Non-Catalytic Microwave Assisted Transesterification of Palm Oil with Dimethyl Carbonate

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Abstract: *A reactor-condenser microwave (600W) was modified as an assisted method for continuous transesterification of palm oil. The high free fatty acid oil was simultaneously neutralized and trans esterified with dimethyl carbonate. With the DMC to oil molar ratio of 10:1, 7:1 and 5:1, with temperature range of 150 degrees to 250 degrees, 2 to 4-hour residence time, the continuous conversion of palm oil to ethyl ester was over 90%. The palm oil biodiesel was analyses using FTIR analysis to determine the conversion yield. Most ideal ratio was figured out to be 1:7 (oil to DMC) and it continue to next 4 hour of heating to obtain the best result. The maximum conversion yield achieve was 95.9% and the density, viscosity also fuel properties achieve ASTM standard.*

Keywords: Microwave reactor; Non-catalytic; Palm Oil; FTIR analysis

1 Introduction

The world is evolving, creating a new developed civilization which make the petroleum or fossil fuel as an important aspect. Nevertheless, the source was limited because it will be depleted. Yet, the energy demand keeps increasing while the power supply industry was neck on neck to fulfill the demand. Even worse, the main feedstock used to generate energy is mostly nonrenewable resources. Hence, the research nowadays have been shifted to renewable energy in order to support sustainable development, especially biofuel (Lin & Chen, 2017). Biofuel is getting more attention,

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particularly biodiesel, to reduce the greenhouse effect and air pollution. Biodiesel, also known as fatty acid methyl esters (FAME), chemically can be synthesized through the chemical reaction between oil (usually vegetable oil) and alcohol in the presence or absence of catalyst (Nomanbhay & Mei Yin, 2017, Sahar *et al.*, 2018). The feedstock that usually used in producing biodiesel are palm oil, rapeseed oil, canola oil, bacteria, algae, waste cooking oil (WCO) etc. As one of the huge producer of palm oil, the research in Malaysia has seemed take interest in palm oil industry. The palm oil industry was developed massively as the demand was increasing for last 25 years, and now, enhancement has been made by focusing on the quality improvement, disease resistant and high increment on palm oil (Masani *et al.*, 2018). A study was done by El-Araby *et al.*, (2018) on the palm oil–biodiesel–diesel fuel blend properties (viscosity, density and flash point) in diesel engine where they found that palm oil properties in engine performance has no difference with regular diesel oil. Hence, this has made the palm oil and blended diesel-palm oil have huge potential in transportation industry.

There were many methods can be used to convert the feedstock into biodiesel or biofuel. Several conversion method and technology have been studied and validated its effectiveness. Among the available technologies, the chemical conversion method has been proven as one of the simplest and widely-used process in producing biofuel. Xiang *et al.*, (2017) studied on the effect of modified coal fly ash as catalyst in increasing the biodiesel yield of waste cooking oil. The highest biodiesel yield of 94.91% was obtained with the ratio of 1:9.67 (oil to methanol). The yield was increased by 90% if the catalyst was used for 8 times. Besides, Sahar *et al.* (2018) also conducted transesterification on waste cooking oil using reactor equipped with reflux condenser and the result getting the achievement of FAME yield up to 94% with 1:3 oil-methanol ratio and 1 percent of catalyst. The microwave-assisted (thermochemical) conversion have become more demanding than other method and it was evolving through quite some time as some study shows its effectiveness in achieve high conversion yield for short of time (Lertsathapornsuk *et al.*, 2008)

Furthermore, superior advantages in term of time and product yield was also observed when microwave processing technology was implemented into transesterification reaction (Li *et al.*, 2018). Electromagnetic waves that emitted from magnetron has the ability to make the material absorb the energy and convert it into heat, providing volumetric heating effect. Study done by Ding *et al.*, (2018) using microwave irritation condition on transesterification process for biodiesel production concluded that biodiesel yield can be achieved up to 98.93% with molar ratio of 1:11 oil to methanol. This has also proven that microwave-assisted transesterification is an efficient and environmental friendly biodiesel production method. The research keep continues to find the best way to convert feedstock to biodiesel but not much on palm oil itself. This paper will focus on the non-catalytic conversion of palm oil into biodiesel with microwave-assisted method at milder condition using dimethyl carbonate.

