Activated *Paphia undulate* Shells Waste (APSW): A Cost-Effective Catalyst for Biodiesel Synthesis from *Rubber* and *Jatropha curcas* Seeds Oil (RSOME & JSOME)

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Abstract—The Paphia undulata shell, which is available in abundance, has no any further processing for value-added materials and is commonly discarded as solid waste, was used as solid catalyst in a transesterification reaction to synthesize biodiesel from Rubber and Jatropha curcas seeds oil (RSOME and JSOME). The biodiesel non-vegetable oil feedstock and catalyst were prepared, characterized, activated and analyzed using GC, FESEM, BET, DTA-TGA, XRF and FTIR analysis. The activated Paphia undulata shells waste (APSW) had a maximum surface area of 2.1290 m²/g, diameters of 17- 3000 Å, pores adsorption and desorption cumulative volume were 0.006841cm³/g and 0.006928cm³/g with the pores size of 312.527 Å, and it reflected the composition of 83.99 - 86.25 % calcium oxide, etc. The resulted Rubber and Jatropha seeds oil biodiesel yields of 75.93% and 93.33% were found by the methanol/oil ratio of 6:1, catalyst amount of 4 wt.%, temperature of 60 °C and reaction time of 3 hours. The synthesized biodiesels were approved by GC-MS, and its properties fulfills the limitation of specification described by ASTM D6751.

Index Terms—Transesterification, Paphia undulata shells, Rubber and Jatropha oil, biodiesel.

I. INTRODUCTION

The shell wastes of *Paphia undulata* were commonly casted away on the restaurants, food and canneries industry in Malaysia without any eminent utilization for valuable products. Estimated potential annual production is 20,000 MT, and it has been steadily increasing since the 1990s to reach a new record of 13.6 million metric tonnes in 2005. Utilization of the heterogeneous catalyst such as *Paphia undulata* shell for biodiesel does not produce soap through free fatty acid neutralization and triglyceride saponification. Otherwise, the use of metal catalysts for biodiesel synthesis from edible sources like palm oil, soybean, etc. which are consumed as food creates versus fuel dilemma and expensive, thus this work aims to circumvent this issues by using the activated *Paphia undulata* shells waste (APSW) for *Rubber* and *Jatropha curcas* oil transesterification to biodiesel

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(RSOME & JSOME). Biodiesel synthesis using CaO base catalyst has some advantages such as higher activity, mild reaction condition and low cost.

The Rubber and Jatropha based biodiesel offers an alternative feedstock due to the total land area of Malaysia rubber plantation about 1,229,940 hectares which resulted about 355,200,000 kg fat and 136,800,000 kg protein waste per year from rubber seeds, and the Jatropha plantation was 1.5 million acres in year 2009 and 2.5 million acres in 2010, and it could be increased yearly. These potential raw materials, catalyst and methods could be a promising process technology for biodiesel synthesis. Biodiesel known as an alternative and renewable energy for diesel engine that more biodegradable, non-toxic and low emission of CO₂ compared to petroleum diesel [1]. It's also chemically known as long chain of fatty acid methyl ester that produced from various vegetables oil. Synthesis of biodiesel is by reaction of triglyceride and alcohol in the transesterification process [2]-[3]. According to [4], the fluctuation price of biodiesel in market contributes from the used raw material. Thus, there has been depressing needs in finding new feedstock sources and more economical. The feedstock from the vegetables oils become a resources in the production biodiesel due to its renewable and eco-friendly, and also have excellent properties. Vegetables oil as raw material in the methyl ester synthesis is able to alleviate the production carbon dioxide in atmosphere where nowadays it is major worldwide environmental problem.

