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The Physicochemical Profile Of *Feun Kase (Thevetia Peruviana)* Oil As A New Feedstock For Renewable Energy

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Abstract

The depletion of world fossil energy has led to the development of renewable energy from vegetable oils, including *Feun Kase* with abundant vegetable oils. The main focus of this research was to identify the physicochemical properties and to explore the potential of Indonesian *Feun Kase (Thevetia peruviana)* oil as renewable energy. *Feun Kase* oil has been isolated by press and soxhlet method. The soxhlet (66.32 % weight) provided a higher yield than the press (25.58 % weight). The oil yielded was directly tested for the physicochemical test according to SNI 7182: 2015 (Indonesian National Standard). The parameters in this research were water content, acid number, saponification number, iodine value, density, viscosity, cetane number, flash point, and cloud point. The GCMS profile showed the presence of several fatty acids such as hexadecenoic acid (palmitic acid), 9,12−octadecadienoic acid (linoleic acid), 9−octadecenoic acid (oleic acid), and octadecenoic acid (stearic acid). Although there was no conclusion in terms of biodiesel, this research could give the report as the basic information that revealed the potential of *Feun Kase* oil. Several tests revealed that *Feun Kase* oil can be used as the feedstock for biodiesel. Unlike palm oil, the non-edible oil of Feun Kase does not compete with the food sector. Thus, it indicated that *Feun Kase* oil is a very competitive feedstock for renewable energy.

1. Introduction

Energy supply in the future is still a popular issue. The evidence could be clearly seen in the development of industrial and technology sectors, economic growth and population growth. Currently, biodiesel is still a popular renewable energy because of its benign and easy to obtain. In addition, using biodiesel could reduce COxand SOx gas emissions (Verma and Sharma, 2015). In the transesterification process, biodiesel is produced from vegetable oil. This oil can be obtained from soybean, canola, palm, corn, coconut, and many more. The United States Department of Energy states that, in 2018 there were 1,857 million gallons of biodiesel produced from edible oil. Soybean oil, corn oil and soybean oil were highest in sales, respectively 7,542; 2,084; and 1.197 million Pound (Energy, 2019).

The raw material for biodiesel which competes with the food sector is still being a controversy (Mekhilef, Siga and Saidur, 2011). The selling price of biodiesel is too high, and the fluctuation in raw material (food) price causes biodiesel to be unpopular. Therefore, the biodiesel sector really needs non-food raw materials (Ajanovic, 2011; Tokgozet *al.*, 2012). *Feun Kase* (*Thevetia peruviana*) seeds contain quite high oil, namely 63 % (A Ibiyemi *et al.*, 2002). However, this oil contains toxic cardiac glycosides such as thevetin A, thevetin B, neriifolin, and ruvoside (Singh *et al.*, 2012). Therefore, this oil is classified as non-edible oil and has not been utilized for biodiesel production. However, Feun Kase (*Thevetia peruviana*) is very abundant in Indonesia and has not received attention to be developed as a source of biodiesel. This research is focused on 10 tests of physico-chemical parameters according to SNI 7182: 2015. In addition, the fatty acid content of Feun Kase oil is shown by GCMS data.

2. Method

Materials

The materials used in this study included *Feun Kase* seeds, ethanol, KOH, NaOH, HCl, $Na_2S_2O_3$, $H_2C_2O_4$, petroleum ether, pp indicator, chloroform, iodine, starch solution, glacial acetic acid, bromine, and KI solution. All obtained from Merck Indonesia.

Sampling and preparation

Samples of Feun Kase seeds were taken from East Nusa Tenggara Province (Indonesia). Sample preparation was carried out at the Laboratory of the Faculty of Agriculture, University of Timor and the Laboratory of Chemistry, Faculty of Science and Engineering, University of Nusa Cendana. The Feun Kase meal was blended and dried in an oven at 60°C with particular time variations. The dried Feun Kase is stored in a closed container.

Oil Isolation

Oil isolation was carried out by pressing (B1) and by means of soxhlet (B2) using petroleum ether which lasted for 4 hours at 60°C.

