



Original Research Paper

Microwave-assisted methyl esters synthesis of Kapok (*Ceiba pentandra*) seed oil: parametric and optimization study

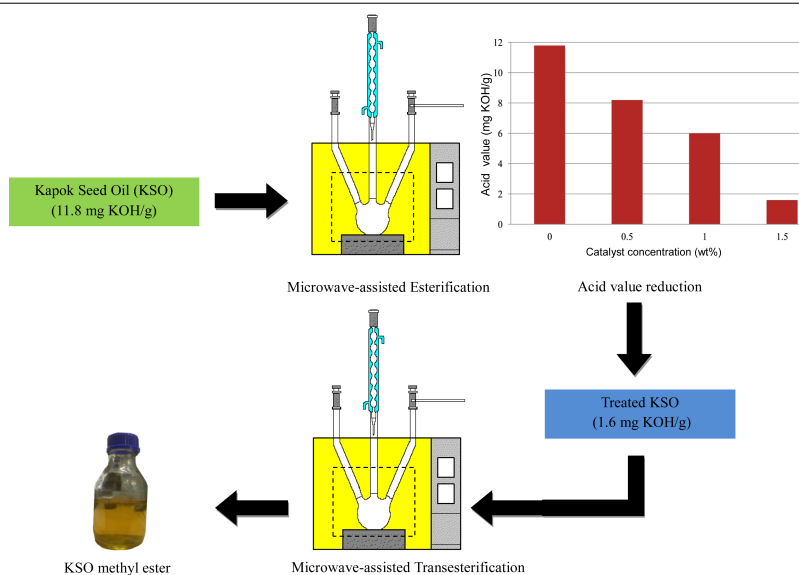
Awais Bokhari, Lai Fatt Chuah, Suzana Yusup*, Junaid Ahmad, Muhammad Rashid Shamsuddin, Meng Kiat Teng

Biomass Processing Laboratory, Centre of Biofuel and Biochemical Research (CBBR), Chemical Engineering Department, Universiti Teknologi PETRONAS, Bandar Seri Iskandar, 32610 Seri Iskandar, Perak, Malaysia.

HIGHLIGHTS

- Kapok oil methyl ester was produced by microwave-assisted technique.
- About 14 to 37 fold lesser reaction time using microwave than mechanical stirring.
- Optimization and parametric study by using response surface methodology.
- The properties of the produced fuels met EN 14214 and ASTM D 6751.

GRAPHICAL ABSTRACT



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ABSTRACT

The depleting fossil fuel reserves and increasing environmental concerns have continued to stimulate research into biodiesel as a green fuel alternative produced from renewable resources. In this study, Kapok (*Ceiba pentandra*) oil methyl ester was produced by using microwave-assisted technique. The optimum operating conditions for the microwave-assisted transesterification of Kapok seed oil including temperature, catalyst loading, methanol to oil molar ratio, and irradiation time were investigated by using Response Surface Methodology (RSM) based on Central Composite Design (CCD). A maximum conversion of 98.9 % was obtained under optimum conditions of 57.09 °C reaction temperature, 2.15 wt% catalyst (KOH) loading, oil to methanol molar ratio of 1:9.85, and reaction time of 3.29 min. Fourier Transform Infra-Red (FT-IR) spectroscopy was performed to verify the conversion of the fatty acid into methyl esters. The properties of Kapok oil methyl ester produced under the optimum conditions were characterized and found in agreement with the international ASTM D 6751 and EN 14214 standards.

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* Corresponding author at: Tel.: +6053687642
E-mail address: drsuzana_yusuf@petronas.com.my

1. Introduction

The fast growing population and industrialization have relentlessly increased the demand for energy (Moradi et al., 2015). Generally, energy demands are mostly met by non-renewable resources, such as petroleum, natural gas, and coal. It is well documented that these resources have serious negative environmental impacts particularly due to the emissions of nitrogen oxides, sulphur oxides, unburned hydrocarbons, and particulate matters (Chuah et al., 2015a; Jaber et al., 2015). Renewable fuels such as bioethanol and biodiesel are amongst the best alternatives to fossil fuels (Atabani et al., 2015).

