

PAPER • OPEN ACCESS

Methanolysis of Ceiba Petandra (Kapok) seed for high yield fatty acid methyl ester (FAME): A parametric study

To cite this article: Indah Thuraya Herman *et al* 2021 *IOP Conf. Ser.: Earth Environ. Sci.* **765** 012075

View the [article online](#) for updates and enhancements.

You may also like

- [Synthesis of kemiri sunan \(*reutealis trisperma \(blanco\) airy shaw*\) H-FAME through partially hydrogenation using Ni/C catalyst to increase oxidation stability](#)
E Ramayeni, B H Susanto and D F Pratama
- [Effects of Metal Ratio and Aging Time of Cr-Ti Mixed Oxides on Catalyst Characteristics and FAME Density](#)
N Yazid, Zuraida Wan, M S Samin et al.
- [A recyclable solid catalyst of KF/Ca-Mg-Al-O using for biodiesel production from *jatropha seed oil*: preparation, characterization, and methanolysis process optimization](#)
Qinghong You, Xiulian Yin, Jun Wang et al.



The advertisement features a dark teal background. On the left, the ECS logo is displayed above the text 'The Electrochemical Society Advancing solid state & electrochemical science & technology'. Below this, it announces the '242nd ECS Meeting' from 'Oct 9 - 13, 2022 • Atlanta, GA, US', highlighting 'Presenting more than 2,400 technical abstracts in 50 symposia'. In the center, a portrait of M. Stanley Whittingham is shown next to a Nobel Prize medal. To the right, a 'Register now!' button with a checkmark icon is positioned above a photograph of a large audience at a conference. Below the audience photo, a person is shown interacting with a futuristic, glowing interface of icons.

ECS The Electrochemical Society
Advancing solid state & electrochemical science & technology

242nd ECS Meeting
Oct 9 – 13, 2022 • Atlanta, GA, US
Presenting more than 2,400
technical abstracts in 50 symposia

ECS Plenary Lecture
featuring
M. Stanley Whittingham,
Binghamton University
Nobel Laureate –
2019 Nobel Prize in Chemistry

Register now!

Methanolysis of *Ceiba Petandra* (Kapok) seed for high yield fatty acid methyl ester (FAME): A parametric study

Indah Thuraya Herman¹, Faridah Mukhrofun¹, Khairuddin Md Isa², Naimah Ibrahim¹ and Mohd Aizudin Abd Aziz³

¹Faculty of Civil Engineering Technology, Universiti Malaysia Perlis, Arau, Malaysia

²Faculty of Chemical Engineering Technology, Universiti Malaysia Perlis, Arau, Malaysia

³Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, Kuantan, Malaysia

E-mail: indahthuraya@gmail.com.my

Abstract. The feedstock that has high potential for biodiesel production was investigated. The *ceiba petandra* (CP) seed was used in this study as the feedstock. The methanolysis of the CP seed to produce high yield of Fatty Acid Methyl Ester (FAME) was studied. Hydrothermal liquefaction (HTL) was used to produce the bio-crude from the CP with high ester yield. The gas chromatography- mass spectrometry was used to analyze the chemical content in the bio-crude. The highest oil yield was 44.68% which was obtained at 60 minutes reaction time, 300 °C temperature and 4:1 methanol/ oil mass ratio. The optimum parameters for the highest ester content (67.33%) was obtained at 60 minutes reaction time, 300 °C temperature and 3:1 methanol/oil mass ratio. It is observed that CP seed has potential for the production of biodiesel.

Keywords: Biodiesel, Methanolysis, Fatty Acid Methyl Ester, Hydrothermal liquefaction, Esterification

1. Introduction

Fatty Acid Methyl Ester (FAME) or also known as biodiesel is a renewable fuel which goes on with extreme concern due to rising oil prices and crude oil crises [1]. Biodiesel is made from vegetables oil and animal fats and it has the potential to alleviate environmental pressures and achieve sustainable development compared with fossil fuels [2]. Researchers are currently searching for new sources of renewable sustainable energy because of inadequate fossil fuels and also because of rising fossil fuel prices [3]. The fast-growing population and industrialization have increased the demand for energy. Generally, non-renewable resources such as petroleum, natural gas and coal are the most energy demand [4].