Moisture Content

This test was carried out by heating the oil in an oven at 105°C for 3 hours. Weight lost was calculated as moisture content (%).

$$Moisture\ content\ (\%) = \frac{a-b}{a} \times 100\%$$

a= sample weight before oven
b= sample weight after oven

Acid Number

Two grams of sample was added with 10 mL EtOH in an Erlenmeyer and shaken for 10 minutes until the oil dissolves in the solvent. After dissolving, 1 drop of 1 % pp indicator was added and titrated with 0.1 N KOH until the pink color did not disappear for 30 seconds. The volume of KOH used was recorded. The procedure was carried out for B1 and B2 twice.

$$Acid\ Number = \frac{V_{KOH} \times N_{KOH} \times 56.11}{\text{sample weight}}$$

Saponification Number

The oil was refluxed with KOH 0.5 N, then titrated with 0.5 N HCl. The saponification number was calculated according to the following equation:

$$BP = \frac{(V_{HCl}\ \text{blanko} - V_{HCl}\ \text{sample}) \times \text{N KOH} \times 56{,}11}{\text{oil weight (g)}}$$

Iodine Value

This test was performed using Hanus reagent and then titrated with $Na_2S_2O_3$. The calculation of iodine value is carried out according to the following equation:

$$Iod\ value = \frac{(B-S) \times N \times 12{,}69}{G}$$

Information:
B : mL $Na_2S_2O_3$ for blank titration
S : mL $Na_2S_2O_3$ for sample titration
N : normality of the $Na_2S_2O_3$ solution
G : sample weight

Density

This test refered to SNI 7182: 2015 using pycnometer and was calculated using the formula below.

$$D = \frac{(A + \text{sample}) - (A)}{\text{Volume sample}}$$

Information :
D = Density
A = Pycnometer weight

Viscocity

This test was done by calculating the oil flow time in the Otswald viscometer and then calculating it by the equation below.

$$\eta = \eta_0 \frac{t.\rho}{t_0.\rho_0}$$

Cloud Point

Oil was put into the test tube (30 mm in diameter) and covered with a rubber cover that has been attached with a thermometer. Then put in a container filled with water and ice cubes. Pay attention to the temperature on the thermometer and the bottom of the tube. When crystals (cloud) begin to appear, the temperature in this state was recorded as a cloud point.

Cetane Number

Cetane numberwere determined by the following calculations (Guil-Laynez, Guil-Guerrero and Guil-Laynez, 2019):

CN = 46,3 + 5.458/SN – 0,225 x IV

Information:
CN = Cetane Number
SN = Saponification Number
IV = Iodine Value

Flash Point

The flash point in this study was carried out by the Pensky Martens. This test was carried out at the Laboratory of Petroleum Gas and Coal, Department of Chemical Engineering, Gadjah Mada University.

Analysis of Fatty Acid

Screening was carried out using GCMS at the Chemical Laboratory of the Department of Chemistry, Gadjah Mada University. The state of the GCMS is as follows: GCMS brand - QP2010S SHIMADZU; AGILENTJ % W DB-1 column; column length 30 m; diameter 0.25 mm; ionizing EI 70 Ev; pressure 12 kPa; and a flow rate of 0.54 mL / min. Before the analysis, the oil was transesterified so that could be analyzed on GCMS.

3. Results and Discussion

Sample Preparation

Removing water content in Feun Kase oil is an important step to obtain Feun Kase oil which is safe for storage. The optimum drying time for Feun Kase has been determined by comparing the amount of weight lost in test plate (C1-C6) at 60°C. The best result is 4 hours with 12.27% weight loss, shown by the graph in Figure 1.

