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Potential of Vegetable Oils for Producing Green Diesel Via Hydrocracking Process

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Abstract

The effects of fatty acid components on the yield of alternative diesel produced by hydrocracking of vegetable oils and its properties were studied in this work. Green diesel synthesis via hydrocracking reaction of 4 different raw materials including palm oil (PO), sunflower oil (SFO), soybean oil (SO), and rice bran oil (RBO) was investigated. Green diesel production was carried out at a constant pressure of 5 MPa and a constant temperature of 450°C in a continuous high-pressure reactor supplied with hydrogen. Feedstocks of vegetable oils were fed to the reactor with a liquid hourly space velocity (LHSV) of 1 h^{-1} over Pd/TiO_2-Al_2O_3 catalyst. The results revealed that RBO gave the highest liquid product yield of 94%, while PO gave the highest diesel selectivity of 82% due to the high content of saturated (C16:0) and monounsaturated (C18:1) fatty acids. Moreover, the green diesel obtained from each feedstock was analyzed for its properties such as kinematic viscosity, net heat of combustion, and density. It was also found that the physical and thermal properties of the synthetic green diesel obtained from all the feedstocks were comparable with those of the standard diesel, except the net heat of combustion which was higher in the synthetic green diesel. Furthermore, the green diesel produced from this process was less contaminated by by-products as compared with the biodiesel obtained by transesterification reaction.

Keywords : Hydrocracking; Green-diesel; Green-fuel; Biofuel

Introduction

Since the world's energy demand is ever-increasing while the petroleum energy resources are on the decrease; energy shortage, as well as the consideration of environmental issues, have been a great concern for energy solutions. Therefore, the interest in alternative renewable fuel production, such as ethanol, biodiesel, and green diesel, has been dramatically increased. Bio-ethanol can be produced by fermentation of substrate-based carbohydrates under the metabolic pathways of suitable bacterial strains [1]. Generally, biodiesel can be obtained by transesterification reaction between alcohol and substrate-based triglycerides by using a catalyst to produce ester and glycerol [2]. Free glycerol, metals, soap, free fatty acids (FFA), methanol, catalyst, and water are impurities that are contaminated in crude biodiesel. These impurities can reduce the life-time of the engine [3]. Therefore, a purification process for removing the impurities from biodiesel is necessary. The traditional method for purifying biodiesel is wet washing by adding hot water to remove soap formation and the remaining catalyst due to their water solubility [4]. However, this treatment has some disadvantages, such as a rise in production cost and time, wastewater discharges, and hard in removing biodiesel from water [5]. Biodiesel washing is recommended to repeat several times, and this can produce a large amount of wastewater approximately 0.2-3 L of the wastewater per liter of the obtained biodiesel [2, 6]. Thus, the production of diesel by an alternative process such as the hydroprocessing process, which yields less amount of wastewater, is of interest. The diesel produced from this process is named as green diesel.

The hydroprocessing process has typically been used in the petroleum refinery plant for a long time for upgrading and improving product quality. Hydroprocessing is divided into two main chemical reactions, hydrotreating, and hydrocracking. Normally, hydrotreating is mostly used for eliminating heteroatom from crude oil in the petroleum industry. Meanwhile, hydrocracking is used to convert a larger molecule of hydrocarbon structure into hydrocarbon compounds of lower molecular weight [7]. Hydrocracking which is usually performed after hydrotreatment is normally operated in severe conditions, i.e. high pressure and temperature, with the attendance of hydrogen to prevent aromatization reaction, which causes the deactivation of the catalyst. The obtained product comprises several types of biofuel like crude petroleum. The fractional distillation method is then used to refine the product to obtain biofuels, such as green diesel, green kerosene, and green gasoline. A few amounts of water are produced, thereby reducing the wastewater problem. Green diesel obtained from hydroprocessing has been gaining attention over the past decade due to a higher cetane number, lower acid number, lower NO_x emissions, and better cold flow properties when compared with biodiesel and also has a lower sulfur and aromatics content when compared with petroleum diesel [8].

