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Microwave-assisted conversion of agro-industrial copra residue oil to diesel engine compatible fatty acid methyl esters

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Abstract. Due to the increasing concern towards fossil fuel reserve and its fluctuating price, many countries shifted towards producing biodiesel as an alternative green fuel. In this study, the main objective is to synthesis biodiesel from coconut copra, an agro-industrial residue, as potential feedstock using different transesterification techniques, namely stirring mechanical-assisted technique (SAT), ultrasonic-assisted technique (UAT), and microwave-assisted technique (MAT). The current study will show comparison between the biodiesel yields, fatty acid methyl ester (FAME) compositions and fuel properties of the transesterification techniques used. The result showed that the copra residue biodiesel (CRB) using SAT produced the highest yield of biodiesel compared to UAT and MAT, which are 96.85% (± 0.69), 93.79% (± 1.00), and 91.76% (± 0.87), respectively. Furthermore, different volumes of solvent and catalyst amount were found to affect the biodiesel yields, relatively. On the other hand, for FAME compositions, insignificant results were observed. The fuel properties in CRB complied with the international standards, thus, showing high compatibility to be used in diesel engine.

1. Introduction

Nowadays, the production of energy is constrained, which could be seen through the decline in fossil fuel reserve and fluctuating price each year [1]. Without any doubt, the utilization of fossil fuel also contributes to emissions of greenhouse gasses (GHG), the main contributor towards global warming [2]. Therefore, finding the sources of new energy becomes more important than before. By 2030, Malaysians' demand for energy will rapidly increase to almost 100 million tons of oil equivalent and there is an urgent need to explore and increase the utilization of renewable energy such as biodiesel [3]. Biodiesel is a type of fuel that is green and environmental-friendly in comparison to petroleum diesel fuels and it is a type of renewable energy that can be regenerated in a short period of time. Thus, the selection of suitable feedstock for biodiesel production is important to make sure that it is sustainable and natural biomass such as *Cocos nucifera* (coconut) can be considered as a potential feedstock for biodiesel.

Cocos nucifera or coconut is derived from the Aracacea family, the subfamily of cocoidea. The coconut oil can be categorized in two types; virgin and refined. The virgin oil is produced from fresh fruit (ripe) while refined oil is extracted from dry coconut named copra [4]. *Cocos nucifera* oil, which



is extracted from copra can be used for many purposes such as cooking oil, hair oil, and lamp oil [5]. The coconut oil yield contains high triacylglycerol composition. The lauric acid makes up the most it, followed by myristic acid, and linoleic acid [6]. These components are essential in biodiesel production.

In this study, oil extracted from copra residue is evaluated for its potential as a renewable biodiesel production alternative. Copra residue is mostly generated from agricultural food industry especially during coconut transportation and food production [7]. This residue was regarded as waste and mostly end up in landfill. Copra residue oil (CRO) are suitable for biodiesel production as the coconut trees normally bear fruit throughout of the year with high copra residue left unattended.

2. Methodology

2.1. Preparation of materials

Matured *Cocos nucifera* copra residue were collected from small and medium agro-industries located in Bangsar, Kuala Lumpur. The sample was washed properly prior for drying. The sample then grinded using a Wonder® blender (WB-1) into powder form after oven dry using mild temperature of 60°C, to prevent thermal deterioration of the unsaturated fatty acids (the drying process stop after the sample reach constant moisture content). The oil extraction was using the ultrasonic-assisted techniques for 30 minutes with 300 mL of hexane as solvent.