Basically, the vegetables oil is divided into two types that is edible oil that able to being eaten as food such as coconut oil, soybean, and sunflower [5]. This type of oil was come up with an issue where it disturbes human food chain when applied as raw material during biodiesel production. Meanwhile, non-edible oil is oil that unable to use as food source such as Jatropha, castor, karanja oil and others. This oil has high potential to be used as raw material. The rubber seed (heveabrasiliensis) oil contains high unsaturated fatty acid (80%), it can be realized in the transesterification process for biodiesel synthesis. Rubber seed has widely mixture of chemical composition such as fat, protein and other mineral that has potential to be used in a number of applications such as biodiesel, biolubricant and feed for animals [6]. The world producers of natural rubber are Thailand (35%), Indonesia (23%), Malaysia (12%), India (9%) and China (7%) [7]. During 2009, the rubber plantation area in Malaysia has been growing up to 1.2 million hectares and there were abundance of rubber seeds could be found [8]. On the other hands, Jatropha curcas belongs to Euphorbiaceae family that known as *Jarak Pagar* in Malaysia was commonly found in tropical country and sub-tropical country in the world such as Malaysia, Indonesia, Hong Kong and India [9]. Thus, availability of *Jatropha* oil as raw material can help to reduce price of biodiesel at international market that influenced from the raw material cost and human food chain disturbance. The *Jatropha* oil has also the potential to be used as feedstock in the biodiesel and biolubricant synthesis due to high unsaturated fatty acid content [10].

The developement of heterogeneous catalyst as alternative heterogeneous catalyst such as metallic or metal oxide has wide potential to be utilized where these catalyst can be recycled easily [11]. Beside that, its much cheaper than enzyme or other biochemical catalyst. Currently, the transesterification process of biodiesel using solid base heterogeneous calayst such as calcium oxide that derived from mollusk shells has been investigating continously [12], [13]. The important reason is a new solid base heterogeneous should be developed due to their ablity for yield improvement at lower temperature, shorter time and easy in separation process [14]. The *Paphia undulata* has been supporting offshore of Malaysia since 1970, where the shell can be developed as solid base catalyst for biodiesel synthesis.

Unfortunately, the oil or fat of alkaline transesterification process should be not more than 1% of free fatty acid (FFA) content. The FFA level that above this threshold level, saponification will take place and results the yield decrease of methyl ester. Thus, there is an alternative suggestion by applying two-step process of esterification and transesterification for biodiesel and biolubricant production from high fatty acid oil [10], such as *Jatropha* and rubber seeds oil. This two step process application can be expected a high yield of biodiesel synthesis.

II. MATERIALS AND METHODS

A. Materials

The *Papia undulata* shells waste was collected from resturants and food processing industries, Kuantan, Pahang. The *Jatropha curcas* oil, was procured from Bionas Sdn Bhd., Kuala Lumpur. The *Rubber* seeds were provided by RISDA rubber plantation, Selangor, Malaysia. Methanol, hydrocloric acid and hexane (GC grade) were purchased from Chemmart Asia Sdn Bhd.

B. Methods

The used rubber seed of 20 kg was dried in the oven at 100°C for 4 hours. The dried rubber seed was decorticated manually from the kernel shell. Then, the kernel were milled based on the particles size for an average diameter 1mm for extraction. The used microwave assisted extraction is shown in Fig.1. Then, the rubber seed of 10 g was placed in 250 ml extraction flask with 100ml of n-hexane as solvent. The microwave assisted extraction of rubber seeds oil was operated by 150 W for about 15 minutes with 5 minutes interval for cooling. The hexane-oil mixture was evaporated using rotary evaporator. The oil was then collected and centrifuged at 1000 rpm for 5 minutes for oil separation from other particles. The gas chromatography (GC) was used for

fatty acid analysis. Next, the *Papia undulata* shells was washed using clean water and dried at 100 °C for overnight. Then, the solid catalysts was crushed and calcinated in the furnace at 680 °C for 2 hours. The catalyst was removed from the furnace and kept in desiccators to prevent the air contact. Finally, the raw material (*Papia undulata* shells) and the solid catalyst were characterized using several methods such as Field Emission Scanning Electron Microscopy (FESEM), X-Ray Florescence (XRF), Fourier Transformed Infra Red (FTIR) Spectroscopy, Brunauer- Emmett-Teller (BET) and Differential Thermal and Thermo-Gravimetric Analysis (DTA-TGA).



