Synthesis of Biodiesel from Ceiba Pentandra using Microwave Assisted Technique

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ABSTRACT

The rapid growth of industrial and human population had encouraged the exploration of renewable fuel such as biodiesel which is produced from plant oil and animal fat. Ceiba Pentandra which is one of the non-edible feedstocks is much explored in Malaysia for biodiesel production. Biodiesel is usually produced by transesterification process using conventional heating which requires large amount of heat energy and long reaction time. This project studies on Kapok oil methyl ester (KOME) production using microwave assisted technique. Studies have been conducted to investigate the optimum operating conditions for the microwave assisted transesterification of Ceiba Pentandra seed oil including the temperature, catalyst concentration, methanol to oil molar ratio and irradiation time. The optimum operating conditions which result in highest KOME yield (95.6%) are found to be at 55°C, 2 wt% KOH catalyst, 10:1 methanol to oil molar ratio and 3.5 minutes of irradiation time. The microwave synthesis reactor which is able to deliver energy directly to the reactant at molecular level has drastically reduced the reaction time and total energy consumption in the transesterification process. The properties of the biodiesel produced under the optimum conditions are characterized and it shows that the KOME meets the international standard of biodiesel (ASTM 6751 & EN 14214).

1. INTRODUCTION

Direct utilization of vegetable oil is not recommended due to viscosity problem [1]. It is reported that Kapok fiber contains 34-64% of cellulose and has high potential to produce cellulosic ethanol [2]. Generally, biodiesel is sysnthesized through transesterification of oil using basic catalyst with heating function. Many studies had been done on producing biodiesel from Kapok seed oil (KSO) using the conventional heating method. Sivakumar et al. [3] reported the biodiesel production from Ceiba Pentandra seed oil. It was able to achieve 99.5% conversion under optimized conditions using conventional heating method. The main problems are large amount of heat energy and long reaction time are needed to achieve high conversion of oil. It will impact the production cost thus the commercialization potential. This project applies a technique to deliver energy directly to the reactant at shorter reaction time which is microwave assisted technique. A microwave synthesis reactor is used to study the optimum conditions for transesterification of KSO by varying different parameters (temperature, catalyst concentration, solvent to oil molar ratio and reaction time). The energy consumption of microwave assisted technique is compared to that of conventional heating method. Besides, the Kapok oil methyl ester (KOME) produced under optimum condition is characterized following the international biodiesel standard (ASTM 6751 and EN 14214).

2. EXPERIMENTAL

A. Acidity test of crude KSO

The acid value of KSO is determined using American Oil Chemists' Society (AOCS) method and it is shown that the acid value of the crude KSO used in this study is 11.19 mg KOH g^{-1} oil which corresponds to 5.59% of free fatty acid (FFA) content in the oil. Higher FFA content (>1%) can lead to soap formation during transesterification reaction [4]. Therefore, the FFA content of KSO is reduced to below 1% using acid catalyzed esterification as the pretreatment of the feedstock.

B. Esterification

The acid esterification reaction of KSO is carried out at 65ºC, 6:1 methanol to oil molar ratio, 1.5 wt% catalyst and 5 minutes reaction time using microwave synthesis reactor. The acid value of KSO is again determined upon completion of esterification to ensure the FFA value is lower than 1% before proceeding to transesterification reaction.

C. Transesterification

Transesterification is performed according to the experiments designed using the response surface methodology (RSM). A total of 30 runs with different conditions of parameters are performed. In each run, 100 g of KSO mixing with specific amount of methanol-KOH solution in a round bottom flask is heated and stirred in microwave for a designed time period. Upon completion, the mixture is left overnight in a separating funnel for complete separation. Two layers of immiscible phase are obtained. The upper layer is the KOME whereas the lower layer byproduct consists of glycerol, excess methanol and unreacted catalyst. The KOME is purified with warm deionized water to remove residual catalyst. Rotary evaporator is then used to remove the residual water in KOME.

D. Fatty acid methyl ester (FAME) analysis

The KOME produced are analyzed using Gas Chromatography (GC) to determine the FAME conversion in each experiment run. The GC used is model 7890A from Agilent-Technologies. The GC system is equipped with a variable split flow injector, a temperature programmable oven, a flame ionization detector and capillary column coated with polyethylene glycol (30 x 0.25 mm; film thickness 0.25µm). Helium is used as the carrier gas at 1.2 ml min^{-1}. Column temperature is adjusted from 60°C hold for 2 minutes, programmed at 10°C min^{-1} up to 200°C, programmed at 5°C min^{-1} up to 240°C, and final temperature is hold for 7 minutes. The injector temperature and detector temperature are set at 250°C. Volume of sample injected is 1 µL.

3. RESULTS AND DISCUSSION

A. *Acid value of KSO after esterification*

The acid value of KSO is tested after the acid esterification process. The results are shown in Table 1.