Biodiesel is advantageous as a replacement for conventional diesel because up to a certain inclusion rate, it does not require any modifications in diesel engines. This green fuel is defined as the mono-alkyl esters of long chain fatty acids derived from triglycerides of vegetable oils and animal fat (Fadhil, 2013). These triglycerides cannot be used directly in diesel engines due to the high viscosity of the oils and their low volatility resulting in incomplete combustion and carbon depositions. Thus, they need to be converted into biodiesel via a number of processes such as transesterification (Sharma et al., 2008). In the transesterification reaction, triglycerides react with an alcohol (methanol or ethanol) in the presence of a catalyst (Chuah et al., 2015b). The catalyst could be either acidic or basic depending on the free fatty acid content of the oil feedstock (Georgogianni et al., 2009). Stoichiometrically, one mole of triglycerides reacts with three moles of methanol to produce three moles of methyl ester and one mole of glycerin (Chuah et al., 2015c).

Malaysia is diversifying its biodiesel feedstock towards non-edible plant oils, such as *Ceiba pentandra* (Kapok), *Calophyllum inophyllum* (bintangor laut / nyamplung / penaga laut), *Jatropha curcas* (Jatropha), *Ricinus communis* (Castor), *Hevea brasiliensis* (Rubber), and waste from palm oil processing. These local non-edible plant oils have drawn attention as biodiesel feedstock due to their potential and abundant supply. *Ceiba pentandra* locally known as Kapok or Kekabu is grown in Malaysia, India and other parts of Asia. It grows naturally in humid and sub-humid tropical regions. Kapok pod contains 17% fiber that is mainly utilized in making pillows and mattresses, whereas the seeds are traditionally considered as waste (Ong et al., 2013). Kapok seeds make up about 25 - 28 wt% of each pod with an average potential oil yield of 1280 kg/ha annually (Yunus Khan et al., 2015). The most common method for extracting oil from the Kapok seeds is mechanical expeller (Vedharaj et al., 2013). The use of Kapok seeds is well in line with the purpose of the second generation biodiesel production, i.e., utilization of non-edible feedstock to avoid direct conflict with human food (Lee et al., 2011).

Various non-edible oils have been used for biodiesel production through transesterification of triglycerides by using different methods, such as mechanical stirring, supercritical procedure (Ong et al., 2013), ultrasonic techniques (Ji et al., 2006), hydrodynamic cavitation (Chuah et al., 2015b), and microwave (Lee et al., 2010). However, only a few studies have reported on biodiesel production from Kapok seed oil (KSO). Among them was the study recently conducted by Yunus Khan et al. (2015) who investigated the fuel properties of a biodiesel obtained from the blends of *Ceiba pentandra* and *Nigella sativa* by mechanical stirring. Sivakumar et al. (2013) also studied the effect of molar ratio of methanol to Kapok oil, temperature, time, and catalyst concentration on biodiesel production process by using mechanical stirring method. In a different study, Vedharaj et al. (2013) reported that biodiesel derived from Kapok oil emitted higher nitrogen oxides compared to diesel fuel. The authors further strived to reduce the nitrogen oxides by using urea based selective non-catalytic reduction system, which was retrofitted in the exhaust pipe (Vedharaj et al., 2014).

Transesterification reaction assisted by mechanical stirring has been widely used for biodiesel production. However, there are a number of problems associated with this technique, i.e., long reaction time, non-uniform heat distribution, and large energy requirements. These drawbacks have rendered researchers to find alternative methods. Microwave-assisted biodiesel production has been investigated using various oil feedstock, e.g., *Camelina sativa* oil, rice bran oil, *Pongamia pinnata*, tallow, yellow horn oil, castor oil, used cooking oil, palm oil, coconut oil, soybean oil, and *Jatropha curcas* (Motasemi and Ani, 2012; Yunus Khan et al., 2014). This technique has advantages over the mechanical stirring method including shorter reaction time, efficient heating, and facilitated separation of glycerol from biodiesel

(Motasemi and Ani, 2012). Despite its advantages, microwave-assisted transesterification has never been attempted on raw KSO.

Therefore in the present study, the design of experiment and optimization of microwave-assisted transesterification of Kkapok oil into biodiesel was conducted by incorporating four reaction parameters, namely methanol to oil molar ratio, catalyst concentration, temperature, and reaction time using response surface methodology (RSM) and four-way analysis of variance (ANOVA). The response (i.e., methyl ester conversion) was fitted by a quadratic polynomial regression model using least square analysis in a five-level-four-factor central composite design (CCD). The quality of Kapok oil methyl ester produced was investigated according to the ASTM D 6751 and EN 14214 standards.