2.2. Transesterification techniques

The conversion of the CRO into biodiesel was done using transesterification which assisted by three different techniques, namely stirring mechanical-assisted (SAT), ultrasonic-assisted (UAT), and microwave-assisted (MAT). The UAT was done by employing ultrasonic water bath with ultrasonic power 120 W, frequency 40 kHz, 3 L capacity and 240 × 140 × 100 mm of internal size dimension. While, MAT used modified domestic microwave which 800 W for power, 200 rpm magnetic stirring, 25 L capacity and 315 × 227 × 349 mm of interior dimension. The solvent used for transesterification reaction is methanol and potassium hydroxide (KOH) served as catalyst. The transesterification reaction will use 3:1, 6:1, 9:1, 12:1, and 15:1 methanol to oil molar ratio (v/w) with 0.5%, 1.0%, 1.5%, 2.0%, and 2.5% of KOH to oil (w/w). Before mixing the sample, the KOH needs to dissolve in methanol first to form sodium methoxide solution. The reaction was set at 65°C temperature and stirred for 30 minutes or until the reaction occurs. Once esterified, the sample was transferred to the separatory funnel for 24 hours. The final product of transesterification reaction produced two layers of compounds known as biodiesel or fatty acid methyl acid (FAME) at the top and glycerol at the bottom. The FAME was siphoned off for analysis. All the 25 experiments being run for all techniques were statistically analysed using Minitab version 18 by employing ANOVA to verify the models' accuracy. All the experiments were performed in triplicate (n=3).

2.3. Test methodology

The copra residue biodiesel (CRB) fuel properties were analysed for ester content, density, kinematic viscosity, cloud point, cold filter plugging point, pour point, and oxidation stability. The ester content was analysed using Gas Chromatography-Flame Ionization Detector (GC-FID) with oven temperature programmed to start and hold at 150°C for 15 minutes, increasing by 210°C at 2°C/min, the 50°C/min to 220°C, and then hold at 200°C for 5 minutes with nitrogen as the carrier gas. Injector and detector temperatures were set at 240°C and 28°C respectively. The sample was prepared according to AOCS Official Method AOCS Ce2-66 [8]. Density was determined using I-type hydrometer while kinematic viscosity was evaluated using Automatic Kinematic Viscosity Measuring System AKV-201 at 40°C. The pour point and cloud point analysis were done using Mini Pour/Cloud Point Tester MPC-102 while cold filter plugging point determined using Automated Cold Filter Plugging Point Tester AFP-102. The oxidation stability was determined by Rancimat 743 (Methrom, Herisau, Switzerland).

3. Results and discussion

3.1. Effect of different transesterification techniques

The comparisons of CRB yield between SAT, UAT, and MAT transesterification techniques are shown in figure 1. Results observed optimal value of 96.85% (± 0.69) of CRB yield when using SAT technique while UAT and MAT are 93.79% (± 1.00) and 91.76% (± 0.87), respectively. Statistically, tables 1-3 showed the fitness of developed models of SAT, UAT and MAT techniques at $R^2=0.9858$, $R^2=0.9860$ and $R^2=0.9884$, respectively. In addition, the table also tabulated the significant level of the developed parameters models for all transesterification techniques which the p-value less than 0.05.

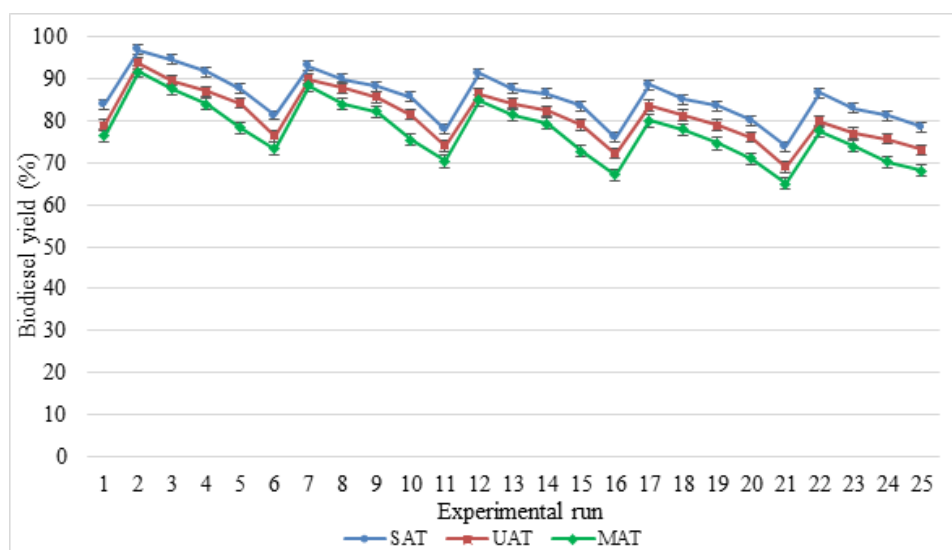


Figure 1. Biodiesel yield through 25 experimental run using different techniques; SAT, UAT, and MAT

