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THE EFFECT OF VACUUM PROCESS ON BIODIESEL PRODUCTION FROM PALM KERNEL FATTY ACID DISTILLATE

Article Highlights

- Producing biodiesel under vacuum condition reduce costs and energy consumption
- The operating temperature is low in producing biodiesel at vacuum pressure
- Production of biodiesel from PFAD at a vacuum pressure by using PTSA catalyst

Abstract

This study examines the operating pressure effect on the manufacture of biodiesel from Palm Kernel Fatty Acid Distillate (PKFAD) by using para-Toluene Sulfonic Acid (PTSA) catalyst. The operating pressures studied are the vacuum pressures of 70 kPa, 80 kPa, 90 kPa, and 100 kPa, which varied with PTSA concentrations of 5%, 10%, 15%, 20%, and 25%, and the molar ratios of methanol and PKFAD are 5:1, 6:1, 7:1, 8:1, 9:1, and 10:1. Biodiesel production from PKFAD was carried out with reaction duration of 120 minutes and reaction temperature at 50 ^{0}C. The amounts of fatty acids converted to biodiesel were calculated based on the initial and acid numbers after the esterification reaction. The results show that a vacuum pressure of 70 kPa to 100 kPa provides a conversion above 96% for all catalyst concentrations and all methanol and PKFAD molar ratios. The highest conversion of PKFAD to biodiesel of 98.6% is obtained at an operating pressure of 80 kPa, a catalyst concentration of 25%, and the molar ratio of methanol and PKFAD is 10:1. The biodiesel characteristics obtained in this study have met the standards of the American Society for Testing Materials.

Keywords: biodiesel production, palm kernel fatty acid distillate, vacuum process.

SCIENTIFIC PAPER

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Biodiesel production as a substitute for diesel from fossil materials has been widely carried out by utilizing vegetable and animal oils [1–5]. This biodiesel production process usually uses a catalyst to speed up the reaction, both homogeneous and heterogeneous catalysts [6–10]. Biodiesel production is generally carried out at operating pressures above 101.325 kPa (1 atm), both esterification and transesterification [6,11,12].

Palm Kernel Fatty Acid Distillate (PKFAD) was examined in the present study at a vacuum pressure to produce biodiesel (methyl ester). PKFAD is a by-product of the physical refining process of crude palm kernel oil [13]. It has the availability and low price so that this waste material can be used to produce biodiesel at low cost [14,15]. This vacuum pressure condition is intended to save energy for making biodiesel that has occurred so far. In the vacuum pressure condition, the operating temperature will be lower because the boiling point of a liquid will be lower so that the energy required for heating is also low. It agrees with the ideal gas equation, which shows that pressure and temperature have a relationship, that is, if the pressure is lowered, the temperature will decrease. The catalyst used in this study is an inexpensive

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homogeneous catalyst, namely *para*-Toluene Sulfonic Acid (PTSA, $C_7H_8O_3S$) [6,9,16–18]. It is a strong organic acid compound that is non-oxidizing, colorless (white), solid, and easily soluble in water, alcohol, and organic solutions.

The reaction of making biodiesel from PKFAD is shown in Reaction (R1):

$$\underset{\text{PKFAD}}{RCOOH} + \underset{\text{methanol}}{CH_3OH} \leftrightarrow \underset{\text{methylester}}{RCOOR} + \underset{\text{water}}{H_2O} \quad \text{(R1)}$$

In this reaction, a decrease in acid number (acid value) is used to describe the amount of fatty acid that is converted into biodiesel because the raw materials used are fatty acids, so the acid number in the reaction product can be used to calculate the amount of fatty acid that is not converted to biodiesel. The lower acid number obtained indicates that the PKFAD conversion to biodiesel will be higher. In this study, the acid number at the end of the esterification reaction reflects the PKFAD content that is not changed to biodiesel.

Several things that affect biodiesel production are also varied in this study, such as the molar ratio of reactants, catalyst concentration, reaction time, reaction temperature, and vacuum pressure. The characteristics of PKFAD as feedstock are shown in Table 1.

Table 1. PKFAD Characteristics.