This paper focus on non-catalytic transesterification process as it provides simpler purification and environmentally friendly post-processes (Cho *et al.*, 2012). Besides, without catalyst, the problems, such as the formation of unwanted soap instead of biodiesel due to the high free fatty acid content in the feedstock, will be eliminated. Diasakou *et al.*, (1998) investigated the transesterification of soybean oil with methanol for non-catalytic thermal method at 220-235°C, They found that diglycexide and triglyceride conversion rates are much higher than the conversion rates of monoglyceride to glycerol. A non-catalytic with supercritical methanol allows a simple process and obtained high yield in biodiesel production despite also having likely same properties with biodiesel and petrodiesel make the transesterified product of vegetable oil considered the most promising one to substitute diesel (Demirbas, 2006). There were also two-step method suggest by Minami and Saka (2006) which include the hydrolysis step in the transesterification process to against the presence of water in oil/fats and it was high tolerant. Asri *et al.*, (2013) figured out that non-catalytic of transesterification process for vegetable oil in supercritical methanol can overcome the flaw of homogeneous catalyst process. Ilham and Saka (2011) in other hand study on the potential of dimethyl carbonate as non-catalytic reactant and found out that it can be a good candidate in the supercritical condition. However, the studies mentioned above required high temperature and pressure operating condition, which lead to high energy input and production cost. Hence, non-catalytic transesterification under milder condition is needed to be explored further. To the best knowledge of the author, there are little information about the noncatalytic microwave-assisted transesterification under subcritical condition. In this paper, the effect of oil-to-dimethyl carbonate (DMC) molar ratio and reaction temperature on biodiesel conversion yield was investigated under microwave irradiation using non-catalytic transesterification method.

2 Material and Methods

This study was devoted to convert the palm oil mixed with dimethyl carbonate (DMC) to biodiesel using most economic method, which is by microwave-assisted reaction. This noncatalytic experiment was conducted at optimum and ideal measured where the transesterification process in the microwave reactor also been studied. The gained sample was been analyzed using Fourier-transform infrared spectroscopy (FTIR) to determine the conversion of yield of ester and decided which sample give the highest percentage of conversion yield. Technically, the palm oil was heated in the microwave before pouring in the DMC according to the decided molar ratio and will in constant heat in the range of 150 degree to 250 degree Celsius. The sample was been drawn out at the end of each experiment and was analyzed.

2.1 Materials

Refined commercial palm oil was obtained from local store located at Bangi, Selangor. This oil has main fatty acid composition: myristic acid 1.1%, lauric acid 0.2%, stearic acid 4.5%, linoleic acid 10.1%, oleic acid 39.2%, palmitic acid 44.0%, arachidic acid 0.1%, and linolenic acid 0.4%. While for the dimethyl carbonate (DMC) was purely analytical and was purchased from Sigma Aldrich company (99%, MW : 90.08 g/mol)

2.2 Preparation of Biodiesel

The biodiesel was prepared in 2L beaker with a magnetic stirrer, a thermometer and watercooled condenser for transesterification reaction. The palm oil (762.57g) and DMC (810.81g) was displaced into the beaker at predetermined temperature with different palm oil and DMC mole ratio (1:5, 1:7 and 1:10).

2.3 Palm Oil Transesterification

The total reaction volume of 1.58 liters was considered as constant for transesterification process. The sample was heated up in range of 150 degrees to 250 degrees for about 2 hours while the microwave was set to 600 watts. Sample was then withdrawn from microwave using pump at every two hours at determined temperature. The sample was then analyze before decided the ideal ratio blend of oil and dimethyl carbonate for heat it up again up to 4 hours.