2. Materials and methods

KSO was purchased from the East Java Province, Indonesia. Solvent and chemical used in the experiments, i.e., anhydrous methanol, 95 % sulphuric acid (H_2SO_4), and potassium hydroxide (KOH) were analytical grade. All chemicals were purchased from Merck (Malaysia) except for H_2SO_4 which was purchased from Sigma Aldrich (Malaysia).

2.1. Kapok seed oil characterization

The properties of KSO including acid value, saponification value, iodine value, density, kinematic viscosity, and flash point were analyzed. All analysis were performed by following the AOCS, DIN and ASTM methods (Chuah et al., 2015a).

2.2. Pretreatment of kapok seed oil

Due to high acid value, the KSO was pretreated to ensure a high conversion rate into methyl esters. To reduce the acid value of the KSO, acid esterification reaction was performed under microwave irradiations by reacting KSO with methanol (1:6) in the presence of sulphuric acid (1.5 wt.%) as catalyst at 60 °C for 5 min. The acid value of the KSO was measured after the pretreatment to ensure that it was lower than 2 % before proceeding with the alkali-catalyzed transesterification reaction (Ramadhas et al., 2005).

2.3. Experimental design

The experimental arrays were designed by CCD. The reaction variables and their respective ranges are shown in Table 1. The independent input process variables were primarily classified in terms of low and high levels. The factors were further distributed into versatile points called axial, center and factorial points. The axial points were coded by the CCD as -2 and +2. The low and high lever factor points were designated as -1 and +1. Whereas the centre points were coded as 0 and the repeated experimental arrays were designed on the centre points.

Table 1.
Design Parameters for transesterification process.

Process parameters	-2	-1	0	+1	+2
Methanol to oil (molar ratio)	2	6	10	14	18
Catalyst loading (wt%)	0	1	2	3	4
Temperature (°C)	25	40	55	70	85
Time (min)	0.5	2	3.50	5	6.5

2.4. Transesterification and microwave configuration

Transesterification was performed according to the experimental design set by RSM and following the procedure described by Yusup and Khan (2010). A total of 30 runs were performed under different combinations of process parameters. In each run, 50 g of KSO was mixed with a specified amount of methanol-KOH solution in a 500 mL three-neck round-bottom reactor, and was heated and stirred in a 3000 W microwave (Fig. 1) for a specified time period set by the design of expert. Upon completion of the reaction, the mixture was transferred into a separating funnel and was left for 6 h for complete separation. Two layers of immiscible phases were obtained. The

upper layer consisted of Kapok oil methyl ester (KOME), whereas the lower layer included the by-product and residues consisting of glycerol, excess methanol and un-reacted catalyst. KOME was separated and washed with warm deionized water to remove residual catalyst. Rotary evaporator was then employed to remove the residual water in KOME.

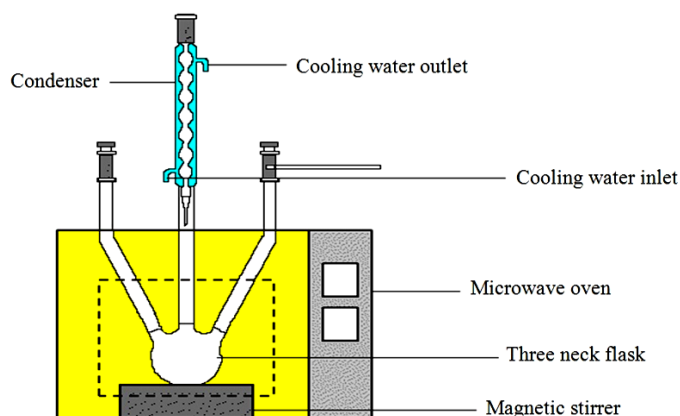


Fig.1. Experimental setup

2.5. Fatty acid methyl ester analysis

The produced KOME was analyzed by using Gas Chromatography (GC) to determine the fatty acid methyl ester (FAME) conversion achieved in each experimental run based on the EN 14013 standard method (Knothe, 2006). The Agilent-Technologies 7890A model GC was used for the FAME determination. The GC system was equipped with a variable split flow injector, a temperature programmable oven, a flame ionization detector, and a capillary column coated with methylpolysiloxane (DB-23) (60 x 0.25 mm; film thickness 0.25 μ m).