Characteristics	Measurement results	Unit	Test Method
Density at 25 °C	0.904	g/cm^3	ASTM D1298
Color	0.9R	5¼ in Lovibond	AOCS Cc 13e-92
	7.9Y	5¼ in Lovibond	AOCS Cc 13e-92
Acid Value	336	mg KOH/g	AOCS Te 2a-64
Iodine Value	4.36	g I_2/100 g	AOCS TI 2a-64
Sulfur	2.05	ppm	ASTM D5453
Water content	0.02	%	ASTM D2705
C_6	0.06	%	ASTM D6584
C_8	31.95	%	ASTM D6584
C_{10}	60.23	%	ASTM D6584
C_{12}	0.50	%	ASTM D6584
C_{14}	0.17	%	ASTM D6584
C_{16}	0.27	%	ASTM D6584
C_{18}	1.89	%	ASTM D6584
C_{20}	1.89	%	ASTM D6584
unknown	2.12	%	ASTM D6584

MATERIAL AND METHODS

The fatty acid used consists of PKFAD; the composition and characteristics can be seen in Table 1. The PKFAD used in this study came from an oleochemical factory in North Sumatra Province, Indonesia. The PKFAD is the result of the refinery and is used directly without further treatment. The majority component of PKFAD consists of C_8 ($CH_3(CH_2)_6COOH$) or caprylic acid and C_{10} ($CH_3(CH_2)_8COOH$) or capric acid. Besides caprylic acid and capric acid, the PKFAD also contains caproic acid (C_6), lauric acid (C_{12}), myristic acid (C_{14}), palmitic acid (C_{16}), stearic acid (C_{18}), and arachidic acid (C_{20}). The alcohol is 99% methanol (Merck Chemicals Ltd), while the PTSA catalyst is purchased from Navdeep Chemicals Pvt Ltd. In this biodiesel production, the raw material and catalyst were heated on a hot plate to 50 ^{0}C and a speed of 300 rpm.

The biodiesel production from PKFAD consists of several variations, including variations in vacuum pressure (70 kPa, 80 kPa, 90 kPa, and 100 kPa) compared to the operating pressure at 110 kPa), variations in the concentration of catalysts on PKFAD (5%, 10%, 15%, 20%, and 25 %), variations in the molar ratios of methanol to PKFAD (5:1, 6:1, 7:1, 8:1, 9:1, and 10:1), reaction temperature (35 ^{0}C, 40 ^{0}C, 45 ^{0}C, 50 ^{0}C, and 55 ^{0}C), and variations in reaction time (60 minutes, 75 minutes, 90 minutes, 105 minutes, and 120 minutes). Eq. (1) is used to calculate the amount of fatty acids converted into biodiesel [19–22]:

$$y(\%) = \frac{BA_0 - BA_1}{BA_0} 100\% \quad (1)$$

where y is the fatty acids conversion into biodiesel, BA_0 is the initial acid number (mg KOH/g), and BA_1 is the acid number after the esterification reaction at a certain time (mg KOH/g). The biodiesel's composition was analyzed using Gas Chromatography-Mass Spectrometry (GC-MS) with the details of the Agilent Technologies 7890A system (SN CN11051074) using hydrogen as a carrier.

RESULTS AND DISCUSSION

Effect of vacuum pressure on the acid number without the addition of a catalyst

Biodiesel can be formed from PKFAD in the esterification reaction at various vacuum pressures. Fig. S1 (Supplementary material) shows the acid number obtained at various operating pressure performed with a molar ratio of 10:1, a reaction temperature of 50 ^{0}C, and a reaction time of 120 minutes. The results showed that the highest acid number of 122 mg KOH/g was obtained at an operating pressure of 80 kPa. Using Eq. (1), the conversion of PKFAD to biodiesel at an operating pressure of 80 kPa reaches 63.69%, while at operating pressures of 70 kPa, 90 kPa, and 100 kPa are 62.50%, 60.71%, and 57.74%, respectively. The absence of a catalyst reduced the ability to enhance the reaction of the reactants by the role of a catalyst. The results obtained in Fig. S1 indicate that biodiesel production from PKFAD under vacuum conditions without the addition of a catalyst can occur but with a low conversion rate.