Biodiesel was highlighted as a desirable green fuel for addressing environmental problems. The emissions of engines generated from the combustion of fossil fuels contain high levels of air pollutants and therefore raise environmental concerns. Biodiesel is more environmentally friendly compared to petrodiesel fuel due to low emissions of air pollution, biodegradable, less toxic, safe to handle and store, has excellent lubricity and provides energy density comparable to traditional petrodiesel [5]. The usage



of biodiesel especially from vegetable oil as an engine fuel is getting much more attention in academic and industrial sector due to the higher availability, non-hazardous, biodegradable, cost effective and also environmentally friendly [6].

Pyrolysis, microemulsion formation, dilution, transesterification and supercritical method are some available techniques that are applied to produce biodiesel as well as reduce the viscosity of vegetables oil [7]. The most common technique used is the method of transesterification. Transesterification is a method of esterification of vegetables or animal fats with alcohol by means of acidic, basic catalysts and enzymes [7]. There are two steps in biodiesel production – esterification and transesterification [8]. To convert solid biomass to liquid hydrocarbons, hydrothermal liquefaction (HTL) or pyrolysis is performed. From the biomass conversion, a product (liquid, gas and solid) that has sufficient potential to be used for fuel purposes or feedstock for chemical industry is obtained [9].

In Malaysia there are agriculture and forestry abundant natural resources that can be used for the production of biodiesel. Between them, kapok tree or also known as silk- cotton tree, is one of the plant abundant natural resources with high potential for biodiesel production. Cotton tree is a large plant from the family Malvaceae which is spreadly distributed in the tropical rainforest [10]. Some parts of this plant that were used as timber and pod have high economic value. Kapok pod consists of fibre, which is mainly used to make pillows and mattresses while the resulting seeds end only as waste [5]. Cotton seeds are brownish black in color, with an average annual oil yield of 1280 kg/ ha occupying 25- 28 per cent by weight for each fruit. This was recommended as an effective feedstock for the development of biodiesel [11].

Gas chromatography- Mass spectrometry (GCMS) is the most commonly used technique for studying in the individual components that make up a fuel [12]. This technique requires a minimal preparation of samples and permits separation and identification of individual FAMES. FAME has physical properties similar to the conventional diesel [13]. This present research provides an insight into the non-catalytic development of cotton seed oil biodiesel, which is not only opens up a broader plan to combat the worldwide depletion of crude oil supplies but also enriches the cutting-edge biodiesel research market.

2. Methodology

2.1 Sample preparation

Ceiba Petandra was collected from Wang Ulu, Perlis. The seed first separated from the fibre, then sun-dried for 6 hours.

2.2 Experimental data sample

The experiment conducted were based on the effect of parameter which involved reaction time (30, 60 and 90 minutes), temperature (200, 250, 300 and 320°C) and the volume of methanol (10, 15, 20 and 24 ml). The effect of each parameters is determined by designing set of experiments with two constant parameters. The experimental designs were carried out based on the following Table 1:

Table 1. Design of experimental run

No of run	Time (min)	Temperature °C	Volume of methanol (ml)
1	60	200	20
2	60	250	20
3	60	300	20
4	60	320	20
5	30	300	20
6	60	300	20

7	90	300	20
8	60	300	10
9	60	300	15
10	60	300	20
11	60	300	24

2.3 Bioreactor set up (HTL Process)

HTL process was conducted in a 100 ml reactor where there was loaded with 5g of sample with the addition of pre-set methanol. The values of the parameter set were based on the experimental data design. The reactor was equipped with a stirrer and the stirring speed was set to 830 rpm. The reaction time was start measured once the temperature reached the desired temperature that had been set. The percentages of bio-crude, bio-char and gas produced were calculated using following formula [14]:

$$\%bio - crude\ yield = \frac{weight\ of\ bio-crude}{weight\ of\ cotton\ seed\ sample} \times 100 \quad (1)$$

$$\%bio - char\ yield = \frac{weight\ of\ bio-char}{weight\ of\ cotton\ seed\ sample} \times 100 \quad (2)$$

