 rpm for 2 hours. The effect of reaction time on efficiency was investigated at 30 minutes to 150 minutes in 30 minutes intervals to optimize the time required to complete the reaction.

Conversely, the products were then poured into a separating funnel and left overnight to allow the separation of excess sulphuric acid, methanol, and impurities that may form on the upper layer of the solution. Then, the acid-esterified oil settled at the bottom of the funnel was heated up at 100°C ± 10°C for 30 minutes to eliminate excess methanol and water from the esterified oil. The remaining amount of FFA content was then determined before conducting the transesterification process.

Table 1. Properties of crude *Hevia Brasiliensis* oil

Oil Properties	
Acid number (mg KOH/g)	104.74
Kinematic viscosity @40°C (mm ² /s)	36.96
Density @15°C (kgm ⁻³)	985
Water content (wt.%)	0.48
Color	Dark brown
Fatty acid composition (%)	
Palmitic (16:0)	9.3
Stearic (18:0)	8.2
Oleic (18:1)	26
Linoleic (18:2)	38.3
Linolenic (18:3)	18

2.4. Microwave transesterification reaction

The biodiesel transesterification process was carried out as a batch process in a modified 1000W, 2450Hz household microwave oven (Model: Samsung). This experimental setup (Figure 1) was placed in Green Technology Vehicle Laboratory in the Faculty of Mechanical Engineering, Universiti Teknikal Malaysia Melaka. On top of the microwave, two holes were drilled; one hole places the overhead mechanical stirrer which was driven by a motor to ensure good mixing and constant temperature while the other hole was drilled to adjust the thermocouple connected to a portable USB-5104 4-channel thermocouple logger inside the microwave. A one-liter beaker and a separating funnel were employed to separate the methyl ester from the reaction mixture. The reaction was optimized using different microwave power inputs, catalyst loading, reaction times, and methanol to oil molar ratios.

The transesterification process was conducted by heating 80 grams of treated *Hevea brasiliensis* at 63 ± 1°C on a hot plate and was continuously agitating at 600 rpm using a magnetic stirrer throughout the process. The methanol and heterogeneous catalyst were mixed according to the pre-determined ratio, where the mixture temperature and oil were raised to 60°C. The changes in mixture temperatures were monitored using a Type-K thermocouple equipped with the data logger and subsequently filtered using a filter paper before being streamed into a separation funnel when it reached a steady-state temperature. The separation of fatty methyl ester (FAME) and glycerol occurred in the separation funnel after 12 hours and then the lower layer glycerol and ester were removed from any excess methanol within its composition.

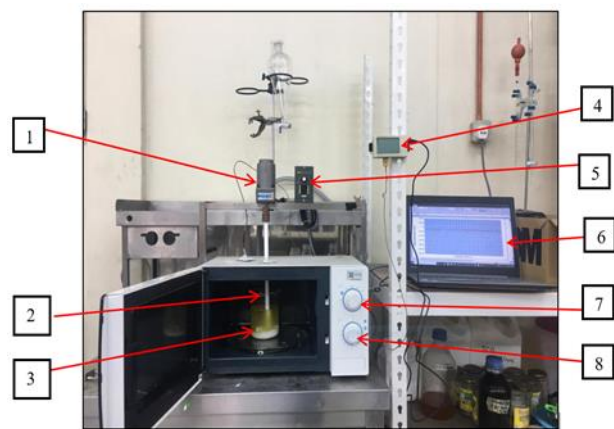


Figure 1 Experimental set up used to perform biodiesel production (1) Overhead mechanical stirrer; (2) Teflon stirrer; (3) batch beaker; (4) thermocouple logger; (5) stirrer controller; (6) monitor display; (7) reaction time controller; (8) microwave power controller

2.5. Fatty acid methyl ester analysis

Gas Chromatography-Mass Spectrometry (GC-MS) was used to analyze the quantity and quality of the produced FAME present in the product sample. To determine the total yield from the produced biodiesel, Eq. (1) was utilized to calculate the yield production [9].

$$FAME(\%) \text{ from GC} \times \left(\frac{\text{Vol. of product}}{\text{Vol. of oil}} \right) \times 100 \quad (1)$$

The conversion of *Hevea brasiliensis* fatty acids to methyl esters *Hevea brasiliensis* was analyzed using GC-MS Agilent 7890A GC system equipped with a 5977B quadrupole mass spectrometer and a 7693 Automatic Liquid Sampler (Agilent Technologies). A 10 mg/mL of methyl ester was dissolved in 1.0 mL of n-hexane for each sample and 1.0 µL of the sample solution was injected into the GC-MS. Helium was used as the carrier gas at a flow rate of 1.0 mL/min and the inlet temperature was set to 250 °C with a split ratio of 50:1.