At a non-vacuum operating pressure of 110 kPa, the acid number was higher than at vacuum pressure, or the conversion value of PKFAD to biodiesel was lower. The acid number obtained at 110 kPa was 148 mg KOH/g or the PKFAD conversion to biodiesel reached 55.95%. The results obtained indicate that it is necessary to make efforts to increase the reaction rate. The treatment to be carried out included adding a catalyst and increasing the temperature of the esterification reaction. These treatments were varied with the molar ratio of methanol to PKFAD.

Effect of reaction temperature on the acid number

Fig. S2 shows the reaction temperature effect on the acid number obtained at a molar ratio of 10:1, an operating pressure of 80 kPa, a catalyst concentration of 25%, and a reaction time of 120 minutes. It can also be seen that the lowest acid number was at 50 ^{0}C, which was 4.72 mg KOH/g, or the PKFAD conversion to biodiesel reached 98.60%, while at 35 ^{0}C, 40 ^{0}C, 45 ^{0}C, and 55 ^{0}C, the conversion of PKFAD to biodiesel were 98.06%, 98.37, 98.51%, and 98.37%, respectively. The absence of a catalyst reduced the ability to enhance the reaction of the reactants by the role of a catalyst. These results indicate that the operating temperature close to room temperature at a vacuum pressure of 80 kPa can provide a conversion result of PKFAD to biodiesel above 98%. Hence, operating pressure in vacuum conditions can save energy consumption in biodiesel production from PKFAD, and the conversions of PKFAD to biodiesel in vacuum pressure conditions are higher than operating in non-vacuum pressure conditions using various catalysts [14–17, 20–23].

Reaction time and catalyst concentration effect on the acid number

Fig. 1 shows the reaction time effect and catalyst (PTSA) concentration effect on the acid number at the operating pressure of 80 kPa, the molar ratio of methanol to PKFAD of 10:1, and the reaction temperature of 50 ^{0}C. The results showed that the acid number decreased with increasing reaction duration. Biodiesel production using a homogeneous catalyst is generally carried out with reaction times between 60 minutes and 120 minutes. However, a longer reaction time can lower the acid value [27]. Likewise, with the catalyst concentration, a decrease in acid number occurs with an increase in the catalyst concentration. It can be seen in Fig. 1 that the reaction time and catalyst concentration greatly affect the acid number obtained. The lowest acid number of 4.72 mg KOH/g was obtained at a reaction time of 120 minutes and a concentration of 25%, which means that 98.6% of PKFAD was converted into biodiesel. For all catalyst concentrations at the reaction time of 120 minutes, the amount of PKFAD converted into biodiesel is above 97%. The results obtained in this study are comparable to those of other researchers who were able to achieve the conversion of fatty acids into biodiesel up to 97.1% by Guan *et al.* [9], 87% by Liu *et al.* [10], 96% by Marchetti [26], and 95% by Metere [19]. All these researchers used acid catalysts to catalyze fatty acids and/or triglycerides as raw materials for biodiesel production, but all these studies were conducted at non-vacuum pressure. Hence, the addition of PTSA catalysts significantly increases the esterification reaction rate of the PKFAD into biodiesel.

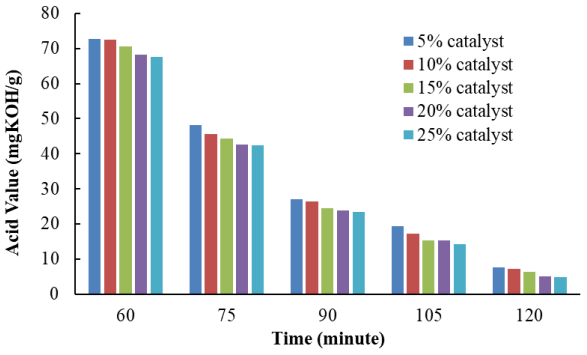

Figure 1. Reaction time and catalyst concentration effect on acid value at a molar ratio of 10: 1 and an operating pressure of 80 kPa.

