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Method Article

Techniques for analysing and monitoring during continuous bio-hydrogenation of kerosene from palm oils



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A B S T R A C T

In this research work, analytical, experimental methods and monitoring techniques of bio-hydrogenated kerosene (BHK) production in continuous mode were presented. Two kinds of raw materials obtained from palm processing plant named as refined bleached deodorised palm oil (RPO) and palm kernel oil (PKO) were converted into BHK via hydrocracking reaction catalysed by Pd/Al₂O₃ catalyst in pilot scale. Firstly, both of RPO and PKO were pretreated by thermal technique. Subsequently, fatty acid compositions of palm oils were analysed by Gas Chromatography (GC). Then, hydrocracking reaction of RPO and PKO were separately conducted in continuous high pressure packed bed reactor (HPPBR). After reaction, crude-biofuel was refined into BHK via fractional distillation. In addition, some properties of BHK obtained from RPO/PKO such as were C, H, O elements, freezing point, flash points, total acid number and carbon distribution were analysed following the ASTM and UOP 915 standards.

- Thermal pretreatment of refined bleached deodorised palm oil (RPO) and palm kernel oil (PKO).
- Continuous hydrocracking reaction of palm oil was conducted in pilot scale.
- Characterisation of bio-hydrogenated kerosene obtained from palm oil.

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Method details

Currently, production of biofuel from bioresource plays important role on reducing dependence fossil resource due to continual petroleum depletion and global warming and climate change in worldwide [1]. The most preferable process for biofuel production from vegetable oils, animal fats and waste oils is hydroprocessing process. In this process, triglycerides (TG) /fatty acids (FAs)/fatty acids methyl ester (FAME) can be converted to shorter chain hydrocarbon fuel under high temperature and pressure in the presence of chemical catalysts [2]. There are various vegetable oils such as palm oil, soybean oil, rapeseed oil, sunflower oil, castor oil, coconut oil and jatropha oil have been typically used as raw materials for biofuel production. According to palm oil, one of important oil crops and also the lowest price oil in South East Asia and Thailand also has very high potential for palm oil production (5000 tons crude palm oil /day) [3]. Accordingly, there are many previous works relates to production of bio-hydrogenated kerosene (BHK), bio-hydrogenated diesel (BHD) or green diesel and bio-hydrogenated gasoline (BHG) from palm oils. Hydrocracking of palm oil to bio- jet fuel over Ni/zeolite SAPO-34 catalyst was reported by Li et al. 2016 [4]. The highest bio-jet fuel (42.03%) was obtained at 390 °C under 3 MPa hydrogen for 8 h. Khel et al. 2019 [5] studied production of bio-jet fuel from palm oil via hydrocracking over Ru-H-Beta-300 catalyst was also reported Bio-jet fuel yield (24%) was obtained at 240 °C under 45 bar hydrogen. In addition, hydrodeoxygenation of palm oil for green diesel production was reported by previous studies [6–12]. However, hydroprocessing of palm oil for hydrocarbon fuel in the ranges of kerosene and diesel were operated in batch mode. Previously, a continual hydrocracking of palm oils for bio-hydrogenated kerosene (BHK)/bio-jet fuel has not been reported.

Recently, bio-hydrogenated kerosene (BHK) production from refined palm oil (RPO) and palm kernel oil (PKO) via hydrocracking reaction in continuous mode was reported firstly by Dujjanutat et al. 2020 [13]. The optimal condition for BHK production for both palm oils was 477 °C reaction temperature, 5.6 MPa hydrogen and 1.5 h⁻¹ of liquid hourly space velocity (LHSV). The highest BHK yields obtained from RPO and PKO were 49.59% and 55.35%, respectively. The BHK products were further characterised based on their properties including carbon distribution, C H O elements, freezing point, flash points and total acid number.

Accordingly, the method details of new catalytic mode to produce kerosene from palm oils can play important role for research development in the field of renewable energy production from refined/unrefined plant oils. Therefore, this article aims to explain experimental analytical as well as monitoring techniques which related to a continual kerosene production from upstream to downstream process such as pretreatment of feedstock, determination of fatty acid composition in palm oils, separation process and characterisation of kerosene to evaluate its performance.