3. RESULT & DISCUSSION

3.1. Catalyst characterization

This study determined that a high temperature of 900°C and above was the most suitable temperature for the optimized transformation of calcium carbonate (CaCO₃) into calcium oxide (CaO). Table 2 shows the XRF analysis done on the catalysts. Based on the results, it was observed that *Perna viridis* catalysts recorded a calcium oxide (CaO) concentration of 94.88% *Perna viridis* and contained a minor concentration of compounds such as sodium oxide (Na₂O) and magnesium oxide (MgO).

Table 2. Analysis of XRF on commercial CaO and calcined *Perna viridis* at 900°C for 4 hours

Compounds	Composition of catalyst (%)	
	Commercial CaO	<i>Perna viridis</i>
CaO	99.44	94.88
Na ₂ O	0.02	1.92
SrO	0.12	0.33
MgO	0.01	0.18
SO ₃	-	0.32
SiO ₂	-	0.08
Others	0.11	0.04

Figure 2 shows the surface morphologies of the calcined *Perna viridis* derived catalyst using a scanning electron microscope (SEM). The particle morphology of the calcined catalyst was uneven in shape, and some of them joined together as aggregates [10]. Via SEM, the images of the surface roughness with small aggregates particles on the surface can be clearly observed.

EDS coupled with SEM analyzed the elemental composition of the calcined catalysts and observed calcium (Ca) and oxygen (O₂). The compositional analysis result was summarized in the form of a histogram of energy vs. signal strength, which was correlated with concentration (Figure 2). Calcium was found to be the most abundant element in the calcined catalyst, with other elements appeared in minor quantities. This result confirmed the XRF data analysis that carbonate minerals were formed as the calcined catalyst consisted of carbon, oxygen, and calcium.

The catalytic activity and stability of the produced catalyst were measured based on the crystalline structure of the catalyst using XRD. Based on the results, intense sharp peaks of CaO were observed at 2θ value of 29.72°, 39.26°, 47.23°, and 48.77° (Figure 3). This is due to CaCO₃ which was completely

transformed into CaO by evolving the carbon dioxide (CO₂) at multiple temperatures [11]. The higher calcination temperature used will contribute toward sharper peaks that will improve the crystallinity of the CaO [12].

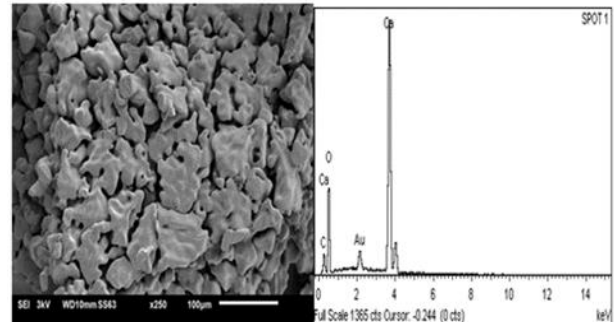


Figure 2 SEM images as well as ED's spectra of calcium oxide catalyst calcined at 900°C for 4 hours for *Perna viridis* (Mussel)

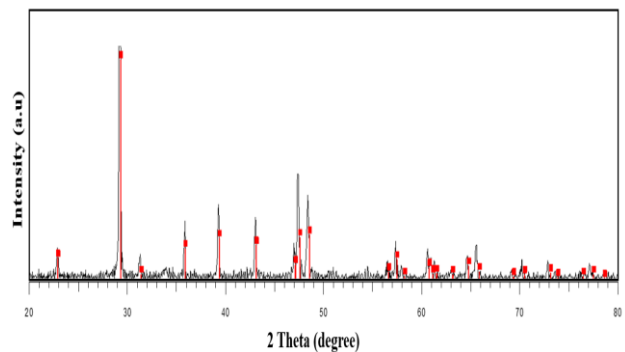


Figure 3 XRD patterns of *Perna viridis* waste shells calcined at 900°C for 4 hours

3.2. Effect of catalyst concentration

The effects of catalyst loading on biodiesel yield production with a ratio of 12:1 methanol to oil are shown in Figure 4. The results indicate that 12 wt.% was the optimum catalyst concentration to yield 96.6% of FAME. Increasing the loading volume to 15 wt.% had caused the biodiesel conversion to decrease to 95.73% and further decrease to 18wt.%, reducing the yield to 95.2% which corresponds to previous work that utilized activated cement clinker and dolomite as a heterogeneous catalyst in their biodiesel production [13]. The FAME yield decreased significantly when the concentration of the catalyst was increased because the equilibrium state had already been achieved and the formation of slurries had made the yield too viscous for sufficient mixing [14].