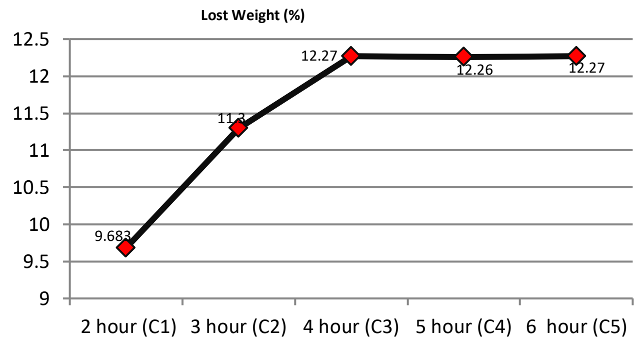


Figure 1. Drying time of test plate

Oil Isolation

The isolation in 2 different ways resulted in a very significant yield calculation. Through the press method (B1), the yield was 25.58 %, while in the sox let method, the yield was 66.32%. This means that the soxhlet method is the best way for extracting the oil out from *Feun Kase*. However, it requires additional treatment such as distillation to separate the solvent from the oil.

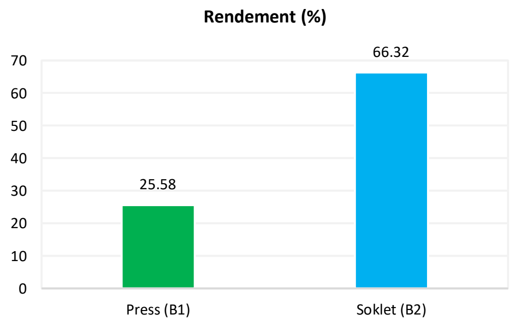


Figure 2. The Comparison of oil isolation rendement

Moisture Content

The Moisture content of Feun Kase oil is lower than the SNI standard. The moisture content of the pressed and soxhlet oil were 0.003% and 0.004%, respectively. The results are shown in the graph in Figure 2. The results indicated that the water content of Feun Kase oil is in accordance with SNI standards.

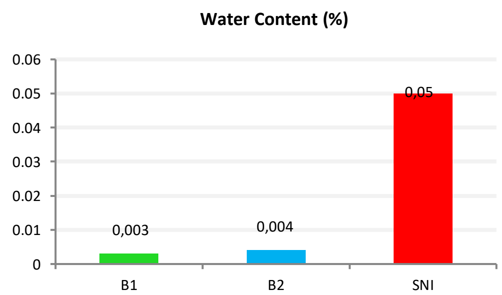


Figure 3. Moisture Content of B1 and B2

Acid Number

The amount of KOH neutralizing the free fatty acids in B1 is 1.94 mg KOH / g sample, while in B2 is 1.38 mg KOH / g sample.

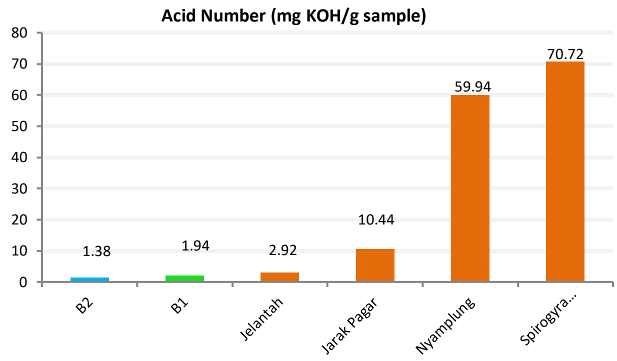


Figure 4. The Comparison of Acid Number of Various Oil.

When compared with the acid number of Nyamplung oil, which is 59.94 mg KOH / g sample (E Atabaniet *al.*, 2011), the acid number of Feun Kase oil gives a better number because it is closest to SNI. Data regarding this acid number is very important in choosing method when this oil is converted into biodiesel. For example, if the acid number is too high, it will produce a lot of soap in the transesterification process.

Saponification Number

The amount of KOH for B1 was 165.65 mg KOH / g sample, while B2 was 166.38 mg KOH / g sample. It can be seen that the saponification number of B1 and B2 do not differ much. This means that the press or soxhlet method does not affect the saponification number.