Table 1. Analysis of variance for SAT

Source	df	Sum of square	Mean of square	F-value	p-value
Solvent ratio	4	984.33	246.083	356.26	0.000
Catalyst	4	1403.37	350.842	507.93	0.000
Solvent ratio*Catalyst	16	12.16	0.760	1.10	0.380
Error	50	34.54	0.691		

Mean – 85.48, standard deviation – 5.74, standard error of mean – 0.66, variance – 32.90, R^2 – 0.9858, adjusted R^2 – 0.9790, predicted R^2 – 0.9681

Table 2. Analysis of variance for UAT

Source	df	Sum of square	Mean of square	F-value	p-value
Solvent ratio	4	1301.12	325.280	415.88	0.000
Catalyst	4	1427.81	356.952	456.38	0.000
Solvent ratio*Catalyst	16	25.58	1.599	2.04	0.028
Error	50	39.11	0.782		

Mean – 81.12, standard deviation – 6.14, standard error of mean – 0.71, variance – 32.75, R^2 – 0.9860, adjusted R^2 – 0.9793, predicted R^2 – 0.9685

Table 3. Analysis of variance for MAT

Source	df	Sum of square	Mean of square	F-value	p-value
Solvent ratio	4	1535.29	383.822	464.79	0.000
Catalyst	4	1948.91	487.228	590.01	0.000
Solvent ratio*Catalyst	16	44.29	2.768	3.35	0.001
Error	50	41.29	0.826		

Mean – 77.44, standard deviation – 6.95, standard error of mean – 0.80, variance – 48.24, R^2 – 0.9884, adjusted R^2 – 0.9829, predicted R^2 – 0.9740

Table 4 depicts the summary of comparisons between transesterification techniques. SAT technique by using heat transferring from the plate to the reactant from outside to inside and stirring ability instigated the conversion reaction of CRO to CRB [9, 10]. Further, the UAT with its ultrasound irradiation produced cavitation impact and, at the same time, it enhanced the reactant surface to facilitate the transesterification. For MAT, the microwave power affected the molecules polarity and ions (alcohol) by altering the magnetic field. In addition, due to high frequency, vibration that caused the molecules to collide and heating occurred from rapid rotation, the reaction of separation methyl ester from glycerol occurred [11]. Furthermore, SAT consists of ability in synthesising biodiesel in high scale compared to UAT and MAT. In addition, UAT contains more advantages such as low-cost production, short time reaction, and can run a few experiments simultaneously without affecting the quantity and quality of yield. In terms of disadvantages, MAT is not recommended since it produces the lowest biodiesel yield, hard to control, and it is not possible to imply in large scale industry [12-15].

Table 4. Comparison of transesterification using different techniques: SAT, UAT and MAT [9-15]

Characteristics	Transesterification techniques		
	Stirring mechanical-assisted (SAT)	Ultrasonic-assisted (UAT)	Microwave-assisted (MAT)
Reaction interaction	a. Heat transfer from outside to inside. b. Stirring enhanced the reaction.	a. Ultrasound waves produced cavity effect and escalated reaction process. b. Optimizing reactant surface.	a. Microwave irradiation affecting molecules of polar and alcohol ions by altering magnetic field. b. Molecules collided due to high frequency of vibration. c. Heating occurred due to rapid rotation.
Yield (%)	High	Moderate	Low
Solvent to oil molar ratio (v/w)	3:1	3:1	3:1
Catalyst concentration	1%	1%	1%
Advantages	Can conduct large amount of transesterification, easily controllable.	Fast reaction, can run a few experiments simultaneously without negative impact, economical.	Fast reaction, consistent.
Disadvantages	Long reaction time	Consumed time to produce heat	Hard to control, not possible to apply in large scale industry.