Materials

1. Refined bleached and deodorised palm oil (RPO)
2. Crude palm kernel oil (PKO)
3. Hydrogen gas (95% purity)
4. Nitrogen gas (99.999% purity)
5. Helium gas (99.999% purity)
6. Oxygen gas (99.999% purity)
7. 0.5% Pd/Al₂O₃ catalyst
8. Potassium hydroxide
9. Toluene
10. Ethanol
11. Heptane
12. Cotton sheet
13. Microfilter paper
14. Distilled water
15. High pressure packed bed reactor (HPPBR)
16. Distillation apparatus (Koehler model K45200)
17. Flash 2000 organic elemental analyser
18. Freezing point apparatus (K29790 / K29795)
19. Tag closed cup tester using Pensky-Martens apparatus
20. Gas chromatography (GC) equipped with a flame ionization detector (FID)

Preparation of feedstocks/pretreatment

Palm oil and crude palm oil were pretreated by thermo-treatment at 80°C until it became to homogeneous phase. Subsequently, homogeneous oils were filtrated through the cotton sheet in order to eliminate undesirable contaminants.

Determination of fatty composition in feedstocks

The fatty acid compositions in both refined palm oil (RPO) and palm kernel oil (PKO) were characterised according to the modified method from Lee et al., 1997 [14] (See in Fig. 1). Oil sample (50 mg) and 0.5 M methanolic sodium hydroxide solution (5 mL) were added into reflux flask connecting with condenser. The reaction mixture in reflux flask was heated to 140-160°C for 5 min for saponification. Then, boron trifluoride-methanol solution (5 mL) was added and heated to 140-160°C for 5 min for methylation. The mixture solution was cooled down to room temperature; hexane (5 mL) and saturated sodium chloride solution (10 mL) were added to obtain fatty acid methyl ester (FAME) in hexanic phase (upper layer). The bi-layer of mixture reaction was observed. The upper layer was dried by adding anhydrous sodium sulfate (1 g) and filtrated through Whatman paper No. 1. Finally, samples were dissolved in heptane and filtrated through micro filter (0.45µm) before injection to GC-MS equipped with FID detector to identify fatty acid composition as described in previous studies [15,16].

High pressure packed bed reactor used for hydrocracking reaction

The high pressure packed bed reactor (HPPBR) was comprised of three systems; feed system, hydrocracking (reactor) system and product separation system (See Fig. 2). Previously, the schematic of reactor has been described by Dujjanutat et al. [13,17,18]. In the first part, palm oil feedstock was continuously fed by hydraulic pump. Level of pump's scale was determined by volumetric flow rate method. Briefly, the scale was varied at 4 stroke levels; the volume of liquid feedstock was measured and compared with the speed of time. The result was provided the linear equation that can be used to determine the desired flow rate. Secondly, hydrocracking reaction was performed in reactor system.

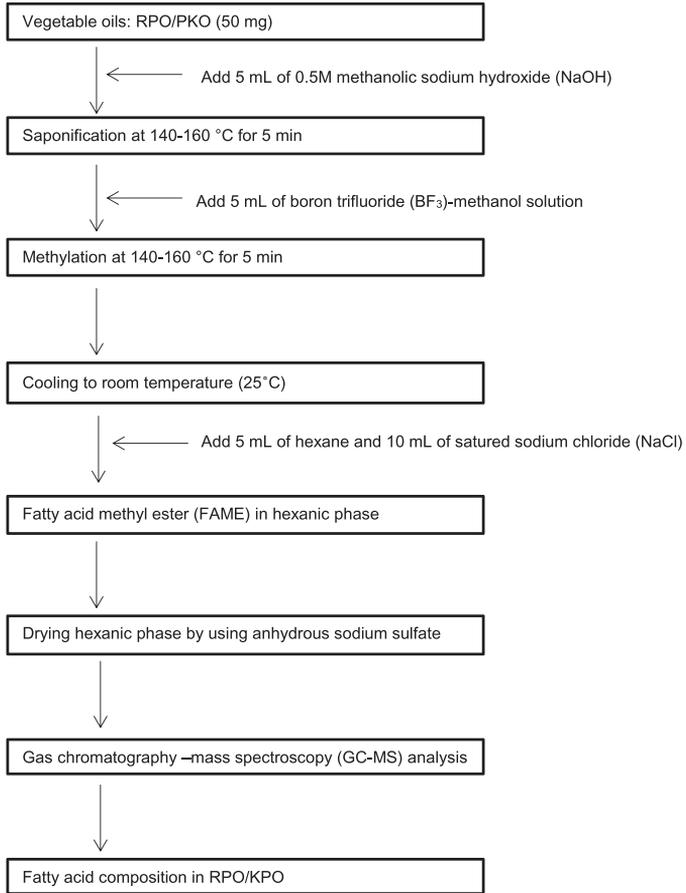


Fig. 1. Flow chart of experimental procedure for determination of fatty composition in feedstocks.

