Parametric Optimization of Kapok *(Ceiba Pentandra)* Oil Methyl Ester Production using Taguchi Approach

N. Norazahar, S. Yusup, M.M. Ahmad, S. Abu Bakar and J. Ahmad

Abstract— Utilization of new feedstock for biodiesel production has been explored. In this research, Kapok seed oil (KSO) with scientific name of *Ceiba Pentandra* has been used for synthesizing the biodiesel. The extraction of KSO gives about 22-25% wt/wt of oil. Pre-treatment of KSO has been done using esterification process with the presence of acid catalyst and transesterification of KOS is carried out after the pre-treatment using potassium hydroxide (KOH) as base catalyst. The biodiesel property of Kapok oil methyl ester (KOME) has been characterized and it shows that KOME meets the properties of biodiesel as stated in standard method of ASTM D6751 and EN14214.

Keywords—Biodiesel, fatty acid methyl esters, kapok seed oil, Transesterification.

I. INTRODUCTION

RECENT growth in population along with industrial development is an important factors that contributes towards the depletion of fossil fuel reserve of the world. Currently, researchers are looking for new sources of renewable sustainable energy due to insufficient fossil fuel to world's fuel requirements and increasing prices of fossil fuels. Ethanol, bio-methanol, and methyl ester from vegetable oils and animals fats are the alternative sources to fossil fuel [1]. Different types of energy sources such as water, solar, wind and biofuels have the potential to replace the fossil fuels. These fuels are largely utilized as transportation fuel [2]. At present, the biofuels such as biodiesel and bio-ethanol are used as the energy sources in different countries especially in Brazil, Germany, Malaysia, Indonesia, USA, Spain, and Argentina. Utilization of biofuels has gained wide attention worldwide [3]. Biodiesel that will be used in diesel engine must meet standards such as ASTM D6751 and EN 14214 [4-6]. The properties of biodiesel depend mainly on two factors, which are the quality of feedstock and the technique used for the biodiesel production. Fatty acid profile, cetane number, oxidative stability, low temperature properties, and viscosity are related to the nature of the feedstock [5].

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In previous studies, it was clear that the feedstock containing high free fatty acids was not suitable to be converted into the biodiesel by using only an alkaline catalyst. Reduction of free fatty acids value of the feedstock by using acid catalyst, prior using the alkaline catalyst is essential to reduce problems associated with biodiesel production [7, 8]. Biodiesel is defined as the mono alkyl ester of long chain fatty acid derived from the vegetable oil and animal fat. Vegetable oils are also used as an alternative fossil fuel as they are renewable, environmental friendly and free from sulfur [9]. Direct utilization of vegetable oil is not recommended due to viscosity problem [10]. Due to this problem, the researchers explore new energy and renewable energy sources. Thus, conversion to biodiesel from different types of edible and non edible oils is explored. Utilizing edible oil as biodiesel feedstock will cause fuel versus food controversy. In current study, non-edible oil is used as biodiesel feedstock. Biodiesel is also called a green diesel because it is renewable, biodegradable and eco-friendly non toxic fuel for diesel engine [11]. Different methods have been used to produce biodiesel but transesterification is the most preferred because of higher

Fig.1 Seed imbedded in Kapok lint [12]

yield obtained. Basically, the transesterification is the reaction of triglycerides (vegetable oil or animal fat) with alcohol to produce ester and glycerin as by-product [13, 14]. Different feedstock are used for biodiesel production around the world, such as soybean oil, canola oil, palm oil, but they are from edible oil sources and its utilization will increase the cooking oil prices. There are many factors that stimulated the researchers to search for new feedstocks for biodiesel production. One of them is "food versus fuel debate". "Food-vs-fuel" now is one of the world's big issue that has been discussed among scientist, researchers and even politicians. Increase in edible oil price such as palm oil and perception of using edible oil for biofuel production is responsible for their increase give initiative to the researchers and government to develop a biodiesel industry using non-edible oil [15]. Thus, new and non edible feedstock for the biodiesel production has been researched such as *Moringa Oleifera* seed oil, tobacco seed oil, *Jatropha Curcas* seed oil [16], castor seed oil [17-19].