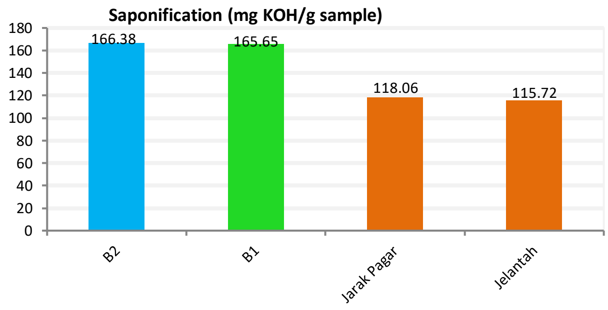


Figure 5. The Comparison of saponification number of Various Oil.

Iodine Value

The iodine valuefor B1from this study was 137.44 mg I_2 / 100g sample, while the B2 was 126.87 mg I_2 / 100g sample. This number shows the number of double bonds in the fatty acid which can react with iodine. In accordance with SNI, the calculated number do not meet the standard, namely a maximum of 115 mg I_2 / 100 g of sample.

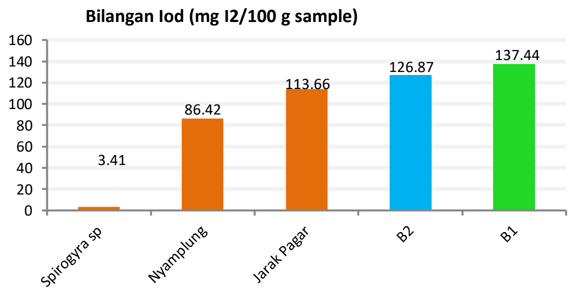


Figure 6. The Comparison of Iod Number of Various Oil

From the above comparison, it can be seen that Feun Kase oil has the highest iodine value among other oils. The number of unsaturated fatty compounds in the oil also makes it easier for these compounds to react with oxygen.

Density

The density of B1 calculated from the results of this study is 884 Kg / m^3, while that of B2 is 855 Kg / m^3. It can be seen that the density of Feun Kase meets the standard by SNI, namely in the range 850 - 890 Kg / m^3.

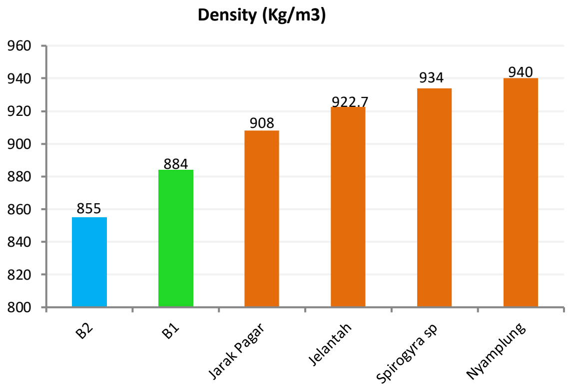


Figure 7. The Comparison of Density of Various Oil.

If the density value between B1 and B2 is related to the acid number, it can be seen that B1 with a higher density has a higher acid number compared to B2. It is also reported by Madhu Agarwal that castor oil (B) with a high free fatty acid content, has a density of 920 Kg / m^3. Meanwhile, castor oil (A) with a low free fatty acid content has a lower density, namely 880 Kg / m^3 (Agarwal*et al.*, 2012). The low content of fatty acids in Feun Kase oil causes a better density than jarak oil.

Viscosity

The viscosity of B1 calculated from the results of this study is 22.25 mm^2 / s, while that of B2 is 19.91 mm^2 / s. From this data, it can be seen that the viscosity of Feun Kase has not met the standards by SNI, namely in the range 2.3 - 6.0 mm^2 / s. Crude palm oil is reported to have a viscosity of 37.5 mm^2 / s at 40°C (Rotimi, 2016). Viscosity is a very important parameter when this oil is applied to diesel engines. If the viscosity is too high, it is difficult to burn the oil in a diesel engine.