Fig. 1. Microwave assisted extraction of rubber seed oil.

The bench scale set-up for synthesis of Rubber seed oil methyl ester (RSOME) and Jathropha seed oil methyl ester (JSOME) was illustrated in Fig. 2. It consists of a round bottom flask reactor that placed in an adjusted temperature water bath. Firstly, acid pre-treatment process was applied to reduce level of free fatty acid. The oil was placed in two neck flask equipped with magnetic stirrer and condenser that used as cooling system to prevent alcoholic loss. Then, the catalyst solution (a specified amount of HCl with 1% w/w of oil was dissolved in methanol at molar ratio of 6:1 methanol to oil) was poured into flask. The mixture was heated up to 60 °C for about 3 hour with constant stirrer. After finishing, the mixture was placed in separating funnel for few hours to separate excess methanol. The excess methanol with HCl and impurities moves to the top surface and removed. Meanwhile, the lower layer was collected and continued with base transesterification process. The transesterification process as the second step was run using the heterogeneous catalyst from the Papia undulata shells waste.

For the optimization reactions of RSOME and JSOME synthesis, the experiment was conducted at various temperature levels (45 to 65 °C), amount of catalyst (1 to 5 % w/w) and time of reaction (1 to 5 h). After final separation, the catalyst with other debris remains accumulated in the bottom layer, meanwhile the upper layer known as RSOME and JSOME was collected and washed using warm water (50°C). The washing process was repeated until the water become clear and the methyl ester production was placed on the rotary evaporator for excess methanol removal by heating up to 75° C with the time of 30 minutes.

The analysis of RSOME and JSOME were performed using Gas Chromatography Masspec (GC-MS). The sample was prepared in 2 ml vial with 0.02 ml, it was mixed with 1.980ml of hexane (GC grade). For this purpose, GC-MS (Agilent GC) with the helium gas by the injector temperature of 200 $^{\circ}$ C at split ratio of 30 ml/min was set up.

The initial oven of 70 $^{\circ}$ C with the hold temperature at 3 minutes was set up. After the condition with the isothermal, the oven temperature was increased up to 230 $^{\circ}$ C at heating rate 7 $^{\circ}$ C/min. This operation condition was run for enhancement biodiesel yield. The yield of biodiesel was calculated by the following formula (refer to "(1)").



Fig. 2. The experimental setup for the RSOME and JSOME synthesis.

Biodiesel Yield (%) =
$$\frac{\text{Biodiesel mass} \times \text{Purity}}{\text{Mass of used oil}}$$
 (1)

where biodiesel mass (g), mass of used oil (g), and purity (%) is methyl ester composition from GC analysis.

III. RESULT AND DISCUSSION

A. Field Emission Scanning Electron Microscopy (FESEM)of Papia undulata Shells

The surface morphology of catalyst from *Papia undulata* shells was analyzed by FESEM. Fig. 3(a) shows that the surface of catalyst before activated is rough and disordered surface with low porosity structure compared after activation process. Otherwise, Fig. 3(b) illustrates a relatively smooth with spherical particles of porous structure. The porous structure is fast similar to the another calcined mollusk shells, like cockle shells, etc. [15]. This invention verify that the grain shapes are almost spherical and grain-neck growth due to the thermal decomposition.

B. X-Ray Florescene (XRF) of Activated Paphia undulata Shells

The detailed chemical composition of the solid catalyst from *Paphia undulata* shells waste using X-ray florescene (XRF) shown in Table I. It indicates that a significant CaO content of 86.25% with small amount of other compounds like Na₂O, SrO, MgO, SO₃, Cl, etc. The minor constituents plays not so important role, but the major component of calcium oxide as heterogenous catalyst has mainly contibuted for biodiesel synthesis. The calcium oxide can be resulted also from calcination of *Pomeca sp.* shell, etc. for transesterification process [12].