II. KAPOK SEEDS AS NEW SOURCE FOR BIODIESEL FEEDSTOCK

Kapok (*Ceiba pentandra*) which is locally known as Kekabu is a native in America and West Africa [14]. The seeds were introduced to Southeast Asia via India. The availability of Kapok seed in Southeast Asia is scarce except for in Western India, Malaysia, Vietnam, Indonesia and Philippines. Kapok is a humid tree of the Malvales and a family of Malvaceae. In Malaysia, Kapok is commonly found in northern parts of peninsular Malaysia. The kapok tree can produce between 500 to 4000 fruits at one time, with each fruit containing 200 seeds. The seed of Kapok are very striking with pointed capsule shape and the woody pods hanging on tree like cucumber in which the flashy silky fiber black color seeds are present [15]. The fiber contains almost 64% cellulose are elastic in nature and use in cushion, pillow, mattresses, life jackets etc [16]. The seed are brownish black in color and surrounded in the lint fiber and it contains 25-28% of oil which its characteristics were found to be close enough to cotton seed oil.

In 1964 up to 1966, several researches had been done by few researchers about the KSO content as well as the fatty acid composition inside the oil [20]. They reported that KSO contain cyclopropenoid fatty acids (CPFA) which can give various types of physiological disorders in farm. Therefore, the seed of Kapok is not suitable for consumption but they discovered that KSO is rich in unsaturated fatty acids [21]. The properties of oil which contained unsaturated fatty acid can be excellent oil in engine performance during cold weather when it converts to biodiesel [21]. The main objective of the current study is to explore the utilization of Kapok seed oil which is non edible oil as potential feed stock for higher quality of biodiesel.

III. FREE FATTY ACID (FFA) CONTENT

Determination of FFA content inside the oil is one of the important parts in transesterification of biodiesel. For feedstock which contains less than 2 to 3 % of FFA content, pre-treatment of the feedstock is not required and one step of process which is direct transesterification reaction is sufficient to produce high quality of biodiesel [22]. In other way, hypothesis can be made that, the higher the FFA content of the feedstock, the lower the conversion of FFA to fatty acid methyl ester (FAME) through base catalyzed transesterification [23, 24]. Therefore, instead of using direct transesterification, two-step, acid-catalyzed esterification and base-catalyzed transesterification process have been introduced to reduce the FFA content to less than 1% and furthermore, maximize the conversion of FFA to FAME [25-27]. Kusdiana and Saka also suggested two-step noncatalytic conversion process as an alternative to direct transesterification reaction [28, 29].

IV. ACID-CATALYZED ESTERIFICATION REACTION

Generally, a one step process can be applied using acid as the catalyst. H_2SO_4 is acid catalyst which commonly used for the reaction but the reaction is relatively slow even though it can give higher biodiesel yield compared to transesterification reaction using base catalyst and the reactor used need to be in acidic environment [30]. Therefore, base-catalyzed transesterification is much more effective in terms of reaction rate. Esterification reaction using acid as catalyst is one of the most important alcoholisms techniques for its ability to neutralize the high FFA content in the biodiesel feedstock. When base catalyst is introduced in the transesterification of feedstock which contain high FFA, this FFA will be converted into soaps and it will further inhibit the separation of the alkyl esters and glycerol [15]. These soap and emulsion formations make separation of product and by-product of biodiesel become longer and harder [31]. In order to achieve good conversion of vegetable oil to ester, the FFA content of the feedstock should not exceed 1% for the alkaline-catalyzed transesterification reaction [32]. Otherwise, saponification reaction of oil and fats with base catalyst will take part in the transesterification reaction as show in the Fig.2.

$$RCOOR' + H2O \rightleftharpoons RCOOH + R'OH$$

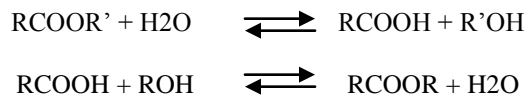$$RCOOH + ROH \rightleftharpoons RCOOR + H2O$$

Fig.2 Saponification reaction of fatty acid alkyl ester

Therefore, to avoid the saponification reaction from occurring, esterification reaction is applied using acid catalyst to reduce the FFA content in the oil below than 1% followed by base-catalyzed transesterification reaction using the treated oil [33, 34]. Determination of acid value as well as the FFA content before pre-treatment is very important to avoid shortcoming after transesterification process.