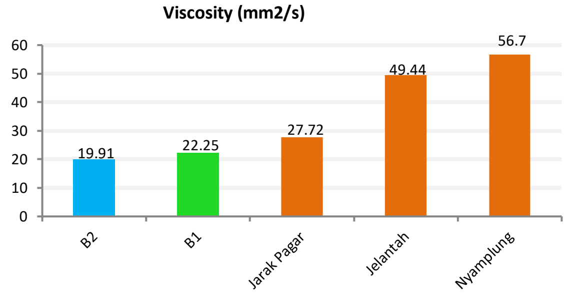


Figure 8. Comparison of Viscosity of Various Oils

Cloud Point

The cloud points of B1 and B2 were the same, which is at 2 °C. Thus, the cloud point of Feun Kase in this study meets the SNI standard, namely a maximum of 18 °C. If the cloud point is too high, then the oil can easily change to solid phase according to the local air temperature, so this is very disturbing during storage and application to the engine. Considering that Indonesia has areas with varying temperatures, the lower the cloud point the better, because this oil can be used in anywhere.

Cetane Number

The calculation results show that the cetane number B1 is 48.32 while B2 is 50.55. SNI provides a standard cetane number of at least 51. When compared with SNI, both B1 and B2 have not entered the criteria for good biodiesel, although B2 with a cetane number of 50.55 is almost close to the standard. If you look at the formula given by AOCS (Guil-Laynez, Guil-Guerrero and Guil-Laynez, 2019), it can be concluded that the low cetane number in Feun Kase oil is caused by the high iodine number. In other words, high levels of unsaturated fatty acids lead to low cetane numbers.

Flash Point

Flash points B1 and B2 were not detected by the Pensky Martens Close Cup (PMCC) device, so it was followed by a Cleveland Open Cup (COC) with a flash point of 324 °C for B1 and 322.5 °C for B2. Methyl esters from palm oil are reported to have a flash point of 170 °C (Ayetor, Sunnu and Parbey, 2015). The flash point of Feun Kase oil does not meet the standards by SNI.

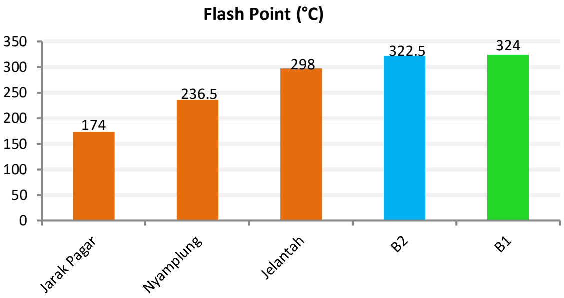


Figure 9. Comparison of flash points of various oils

From the above comparison, it can be seen that Feun Kase occupies a very high flash point. This is very unfortunate if directly applied to diesel engines. Combustion will be very slow because it takes a long time to reach that temperature. As a comparison, Madhu Agarwal in 2011 stated that the transesterified castor oil has a flash point of 174 °C (Agarwal*et al.*, 2012). Feun Kase oil may provide a decent flash point number if it is continued with a transesterification method such as that of Agarwal.

Fatty Acid Content

It should be noted that this Feun Kase oil is transesterified first before being put on GCMS. The decrease in viscosity of the transesterified oil really helps the analysis process at GCMS. Therefore, the molecular weight of the spectra data below includes methyl from the transesterification results.At the retention time of 36.301 with an abundance of 26.73%, the mass spectra data showed that the molecular weight was at the peak m / z 270 [M$^+$]. If you subtract the weight of CH_2 (methyl from the transesterification result), then a compound with a molecular weight of 256 g / mol is obtained. This compound is hexadecanoic acid or better known as palmitic acid with the molecular formula $C_{16}H_{32}O_2$ (figure 9 and 10). Palmitic acid is also found in palm oil with a content of 39.1% (Anguebes *et al.*, 2016).