C. Differential Thermal Analysis and Thermo-Gravimetric Analysis (DTA-TGA) of Paphia undulata Shells

The waste shell was analyzed using DTA-TGA to

determine the suitable calcination temperature during the catalyst preparation for transesterification process that shown in Fig. 4. From the TGA graph, it reflects that 41.76% weight loss is observed on the samples at temperature range 610 to 760 °C. If the temperature is higher than 760 °C, the weight loss is remain almost constant and become ash. Meanwhile from the DTA curve, maximum temperature is found around 600 to 770 °C. From this with taking account the preparation time and energy consumption, the temperature is selected to produce the CaO from the *Paphia undulata* shell at 680 °C.





Fig. 3. FESEM micrograph of *Paphia undulata* shells at 5,000 x, prior to activation (a), post-activation (b).

TABLE I: XRF A	NALYSIS	OF APSW
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Composition	Weight (%)
CaO	86.2
Na ₂ O	1.54
SrO	0.29
MgO	0.17
SO ₃	0.15
Cl	0.07
Fe ₂ O ₃	0.06
P ₂ O ₅	0.06
SiO ₂	0.05
K ₂ O	0.03
Al ₂ O ₃	0.03

D. Brunauer-Emmett-Teller (BET) of Paphia undulata Shells

The major roles in catalyst activity are the specific surface

area and active sites. The BET analysis gave the surface area of *Paphia undulata* shells from 0.9482 m⁷g before calcinations process and after calcination increases up to 2.1290 m⁷g. It is reported that the surface area of calcined cockle shell is 1.56 m⁷g which much lower than calcined *Papia undulata* shells [15]. These results prove that the calcinations process increases the sites of catalyst activity.

E. Fourier Transformed Infra Red (FTIR) Spectroscopy of Paphia undulata Shells

The FTIR spectra of both *Papia undulata* shell before and after calcination was conducted based on the last experiments principles [12]. In this invention, the OH⁻ band was observed at 3643.23 cm^{-1} . The results shown in Fig. 5 and 6. The major peak of CaCO₃ in the uncalcined catalyst occures at 1 471, 856 and 712 cm⁻¹. Meanwhile after calcination major band takes place at 1471.05 876.42 and 558.07 cm⁻¹.









F. Recyclability of Activated Paphia undulata Shells The prepared catalyst is able to be recycled at least five times, with a biodiesel yield nearly 73.52 % and 90 % for RSOME and JSOME (Fig. 7). Before reuse, the spent catalyst was washed with methanol and n-hexane to remove the adsorbed materials. After being used more than five times, the performance of activated *Paphia undulata* shells waste derived catalyst descended gradually. The activation of *Paphia undulata* shell catalyst could be ascribed to its structure change. The structure change of catalyst might be resulted from the reactants (*Rubber* and *Jatropha* seeds oil with hydroxyl group and methanol) contain a little amount of water. However, the heterogenous catalyst is proved to be stable, it can be reused and regenerated by simple calcination for biodiesel synthesis [16]-[18].



Fig. 7. Effect of catalyst recyclability on the biodiesel yield.

G. Effect of Catalyst Loading on the Biodiesel Yield

The catalyst plays a crucial role to create an alternative pathway of reaction besides tendency to lower the activation energy for reaction of molecules reactant [16]. Fig. 8 shown the increase of catalyst amount, also the improvement of biodiesel yield, and the maximum yield of JSOME and RSOME is achieved at 4% w/w catalyst loading. This result is supported by [12] that the obtained optimum biodiesel yield also by using 4% w/w calcium oxide.



Fig. 8. Effect of catalyst loading on the biodiesel yield.