Temperature program included holding for 2 min at 100 °C, heating at 10 °C/min until 200 °C, heating at 5 °C/min until 240 °C and holding for 7 min. Helium was used as the carrier gas at a flow rate of 4 mL/min. Hydrogen and air were used at flow rates of 50 and 400 mL/min, respectively, for flame. The injector temperature and detector temperature were set at 250 °C. The volume of the sample injected was 1 μ L. All the experiments were conducted in three replicates and the reported values are averages of the individual runs and the inaccuracy percentage was less than 2% of the average value. The properties of the purified biodiesel were analysed according to both ASTM and EN standards.

2.6. Fourier Transform Infra-Red Spectroscopy

Fourier transform infra-red (FTIR) spectroscopy was used to analyze the conversion of the KSO into KOME.

3. Results and discussion

3.1. Kapok seed oil characterization

Physiochemical analysis was performed on KSO to analyze the quality of the feedstock. The results were given in Table 2. The acid value is an important parameter to indicate the quality, age and purity degree of an oil during processing and storage. Oil samples possessing acid values > 4 mg KOH/g require a two-step processing, e.g. acid esterification followed by alkaline transesterification. The results of the present study revealed that the acid value of the KSO was 11.8 mg KOH/g, which was higher than the set point of 4 mg KOH/g. Saponification is a process by which the fatty acids in the glycerides of oil are hydrolysed by an alkali. The results obtained revealed that the saponification value of the KSO was 194 mg KOH/g. The density (at 20 °C), kinematic viscosity (at 40 °C) and flash point of the KSO were 0.91 g/cm³, 36.21 mm²/s and 210 °C, respectively.

Table 2. Physiochemical properties of the kapok seed oil.

Analysis	Crude kapok seed oil
Acid value (mg KOH/g)	11.80
Saponification value (mg KOH/g)	194.00
Iodine value (g I ₂ /100 g)	102.40
Density at 20 °C (g/cm ³)	0.91
Kinematic viscosity at 40 °C (mm ² /s)	36.21
Flash point (°C)	210

3.2. Pretreatment of kapok seed oil

It can be observed from Figure 2 that a significant reduction in acid value from 11.8 to 1.6 mg KOH/g was achieved by using 1.5 wt% of H₂SO₄ as a catalyst, methanol to oil ratio molar ratio of 6:1, and reaction time of 5 min at 60 °C.

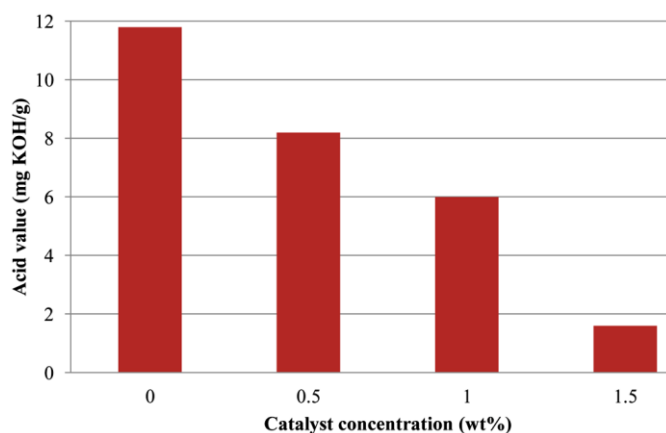


Fig.2. Acid value reduction after pretreatment.

3.3. Optimization study of transesterification process variables

RSM was used to optimize the four process variables, i.e., methanol to oil molar ratio, catalyst concentration, reaction temperature, and reaction time. Thirty runs at points were set based on the Design Expert. The response FAME conversion was calculated by the GC-FID and the results were compared with the predicted response (Table 3). The FAME conversion was observed in the range of 37.40 to 98.98 %. More specifically, the lowest conversion of 37.40 % was associated with the 10:1 methanol to oil molar ratio in the absence of catalyst at 55 °C for 3.5 min of reaction time, while the maximum FAME conversion of 99.98 % was obtained at 10:1 methanol to oil molar ration in the presence of 2 wt% catalyst at 55 °C for 3.50 min of reaction time.