V. TRANSESTERIFICATION REACTION

Transesterification is a chemical reaction in which triglyceride reacts with alcohol in the presence of catalyst. During this reaction, mono-ester is produced. The long chain and branched chain fatty acid molecules are converted to mono-ester by transesterification [6]. The transesterification reaction consists of three consecutive reversible steps; firstly conversion of triglycerides to diglycerides, followed by diglycerides to mono-glycerides. The glycerides are converted into glycerol and one ester molecule in each step. The properties of the ester produced during this reaction are similar to that of diesel. The reaction is as shown in Fig. 3.[35].
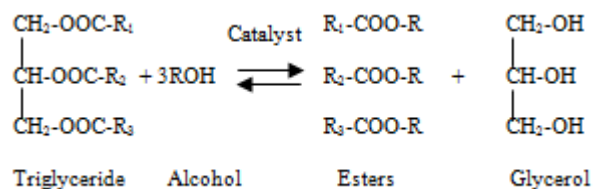
Fig.3 Transesterification of triglycerides with alcohol [35]

Theoretically, three moles of alcohol are required for one mole of triglyceride, but in actual practice a higher molar ratio of methanol is required to establish the equilibrium and to obtain higher percentage yield of ester. Commonly, short chain alcohol such as methanol, ethanol and propanol are used as the solvent. The yield of ester is independent of the type of alcohol. Mostly methanol is used as a solvent because it is the cheapest among others. Both types of acidic and basic catalyst are used for feedstock having high content of free fatty acid and most common basic catalysts are potassium hydroxide.

Numerous studies have been carried out on acid-catalyzed esterification and base-catalyzed transesterification on extracted seed oil from edible sources and non-edible sources. Biodiesel production from rubber seed oil (RSO) using two-step process had been analyzed by Ramadhas *et.al* [10]. In the first step of acid esterification, free fatty acid (FFA) content of RSO was reduced to less than 2% using 0.5wt% of sulfuric acid (H_2SO_4) as acid catalyst, reaction time of 20-30mins and reaction temperature at 50^oC[10]. Modhar Khan *et.al* [36] had tested acid esterification on crude palm oil (CPO) and crude rubber seed oil (CRSO) blend. The optimum conditions for the acid esterification reaction to lower the FFA value to 0.65% were 65^oC, 15 to 1 molar ratio of methanol to oil, 0.5 wt% of H_2SO_4 and 3hr of reaction time. Temperature was the most significant effect on the reduction of FFA in the analysis.