2.4 FTIR Analysis

The sample was undergone Fourier-transform infrared spectroscopy (FTIR) to obtained infrared spectrum of emission of absorption of a sample. The measurement was performed simply by pouring a droplet of liquid sample onto crystal surface. Before that, background spectrum has to be obtained to avoid any disruption from the outside factor on the result. Cleaning was done with trisolvent mixture of acetone – toluene – methanol, this was employed to clean the sample crystal before background scan. The scan results were obtained on the incorporated computer system as spectra. Total require time for spectral collection takes approximately 5 minutes per sample. The spectra were recorded within the range of 4000 to 600 cm^{-1} with 4 cm^{-1} and HappGenzel appodization (Rabelo *et al*., 2015). 20 scans were calculated on each spectrum subjected to background subtraction. Analyses were carried out in triplicates and the average of the three were used to construct the models of spectra

2.5 Analysis of Sample

Sample prepared as described above were analyzed to determine its conversion yield. Mixture of fatty acid methyl ester, FAME was calculated using calibration method with the sample gain and find the percentage difference between actual value of biodiesel and FAME

$$Percentage\ difference = 100 * |a - b| / \left(\frac{(a+b)}{2}\right) \quad \textbf{(1)}$$

$$Yield\ (\%) = 100\% - Percentage\ Difference \quad \textbf{(2)}$$

3 Result and Discussion

3.1 Effect of molar ratio of oil to DMC

The molar ratio between DMC and palm oil played an important measure in determine the ester conversion yield. The stoichiometry of this reaction requires at least three moles of methanol or two moles of DMC per mole of vegetable oil to yield three moles of fatty ester and one mole of glycerol (Anastopoulos *et al.*, 2009). It was proven that increasing of DMC concentration in molar ratio helps the mass transfer to be increase as the reaction mixture overall viscosity decreased (Panadare & Rathod, 2016). Nevertheless, it required more power to increase the working volume of overall reaction. In nature itself, FAME are reversible which leads to more reactant need to be added to counter this forward reaction. Considering this facts, the sample was made of three different molar ratio palm oil to DMC with range of 1:5, 1:7 and 1:10 where DMC should be more than oil ratio itself. Logically, glycerol will be formed as impart product because of the enzyme binding active site when using other solvent. To combat this, DMC was used to partially solved this problem by making immediate conversion of glycerol to glycerol carbonate

The effect of molar ratio of oil to DMC was studied at various amounts of DMC (molar ratios of methanol to oil = 5-10), and the others variables were fixed (1 molar of palm oil, temperature range of 150 - 250°C, 2 hour reaction time, 600-watt microwave power, and 300 rpm stirring rate). Graphical presentation of the results from **Figure 1, Figure 2** and **Figure 3** shows result of FTIR analysis and all of the sample has been compared to biodiesel. The peaks at 1427 cm^{-1} indicated the deformation vibrations of CH_2. The highest conversion yield achieved for each ratio were 90.1%, 95.6% and 89.6% respectively at the peaks of 1427 cm^{-1} after 2 hour of heating. Result tells that with the increase dimethyl carbonate to palm oil mole ratio, the yield increased but it decreased back after 1 to 7 ratio. At higher molar ratios, excessive DMC dilutes the concentration of oil and reduces the collision frequency of reactants and catalyst. Therefore 7:1 was considered as optimum ratio for given process so as to give maximum yield of 95.6%. Reaction studied without application of microwave was reported 7:1 as the optimized ratio which is more than the optimized value for microwave assisted reaction. As discussed before, power consumption is low at lower viscosity, which is maintained by high reactant ratio. In addition, as DMC was more polar than the oil, it improved the dielectric constant in if using it in high ratio which will be favor the absorption of electromagnetic vibration and heat transfer. Result for this section will be summarized in **Table 1**, **Table 2** and **Table 3** where shows every conversion yield at each temperature for all ratio in this experiment

Table 1: Conversion yield at each determined temperature for oil to dimethyl carbonate ratio of 1:5

Temperature (°C)	Conversion Yield at Peaks of 1427 cm^{-1} (%)
200	92.6
250	90.1

Table 2 : Conversion yield at each determined temperature for oil to dimethyl carbonate ratio of 1:7