3.4. ANOVA analysis of base transesterification

Table 4 shows the ANOVA results of the base transesterification. P-value of this model was <0.0001 showing that the model was significant. P-value represents the significance of the model and F-value represents the most influencing factor in a study (Lee et al., 2005). The significance of the reaction parameters with regard to FAME conversion was in the order of catalyst concentration > temperature > reaction time > methanol to oil molar ratio.

The FAME was the response of the process variables in this study and the factor methanol to oil molar ratio (A), catalyst concentration (B), reaction temperature (C), and reaction time (D) were the process variables. The R² value was measured at 0.9085 (Table 4), revealing that the experimental data validated 90.85 % of the model. The regression analysis resulted in a response surface equation for the output response, i.e., FAME conversion.

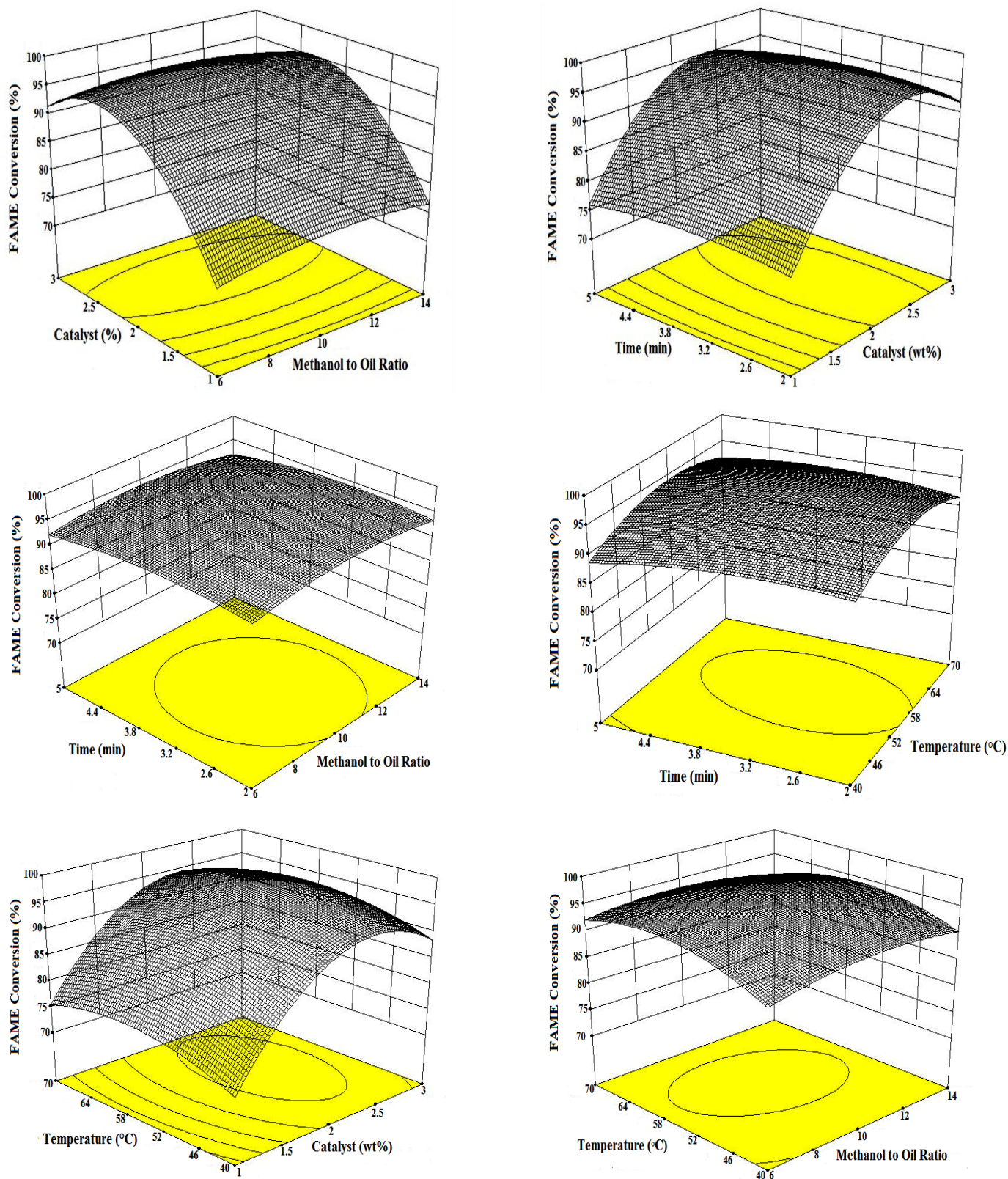


Fig.3. 3-D plots of process variables with respect to fatty acid methyl ester conversion.