VI. MATERIALS AND METHODS

Fig.4 shows the flow chart of research activities which cover the following steps:
a) Drying of Kapok seeds
b) Extraction of KSO
c) Acid value test of KSO using titration method
d) Base catalyzed transesterification of esterified KSO
e) Separation product from byproduct
f) Quality analysis of biodiesel using ASTM D 6751 and EN 14214 standard.
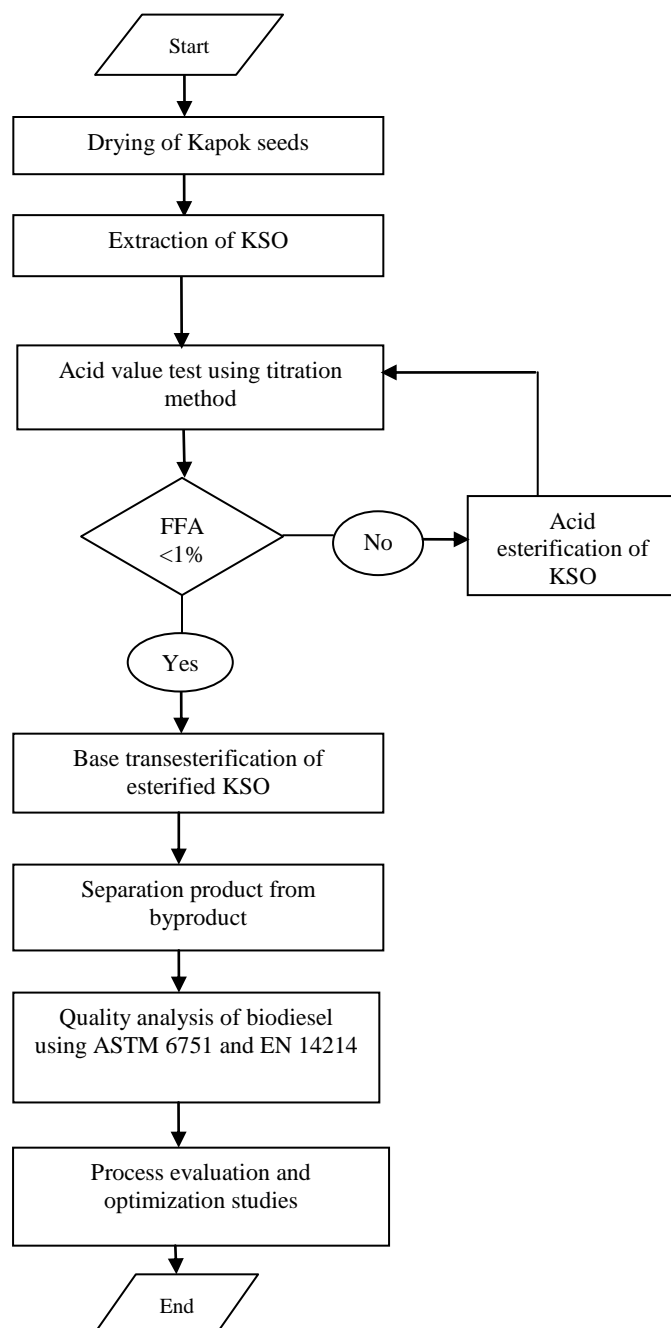g) Process evaluation and optimization studies

Fig. 4 Flow chart of the research activities

A. Materials

Kapok seed was obtained from Bota, Perak, Malaysia. Analytical grade methanol, sulfuric acid and potassium hydroxide were used for esterification and transesterification reaction and were supplied by Merck Chemical Company (Darmstadt, Germany). Pure standard of FAME was purchased from Sigma Chemical Company (St. Louis, USA).

B. Extraction of Kapok seeds oil

1500 g of Kapok seeds were crushed using a grinder. The extraction of Kapok seed oil was carried out in soxhlet extractor using n-hexane as the solvent at 60°C for 6 hr. The solvent was then distilled at 45°C under vacuum using lab scale rotary evaporator. The extracted oil was reheated at 45°C to eliminate excess solvent. The yield of KSO extracted was calculated using formula as shown in (1):

$$KSO\ (wt\%) = \frac{Mass\ of\ oil\ extracted}{Mass\ of\ Kapok\ seed}\ x\ 100\% \qquad (1)$$

C. Analysis of raw materials

The acid value for Kapok seeds oil (KSO) was determined using AOCS Cd 3d-63 method [37] and it was shown that the acid value of KSO used in this study which corresponds to 36.03% of free fatty acid (FFA) content in the oil was 72.05 mg KOH/ g oil. Higher FFA content (> 1%) can lead to soap formation during transesterification reaction. Therefore, the FFA content of KSO was reduced below 1% using acid catalyzed esterification as pre-treatment of the feedstock.

D. Acid Esterification (pretreatment)

The acid esterification reaction of KSO was carried out at 65°C for 3 hr in sohxlet extractor using 6 to 1 methanol to oil ratio as well as 0.5 and 1.0 wt/wt of sulfuric acid as catalyst. This process was repeated until the acid value of the sample was less than 2 mg KOH/ g of oil or below 1% of FFA content before proceeded to transesterification reaction.