Effect of operating pressure and a molar ratio of methanol and PKFAD on the acid number

In Fig. 2, the operating pressure effect on acid number is presented at various molar ratios of methanol and PKFAD with a catalyst concentration of 5% and a

reaction time of 120 minutes. Operating pressure did not significantly affect the acid number, but the acid number under vacuum gave better results than in non-vacuum conditions. The higher molar ratio generally produces a lower acid value [28]. In all molar ratios of methanol and PKFAD for all vacuum pressures, the conversion of PKFAD to biodiesel was above 96%, and the highest was at a molar ratio of 10:1, which could reach above 97%.

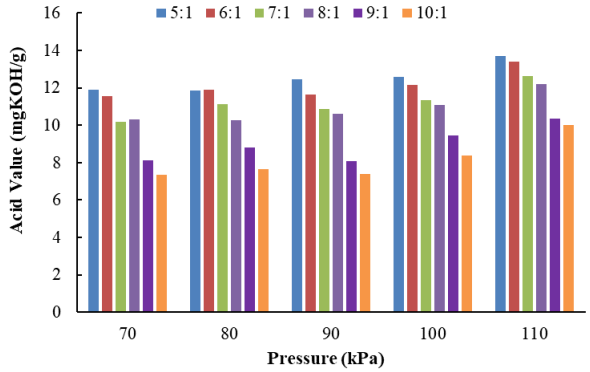

Figure 2. Effect of operating pressure and a molar ratio of methanol and PKFAD on acid value at a catalyst concentration of 5% in a reaction time of 120 minutes.

Fig. 3 shows the effect of operating pressure on the acid number at various methanol and PKFAD molar ratios with a catalyst concentration of 10% and a reaction time of 120 minutes.

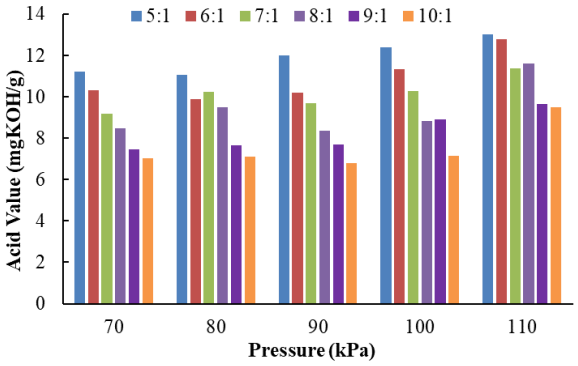

Figure 3. Effect of operating pressure and a molar ratio of methanol and PKFAD on acid value at a catalyst concentration of 10% in a reaction time of 120 minutes.

Similar to Fig. 2, the operating pressure did not significantly affect the acid number, but the acid number obtained under vacuum pressure gave better results than the non-vacuum pressure condition. In all molar ratios of methanol and PKFAD for all vacuum pressures, the conversion of PKFAD to biodiesel was above 96%, and the highest was at a molar ratio of 10:1, which could achieve conversions above 97%.

In Fig. 4, the effect of operating pressure on the acid number is presented at various molar ratios of methanol and PKFAD with a catalyst concentration of 15% and a reaction time of 120 minutes. In all molar ratios of methanol and PKFAD for all vacuum pressures, the conversion of PKFAD to biodiesel was above 96%, and the highest was at a molar ratio of 10:1, which could reach above 98% for all operating pressure variations.

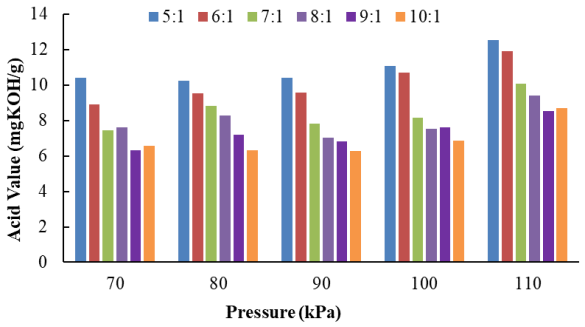

Figure 4. Effect of operating pressure and a molar ratio of methanol and PKFAD on acid value at a catalyst concentration of 15% in a reaction time of 120 minutes.