Table 3.
Transesterification experimental designed and response of fatty acid methyl ester conversion.

Run	Methanol to oil molar ratio	Catalyst concentration (wt%)	Temperature (°C)	Time (min)	Experimental FAME conversion (%)
1	6.00	1.00	40.00	2.00	74.14
2	6.00	1.00	70.00	2.00	77.57
3	6.00	1.00	40.00	5.00	72.52
4	6.00	1.00	70.00	5.00	76.34
5	6.00	3.00	40.00	2.00	79.75
6	6.00	3.00	70.00	2.00	80.25
7	6.00	3.00	40.00	5.00	75.00
8	6.00	3.00	70.00	5.00	80.96
9	14.00	1.00	40.00	2.00	76.03
10	14.00	1.00	70.00	2.00	77.56
11	14.00	1.00	40.00	5.00	73.87
12	14.00	1.00	70.00	5.00	77.27
13	14.00	3.00	40.00	2.00	79.70
14	14.00	3.00	70.00	2.00	77.52
15	14.00	3.00	40.00	5.00	75.10
16	14.00	3.00	70.00	5.00	75.32
17	10.00	2.00	25.00	3.50	82.69
18	10.00	2.00	85.00	3.50	85.94
19	10.00	2.00	55.00	0.50	91.65
20	10.00	2.00	55.00	6.50	93.89
21	10.00	0.00	55.00	3.50	37.40
22	10.00	4.00	55.00	3.50	72.77
23	2.00	2.00	55.00	3.50	89.65
24	18.00	2.00	55.00	3.50	92.87
25	10.00	2.00	55.00	3.50	98.80
26	10.00	2.00	55.00	3.50	98.90
27	10.00	2.00	55.00	3.50	98.96
28	10.00	2.00	55.00	3.50	98.98
29	10.00	2.00	55.00	3.50	98.02
30	10.00	2.00	55.00	3.50	98.64

Table 4.
ANOVA analysis of the Transesterification experiment.

Source	Sum of Squares	DF	Mean Square	F value	P value
Model	4269.52	14	04.97	10.64	<0.0001 significant
A (Methanol)	0.22	1	0.22	0.03	0.9319
B (Catalyst)	330.34	1	330.34	11.52	0.0040
C (Temperature)	22.39	1	22.39	0.78	0.3908
D (Time)	5.66	1	5.66	0.20	0.6630
AB	9.73	1	9.73	0.34	0.5688
AC	7.21	1	7.21	0.25	0.6234
AD	0.35	1	0.35	0.012	0.9137
BC	3.69	1	3.69	0.13	0.7249
BD	1.92	1	1.92	0.067	0.7994
CD	6.40	1	6.40	0.22	0.6434
A ²	176.87	1	176.87	6.17	0.0253
B ²	3680.06	1	3680.06	128.34	<0.0001
C ²	501.42	1	501.42	17.49	0.0008
D ²	128.19	1	128.19	4.47	0.0516
Residual	430.12	15	28.67		
Lack of Fit	429.46	10	42.95	325.18	< 0.0001 significant
Pure Error	0.66	5	0.13		
R ² = 0.9085	R ² _{adj} = 0.8231		Adeq. precision = 14.19		

This equation represented a second order polynomial regression model as shown in (Eq. 1).

$$\text{FAME Conversion (\%)} = +98.72 + (0.095 \times A) + (3.71 \times B) + (0.97 \times C) - (0.49 \times D) - (0.78 \times A \times B) - (0.67 \times A \times C) - (0.15 \times A \times D) - (0.48 \times B \times C) - (0.35 \times B \times D) + (0.63 \times C \times D) - (2.54 \times A^2) - (11.58 \times B^2) - (4.28 \times C^2) - (2.16 \times D^2)$$

(Eq. 1)