The CaO basic site tendency to transform alcohol group into nucleophile molecule that more active and attach carbonyl group in triglyceride to form alkyl ester and glycerol. If refer to the Fig. 8, the optimum catalyst is higher than loading point, the yield of biodiesel descends. The amount of ester yield is slightly with the increase of amount base catalyst that used KOH due to formation of high soap in the product. Thus, the optimum catalyst loading for tranesterification of *Rubber* and *Jatropha* seeds oil is reflected at 4% w/w activated Papia undulata shells waste.

H. Effect of Time on the Biodiesel Yield

The influence of reaction time on the RSOME and JSOME yield was observed at the temperature of 60°C with the methanol to oil ratio of 6:1 and 4% w/w catalyst loading. Fig. 9 reflects that the optimum transesterification time for RSOME and JSOME yields are obtained at 3 hours. At first few hours of reaction time, the reaction is in slow rate due to dispersion of interaction between molecules still in progress [16]-[18]. Above than that point, the yield decreases slightly from 75.93% to 75% for RSOME and 93.33% to 91.09% for JSOME (Fig. 9).The yield reduction of biodiesel synthesis is due to backward reaction which causes more soap formation.



Fig. 10. Effect of temperature on the biodiesel yield.

I. Effect of Temperature on the Biodiesel Yield

The effect of temperature on the RSOME and JSOME synthesis was tested by the methanol to oil ratio 6 to 1 with 3 hours of reaction time and 4% w/w (catalyst/oil weight ratio). Another catalyst, like egg shell waste has been approved for biodiesel synthesis from non-vegetables oil by various temperatures [16]. By the maximum temperature of $60 \,^{\circ}{\rm C}$ gives the maximum yield of RSOME and JSOME are 75.93% and 93.33% (Fig. 10). The yield of RSOME and JSOME decreases slightly above this temperature. The high temperature is able to supply sufficient energy for collision between reactant molecules that promote towards maximum reaction. Unfortunately, above $60 \, \mathrm{C}$ favorable tends to accelerate the saponification of the triglyceride before completion of the transesterification process. The process condition of 60 °C has also been obtained for biodiesel synthesis from waste frying oil through two-step catalyzed process using methanol [17]. The use of methanol for transesterification of soybean oil has also been done under batch condition [18]. As the temperature was more than the methanol boiling point $(64.7 \,^{\circ}\text{C})$, the biodiesel yield decreased due to vaporization. The percentage yield of RSOME is slightly lower than JSOME. The optimum temperature for biodiesel synthesis is depends on the fat and the oil used. Thus, this invention shows that RSOME and JSOME shares with the same optimum temperature of $60 \,^{\circ}\text{C}$, but the different synthesis yield due to fatty acid content.

IV. CONCLUSION

The activated Paphia undulata shells waste can be utilized as a cost-effective catalyst for biodiesel synthesis from non-vegetable oil. The characterization and analysis results of activated catalyst shown a large surface area compared non-activated catalyst. The catalyst weight loss after activation occurred 41.72 % at temperature of 680 °C, and it causes the chemical structure and composition change of the catalyst. The calcined catalyst gave the maximum calcium oxide of 86.25%. This activated catalyst containing high calcium oxide exhibited a good performance in synthezing biodiesel. The highest biodiesel yield (75.93% of RSOME and nearly 93.33% for JSOME) was observed at temperature of 60 °C by catalyst addition of 4 wt.% to a mixture of methanol-oil ratio of 6:1 via batch system for 3 hours. Besides that, the catalyst still maintained the activity and crystalline phase after being reused 5 cycles, and it had high recyclability up to 73.52 % and 90 % for RSOME and JSOME. The resulted JSOME and RSOME properties fulfills the limitation of biodiesel specification, and the biodiesel yield can be improved by the development of various another process parameters, like methanol-non edible oils mixture ratio, etc. and heterogenous catalysts via the multi-step purification process.

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