E. Transesterification of Kapok seed oil

100 g of KSO was heated in a round bottom flask at 55°C using heating plate. The methanol-KOH solution at specific amount was then added into the round bottom flask and stirred for 3 hr. Once the transesterification reaction completed, the mixture was left overnight for complete separation. Two layers of immiscible phase were obtained. The upper layer consisted of fatty acid methyl esters (FAME) and the lower layer contained glycerol, excess methanol, and unreacted catalyst. The FAME was purified with warm deionized water to remove the residual catalyst and anhydrous sodium sulphate (Na_2SO_4) was then added to the methyl esters to absorb the residual water. The mixture was then filtered to get pure biodiesel.

VII. RESULTS AND DISCUSSION

A. Kapok seed oil yield after extraction

Equation (1) was used to calculate oil yield extracted from Kapok seeds. From the extraction analysis, about 15 g of oil was extracted from 80 g of Kapok seeds which equivalent to 18.75 wt%.

B. Acid value test of Kapok seeds oil before and after esterification

Acid value of KSO can be calculated using AOCS Cd 3d-63 method [37] by referring to (2):

$$Acid\ value\ \left(mg\ \frac{KOH}{g\ of\ oil}\right) = \frac{(A - B)x\ Nx\ 56.11}{W} \qquad (2)$$

Where:
A   : Volume of titrant used for sample (ml)
B   : Volume of titrant used for blank (ml)
N   : Normality of KOH which is equal to 2
56.11 : Molecular weight of KOH (g/mol)
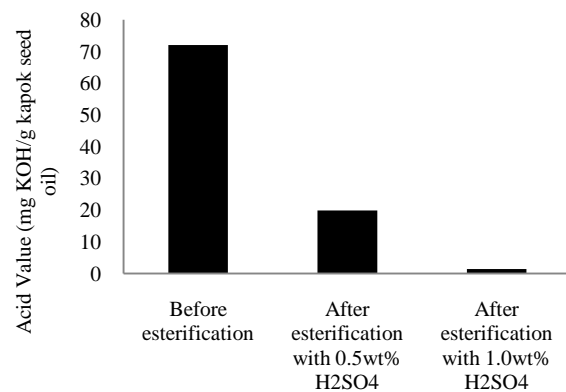W   : Weight of sample

Fig.5 Comparison of acid value for KSO before and after esterification reaction
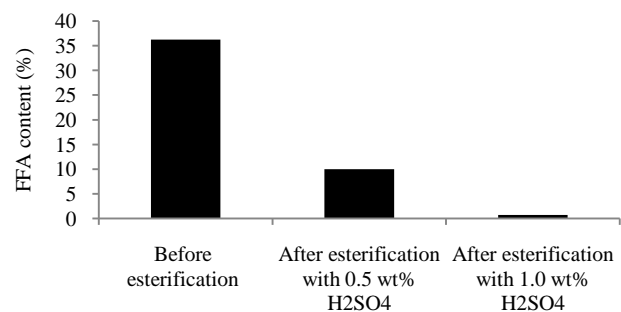

Fig.6 FFA content for KSO before and after esterification reaction

Fig.5 and Fig.6 showed the acid value and FFA of KSO before and after the esterification process. The acid value was reduced from 72.05 mg KOH/ g oil to 1.37mg KOH/ g oil. About 98% reduction of acid value was achieved when 1 wt% of sulfuric acid as catalyst was added into the solution. It corresponded to 0.69% of FFA content of KSO. Treated KSO was then used for transesterification process.