Table 1. FFA value of KSO after esterification

No. of Titration	Acid Value (mg KOH/g oil)	Free Fatty Acid (FFA) (%)
1	1.15	0.57
2	1.17	0.58
	Average FFA	0.575

B. *KOME yield from transesterification*

A research done by Norazahar et al. on KOME production using conventional heating, the highest yield of KOME (98%) was obtained at 55°C, 2 wt% of KOH, 8:1 methanol-to-oil molar ratio, and 3 hours reaction time [4]. The result of current project is comparable with the project done on KOME production using conventional heating. The significant achievement is the drastic reduction of reaction time from 3 hours to 3.5 minutes although the yield obtained using microwave system (95.6%) is slightly less than 98% yield obtained using conventional heating. Azcan et al. [6] reported that a FAME conversion of 98.87% can be achieved at 60°C, 1 wt% of sodium methoxide, 6:1 methanol-to-oil molar ratio, and 5 minutes reaction time.

C. *FAME profile of KOME*

The fatty acid profile of KOME is presented in Table 2. It shows that KSO contains high unsaturated fatty acid which is linoleic acid and this makes it an excellent oil for engine performance during cold weather after conversion to biodiesel. Hilditch et. al. [5] reported that KSO consists mainly of oleic acid and linoleic acid forming together about 70 wt% of the total fatty acids.

Table 2. Fatty acid methyl ester profile of KOME

KOME	Percentage (wt%)	
	Current work	Norazahar et al., 2012
Caproic acid (C6)	-	9.42
Palmitic acid (C16)	20.44	23.17
Stearic acid (C18)	12.47	4.73
Oleic acid (C18:1)	19.32	22.88
Linoleic acid (C18:2)	39.14	30.00
Linolenic acid (C18:3)	1.61	-
Arachidic acid (C20)	-	1.18

D. *Energy consumption of microwave synthesis reactor*

The irradiation time for production of KOME using microwave synthesis reactor takes only 3.5 minutes whereas conventional heating requires 3 hours to achieve 98% of KOME yield [4].Total energy consumption for microwave heating is 0.075 KWh whereas total energy consumption for conventional heating is 3.021 KWh. This shows that conventional heating consumes almost 40 times more energy compared to microwave system.

E. *Fuel properties*

Characterizations are done on the KOME and the results are shown in Table 3.

Table 3. Properties of KOME in comparison to ASTM 6751 and EN 14214 standards

Properties	KOME	ASTM 6751	EN 14214
Density@25ºC (kgm^{-3})	874	-	-
Cloud point (ºC)	2	5	[a]
Pour point (ºC)	0	-15	[b]
Cold filter plugging point (ºC)	3	-	[b]
Flash point (ºC)	149	93 min	120 min
Kinematic viscosity @ 40ºC (mm^2s^{-1})	1.9	1.9 – 6.0	3.5 – 5.0
Oxidative stability (hr)	3.69	3 min	6 min
Moisture content (%)	0.03	< 0.05	< 0.03
Acid value ($mgKOH\ g^{-1}$)	0.3	< 0.5	< 0.5
Cetane number	57.08	47 min	51 min

[a] Not specified

[b] Not specified. EN 14214 use time and location dependant values for cold filter plugging point instead

4. CONCLUSION

Esterification followed by transesterification has successfully reduced the FFA value of the KOME to 0.15 %. The optimum operating conditions for transesterification process to synthesize biodiesel are at 55°C, 2 wt% KOH catalyst, 10:1 methanol to oil molar ratio and 3.5 minutes of irradiation time. The highest KOME yield at this condition is 95.6 %. Reducing the reaction time can eventually reduce the energy consumption. Conventional heating consumes almost 40 times of energy compared to microwave system to produce comparable yield of KOME. Ceiba Pentandra biodiesel is a potential sources of biodiesel, since its properties are comparable and satisfy the standard for biodiesel (ASTM 6751 and EN 14214).

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REFERENCES

[1] Ramadhas, A., Jayaraj, S., & Muraleedharan, C. (2003). Chemical composition and nutritive value of kapok seed meal for broiler chickens. *British Poultry Sci* , 4:505-509..

[2] Tye, Y. Y., Lee, K. T., N., W. A., & Leh, C. P. (2012). Potential of Ceiba pentandra (L.) Gaertn (kapok fiber) as a resource for second generation bioethanol: effect of varios simple pretreatment methods on sugar production. *Bioresour Technol* , 116:536-539.

[3] Sivakumar, P., Sindhanaiselvan, S., Ghandi, N. N., Devi, S. S., & Renganathan, S. (2013). Optimization and kinetic studies on biodiesel production from underutilized Ceiba Pentandra Oil. *Fuel* , 103:693-698.

[4] Norazahar, N., Yusup, S., Ahmad, M. M., Bakar, S. A., & Ahmad, J. (2012). Parametric Optimization of Kapok (Ceiba Pentandra) Oil Methyl Ester Production using Taguchi Approach. *International Journal of Energy and Environment* , 6:541-548.

[5] Hilditch, T., & Williams, P. *The Chemical Constitution of Natural Fats.* London: Champman and Hall.

[6] Azcan, N. & Yilmaz, O. (2013). Microwave assisted transesterification of waste frying oil and concentrate methyl ester content of biodiesel by molecular distillation. *Fuel*, 104:614-619.