$$\%gas + loss = 100\% - (\%bio - crude) - (\%bio - char) \quad (3)$$

2.4 Sample characterization

The oils produced from the HTL process were analyzed using gas chromatography- mass spectrometry (GCMS). 0.5g of bio-crude was diluted with HPLC methanol. Then, this solution was filtered into a small vial to test the GCMS. The chemical composition will be analysed following to the National Institute of Standards and Technology (NIST) mass spectral library standard reference database. From this analysis, percentages of chemicals (ester, alcohol, acid, phenol, benzene) were identified using following formula:

$$\%chemical = \frac{area\ of\ chemical}{total\ area} \times 100 \quad (4)$$

3. Results and Discussion

3.1 Parametric studies on methanolysis of cotton seed

Parameters that involved in methanolysis of cotton seed were temperature, reaction time and volume of methanol. The products from HTL process were bio-crude or bio-oil, bio-char and gas. The effect of parameters on methanolysis were study by hold one parameter and alter another two parameters with 5g of cotton seed used as feedstock.

The condition experiment on effect of temperature were 200, 250, 300 and 320°C with constant time and volume of methanol were 60 min and 20 ml respectively. The product yield on effect of temperature was shown in Figure 1. It was observed that the oil yield continuously increases from 8.35% to 48.53% as the temperature increases from 200 to 300°C. Further increases in temperature to 320°C decrease the bio-oil yield to 16.57%. For bio-char yield, the experimental run at 200°C produced the highest residues (64.95%). The experimental run with the temperature of 250°C produced the highest amount of gas which is 38.70%. Similar oil yield trend as a function of temperature was observed in the study of hydrothermal liquefaction in sub-critical water/ethanol[14]. Whereas, the oil yield increasing from 240 to 300°C and slightly decrease at 320°C. Therefore, it is concluded that 300°C is sufficient to achieve the highest oil yield from methanolysis of CP.

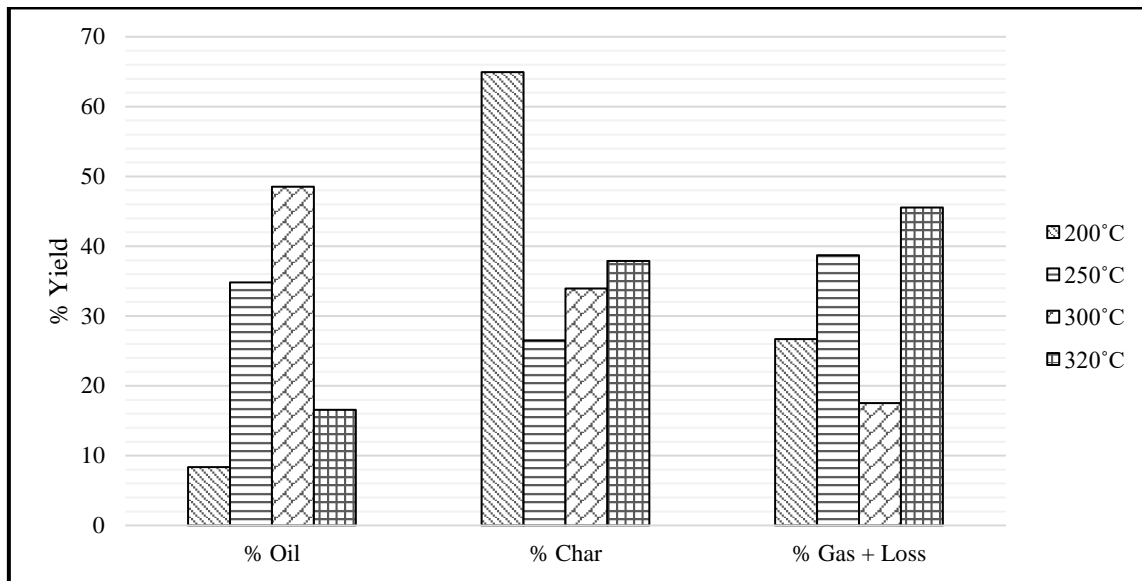


Figure 1. Effect of temperature on the product distribution of yield

Figure 2 shows the effect of time on the products yield (%). Three different experiments with 30, 60 and 90 minutes of reaction time were conducted. The temperature and volume of methanol were constant at 300°C and 20 ml respectively. From the experiment, it is observed that the highest oil yield obtained at reaction time 60 minutes. The oil yield decreased to 18.56% as the reaction time was increased to 90 minutes. The experimental run at 30 minutes produced the highest amount of gas which was 63.08%.