Vegetable oil, an attractive renewable resource, has the potential to use as a raw material for producing biofuel with properties comparable with fossil-based fuel [9]. The variety of fatty acids in vegetable oil usually contains carbon atoms ranging from 2-28, and this is related to the carbon composition of the biofuel [9]. In addition, most of the primary fatty acids of several natural oils are in the range of diesel characteristics. For example, palm oil (PO) contains C16 saturated and C18 mono-unsaturated fatty acids as significant components. Also, soybean oil (SO)), rice bran oil (RBO and sunflower oil (SFO) contain mono-unsaturated and di-unsaturated C18 fatty acids as the main components with different ratios [10-12]. Therefore, different types of fatty acids may influence the formation of products from hydrocracking processes.

Triglyceride (TG) is the main component that can be found in vegetable oils. The variation of hydrocarbon of TG is related to several types of biofuel. Renewable diesel contains hydrocarbon ranging between C_9 and C_{18} in its structure [13]. The catalytic hydrocracking can transform the structure of TG into hydrocarbons by a variety of different pathways; decarboxylation, decarbonylation, hydrodeoxygenation, hydrogenation, and random cracking. These reactions can produce hydrocarbon products that have a carbon atom number less than the original TG. Also, CO_2 and CO are produced as by-products of decarboxylation and decarbonylation reaction, while H_2O is formed from oxygen removal by hydrodeoxygenation [14, 15].

In this work, the potential of four vegetable oils (rice bran oil, sunflower oil, palm oil, and soybean oil) for producing green diesel via hydrocracking process were studied. The reactions were performed at 450°C, 5 MPa, and liquid hourly space velocity (LHSV) of 1.0 h^{-1} over a commercial catalyst of 0.5%Pd/Al_2O_3.

Materials and Methods

1. Feedstocks and chemicals

Refined palm oil, soybean oil, sunflower oil, and rice bran oil were used as feedstocks throughout this work, and their fatty acid compositions were presented in Figure 1 [10, 11]. In addition, 95% of hydrogen gas was used as the reactive gas, while nitrogen gas with 99% purity was applied for checking gas leaks and cooling down the system.

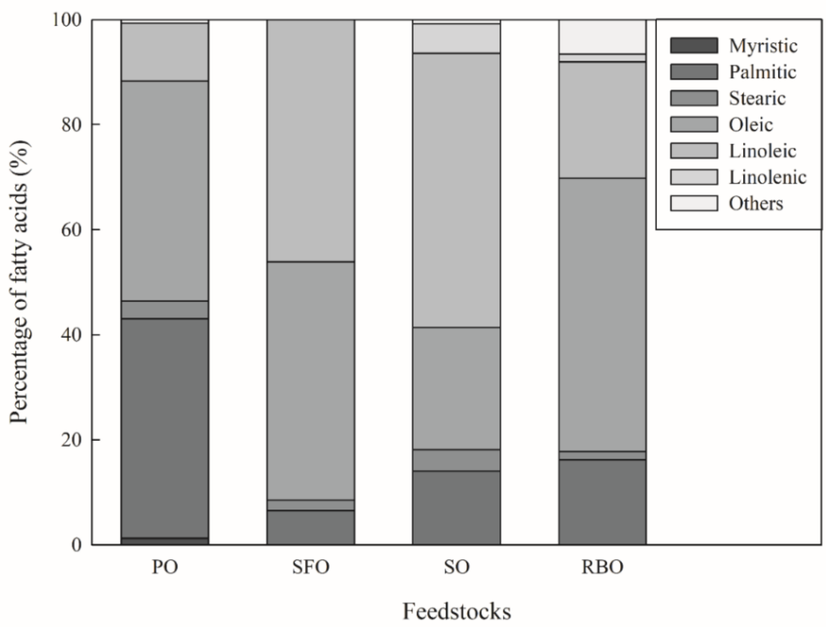

Figure 1 Fatty acid components of PO, RBO, SO, and SFO [10, 11]

2. Catalyst selection and characterization

Palladium (Pd) is a well-known catalyst that offers high selectivity for hydrogenation reaction. Therefore, a 0.5% Pd/Al_2O_3 commercial-grade catalyst (Haze Development Zone Dayuan Chemical Co., LTD, China) was selected to use for hydrocracking reaction.