Table 5 shows the comparison of biodiesel yield using coconut oil with different feedstock from the previous studies. Most of the feedstock in the table was commercially produced in different countries. The study revealed that coconut oil produced relatively high biodiesel yield (98.00%) but slightly lower compared to waste vegetable oil (100.00%), soybean oil (99.00%), and castor oil (99.00%) compared to the other feedstock. However, waste cooking oil (96.78%), rapeseed oil (96.00%), algae oil (94.27%), sunflower seed oil (90.00%), and used frying oil (85.30%) depicted lower yield of biodiesel produced. In brief, it can be noticed that differences in transesterification techniques do give different results of biodiesel conversion yield [16-19].

Table 5. Comparison of biodiesel yield with other sources [16-19]

Feedstock	Technique	Biodiesel yield (%)
Coconut oil	Ultrasonic	98.00
Soybean oil	Ultrasonic	99.00
Soybean oil	Mechanical stirring	97.00
Castor oil	Ultrasonic	99.00
Rapeseed oil	Mechanical stirring	96.00
Sunflower seed oil	Ultrasonic	90.00
Waste vegetable oil	Microwave	100.00
Waste cooking oil	Ultrasonic	96.78
Used frying oil	Mechanical stirring	85.30
Algae oil	Mechanical stirring	94.27

3.2. Effect of solvent molar ratio

Figure 2 shows the effects of the solvent to oil molar ratio (v/w) for all transesterification techniques: SAT, UAT, and MAT. From the results, it was shown that the optimum solvent to oil molar ratio (v/w) was 3:1 as validated by all techniques. The results depicting 3:1 solvent to oil ratio (v/w) synthesised the highest CRB yield for SAT, UAT, and MAT with 96.85%, 93.79%, and 91.76%, respectively. On the other hand, the results displayed that if the solvent ratio increased, the biodiesel yield showed a reversed trend. The results were supported by previous studies, which stated that solvent to oil ratio is an important parameter since it gives major effect in producing biodiesel because low ratio could cause the transesterification reaction to be incomplete whereas if the ratio is too high, it would decrease the yield [11, 14]. The usage of methanol as solvent for transesterification was suggested by the previous studies because of its reactivity characteristic, but excessive ratio could lower the yield because of the solubility of glycerol that is affected by increasing polarity, which makes the separation process of methyl ester become harder [13, 15].

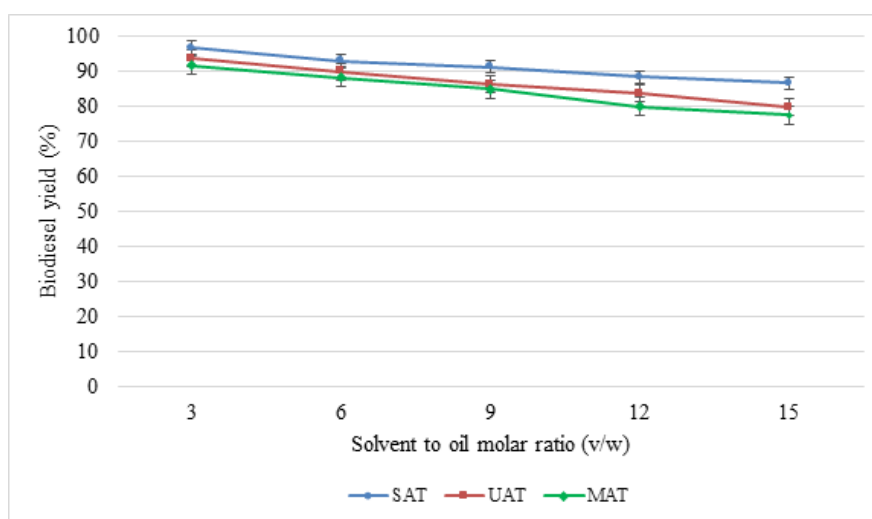


Figure 2. Effect of solvent amount to oil molar ratio (v/w) on biodiesel yield at 1% of catalyst

3.3. Effect of catalyst concentration

The employment of catalyst concentration as parameter in synthesis of biodiesel is important because the percentage of catalyst could affect the transesterification by which it could cause saponification reaction to occur [20]. Figure 3 depicts the effect of catalyst concentration used in the experiment. The results displayed consistency of optimal CRB yield for all of the techniques by employing 1% catalyst to oil (w/w). Overall, the results observed decrease in the pattern of CRB yield after optimal value was reached. This is supported by previous studies, which mentioned that too low or too high catalyst concentration could cause incomplete reaction and give lower yield [21-23]. The results of reactions without using any catalyst was not shown but prior research showed that without catalyst, no reaction occurred, unless the reaction was done at supercritical condition [2].