C. Kapok oil methyl ester (KOME) from transesterification process

Analysis had been done for fatty acid methyl ester of KSO produced from transesterification process. Experimental work was designed using Taguchi approach to find the optimum conditions as shown in Table 1:

Table 1: Design of Experiment for transesterification of KSO

Parameters	Level		
A (Molar ratio of alcohol to oil)	6:1	8:1	10:1
B (Temperature - ^{o}C)	45	55	65
C (Catalyst weight - wt %)	10.5	1.0	2.0

From the experimental analysis above, the highest yield of KOME (98%) was obtained at 55°, 8:1 of methanol-to-oil molar ratio, and 2 wt% of KOH. Modhar, 2009 [38][38] analyzed the same design of experiment for transesterification of rubber seed oil (RSO) and discovered that the optimum conditions for rubber seed oil methyl ester (RSOME) conversion were at temperature of 55°C, methanol-to-oil molar ratio of 8:1 and 2 wt% of base catalyst based on weight percent of oil [38].

Fig.7 Kapok oil methyl ester (KOME) after separation from by-product

D. Kapok oil methyl ester (KOME) profile by GC-MS

Kapok seed oil methyl esters (KOME) were analyzed using Gas Chromatography Mass Spectroscopy (GC-MS) from Agilent-Technologies. GC system was equipped with a Triple Axis inert XL EI/CI mass selective detector and Agilent-technologies capillary column RT-2500 (100 x 0.25 mm; film thickness 0.20 μmeter). 1.0 μL of sample was injected to the column using the split mode (split ratio 1:100). Helium was used as a carrier gas at 1.2 ml/min. Column oven temperature was adjusted from 150°C to 250°C at a linear ramp rate of 4°C/min. The initial and final hold up time was 1 and 5 min respectively. For GC/MS detection, an electron ionization method was used with the ionization energy of 70 eV. The determination of KOME on GC/MS was analysed in quantitative and qualitative analysis. The identification of the unknown KOME was compared with the standards of FAME based on their relative retention times. The fatty acid methyl esters obtained is shown in Table 2:

Table 2: Fatty acid methyl esters profile of KOME

KOME	Percentage (wt %)
Methyl Palmitate	23.17
Methyl Caproate	9.42
Methyl Linoleate	30.00
Methyl Oleate	22.88
Methyl Stearate	4.73
Methyl Sterculate	8.64
Methyl Arachidate	1.18

Based on the FAMEs profile in Table 2, it showed that KSO contained high unsaturated fatty acid which is linoleic acid followed by palmitic acid (saturated fatty acid). Hilditch *et.al* [39] also reported that KSO consists mainly oleic and linoleic acids forming together about 70 wt% of the total fatty acids [39].

VIII. FUEL PROPERTIES

The fuels properties of the KOME such as cetane number, density and oxidative stability were analyzed and compared with ASTM D6751 and EN14214 standard methods as shown in Table 3:

Table 3: Properties of KOME in comparison to ASTM D6751 and EN 14214 standards

Property	KOME	ASTM D6751	EN 14214
Cetane number	57.52	47 min	51 min
Density (25°C) kg m^{-3}	860	-	-
Oxidative stability (hr)	12.3	3 min	6 min
Cloud point (^{o}C)	4.4	5	[a]
Pour point (^{o}C)	1	-15	[b]
Cloud filter plugging point (^{o}C)	4	-	[b]
Flash point (^{o}C)	148	93 min	120 min
Kinematic Viscosity (mm^{2}s^{-1} at 40°C)	1.8	1.9-6	3.5-5.0
Sulphur content (wt %)	0.024	0.05 max	-
Moisture content (%)	0.03	-	-
Higher heating value (MJ kg^{-1})	39.4	-	-
Acid value (mg KOH g^{-1})	1.37	0.5 max	0.5 max

[a] Not specified

[b] Not specified. EN14214 use time-and location dependant values for the cold filter plugging point (CFPP) instead.

A. Cetane number

The cetane number expresses the ignition property of biodiesel similar to cetane number in fossil base diesel. It is related to the ignition delay time a fuel experiences upon injection into the combustion chamber. The cetane number decreases with decreasing chain length and increasing degree of un-saturation and branching. Cetane number measures the biodiesel ignition delay with higher cetane number indicates shorter time between initiation of fuel injection and ignition. In this study, the cetane number for KOME is 57.52. The minimum cetane number prescribed in ASTM D6751 and EN 14214 is 47 and 51 respectively.