The selected catalyst was imaged by scanning electron microscopy (SEM) using LEO 1450VP (ZEIZZ International, Germany). The specific surface area was estimated following the Brunauer-Emmett-Teller (BET) method. Nitrogen adsorption-desorption isotherm was performed at 77 K in an ASAP 2020 instrument (Micromeritics, USA). Before the analysis, the catalyst was evacuated at 120℃ for 3 hrs. In addition, the X-ray diffraction (XRD) method was used to analyze the crystalline structure of the catalyst by using Empyrean XRD diffractometer (Malvern PANalytical, United Kingdom) under Cu Kα radiation at λ = 1.542 Å, operating at 40 kV, 40 mA, and increasing a step size of 0.0167° with a step time of 100 s in the range 10° to 90°.

3. Biofuel production

Hydrocracking reaction was performed by using a high-pressure packed bed reactor (HPPBR) for producing biofuels. This reactor contained three main systems; feed system, reaction system, and product separation system.

The simple diagram of HPPBR was illustrated in Figure 2, which was concisely redrawn from the previous work [16]. A 100 ml of 0.5%Pd/Al_2O_3 catalyst was packed inside a tubular reactor which had an inner diameter of 0.96 cm and 43 cm long. For starting up the reaction, the catalyst was activated under a flow of H_2 at 5 MPa and 400°C for 4 hours. Then, the reaction was performed by heating the reactor to 450°C under the same pressure. A feedstock was fed into the reactor by a high-pressure liquid pump which controlled the feedstock flow rate at 1.0 h^{-1} of LHSV. H_2 and feedstock were mixed before entering the tubular reactor where the hydrocracking reaction took place. After that, the hydrocracking reaction product continuously flowed to the heat exchanger for separating

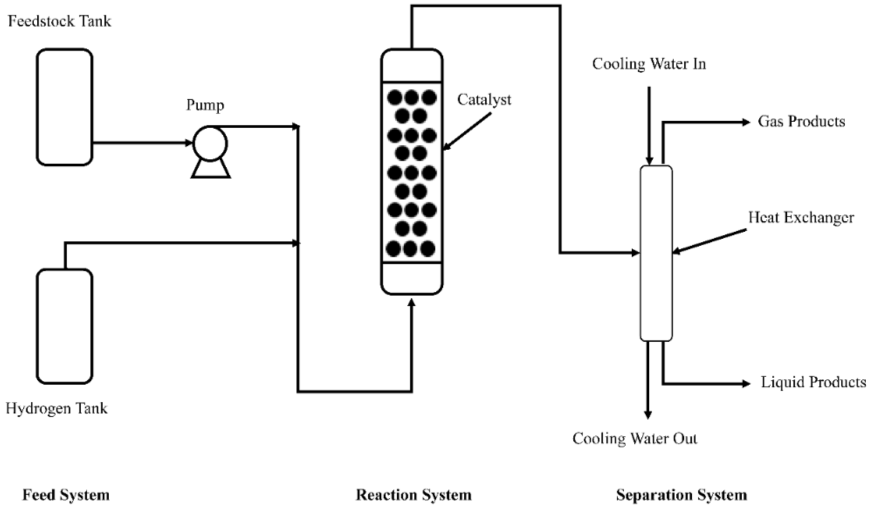

Figure 2 Simple diagram of high-pressure packed bed reactor

liquid and gaseous products. The biofuel product was collected from the product storage tank and further distilled by a distillation unit to obtain green gasoline and green diesel.

Furthermore, green diesel from each feedstock was characterized by its properties and compared with the diesel fuel specifications. The density was analyzed at 15°C, kinematic viscosity at 40°C, and net heat of combustion was also analyzed by a bomb calorimeter.

Results and Discussion

1. Catalyst characterization

The images of the selected catalyst were presented in Figure 3. The whole pellet of the catalyst was captured with nine times magnification and shown in Figure 3 (a). Also, the close-up image of the catalyst was captured and presented in Figure 3 (b). In this figure, the small particles diffused on the surface were obviously seen and this indicated that the Pd particles were adsorbed and distributed on the surface of the supporting material.