Temperature (°C)	Conversion Yield at Peaks of 1427 cm^{-1} (%)
150	72.4
200	89.3
250	95.6

Table 3 : Conversion yield at each determined temperature for oil to dimethyl carbonate ratio of 1:10

Temperature (°C)	Conversion Yield at Peaks of 1427 cm^{-1} (%)
150	88.8
200	89.6
250	82.4

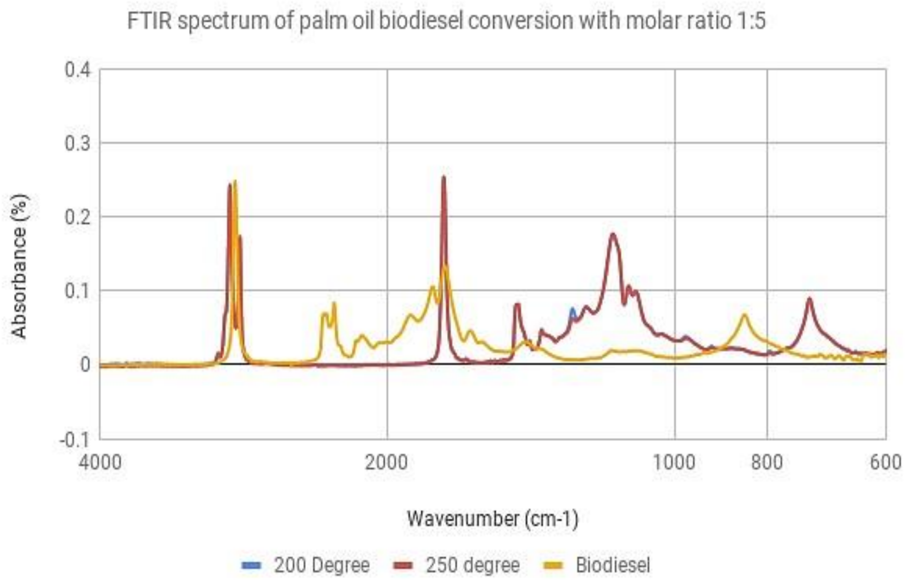

Figure 1: Spectrum of FTIR analysis on the palm oil biodiesel sample with oil to DMC ratio 1***:5***

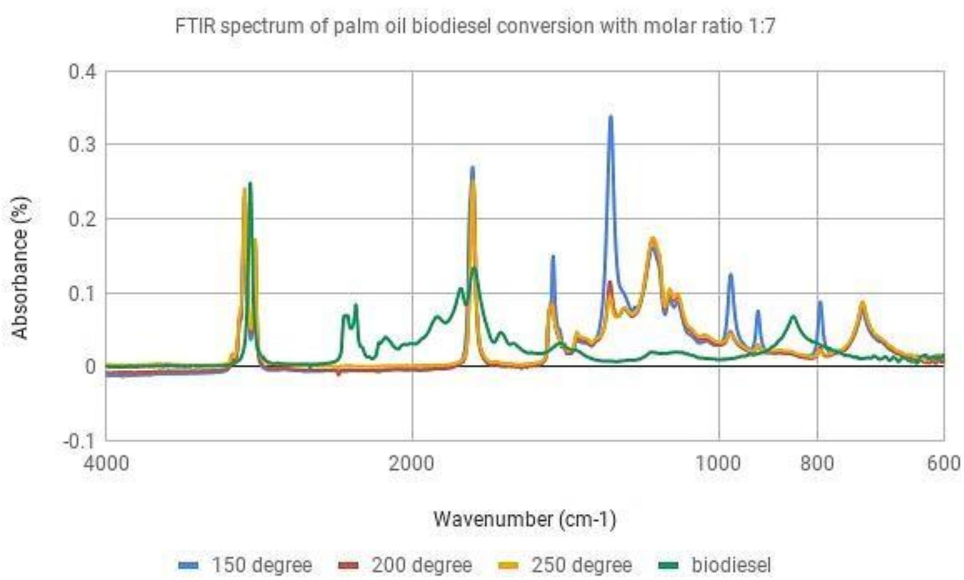

Figure 2: Spectrum of FTIR analysis on the palm oil biodiesel sample with oil to DMC ratio 1:7