B. Density

Density specification for biodiesel is included in the European standard (EN 14214). Density of KOME in this study is 860 kg/ m^3 at 25°C using Density Meter, Anton Paar (DMA 4500M). Chettri et.al [40] also determined the density of jatropha, canola and soapnut biodiesel using densitometer at elevated temperatures and pressure and reported the density value of three different types of biodiesel were 886, 884 and 880 kg/ m^3 respectively [40]. Yusup.S et.al [41] reported that rubber seed oil which has higher density (910 kg/m^3) was reduced to 874 kg/ m^3 when converted to rubber seed oil methyl ester (RSOME) during transesterification process [38]. It shows that transesterification of the oil is the solution to match the acceptable range with the standard biodiesel.

C. Flow Properties of biodiesel

The flow of biodiesel consists of cloud point (CP), pour point (PP) and cloud filter plugging point (CFPP). These properties are temperature dependant and the limits of cloud point are not specified in both the ASTM and EN standards since the value is associated with the weather condition and the place where the test is conducted. Furthermore, the biodiesel blend standard of ASTM D7467 does not contain any temperature limits. The EN 14214 standard also does not mention a low-temperature parameter in its specifications. However, it discusses the use of a low-temperature filterability test which is cold filter plugging point (CFPP). Table 3 showed the CP, CFPP and PP values of KOME were 4.4, 4 and 1°C respectively. From previous research work which had been done on rapeseed biodiesel, it showed that rapeseed biodiesel had lower values of CP and PP which were CP -3°C and PP -9°C [42]. Jatropha, pongamia and palm biodiesel had been tested on its cloud point and pour point by Sarin et.al[43] and it show that Palm, Jatropha, and Pongamia biodiesels had cloud points of 16.0, 4.0, and −1.0 °C and pour points of 12.0, −3.0, and −6.0 °C respectively. The low temperature properties of KOME can be improved by using different types of additives.

D. Oxidative stability

The oxidative stability of biodiesel was analyzed by standard in rancimat method following EN 14112 which is 6 and 3hr (minimum) based on EN 14214 and ASTM D6751 respectively. Both standards are automated and sample heating is conducted to a specified temperature (normally 110°C) and air bubble is performed through the sample .The oxidation of biodiesel contributes to major problem during storage. The presence of unsaturated bonds influenced this property largely. The oxidation occurs due to presence of air, eminent temperature and presence of metals content which promote the oxidation of the unsaturated bonds. Generally, oxidation is slow at the beginning of the reaction. The point at which the rate of oxidation increases is the induction period which is the maximum change of rate of oxidation. The induction time of KOME was determined to be 12.3 hr which can be classified as good and stable compared to the values in both standards.

E. Higher heating value (HHV) and sulphur contents

HHV is the measurement of the energy produced during biodiesel burning. This property is related to the stability of biodiesel. In this analysis, HHV of KOME was 39.4 MJ/kg. The sulphur content of KOME was analyzed by LECO CHNS-932 USA model and the value was 0.024 wt% which was less than the standard value.

IX. CONCLUSION

From the research analysis, it can be proven that KSO is one of the potential feedstocks for biodiesel production due to its abundance cultivation, high oil content ratio and no competitive issue between other edible oil resources. The biodiesel production from high FFA feedstock of KSO using direct transesterification without pre-treatment is difficult because high FFA creates problem and large amount of oil are converted into soap. In this study, esterification was done as pre-treatment for the feedstock followed by transesterificaion process. Acid value of KSO reduced to 98% reduction from 72.05 mg KOH/ g oil to 1.37 mg KOH/ g oil. Biodiesel production from KSO was done using transesterification process and the highest yield of KOME which is 98% was obtained at 55°C, 8:1 of methanol-to-oil molar ratio, and 2 wt% of KOH. Characterizations of KOME according to international standard method (ASTM D6751 and EN 14214) showed that the properties of KOME such as cetane number, oxidation stability, cold flow properties and viscosity meet the standards. Nevertheless further research and development is required to improve its fuel properties.

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