Furthermore, the results of the BET method showed that the surface area of the Pd/Al_2O_3 catalyst was found at 71.23 m^2/g, and the average pore diameter at about 7.25 nm was also obtained, these could be classified as mesoporous material according to IUPAC classification [17]. Dicks et al. [18] studied a series of commercial catalysts for testing hydrodesulfurization (HDS) activity by using an atmospheric gas oil as feedstock, and it has been reported that a mesopore diameter in the range of 7.5-11 nm empowers the highest hydrogenation activity.

Also, the mesoporous property provides an advantage as it readily allows the diffusion of bulky TG molecules of palm oil. This is in agreement with Tiwari et al. [19], who reported that the dimension of TG is approximately 0.6-2.5 nm in length. Therefore, the TG molecule and fatty acid chain can pass through the catalyst's pore. The pore structure of the catalyst is important to encourage the activity of the reaction [20]. Nevertheless, the BET result provided a pore volume of the Pd/Al_2O_3 at 0.14 cm^3/g, this could interrupt the accessibility of the TG.

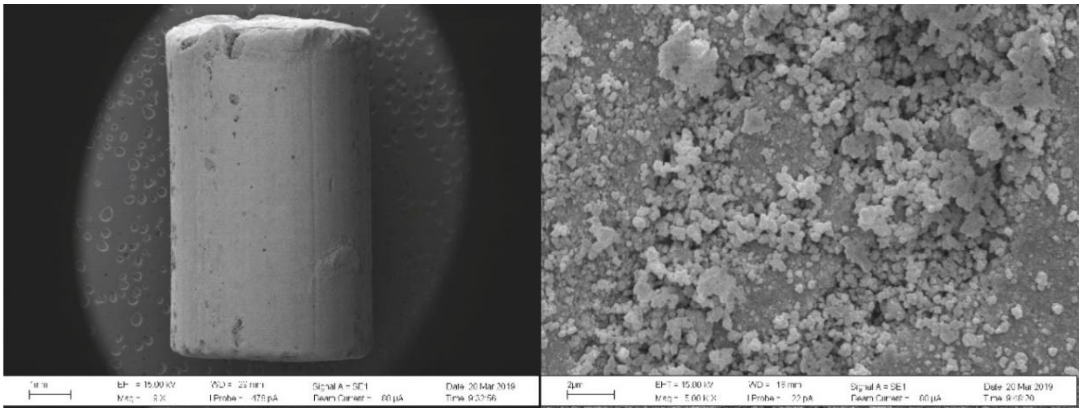

Figure 3 Scanning Electron Microscope (SEM) images of $Pd/TiO_2-Al_2O_3$ catalysts at ×9 magnification (a) and ×5000 magnification (b)

The XRD patterns showed narrow diffraction peaks and intensity as depicted in Figure 4. This figure indicated a crystalline structure of the catalyst. The diffraction peaks of PdO, TiO_2, and Al_2O_3 were remarkable. Palladium is the most exclusive metal used in hydrocracking reaction due to its highly active function that can promote hydrogenolysis. Alumina is an inexpensive and highly stable material that contains basic and acidic sites of supporting structure [21]. Many researchers tried to use titania combined with other oxides, and they reported that the cooperation of mixed oxides in the support system could improve beneficial features of both oxides (for example; TiO_2-Al_2O_3), and provided a higher activity [22, 23].

In addition, titania can improve the hydrogenation efficiency in the hydrocracking reaction. However, the synergy between support and the active metal component of the hydrocracking catalyst usually occurs with the use of titania. Therefore, it is recommended that the support should be composed of mixed oxides between TiO_2-Al_2O_3 to gain beneficial features of both oxides and improve a higher catalytic activity [23].

2. Biofuels production

Hydrocracking reaction was operated by HPPBR reactor to produce liquid biofuel. Typically, the collected biofuel product could contain water that is a by-product obtained from the oxygen removal by hydrodeoxygenation reaction. However, it can be easily separated by a fractional funnel. The biofuel product yield can be calculated by Equation (1).

$$\text{Biofuel product yield (\%vol.)} = \left[\frac{V_p}{V_f}\right] \times 100 \quad (1)$$

where V_p represented the volume of product distillate obtained between 50°C-300°C, V_f is the volume of the feedstock used.