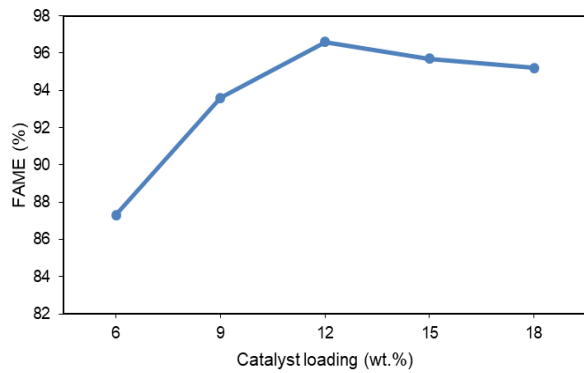


Figure 4 Catalyst concentration influence on FAME yield

3.3. Effect of the molar ratio of the methanol to oil

The effects of methanol to oil molar ratio is one of the most important factors affecting the yield of ester production. Based on the study, it was observed that the yield conversion was starting to increase from 88.7% at the 6:1 ratio to the optimum yield conversion of 96.6% at a 12:1 molar ratio (Figure 5). The molar ratio of methanol to oil at 12:1 is considered the optimum ratio due to the minimum methanol consumption [15]. Once it reached the highest peak of conversion, the conversion is starting to decrease due to the reversible transesterification process [16].

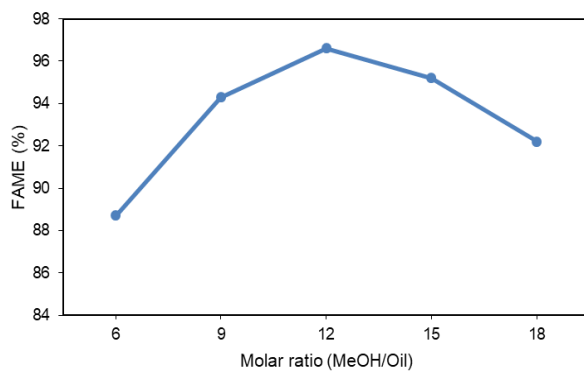


Figure 5 The methanol to oil molar ratio

3.4. Effect of reaction time

A similar pattern was observed with the effects of different reaction times on the FAME yield percentage (Figure 6). Using a 12:1 methanol to oil molar ratio and 12 wt% catalysts loading, the effect of reaction time varied from 3 to 9 minutes. The FAME conversion increased as the reaction time increased, where it was observed that 96.6% of FAME was converted when it was reacted for 7 minutes and subsequently decreased as the reaction time is prolonged. The decrease of FAME yield is contributed by the extension of the

transesterification process, the acceleration of ester formation, and the reconversion of oil to alcohol [17].

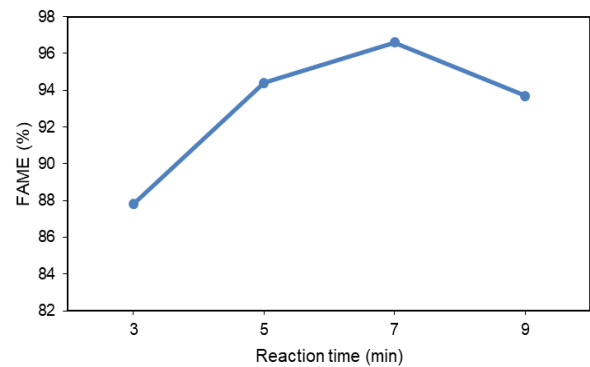


Figure 6 Effect of reaction time on FAME yield

3.5. Effect of microwave power

As shown in Figure7, the biodiesel yield increased as the microwave power increased where 400 Watt produced the highest biodiesel yield at 96.6%. However, caution must be placed to ensure that the microwave power is not too high as it may cause damage to organic molecules (triglycerides) [18].