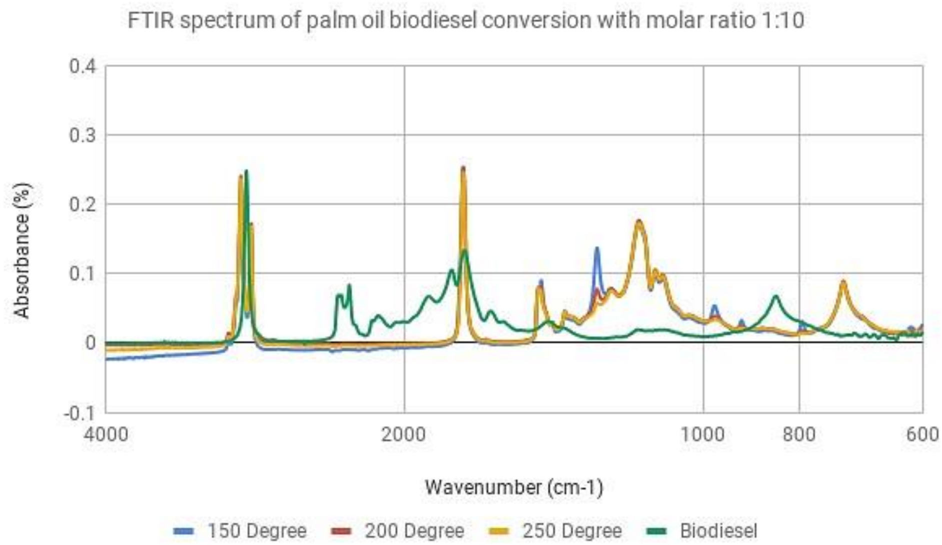

Figure 3: Spectrum of FTIR analysis on the palm oil biodiesel sample with oil to DMC ratio 1:10

3.2 Effect of reaction time on yield

Next experiment have been made to few sample while prolong the period of heating. The influence of reaction time on the biodiesel yield was investigated, while the reaction time was prolonging from 2 hours to 4 hours, mole ratio of DMC to oil was 7:1 (optimum ratio for microwave assisted reaction) and microwave power was constant to 600 watts. According to **Figure 4**, the yield increased slightly when the reaction time prolonged and it achieve 95.9% of conversion yield compare to before. At 2 hour of heating, the conversion yield achieve was just 95.6% and it was increase by 0.3 percent by prolong the heating to 4 hours. Therefore, it could be considering the longer the heating period, it may not effect much on the conversion yield as it also may be ideal for reaction time below 2 hour as based on previous study stated that reaction time of 50 min was found suitable for higher FAME yield and by increasing the reaction time, there no significant change in FAME yield (Sahar *et al.*, 2018)

Table 4 : Change in conversion yield after prolong the heating for 4 hour for ratio of 1:7

Temperature (°C)	Conversion Yield at Peaks of 1427 cm^{-1} (%)
150	88.3
200	70.3
250	95.9