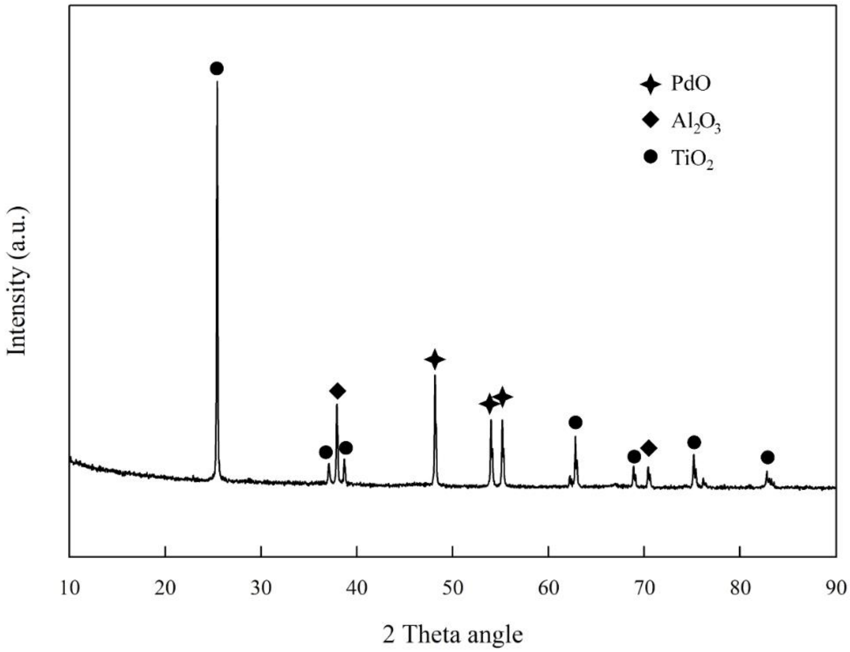

Figure 4 X-ray diffraction pattern of Pd/Al_2O_3 catalyst

In this work, four vegetable oil feedstocks were used to react with H_2 via hydrocracking reaction. The biofuel yield from each feedstock was calculated from Equation (1) and shown in Figure 5. The results revealed that RBO gave the highest biofuel product yield of 94% while PO provided the second-highest of 93%. In addition, SO and SFO also gave relatively similar values of 78% and 72%, respectively. It should be noted that the high contents of oxygen in the feedstocks affect the amount of consumed hydrogen. The excess hydrogen can promote the hydrocracking reaction and give a high yield because hydrogen will be matched with the cracked fatty acid at the terminal of the hydrocarbon structure and hence the shorter hydrocarbon chain can be obtained. PO and RBO contain high amounts of saturated and monounsaturated fatty acids (see Figure 1) which matched the hydrogen feeding rate used in this study. This explained the highest liquid product yields in the cases of PO and RBO. In contrast, SO and SFO consisted of very high unsaturated fatty acids and required a higher hydrogen feeding rate. If the feeding rate of hydrogen was not suitable for the system, hydrocracking might be inhibited, and polymerization could occur. This can be confirmed by the presence of gums and solid greases found in the tubular line of the reactor system after the reaction [12].

Furthermore, the liquid products were separated by the distillation process to obtain the green gasoline and green diesel based on the range of boiling points following ASTM D86 by using Petrotest Instrument (Anton Paar, Germany) [24]. In brief, the liquid sample was heated up from the ambient atmosphere. The vapor temperature of the system was exhibited by a thermometer that connected to the distillation flask. Since we were interested in the quantities of gasoline and diesel obtain from the distillation process, the collection of the distillation products in a measuring cylinder began when the temperature reading was 50°C until 170°C. The measuring cylinder was then put away and replaced by a new one, and the distillation process continued until the temperature reading was 300°C. The distillation