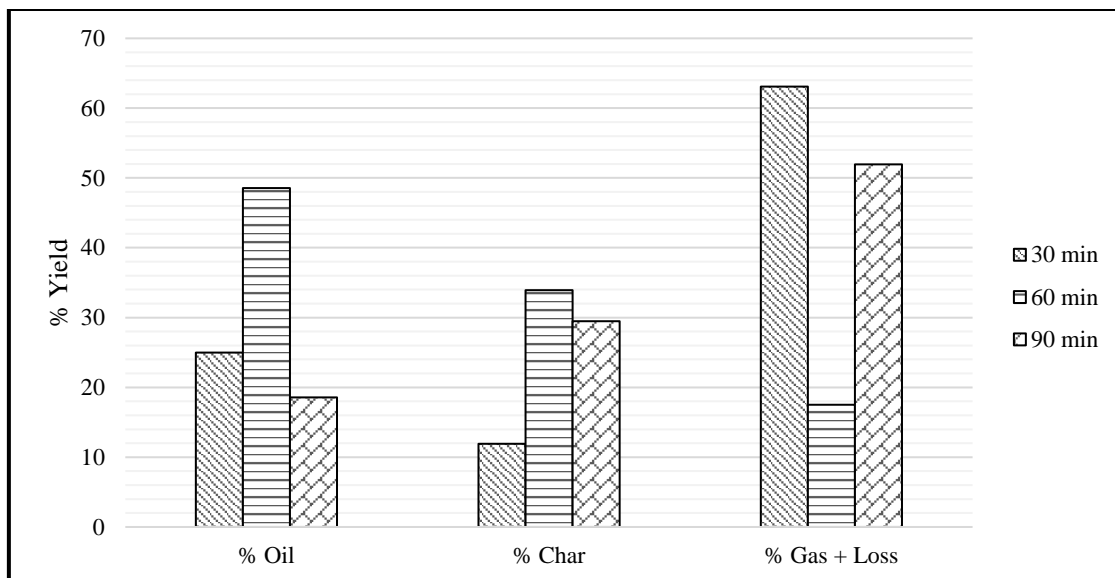


Figure 2. Effect of reaction time on the product distribution of yield

To identify the effect on the volume of methanol, four sets of experiment were conducted with 10,15,20 and 24 ml methanol respectively. All the experiments were run for 60 minutes with temperature 300°C as a constant condition. The results obtained from the experiments presented in figure 3. From the experiments, it is identified that 20 ml methanol gave the highest yield of bio-crude and the lowest

yield of gas which is 48.53% and 17.5% respectively. The experimental run that used 10 ml of methanol produced the lowest yield of bio-crude (5.15%) but the highest yield of gas (53.69%). The highest bio-char produced was obtained using 15 ml which is 44.68% while the lowest was with 24 ml (26.71%).