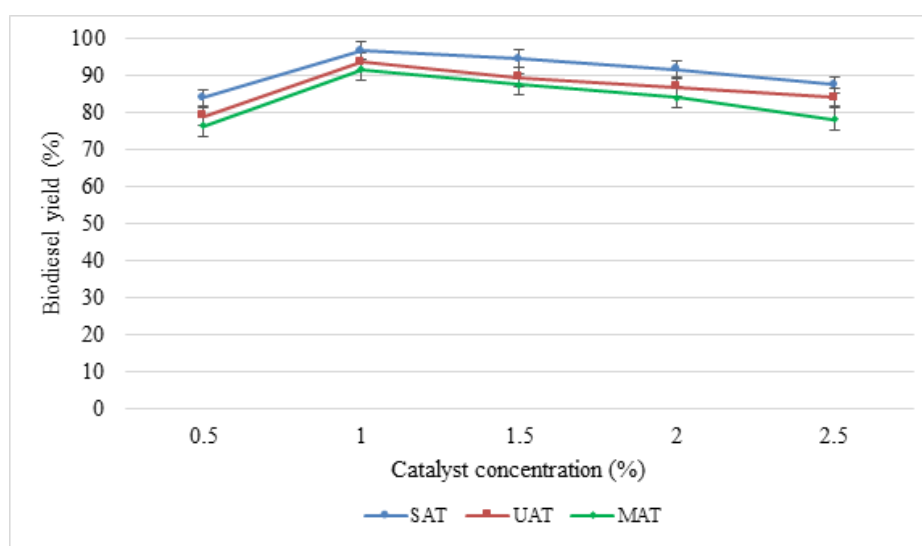


Figure 3. Effect of catalyst concentration on biodiesel yield at 3:1 solvent to oil molar ratio (v/w)

3.4. Fatty acid methyl esters (FAME) composition

Table 6 show FAME composition of CRB from different transesterification techniques, which are SAT, UAT, and MAT. All the analyzed FAME used CRB derived using 3:1 solvent to oil molar ratio

(v/w) and 1% catalyst concentration to oil (w/w). The results indicated that there were 9 types of FAME found in CRB from all techniques, namely methyl caproate (C6:0), methyl caprylate (C8:0), methyl caprate (C10:0), methyl laurate (C12:0), methyl myristate (C14:0), methyl palmitate acid (C16:0), methyl stearate (C18:0), methyl oleate (C18:1), and methyl linoleate (C18:2). All transesterification techniques depicted methyl laurate as the highest compound of FAME with 47.8% (SAT), 48.1% (UAT), and 47.9% (MAT) in comparison to other compounds. On the other hand, methyl caproate was displayed as the lowest compound in CRB as converted using all the techniques with approximately $\leq 0.8\%$ [17, 24].

3.5. Copra residue biodiesel fuel properties

Table 7 shows the CRB fuel properties and comparison with international standard: ASTM D6751-07 and EN 14214 [17, 25, 26]. The fuel properties such as cetane number, cloud point, oxidation stability, and so on play major roles in determining the quality of produced biodiesel. The kinematic viscosity (K.V.) tabulated good content which in the range both of international standard EN and ASTM, the K.V. content could lead to fuel atomization in the engine's combustion chamber. The cetane number (CN) affects the ignition of fuel delay; the higher the number of cetane, the less occurrence of delay in fuel ignition. The table shows that ASTM and EN put the standard for cetane number where the value should be more than ≥ 47 to have good ignition delay. From the study, it was showed that all of CRB yield contained cetane number of more than 64. The cetane number value is influenced by the presence of saturated fatty acid such as palmitic acid. Next, the results displayed the predicted cloud point (CP) result for CRB yield (-0.3 to 0°C). The cloud point is the point where solidification occurred. As CP, the cold filter plugging point (CFPP) is a temperature where crystallization occurs and will cause clogging inside the fuel filter. Hence, as cetane number, the saturated fatty acids found in CRB yield gave high impacts to CP and CFPP values [16, 27].