3.5. Parametric analysis

Figure 3 depicts the 3-D plots of the transesterification process parameters with respect to the response (i.e., FAME conversion). Excess methanol was used to shift the reaction towards equilibrium. Methyl esters conversion was increased by increasing methanol ratio up to 10, but further increases in this parameter led to no remarkable effect on the response and hindered the glycerol separation. Catalyst loading was observed as the most significant variable effecting the FAME conversion. Maximum conversion of 98.98 % was achieved using 2.0 wt% of the catalyst and further increases in catalyst loading decreased the methyl esters conversion. Reaction rate was increased by increasing the reaction temperature up to 55 °C. The maximum FAME conversion was achieved within minimum reaction time of 3.5 min under microwave irradiations. Hence, the microwave-assisted transesterification method investigated herein proved to be effective in terms of enhancing methyl esters conversion and decreasing reaction time.

3.6. Fatty acid methyl ester profile of Kapok oil methyl ester

The fatty acid profile of KOME is presented in Table 5 and compared with those of other studies conducted on KSO and some non-edible oil feedstock. As shown, the KSO contained higher amounts of unsaturated fatty acids than saturated ones. This attribute of non-edible oil feedstock would result in favourable cold flow properties, but on the other hand, lead to poor oxidation stability. On the contrary, palm oil methyl ester or soybean methyl ester do not possess suitable cold flow properties, but have a good oxidation stability (Ma and Hanna, 1999).

3.7. Fourier Transform Infra-Red analysis

The FT-IR analysis was also performed on the KOME. Figure 4 shows the FT-IR spectrum of KOME confirming the successful transesterification of the KSO. The band range from 2854 – 3008 cm⁻¹ represents the asymmetric stretching of the methyl group. The bands appearing in the range of the 1436 - 1741 cm⁻¹ are associated with aldehyde, ketone, and fatty acids. Symmetric stretching and vibration of the hydroxyl group could be observed in the range of the 1169 - 1246 cm⁻¹ (Zhang et al., 2012).

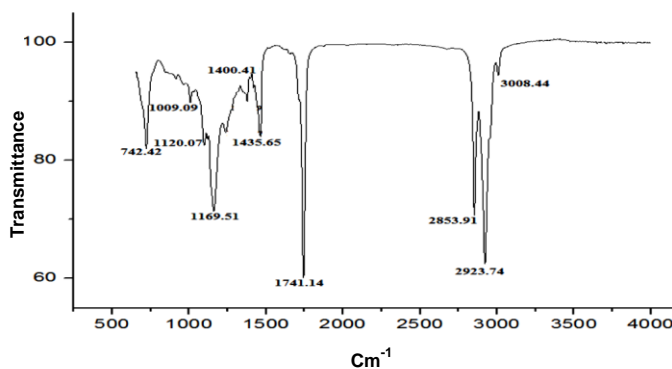


Fig.4. FT-IR spectrum of the Kapok oil methyl ester.

3.8. Optimized conditions and fuel properties

Optimum conditions for KOME production using microwave-assisted method conducted in the present study and mechanical stirring procedure (Sivakumar et al., 2013; Yunus Khan et al., 2015) are presented in Table 6. The optimum conditions were determined by numerical optimization tool in the Design Expert 8.0 software. Basically, numerical optimization selects the most appropriate and optimized values between the ranges of input variables and its associated output response designated arrays which were designed by CCD. Comparison of the optimized values of the current work, i.e., microwave assisted technology and those of Sivakumar et al. (2013) and Yunus Khan et al. (2015) who investigated the mechanical stirring method reveals that biodiesel production using the microwave assisted technology required less reaction time and temperature.

Table 5.
Kapak seed oil fatty acid profile

Fatty acid	Kapak oil methyl ester (This study)	Kapak oil methyl ester Sivakumar et al. (2013)	Kapak oil methyl ester Yunus Khan et al. (2015)	Jatropha oil methyl ester Lee et al. (2011)	Rubber seed oil methyl ester Ahmad et al. (2014)	Rapeseed methyl ester Kusdiana and Saka (2001)
Palmitic (C16:0)	25.75	23.20	20.80	11.00	9.89	3.49
Stearic (C18:0)	5.43	5.68	2.70	4.00	9.90	0.85
Oleic (C18:1)	24.32	29.69	20.10	22.00	24.89	64.40
Linoleic (C18:2)	42.46	35.11	38.10	53.00	35.00	22.30
Linolenic (C18:3)	-	-	1.70	8.00	16.78	8.23
Arachidic (C20:0)	2.04	1.89	0.50	-	-	-