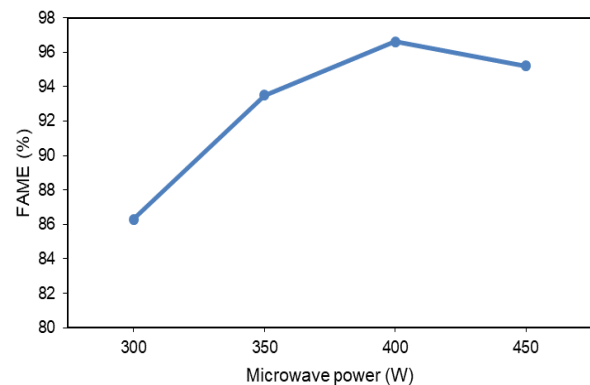


Figure 7 Effect of microwave power on FAME yield

3.6. FAME and fuel analysis

The fuel characteristics of the produced biodiesel have been calculated and compared against the properties of conventional diesel tabulated in Table 4 according to the requirements of ASTM D6751 and EN 14214 standards. The evaluation of fuel properties is very significant to ensure the biodiesel produced can function properly and within the standards required. The results indicated that the flashpoint of the produced methyl ester is higher than that of petroleum diesel, making it safer to store and handle [19]. The kinematic viscosity and fuel acid value of the biodiesel are higher than those of petroleum diesel by about 37% and 47%, respectively. The increase in the value for each parameter shows high water content in the methyl ester that can cause corrosion and engine knocking. The

Table 3. Physical-Chemical Properties of *Hevea brasiliensis* biodiesel

Properties	Unit	ASTM Limits	Testing method	Diesel	<i>H. Brasiliensis</i> , B100
Acid number	mg KOH/g	0.5 max	AOCS-Cd 3d-63	0.17	0.32
Kinematic viscosity @40°C	mm ² /s	1.9-6.0	ASTM D445	2.91	4.63
Density @15°C	kg/cm ³	NS	ASTM D1298	839	894.12
Water content (wt.%)	%. vol	0.05 max	ASTM D2709	NS	0.042
Calorific value	MJ/kg	NS	ASTM D975	45.83	39.19
Flash point	°C	130 min	ASTM D 93	71.5	181.2

Table 4. Main constituents of synthesized *Hevea Brasiliensis* biodiesel

FAME	Compound name	GC-MS yield (%)
C14:0	Myristic acid	0.196
C16:0	Palmitic acid	8.733
C16:1	Palmitoleic acid	0.474
C18:0	Stearic	7.771
C18:1	Oleic	30.268
C18:2	Linoleic	37.092
C18:3	Linolenic	10.653

calorific value recorded 14.5% lower than diesel. The lower caloric value contributed to lower torque and compensate to low energy content in the engine. Density is one of the important parameters to ensure an ideal air to fuel ratios can be achieved. Failure to comply with, will result in incomplete combustion and particulate matter emissions. The density shows quite high value but within the specified range. The composition of *Hevea brasiliensis* biodiesel was identified by GC-MS (Table 4). The two major FAMES were identified as linoleic acid (18:2) and oleic acid (C18:1). The total FFA conversion recorded was 95.19%. The *Hevea brasiliensis* methyl ester has consisted of 10.653% linolenic acid which is complied with the EN 14212 that stated the standard limits for linolenic acid (C18:3) content in the methyl ester should not exceed 12% which can cause a significant decrease to cetane number [20].

4. CONCLUSION

A reusable solid catalyst from waste *Perna viridis* mussel shells was developed by the calcination process for effective biodiesel synthesis. A high *Hevea brasiliensis* biodiesel yield of 96.6% was obtained under the 12:1 MeOH to oil ratio, catalyst concentration with 12 wt%, and 7minute reaction time using 450 Watts microwave power. The biodiesel's physicochemical properties were all within the specified range for biodiesel. *Perna viridis* shells have the potential to be an

environmentally friendly, heterogeneous catalyst for the development of biodiesel production.

AUTHORS' CONTRIBUTIONS

M.M. Zamberi designed and executed the experiments, analyzed the data, and authored the article in consultation with F.N. Ani and M.F.M. Said. N.F. Haminudin assisted H. Bakri and F.S. Anuar helped in interpreting the results and worked on the manuscript.

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