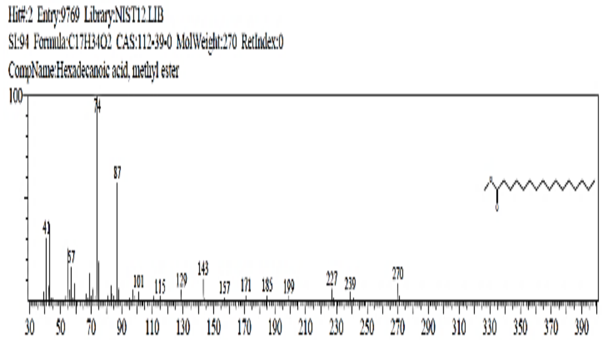


Figure 9. GCMS mass spectra at retention time of 36.301 for palmitic acid

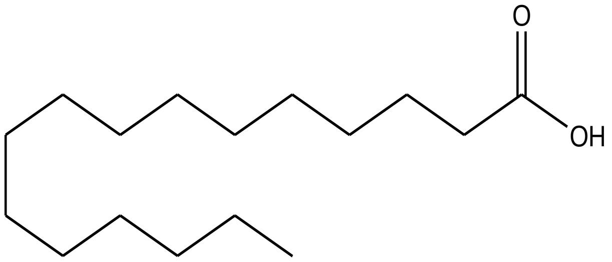


Figure 10. Structure of palmitic acid

At the retention time of 39,665 with an abundance of 12.41 %, 9,12-octadecadinoate was detected with the molecular formula $C_{18}H_{32}O_2$ where there are 2 double bonds in the alkyl chain (figure 11 and 12). This compound is also called linoleic acid which is the largest component in corn oil (Ni*et al.*, 2016; Veljkovićet al., 2018), and flower oil (Zheljazkov*et al.*, 2009).

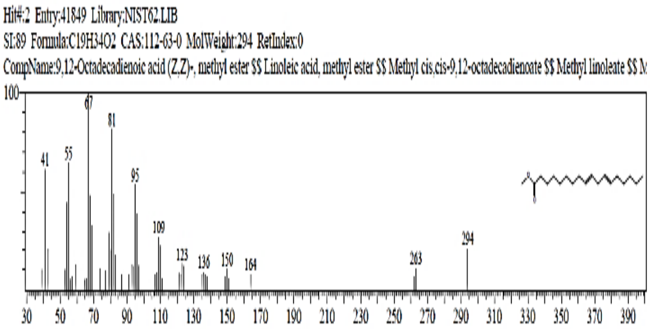


Figure 11. GCMS mass spectra at retention time of 39.665 for linoleic acid

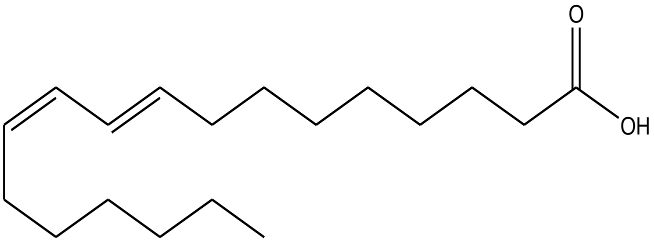


Figure 12. Structure of linoleic acid

At the retention time of 39.764 with an abundance of 55.83 %, 9-octadecanoic or better known as oleic acid with the molecular formula $C_{18}H_{34}O_2$ was also detected (figure 13 and 14). Oleic acid is also very abundant in olive oil at 69.58% (Dehghan, Golmakani and Hosseini, 2019).

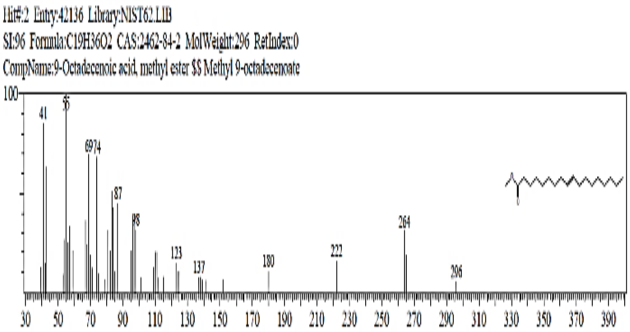
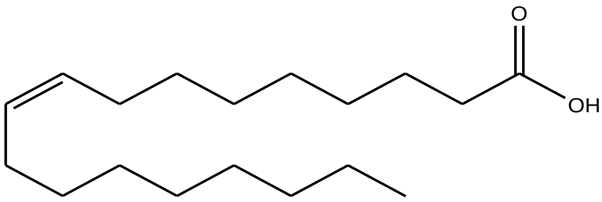