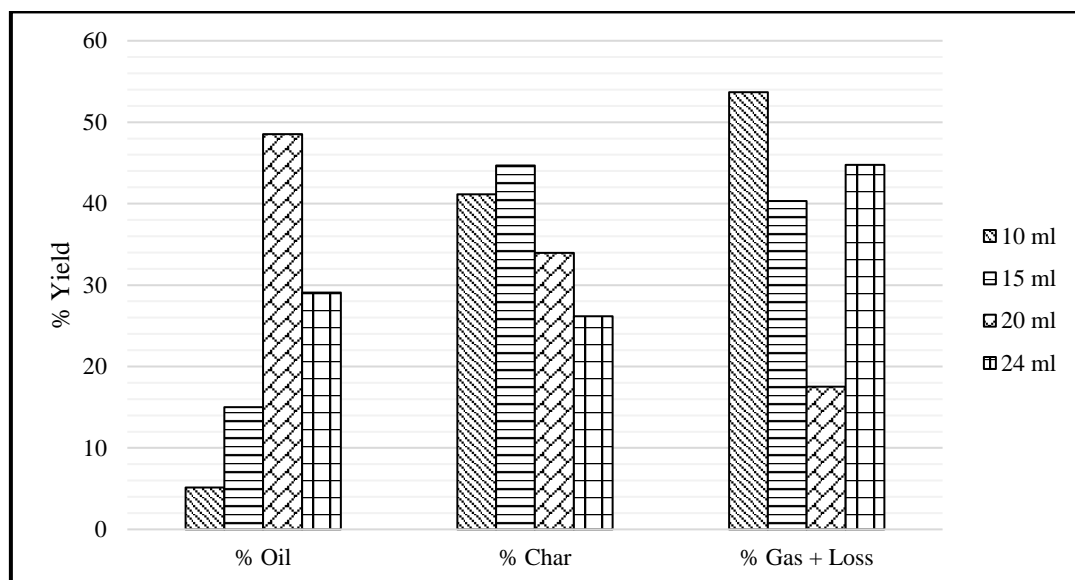


Figure 3. Effect of volume of methanol on the product distribution of yield

HTL is a thermochemical process where high pressure (5- 20 MPa) and moderate temperature (200-374°C) are used to treat biomass [15]. This condition was known as subcritical due to the high temperature (>100°C). At this phase; bio-char, bio-oil, aqueous phase fraction and gaseous materials are produced due to the depolymerization reactions.

From this experiment, it is observed that the highest yield of bio-crude is 48.53% obtained at the temperature of 300°C, the reaction time of 60 minutes and the volume of methanol of 20 ml. The pressure recorded was 7.5 MPa. Compared to the previous study, the highest liquid oil yield is 38.4% which obtained at 300°C, 2 MPa and 30 minutes [14].

3.2 Chemical composition analysis for the produced oil

GCMS analysis was conducted to determine the chemical composition in the produced oil. Each chemical composition was analysed based on their chemical groups. Percentages of ester, benzene, alcohol, phenol and acid were then calculated. Figure 4 shows the effect of temperature on the percent of chemical obtained. The experiment conducted at different temperature (200, 250, 300 and 320°C) for reaction time of 60 minutes and 20 ml volume of methanol.

From the graph it is found that at 300°C gave the highest percentage of ester (62.95%) and the lowest percentage of acid (1.66%). Experiment with 320°C gave the lowest ester content and highest benzene content which 43.47% and 4.42% respectively. For the experiment at 200°C, although it gave a higher of ester (61.09%) but it is also giving a higher yield of acid (18.72%). For 250°C, it is seen that it had high amount of alcohol and phenol which were 15.57% and 1.94% respectively.

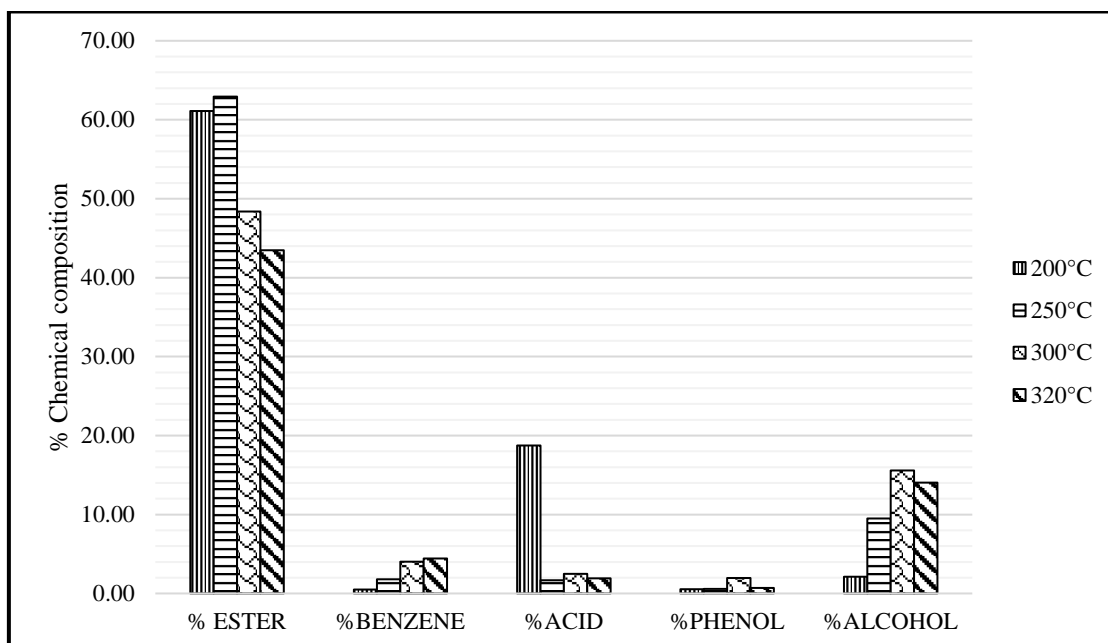


Figure 4. Effect of temperature on the chemical composition at 60 minutes and 20 ml methanol