Table 7 depicts the fuel properties of the produced KOME with microwave-assisted and mechanical stirring methods. All the fuel properties (except oxidation stability) were in agreement with the international standards, i.e., ASTM D 6751 and EN 14214. Compared to the studies in which mechanical stirring method was used, the present study led to improved fuel properties. For instance, cetane number, which plays a significant role in fuel ignition, was significantly higher in the present study than those of the previous investigations on KOME production using mechanical stirring (Sivakumar et al., 2013). Oxidation stability and cold flow properties of KOME showed significant improvements as compared to the previous studies as well (Yunus Khan et al., 2015). More specifically, the oxidation stability of the KOME herein study was measured at 3.69 h, whereas Yunus Khan et al. (2015) reported a much lower value of 1.14 h. The cloud, pour, and cold filter plugging points of the KOME produced in the present study were also improved compared to those of the mechanical-stirring based study performed by Yunus Khan et al. (2015); 2, 0, and 3 °C, compared to 3, 5, and 4 °C, respectively.

Table 7.
Fuel properties of Kapok oil methyl ester.

Properties	This study	Sivakumar et al. (2013)	Yunus Khan et al. (2015)	Methods	ASTM D 6751	EN 14214
	Microwave	Mechanical stirring	Mechanical stirring			
Density 25 °C (kg m ⁻³)	874	-	885 (at 15 °C)	ASTM D 5002	-	0.86-0.90
Cloud point (°C)	2	1	3	ASTM D 97	-	-
Pour point (°C)	0	-	5	ASTM D 2500	-	-
Cold filter plugging point (°C)	3	-	4	ASTM D 6371	-	-
Flash point (°C)	149	169	202.5	ASTM D 93	≥ 93	≥ 120
Kinematic viscosity 40 °C (mm ² s ⁻¹)	1.90	4.17	4.42	ASTM D 445	1.9 – 6.0	3.5 – 5.0
Oxidative stability (h)	3.69	-	1.14	EN 14112	-	≥ 6
Moisture content (wt%)	0.03	0.03	-	ASTM D 2709	< 0.05	< 0.03
Acid value (mg KOH g ⁻¹)	0.3	0.04	0.16	Cd 3d-63	< 0.8	< 0.5
Cetane number	57.08	47	-	ASTM D 613	≥ 47	≥ 51
Higher heating value (MJ kg ⁻¹)	39.7	-	39.4	ASTM D 4868	-	-
Free glycerin (wt%)	0.016	-	-	ASTM D 6584	≤ 0.020	≤ 0.020
Total glycerin (wt%)	0.24	-	-	ASTM D 6584	≤ 0.240	≤ 0.240
Ester Content (wt%)	98.9	99.5	-	EN 14103	-	≥ 96.5

4. Conclusions

High free fatty acid content of the KSO was significantly reduced by acid esterification. RSM was used to optimize the process variables for base transesterification. The optimum operating conditions corresponding to 98.90 % KOME conversion were 57.09 °C, 2.15 wt% KOH catalyst loading, 1:9.85 molar ratio of oil to methanol, and 3.29 min of reaction time. A significant reduction (~ 14 - 37 folds) in the optimum reaction time for transesterification was achieved; i.e., from 45 to 120 min for the mechanical stirring approach to 3.29 min for the microwave-assisted approach. The conversion of the fatty acid into methyl ester was verified by The FT-IR analysis and the fuel properties of KOME met the ASTM D 6751 and EN14214 standards.

Table 6.
Numerical optimization for biodiesel production from Kapok seed oil.

Optimum conditions	This study	Sivakumar et al. (2013)	Yunus Khan et al. (2015)
	Microwave	Mechanical stirring	Mechanical stirring
Oil to methanol ratio	1:9.85	1:6	1:4
Catalyst loading (wt%)	2.15	1	1
Temperature (°C)	57.09	65	60
RPM	-	600	700
Time (min)	3.29	45	120
Predicted conversion (%)	99.07	-	-
Actual conversion (%)	98.90	99.5	-

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