Figure 13. GCMS mass spectra at retention time of 39.764 for oleic acid



Gambar 14. Structure ofoleic acid

At the retention time of 40.262 with an abundance of 5.03 %, the mass spectra data showed that the molecular weight was at the peak m/z 298 [M^+]. If the molecular weight of CH_2 (transesterified methyl) is subtracted, a compound with a molecular weight of 284 g / mol is obtained (figure 15 and 16). This compound is octadecanoic acid or also known as stearic acid, which is known as a raw material for making soaps, cosmetics, candles, plastics and other household uses (Kumar, Sharma and Upadhyaya, 2016).

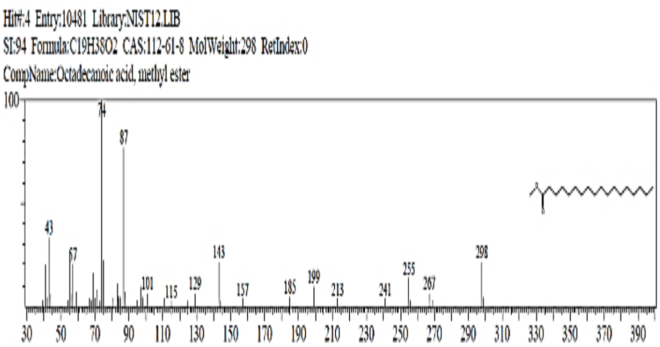


Figure 15. GCMS mass spectra at retention time of 40,262 for stearic acid

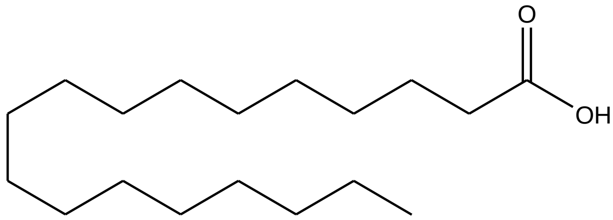


Figure 16. Structure of stearic acid

4. Conclusion

Oil isolation by means of soxhlet gave a better yield than press method. Soxhlet (B2) gave 66.32% and press (B1) gave 25.58% yield.Physico-chemical properties of Feun Kase (Thevetia peruviana) oil in this study were shown by the moisture content of 0.003% (B1) and 0.004% (B2), the acid number 1.94 mg KOH / g sample (B1) and 1.38 mg KOH / g sample (B2), lathering number 165.65 mg KOH / g sample (B1) and 166.38 mg KOH / g sample (B2), the ester content is 98.82% (B1) and 99.16% (B2), iodine value 137.44 mg I2 / 100 g sample (B1) and 126.87 mg I2 / 100 g sample (B2), density 884 Kg / m3 (B1) and 855 Kg / m3 (B2), viscosity 22.52 mm2 / s (B1) and 19.91 mm2 / s (B2), cetane numbers 48.32 (B1) and 50.55 (B2), flash points 324 °C (B1) and 322.5 °C (B2), and cloud point of 2 °C both B1 (press) and B2 (soxhlet). The fatty acids content in Feun Kase oil are hexadecanoic acid (palmitic acid), 9,12-octadecadinoic acid (linoleic acid), 9-octadecanoic acid (oleic acid), and octadecanoic acid (stearic acid).The physico-chemical properties of Feun Kase oil that meet the standards according to SNI 7182: 2015 are water content, ester content, density and cloud point. Meanwhile, those that do not meet SNI standards are the acid number, iodine value, viscosity, cetane number and flash point. From some of the physico-chemical properties of Feun Kase oil and its fatty acid content, it can be concluded that Feun Kase oil can be used as a raw material for making

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