For the effect on the reaction time, three experiments with 30, 60 and 90 minutes were conducted at 300°C and 20 ml methanol. From the figure 5, it is observed that experiment with 60 minutes gave the highest percentage of ester. The experimental run at 30 minutes produced the lowest ester content but the highest acid content and alcohol content with 42.06%, 10.09% and 18.81% respectively. For 90 minutes, there was no content of phenol detected. Experiment with 60 minutes also recorded the lowest amount of benzene, acid and alcohol with 1.78%, 1.66% and 9.48% respectively.

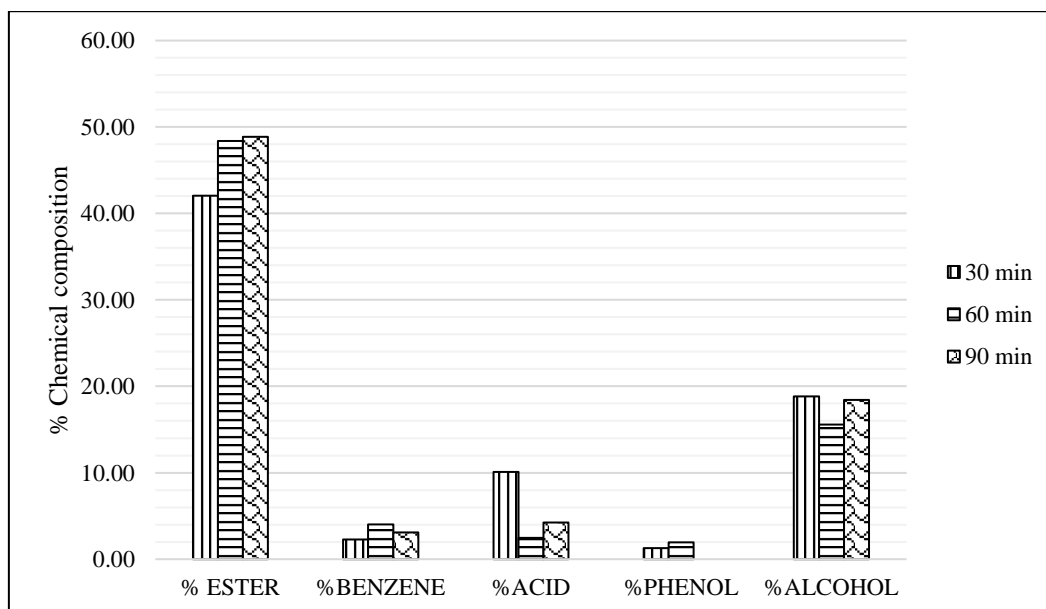


Figure 5. Effect of time on the chemical composition at 60 minutes and 20 ml methanol

To identify the effect on volume of methanol, four experiments were run with different volume of methanol (10,15,20 and 24 ml) at 60 minutes and 300°C. the result of the experiment presented in figure 6. From this experiment, it is observed that the highest amount of ester content was obtained when the

volume of methanol is 15 ml which was 67.33%. It also had the highest amount of alcohol content which was 11.13%. Experiment with 10 ml methanol had the lowest ester content and the highest acid content which were 53.80% and 11.03% respectively.

Generally, from this experiment it is observed that the highest amount of ester content is 67.33% which was obtained at 60 minutes reaction, 300°C temperature and 15 ml volume of methanol. However, from the previous study, it is reported that the FAME content for cotton seed is 97% [16]. They used 7:9 methanol/oil molar ratio with 53°C, 45 minutes and 268 rpm mixing rate with catalyst loading of 1%. The experiment was conducted using ethanol instead of methanol. Another study also shows an excellent yield of methyl ester (96.9%) with presence of 0.75% catalyst concentration at 65°C, methanol to oil molar ratio 6:1 and 600 rpm mixing rate [17].