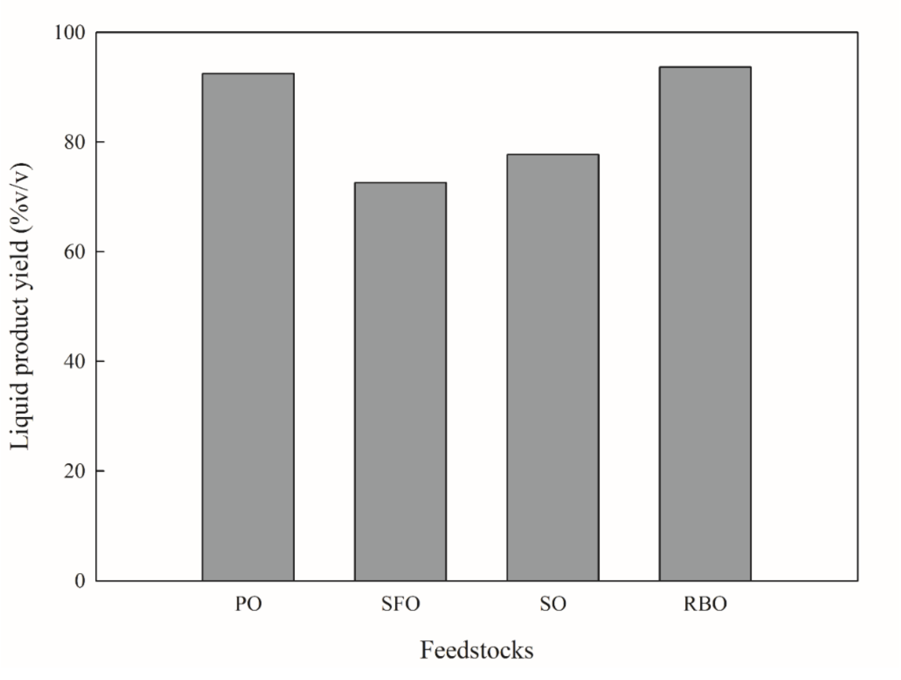

Figure 5 Liquid product yields from the hydrocracking process of PO, SFO, SO, and RBO

product in the first measuring cylinder was gasoline and that in the other cylinder was diesel. It is worth mentioning here that, based on our preliminary distillation test, we found that the distillation of the product could be performed at a maximum temperature of 300°C. After this point, the remaining product became very thick and highly viscous and no more distillation product could be obtained. Product selectivity was defined by Equation (2).

$$\text{Selectivity (\%vol.)} = \left[\frac{V_i}{V_p}\right] \times 100 \qquad (2)$$

where V_p represented the volume of product distillate obtained between 50°C – 300°C and V_i is the volume of the selected products (green gasoline or green diesel) which were defined by their boiling temperature ranges (50°C – 170°C for gasoline and 170°C – 300°C for diesel).

The selectivity of both green fuels is demonstrated in Figure 6. It was found that the highest green diesel was derived from PO, with the selectivity of 82%, and that RBO yielded the lowest green diesel selectivity of 65%. On the contrary, RBO gave the highest green gasoline with the selectivity of 35%, while the PO gave the lowest gasoline selectivity of about 18%. It should be noted that SFO and SO yielded similar values of diesel selectivity of about 68%. Product selectivity of the tested feedstocks varies according to the fatty acid composition in the feedstocks. For example, PO consists of the high content of saturated and mono-unsaturated fatty acids, which promoted hydrogenation and hydrodeoxygenation to change unsaturated fatty acids into the saturated chain and remove the oxygen from the chain in the form of water, respectively, resulting in the highest diesel selectivity. On the other hand, SFO, SO, and RBO had mono-unsaturated and di-unsaturated fatty acids as the main components which gave rise to similar gasoline selectivity values. This can be explained by the fact that the double bond position of di-unsaturated fatty acids can be cracked to a shorter chain to give the product in the gasoline range. In consideration of SFO, SO, and RBO for use in the production of gasoline, RBO is the best candidate because it gave the highest product yield. Therefore, it could be deduced that RBO has the potential to produce shorter chain gasoline hydrocarbons by hydrocracking reaction with higher reaction temperature and pressure. In the case of green diesel, PO is the most suitable raw material to use for the production of this biofuel.