In Fig. 5, the effect of operating pressure on acid number is presented at various molar ratios of methanol and PKFAD with a catalyst concentration of 20% and a reaction time of 120 minutes. In all molar ratios of methanol and PKFAD for all vacuum pressures, the conversion of PKFAD to biodiesel was above 96%, and the highest was at a molar ratio of 10:1, which could reach above 98%.

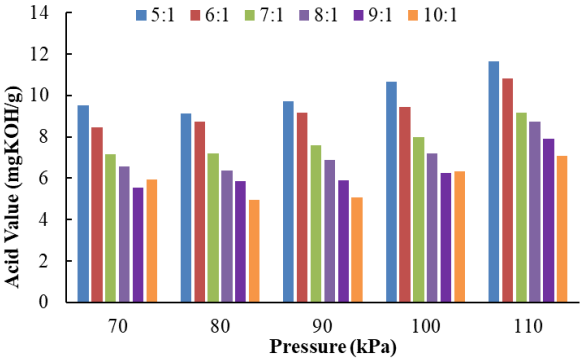

Figure 5. Effect of operating pressure and a molar ratio of methanol and PKFAD on acid value at a catalyst concentration of 20% in a reaction time of 120 minutes.

In Fig. 6, the effect of operating pressure on acid number is presented at various molar ratios of methanol and PKFAD with a catalyst concentration of 25% and a reaction time of 120 minutes. In all molar ratios of methanol and PKFAD for all vacuum pressures, the conversion of PKFAD to biodiesel was above 97%, and the highest was at a molar ratio of 10:1, which reached above 98%.

The lowest acid number of 4.72 mg KOH/g was obtained at an operating pressure of 80 kPa and a

molar ratio of methanol and PKFAD of 10:1, or the conversion of PKFAD to biodiesel reached 98.60% (Fig. 6). This conversion was higher than at other pressures including non-vacuum pressures. At 110 kPa, the lowest acid number of 6.54 mg KOH/g was obtained at a catalyst concentration of 25%, and the molar ratio of methanol and PKFAD was 10:1. Under this operating pressure condition, the conversion of PKFAD to biodiesel reached 98.05 %.

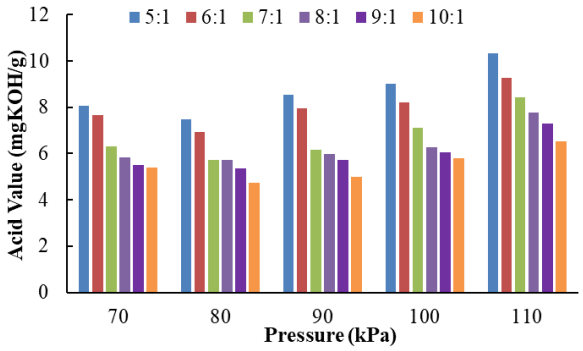

Figure 6. Effect of operating pressure and a molar ratio of methanol and PKFAD on acid value at a catalyst concentration of 25% in a reaction time of 120 minutes.

According to Figs. 2–6, a vacuum pressure of 70 kPa to 100 kPa gave a conversion that was not much different for each of the same methanol and PKFAD molar ratios. However, the vacuum pressure that gave the highest conversion of PKFAD to biodiesel was a pressure of 80 kPa. The conversion results of PKFAD to biodiesel for all vacuum pressures, all catalyst concentrations, and all molar ratios of methanol and PKFAD in the present study were above 96%. Figs. 2–6 show that the acid number obtained under vacuum pressure is better than that obtained at non-vacuum pressure (110 kPa).

Based on the results of the GC-MS analysis in Table 2, it can be seen that the composition of the biodiesel obtained is similar to the composition of PKFAD used as raw material (Table 1), where C_8 and C_{10} are the main compositions of biodiesel. The composition of C_{14} and C_{16} at a pressure of 90 kPa was not obtained, while the composition of the raw material was 0.17% of C_{14} and 0.27% of C_{16}. The loss of C_{14} and C_{16} at 90 kPa was caused by the decomposition of carbon atoms in the esterification reaction, which caused the reading on GC-MS to increase the number of unknowns. In contrast, at 90 kPa, the number of unknown compositions in biodiesel increased to 3.11% from 2.12% on raw materials.