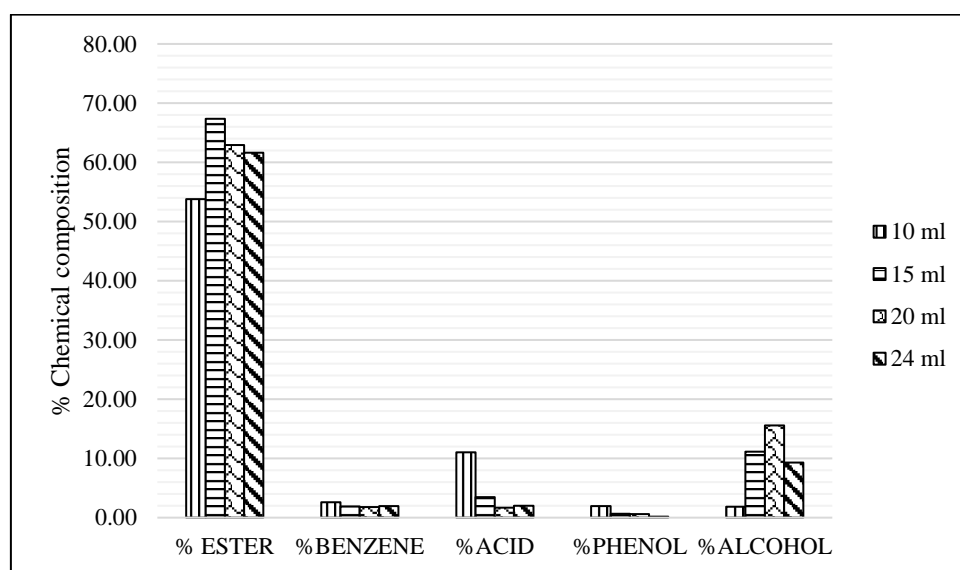


Figure 6. Effect of time on the chemical composition at 60 minutes and 20 ml methanol

High content of methanol indicates that the excess used of methanol were not fully removed. Excess methanol can affect fuel pumps and reduce the flash point. The limit of alcohol content is 0.2 wt% whereas acid number higher than 0.5 were associated with fuel system deposits and reduced life of fuel filters and pumps [18]. A lower yield of ester in this experiment compared to other studies maybe due to the absent of catalyst used.

3.3 The analysis of ester content for the produced oil

From the previous discussion, it was observed that the highest ester content was obtained from the experimental run at 60 minutes reaction time, 300°C temperature and 15 ml methanol. The result obtained for this set of parameters is shown in Figure 7. The ester content obtained was 67.33%. For a biodiesel, it is significant to have high content of FAME. Normally, an excellent biodiesel produced >96.5% of FAME [19].

For this set of parameters, the methanol/CSO ratio is 3:1 and the mixing rate used is 832 rpm. It can be said that the result obtained for this study is low as they reported in their study which the maximum yield of biodiesel obtained is 92% [20].