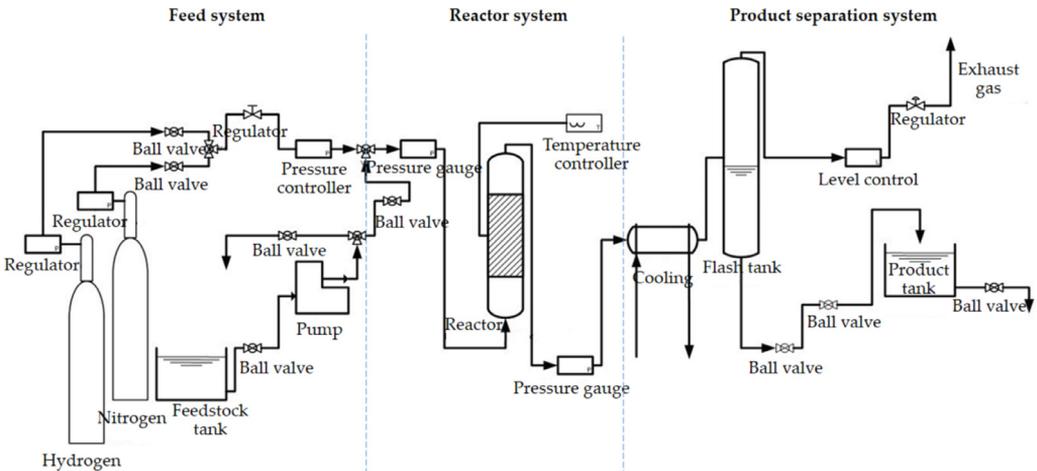


Fig. 2. Diagram of high pressure packed bed reactor (HPPBR).

Feedstock and hydrogen gas was mixed before entering into the stainless steel HPPBR reactor by up-flow current and the reaction was occurred under severe condition. The operating temperature was controlled by thermocouples installed at the top and bottom of the reaction zone. High temperature and pressure were strictly controlled under a severe condition. The mixture of feedstock and H₂ was fed into the reactor at the bottom and the product was transported from the top of the reactor (up-flow current). The product was continuously condensed and separated the liquid/gas products at the heat exchanger which belong to the product separation system. The liquid product could be accumulated in the product tank. Finally, the product was flown to the third part system which the liquid and gas were separated and liquid product was store in the product tank.

Hydrocracking reaction

Hydrocracking reaction was operated in a high-pressure packed bed reactor (HPPBR). There are three main parts (i) feeder (ii) reactor (iii) product separator. The catalyst (100 mL) was packed inside the stainless steel tube which was located in the reactor. Some operating parameters; temperature, hydrogen pressure and feedstock flow rate were controlled by temperature controller, pressure controller and feed pump, respectively. Firstly, refined palm oil (RPO)/ crude palm kernel oil (PKO) and hydrogen gas (H₂) were fed into the reactor system in order to conduct hydrocracking reaction. The product (gas phase) continually flew through the cooling system and condensed into liquid phase in flash tank. The incondensable gas was released from the system by separation unit. Finally, the liquid product was collected from product tank and refined to bio-hydrogenated kerosene (BHK) via fractional distillation.

Fractional distillation

A commercially manual distillation apparatus (Koehler model K45200, USA) was used to perform fractional distillation in this work. The apparatus components met standard of ASTM D86 specifications including a 125-mL sidearm distillation flask (borosilicate glass), a 100-mL graduated cylinder with 1.0 mL graduation intervals and mercury thermometer. The graduate cylinder was centered under the condenser tube exit to allow drop of condensate to fall to the bottom of the cylinder. Three kinds of refined- biofuels were separated by different boiling points, bio-hydrogenated gasoline (50–150 °C), bio-hydrogenated kerosene (150–280 °C) and bio-hydrogenated diesel (280–360 °C). Biofuel yield (%), Refined-biofuel yield (%) and BHK yield (%) could be calculated as shown in Eqs. (1), (2) and (3), respectively.

$$\text{Biofuel yield (\%)} = \frac{\text{Total crude} - \text{biofuel (mL)}}{\text{Feedstock feeding (mL)}} \times 100 \quad (1)$$

$$\text{Refined} - \text{biofuel yield (\%)} = \frac{\text{Total biofuels obtained from distillation (mL)}}{\text{Total crude} - \text{biofuel used in distillation (mL)}} \times 100 \quad (2)$$

$$\text{Bio} - \text{hydrogenated kerosene (BHK) yield (\%)} = \frac{\text{BHK obtained from distillation (mL)}}{\text{Total crude} - \text{biofuel used in distillation (mL)}} \times 100 \quad (3)$$

Characterisation of BHK

The properties of BHK were analysed according to the global standard of American Society for Testing and Materials (ASTM) for approval of alternative aviation fuel as shown in Table 2. In addition, the distribution of carbon of BHK was determined by GC-FID followed UOP 915 standard method.