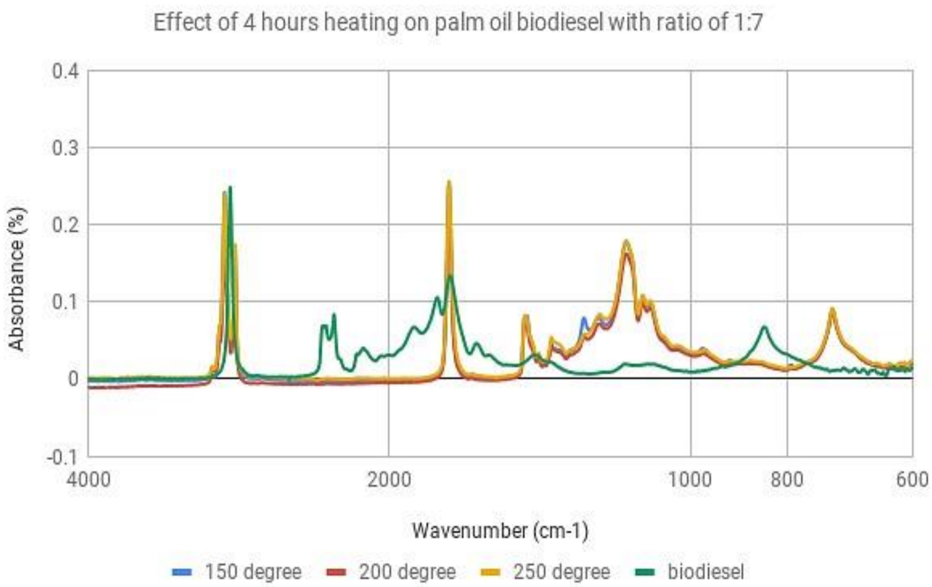

Figure 4: Spectra from FTIR analysis that was done on sample ratio of 1:7 after prolong the heating to 4 hours

3.2 Effect on viscosity and density

Conversion of palm oil to methyl ester will be effect on its viscosity and density as we are using different method. The **Table 5** presented the data gained after the sample has been measured and compared to the diesel and biodiesel based on ASTM standard (El-Araby *et al.*, 2018). For palm oil methyl ester, the density and viscosity result was fall in between the range of biodiesel properties while for flash point was bit off from the range. Because of the molecular weight of the palm oil itself make the flash point of palm oil methyl ester is higher than diesel and biodiesel. As the flash point are higher it may takes time for palm oil methyl ester to ignite in combustion engine.

Table 5: Properties of diesel fuel, biodiesel and palm oil biodiesel.

Fuel Property	**Diesel**	**Biodiesel**	**Palm Oil Methyl ester**
Density @15 °C, g/ml	0.848	0.978	0.878
Kin. Viscosity @40 °C, mm^2/s	1.3–4.1	1.9–6	4.55
Flash point, °C	60–80	100–170	197

Previous study has been made on different molar ratio, heated under 100 degrees for short period of time. The conversion yield achieves almost the same with this study. Specifically, the yield of biodiesel was only 52.69% after 1 hour while the yield reached 97.85% after 6 hour (Ding *et al.*, 2018). However, the yield was maintained at approximately 98%, even when the reaction time continuously enhanced to 8 hours. The temperature not playing big role in manipulate the conversion yield whilst it may affect the viscosity and density of the product itself. Fuels with higher viscosity increases the problems in atomization and damages the fuel injector, thus ultimately results in incomplete combustion and poor engine performance leads to damaging of the engine and also the deposition of solid unburned particles. The fuels with lower viscosity lacks in providing lubrication to the pump and injector, so there also damaging takes place hence optimum viscosity is needed that lies within the range prescribed by ASTM and EN standards. The viscosity of the biodiesel must be at optimum level for it to work ideally. While for density, it seems impossible to achieve 100% of exact conventional diesel but for biodiesel will be varied 37.27 MJ/kg for its calorific value. This is 9% lower than regular Number 2 petro diesel. Variations in biodiesel energy density is more dependent on the feedstock used than the production process. At the end, we can conclude even though the process was varied by temperature and time, it may effect on viscosity difference for biodiesel produce even if it achieves 100 percent conversion yield. As being said, assumption was made that better viscosity of biodiesel can be produce at higher temperature of reaction.

3.3 Fuel properties

Biodiesel that was produced through the conversion from palm oil using dimethyl carbonate method must ensure that it fulfill the standard properties for biodiesel. Fuel properties were studied as shown in **Table 6**. The biodiesel conversation in this was prepared in ideal condition (150°C250°C/120 min/1:10, 1:7, 1:5 molar ratio of oil to dimethyl carbonate). The result was obtained been compared with EU, US and Japanese standard (Ilham & Saka, 2012). Generally, the result from this method following all the requirement based on international standard excluding oxidation stability. Oxidation stability was indicator for chemical reaction that happened when there were interaction between oxygen and lubricating oil. High temperature, acids, water and catalyst can affect the rate of oxidation (*Machinerylubrication.com*, 2018). It is one of the very important measure to avoid deterioration and it could be enhance by utilizing the oils with lower level of unsaturated fatty acid or by adding the antioxidant.