3. Green diesel properties

Table 1 show the properties of green diesel produced from hydrocracking of the feedstocks. It is seen from the table that the kinematic viscosity of all products conformed to the standard specifications. The product of SFO gave the lowest kinematic viscosity (2.04 mm^2/s), while PO gave the highest (2.89 mm^2/s). Additionally, PO and RBO provided closed viscosity of green diesel which higher than the others due to their fatty acid composition. The unsaturated fatty acid of vegetable oil expressed the high viscosity of the product. This is agreed with Anand and his colleague who reported that the high viscosity due to the polyunsaturated character of the raw vegetable oils restricts their use in diesel engines [25]. Also, RBO exhibited the highest density at 822 kg/L and PO gave the second-highest density, and both feedstocks gave the density in the range of the standard specifications while others did not. The net heat of combustion of green- diesel products of PO, SFO, SO, and RBO were acceptable because all the products have higher net heat of combustion than the minimum requirement of diesel fuel.

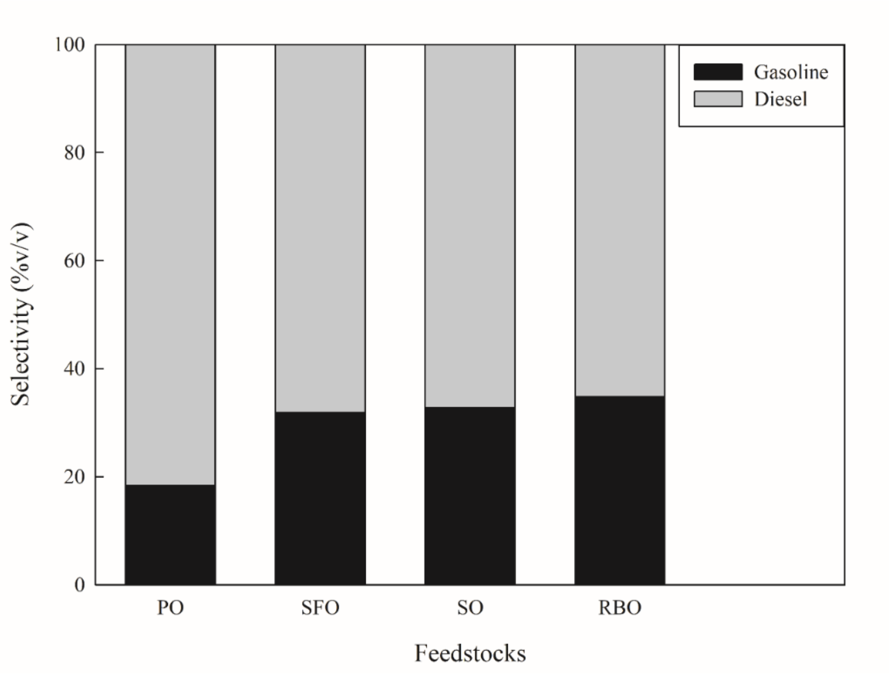

Figure 6 Products selectivity from hydrocracking process of PO, SFO, SO and RBO

Table 1 Fuel properties of green diesel from hydrocracking reaction of PO, SFO, SO, and RBO

Fuel properties	Diesel*	PO	SFO	SO	RBO
Kinetic viscosity at 40°C (mm^2/s)	2 – 4.50	2.89	2.04	2.29	2.69
Density at 15°C (kg/L)	820 – 845	815	793	786	822
Net heat of combustion (MJ/kg)	>42.80	46.40	46.70	48.00	46.70

*Diesel specifications

Conclusion

Different fatty acid components from each vegetable oil feedstock had influenced the biofuel yield and selectivity because the decarboxylation and decarbonylation changed the fatty acid structures to produce an odd carbon atom number of hydrocarbon chains. While hydrodeoxygenation eliminated oxygen from the fatty acids and produced even hydrocarbon chains with an equal number of carbon atoms as the original fatty acids. The effects of all the aforementioned reactions led to the conclusion that, PO has the best potential as a feedstock to produce green diesel due to the highest diesel selectivity of 82% and biofuel product yield of 93% and that RBO is more suitable for bio-gasoline production with the highest gasoline selectivity of 35% and liquid product yield of 94%. Moreover, green diesel synthesis by hydrocracking process of PO, SFO, SO, and RBO can meet the diesel fuel requirements. PO and RBO exhibited high viscosity and density due to their fatty acid composition. Both feedstocks contained unsaturated fatty acids that can express the high viscosity of the product.

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