Furthermore, oxidation stability is also important in determining biodiesel quality because it will determine the extensiveness of biodiesel usage. The stability of oxidation is influenced by the presence of air, heat, light, and especially the fatty compound structure itself (double-bond structure). Previous studies mentioned that high presence of saturated fatty acids concentration could give high stability [16, 27, 28]. In brief, the presence of saturated fatty acids compound in CRB potentially produced high quality of biodiesel production [29, 30]. In addition, the properties contents were mostly in agreement with the previous study of coconut biodiesel [17]. However, the produced biodiesel has not been run in diesel engine and tested for its emission, and those points will be interesting to be studied in the future.

Table 6. Comparison of fatty acid methyl ester profiles using different techniques [17, 24]

FAME Composition		Techniques			Coconut biodiesel
		SAT	UAT	MAT	
C6:0	Methyl Caproate	0.5	0.6	0.6	0-0.8
C8:0	Methyl Caprylate	7.9	8.6	8.1	5.0-9.0
C10:0	Methyl Caprate	6.3	6.7	6.3	6.0-10.0
C12:0	Methyl Laurate	47.8	48.1	47.9	44.0-52.0
C14:0	Methyl Myristate	18.0	17.5	17.9	13.0-19.0
C16:0	Methyl Palmitate	9.4	9.0	9.3	8.0-11.0
C16:1	Methyl Palmitoleate	-	-	-	2.5
C18:0	Methyl Stearate	3.1	2.9	3.0	1.0-3.0
C18:1	Methyl Oleate	5.8	5.5	5.7	5.0-8.0
C18:2	Methyl Linoleate	1.2	1.1	1.2	0-1.0
C20:0	Methyl Eicosanoate	-	-	-	0-0.5

Table 7. Fuel properties of copra residue biodiesel in comparison with international standards [17, 25, 26]

Properties	Unit	EU (EN14214)	US (ASTM D6751-07)	Techniques			Copra Residue biodiesel
				SAT	UAT	MAT	
Density	g/cm ³	0.860-0.900	-	0.871	0.871	0.871	0.807
K.V. (40°C)	mm ² / s	3.50-5.00	1.90-6.00	2.27	2.23	2.26	2.73
Cetane number	-	≥51.00	≥47.00	64.71	64.67	64.68	60.00
Cloud point	°C	-	-3.00 to 12.00	-0.05	-0.26	-0.10	-
CFPP	°C	-	-	-8.65	-9.09	-8.84	-4.00
Flash point	°C	≥101	≥93	-	-	-	110
Pour point	°C	-	-15.00 to 10.00	-6.87	-7.10	-6.93	-
Oxidation stability	h	≥6	≥3	100.87	109.80	100.87	35.5

4. Conclusion

The study emphasized the effect of transesterification using different techniques, namely SAT, UAT and MAT with different parameters models on biodiesel yield, FAME composition and physicochemical properties. The study observed that SAT produced highest yield compared to UAT and MAT which 96.85% (± 0.69), 93.79% (± 1.00), and 91.76% (± 0.87), respectively. For Solvent molar ratio, the study showed that 3:1 solvent to oil molar ratio (v/w) and 1% catalyst to oil (w/w) are the most suitable condition to convert the CRO to CRB. On the other hand, the FAME composition evaluation showed insignificant results since biodiesel yield produced from all the techniques depicted almost the same compound types and amount which the methyl laurate is the highest compound (47-48%). Fuel properties of biodiesel which produced from CRO also complied with most of the properties listed in international standards showed high potential to be used in diesel engine. In addition, the high oxidation stability content on the CRB give an advantage for a long period storage, thus, potentially to be used as feedstock for sustainable biodiesel feedstock.

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