In non-catalytic transesterification process, especially using microwave assisted method, can be more efficient to support the reaction due to minimizing the mass transfer resistance as to commercialize grade biodiesel product with little lost (Tran *et al.*, 2017). Low grade fatty materials such as waste cooking oil can be used as feedstock where traditional transesterification cannot be permitted due to high content of fatty acid. Nevertheless, quality of biodiesel produced from FAME may be varied in each method of conversation. Quality problem may be resulted ineffective and low performance of the biodiesel itself or in some cases may broke the engine. The comparison between the properties gained from the sample with the international standard for biodiesel are very important to ensure the biodiesel gained from this conversion process can be used safely and perform same as conventional biodiesel. Non-catalytic transesterification also have to be commercialized as to reduce the cost of biodiesel production thus this study was very important. Due to this, next study should be focus on effectiveness of this method in large production for it to be commercialize in the oil market as well as how it going to effect the plantation of the palm oil itself.

In other similar studies in supercritical condition, it has been found that using dimethyl carbonate as in non-catalytic transesterification will produce no glycerol for biodiesel production as it was more convenient rather than normal transesterification (Ilham & Saka, 2011).

Table 6: Fuel properties of fatty acid methyl esters (FAME) from palm oil as treated in supercritical dimethyl carbonate method compared with international standards.

Properties	Method	Unit	FAME (Palm oil)	International Standard		
				Japan (JIS K2390)	EU (EN 14214)	US (ASTM D6751-07)
Kinematic viscosity (40 °C)	ASTM D445	mm^2/s	4.5	3.5–5.0	3.5–5.0	1.9–6.0
Carbon residue	ASTM D4530	wt%	0.09	⩽0.30	⩽0.30	⩽0.05
Pour point	ASTM D2500	°C	−7.0	–	–	–
Cold filter plugging point	ASTM D6371	°C	−7.2	–	–	–
Ignition point	ASTM D93	°C	163.6	⩾120	⩾101	⩾130
Cloud point	ASTM D6749	°C	−7.0	–	–	–
Oxidation stability	EN 11442	h	5.7	–	⩾6	–
Ester content	EN 14103	wt%	98.5	>96.5	>96.5	_
Monoglyceride	EN 14105	wt%	0.9	<0.80	<0.80	_
Diglyceride	EN 14105	wt%	0.07	<0.20	<0.20	_
Total glycerol content	EN 14105	wt%	0.04	<0.25	<0.25	<0.24
Water content	EN ISO12937	mg/kg	230	<500	<500	<500
Acid number	EN 14104	mg(KOH)/g	0.18	<0.50	<0.50	<0.50
Iodine value	EN 14111	g(I_2)/100 g	110	<120	<120	_

4 Conclusion

In this paper, three different ratio of oil to DMC was put into test to determine the conversion yield compare to the original biodiesel. It was reveal that using microwave-assisted method could possibly achieve higher conversion yield among other conversion method as the entire sample achieves more than 85%. Ratio of 1:5 achieve 89.5%, for 1:7 ratio achieve 95.5% while for 1:10 ratio gained 98.85% of conversion yield for 2 hour of heating. The next phase using 1:7 ratio as ideal ratio achieved the highest conversion yield which is 99.7% make it more similar to original biodiesel after being heat for 4 hour. Density, viscosity and flash point where been compared with ASTM standard and it achieve between the range except for flash point for it has more molecular weight. Fuel properties also been studied and most of it attain the ASTM standard except for oxidation stability. Real test on combustion engine was suggested to be made for further effectiveness of biodiesel that was extract from palm oil to be commercialize in the market especially transportation.

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