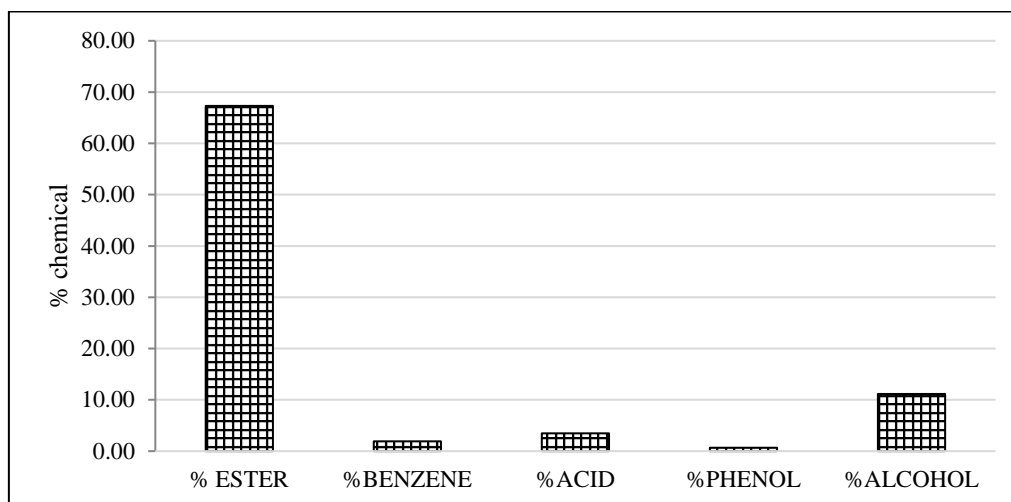


Figure 7. Chemical composition (%) of high yield bio-crude

It is reported that the high temperature affects the hydrocarbon bonding. The mixture of methanol-oil becomes homogenous due to the negative effect of the bonding [21]. Increasing of reaction time led to high conversion of oil into methyl ester due to the longer contact between oil and methanol [5]. To conclude, the optimum parameter obtained to produce high conversion of ester is 60 minutes of reaction time at temperature of 300°C and methanol/oil ratio of 1:3.

4. Conclusion

HTL is one of the methods to produce bio-crude from biomass. From this study, it is observed that the highest CP seed bio-crude value is 44.68% which obtained at 300°C for 60 minutes with 20 ml volume of methanol. A good biodiesel contains high amount of ester content. From this study, the optimum parameter for ester content obtained 67.33% at 300°C for 60 minutes and 15 ml of volume methanol. CP seed has potential to be explore for biodiesel feedstock.

References

- [1] R. Mahesh, V. R. Naira, and S. K. Maiti 2019 *Bioresour. Technol.*, vol. 278
- [2] T. M. I. Mahlia *et al.* 2020 *Renew. Sustain. Energy Rev.*, vol. 118
- [3] J. Ahmad, 2014
- [4] A. Bokhari, L. F. Chuah, S. Yusup, J. Ahmad, M. R. Shamsuddin, and M. K. Teng 2015 *Biofuel Res. J.*, vol. 2, no. 3
- [5] L. K. Ong, C. Effendi, A. Kurniawan, C. X. Lin, X. S. Zhao, and S. Ismadji 2013 *Energy*, vol. 57
- [6] A. Saravanan, M. Murugan, M. Sreenivasa Reddy, and S. Parida 2020 *Fuel*, vol. 263
- [7] T. Şahin, 2018.
- [8] G. Khoobbakht, K. Kheiralipour, H. Rasouli, M. Rafiee, M. Hadipour, and M. Karimi 2020 *Energy*, vol. 196
- [9] R. Kaur, B. Biswas, J. Kumar, M. K. Jha, and T. Bhaskar 2020 *Ind. Crops Prod.*, vol. 149
- [10] F. Kusumo, A. S. Silitonga, H. H. Masjuki, H. C. Ong, J. Siswanto, and T. M. I. Mahlia, 2017 *Energy*, vol. 134
- [11] T. M. Yunus Khan, A. E. Atabani, I. A. Badruddin, R. F. Ankalgi, T. K. Mainuddin Khan, and A. Badarudin 2015 *Ind. Crops Prod.*, vol. 65
- [12] M. E. Flood, M. P. Connolly, M. C. Comiskey, and A. M. Hupp 2016 *Fuel*, vol. 186
- [13] European Technology and Innovation Platform, "FAME / Biodiesel."
- [14] X. Yan, B. Wang, and J. Zhang 2015 *Bioresour. Technol.*, vol. 197
- [15] V. K. Ponnusamy *et al.* 2020 *Bioresour. Technol.*, vol. 310

- [16] X. Fan, X. Wang, and F. Chen 2014 *Open Fuels Energy Sci. J.*, vol. 4
- [17] U. Rashid, F. Anwar, and G. Knothe 2009 *Fuel Process. Technol.*, vol. 90
- [18] K. Shaine Tyson, 2001 “Biodiesel Handling and Use Guide Fourth Edition,”
- [19] R. Goosen, K. Vora, and C. Vona, *Establishment of the Guidelines for the Development of Biodiesel Standards in the APEC Region*, no. April. 2007.
- [20] D. Sinha and S. Murugavelh 2016 *Perspect. Sci.*, vol. 8
- [21] N. A. Handayani *et al.* 2013 *Int. J. Renew. Energy Dev.*, vol. 2

Acknowledgement

FRGS grant of 900- 00764 is acknowledged. The authors gratefully acknowledge the facility and experimental work support provided by the Faculty of Civil Engineering and Technology, Universiti Malaysia Perlis.