Table 3 shows biodiesel's neutralized characteristics, namely biodiesel obtained from the experiment at an operating pressure of 80 kPa, 10:1 of methanol and PKFAD molar ratios, the amount of catalyst 25%, and the reaction duration of 120 minutes. These results indicate that the biodiesel obtained from this study has met the American Society for Testing Materials (ASTM) standards.

Table 2. Composition of biodiesel by using GC-MS Analysis at a molar ratio of methanol and PKFAD 10:1 and a catalyst concentration of 25%.

Pressure (kPa)	Component (%)								
	C_6	C_8	C_{10}	C_{12}	C_{14}	C_{16}	C_{18}	C_{20}	Unknown
70	0.05	32.31	61.49	0.23	0.15	0.21	1.29	2.18	2.09
80	0.04	32.91	61.95	0.42	0.03	0.31	0.82	1.38	2.14
90	0.03	32.79	62.08	0.26	0	0	0.66	1.07	3.11
100	0.03	32.64	61.53	0.41	0.07	0.20	0.80	1.27	3.05
110	0.04	32.91	62.26	0.27	0.03	0.16	0.94	1.56	1.83

Table 3. Characteristics of biodiesel at an operating pressure of 80 kPa, a molar ratio of methanol and PKFAD of 10: 1, and a catalyst concentration of 25%.

Characteristic	Measurement results	ASTM Standard
Ester content	97.8% (m/m)	>96.5%
Density at 25°C	895 kg/m^3	860-900 kg/m^3
Viscosity at 40 °C	2.3 mm^2/s	1.9-6.0 mm^2/s
Flash point	156 °C	≥130 °C
Water content	0,02% (v/v)	≤0.05% (v/v)
Acid value	0.35 mg KOH/g	≤0.50 mg KOH/g
Methanol content	0.01% (m/m)	≤0.02% (m/m)

CONCLUSION

Biodiesel production from PKFAD at a vacuum pressure of 70 kPa to 100 kPa achieves conversions above 96% for all PTSA catalyst concentrations and all methanol and PKFAD molar ratios. The highest conversion of PKFAD to biodiesel of 98.6% was obtained at a vacuum pressure of 80 kPa, with a catalyst concentration of 25%, a reaction time of 120 minutes, and the molar ratio of methanol and PKFAD is 10:1. The characteristics of biodiesel obtained in this study met the American Society for Testing Materials (ASTM) standards. The results show that the ability to produce biodiesel under vacuum conditions will reduce production costs and energy consumption because the operating temperature is lower than in non-vacuum conditions.

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NAUČNI RAD

EFEKAT VAKUUMA NA PROIZVODNJU BIODIZELA OD DESTILATA MASNIH KISELINA PALMI

Ova studija istražuje efekat radnog pritiska na proizvodnju biodizela iz destilata masnih kiselina palminog jezgra (PKFAD) korišćenjem para-toluen sulfonske kiseline (PTSA) kao katalizatora . Istraživannja su sprovedena pri radnim pritiscima (vakuum) od 70 kPa, 80 kPa, 90 kPa i 100 kPa, koncentracijama PTSA od 5%, 10%, 15%, 20% i 25% i molskim odnosima metanola i PKFAD od 5:1, 6:1, 7:1, 8:1, 9:1 i 10:1. Proizvodnja biodizela iz PKFAD-a je trajala 120 minuta na 50 °C. Količine masnih kiselina pretvorenih u biodizel izračunate su na osnovu početnog i kiselinskog broja nakon reakcije esterifikacije. Rezultati pokazuju da vakuum od 70 kPa do 100 kPa obezbeđuje konverziju iznad 96% za sve koncentracije katalizatora i sve molske odnose metanola i PKFAD. Najveća konverzija PKFAD u biodizel od 98,6% se postiže pri radnom pritisku od 80 kPa, koncentraciji katalizatora od 25%, a molskom odnosu metanola i PKFAD od 10:1. Karakteristike biodizela dobijene zadovoljavaju standarde Američkog društva za ispitivanje materijala.

Ključne reči: proizvodnja biodizela, destilat masnih kiselina palminog jezgra, process pod vakuumom.