Determination of carbon hydrogen and oxygen

The concentrations of carbon hydrogen in BHK were determined by combustion method according to ASTM D5291, while the oxygen content was obtained by calculation. The BHK sample (2 µL) was

Table 2

The official methods for characterization of BHK obtained from RPO/PKO.

Parameters	Methods
C H O elements	ASTM D5291 [19]
Freezing point	ASTM D2386 [20]
Flash point	ASTM D56 [21]
Total acid number	ASTM D3242 [22]
Carbon distribution	UOP 915 [23]

injected through the inlet-port septum into a flash 2000 organic elemental analyser (Thermo Fisher Scientific, Italy). The operating condition was set as described in previous study [24] [Determination of the carbon and hydrogen contents of gasoline using a combustion method and the estimation of their measurement uncertainty], combustion temperature (900 °C), separation column (70 °C), and detector oven temperature (70 °C). Helium was used as carrier gas and set flow rate at 100 mL/min for reference and 130 mL/min for detector. The flow rate of oxidation gas (O₂) was 240 mL/min. The concentration of carbon and hydrogen can be calculated as shown in Eq. 4. In addition, the calculation can also be automatically provided by the instrumental system.

$$A = \frac{B \times E \times F}{C \times D} \quad (4)$$

Where

A = the percent of the carbon, hydrogen or nitrogen in the sample, mass %

B = the detector response for carbon, hydrogen or nitrogen in the sample minus response from blank

C = the detector response for carbon, hydrogen or nitrogen in the standard minus response from blank

D = the mass of the sample, mg

E = the mass of the standard, mg

F = the percent of the carbon, hydrogen or nitrogen in the standard, mass %.

Measurement of freezing point

The freezing point of BHK was measured by using freezing point apparatus (K29790 / K29795) which conforms to ASTM D2386 and related specifications. The sample is cooled with continuous stirring in a Dewar-type sample tube until crystals appear. Then, the temperature was recorded.

Measurement of flash point

The flashpoint of BHK was measured by tag closed cup tester using Pensky-Martens apparatus according to ASTM D56. BHK sample (2 mL) was loaded into sample cup and close lid. Then, it was heated with the enclosed air until liquid can gives off vapor to form an ignitable mixture in air near the surface of the liquid. Subsequently, the temperature was recorded.

Determination of total acid number

The total acid number of BHK was determined by titration method according to ASTM D3242. Firstly, the titration solvent was prepared by mixing of toluene (500 mL) and 5 mL of water to 495 mL of anhydrous isopropyl alcohol. The mixture solution of toluene/water/anhydrous isopropyl alcohol is neutral solvent which leads to high accurate determination of acidity in samples. Pure solid phenolphthalein (0.1 ± 0.01 g) was dissolved in 50 mL of water and 50 mL of ethanol to obtain phenolphthalein indicator solution. Subsequently, BHK sample (100 ± 5g) was loaded into 500 mL-Erlenmeyer flask followed by 100 mL of titration solvent and 0.1 mL of the indicator solution. The mixture solution was titrated with 0.1 N KOH until appearance of permanent pink. A blank titration

was a mixture solution between 100 mL of the titration solvent and 0.1 mL of the indicator solution. The total acid number can be calculated as shown in Eq. 5.

$$\text{Acid number, mg of KOH/g} = \frac{(A - B)N \times 56.1}{W} \quad (5)$$

Where

A = KOH solution required for titration of the sample (mL)

B = KOH solution required for titration of the blank (mL)

N = Normality of KOH solution

W = Sample used (g).

Carbon distribution analysis

The bio-hydrogenated kerosene from RPO/PKO was dissolved in heptane with 20 times dilution and filtrated through micro filter. The samples (1 μ L) were injected into the gas chromatography (GC) equipped with a flame ionization detector (FID) in order to analyse carbon distribution according to UOP 915 standard method. The operating condition was described as previous study [25]. The liquid products obtained from the continual hydrocracking reaction were analyzed offline by GC-FID (Agilent 7890A Series) using a HP-5 capillary column (30 m \times 0.32 mm, 0.25 μ m). The n-alkanes and n-alkenes (C8-C20) was used as chemical standard for calibration curves to identify the composition of the liquid product. The internal standard was 1-bromoheptane.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi: [10.1016/j.mex.2020.101128](https://doi.org/10.1016/j.mex.2020.101128).

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