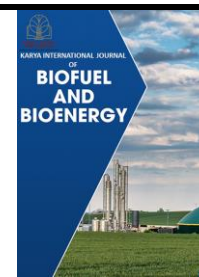




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Optimization of Biodiesel Production Conditions from Waste Cooking Oil using *Parkia speciosa* Hassk. Peel Extracts - Modified Magnetite (Fe_3O_4 -PSH) as Catalyst

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ABSTRACT

The world's energy needs continue to increase while availability decreases, so it is necessary to develop alternative energy sources. Biodiesel with waste raw materials is an alternative energy that attracts attention. This study produced biodiesel using raw materials from waste cooking oil with *Parkia speciosa* Hassk. peel extracts - modified magnetite (Fe_3O_4 -PSH) catalyst. The Fe_3O_4 -PSH catalyst was synthesized using the reverse coprecipitation method and characterized using FTIR, XRD, VSM, PSA, and SEM instruments. Biodiesel production was performed at various reaction conditions such as temperature, time, mole ratio of methanol and oil, and catalyst concentration to obtain biodiesel with the best yield and quality. The biodiesel quality parameters analyzed included density, water content, acid number, flash point, and calorific value. The results showed that the catalyst was successfully synthesized with good crystallinity and had a rough surface morphology. The crystallite size, magnetization saturation value, and average particle size were 14.5 nm, 28.33 emu/g, and 2048.6 nm, respectively. The optimum production conditions were obtained using catalyst concentration (0.5% w/w oil), methanol: oil ratio = 3:1 at a temperature of 60 °C for 90 minutes. The quality test showed that biodiesel quality was by SNI 7182:2015.

1. Introduction

Energy is a basic need for various fields, such as industry, households, and others. Current energy needs are dominated by non-renewable petroleum energy sources. Over time, energy needs have increased along with dwindling energy sources. Currently, vehicle fuel still relies on petroleum and is the energy most consumed by the world's population [1]. It is necessary to develop alternative energy to replace fossil fuels to prevent an energy crisis.

Biodiesel is a widely developed alternative fuel. Biodiesel can be produced from vegetable oil or animal fat through esterification and transesterification reactions [2]. This fuel is environmentally friendly because it is non-toxic, free from sulfur, has a fragrant odour, and produces lower exhaust

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emissions than fossil fuels [3]. The weakness of biodiesel production using raw vegetable and animal materials is competing with food security. Therefore, it is necessary to develop biodiesel production using raw materials that do not interfere with food security. The use of waste containing vegetable oil and animal fat is interesting to study further.

In addition to the selection of the primary raw material, the use of catalysts in biodiesel production is no less critical. Homogeneous catalysts such as sodium hydroxide and potassium hydroxide are widely used in biodiesel production. Homogeneous catalysts produce high yields and activities but need to improve the purification process, which is relatively complex and produces much waste [4]. In addition, soap will be easily formed because fatty acids or glycerides in used cooking oil can react with bases through saponification reactions [5]. To overcome this problem, using heterogeneous catalysts is an alternative solution. Heterogeneous catalysts are environmentally friendly, are not corrosive, can be separated from the product by filtration, and can be used repeatedly for a long time [6]. However, the filtration process can increase operating time and production costs [7], so another alternative is needed, namely using a heterogeneous catalyst in the form of a magnetite catalyst (Fe_3O_4). Magnetite catalysts are magnetic, so the separation process can be carried out using an external magnetic field. This process can save reaction time [8]. The latest innovation in the synthesis of magnetite catalysts is using plant waste extracts. Plant waste extracts have high phenolic and flavonoid compound content, so they can act as reductants and stabilizing agents and prevent particle agglomeration [9-11]. One of the plant wastes that can be used to synthesize magnetite catalysts is *Parkia speciosa* Hassk. peel extracts are used because they have a total of phenol and flavonoids of 90.67 mgGAE/g and 5.86-5.38 mg CE/g DW, respectively [12].

The explanation highlights some of the novelties of this research, namely: First, the use of Fe_3O_4 catalyst from *Parkia speciosa* Hassk. peel plant extract in biodiesel production. Second, optimization of biodiesel production conditions, including temperature, time, methanol and oil mole ratio, and catalyst concentration, was carried out. Third, oil quality tests were carried out, including density, water content, acid number, flash point, and calorific value parameters.

2. Methodology

2.1 Materials

This study's primary raw material for biodiesel production was cooking oil waste from domestic waste. The chemicals used were proanalyst chemicals, including methanol (CH_3OH), Sulfuric acid (H_2SO_4), Sodium hydroxide (NaOH), Iron (III) Chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), and Iron (II) Sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) from Merck Company, Darmstadt, Germany. At the same time, distilled water was produced by Ikapharmindo Putramas Company in Jakarta, Indonesia. The instruments used for catalyst characterization were Fourier-Transform Infrared (FTIR) Spectrophotometer Shimadzu Prestige 21, Japan; X-ray Diffraction, 6000 Shimadzu, Japan; Particle Size Analyzer, Horiba SZ-100, Japan; Surface Area Analyzer, Quantachrome NOVA 1200e, Florida and Scanning Electron Microscope – Energy Dispersive X-ray (SEM-EDX) JEOL JSM-6510LA. The chemical composition of pyrolysis oil was analyzed using Gas Chromatography and Mass Spectroscopy (GC-MS) GC2010 MSQP 2010S Shimadzu, Japan.

2.2 Research Work Procedure

2.2.1 Preparation of *Parkia speciosa* Hassk peel extract.

The *Parkia speciosa* Hassk (PSH) peel is chopped into small pieces and then dried in the sun until dry. After that, it is dried again using an oven at a temperature of 60 °C for 2-3 hours. The dried PSH Peel is ground into powder. Ten grams of PSH peel powder is transferred to a Beaker glass, and 200

mL of aquabidest is added. The mixture is heated at a temperature of 60 °C for 20 minutes. The pH is checked at the end of the extraction. Furthermore, the filtrate is filtered and stored at a temperature of 4 °C.

2.2.2 Synthesis of Fe₃O₄-PSH catalyst

A total of 0.8 grams of NaOH is dissolved in 20 mL aquabidest. A total of 1.082 g of FeCl₃.6H₂O/50 mL of aquabidest was reacted with 0.556 grams of FeSO₄.7H₂O/50 mL of aquabidest then 5 mL of PSH peel extract filtrate was added. The mixture was slowly added to 1 M NaOH solution. The mixture was stirred constantly for 60 minutes at 60 °C. The pH of the mixture was measured at the end of the reaction. The precipitate was filtered using filter paper and a Buchner vacuum pump. The precipitate was dried in an oven at 60 °C and ground into powder. The powder was weighed and stored in a tight container.

2.2.3 Characterization of Fe₃O₄-PSH magnetic catalyst

The Fe₃O₄-PSH catalyst was characterized using FTIR, XRD, VSM, PSA, and SEM instruments.

2.2.4 Esterification reaction of waste cooking oil

300 ml of waste cooking oil was added with 1.5 ml of 98% H₂SO₄ catalyst and then heated to 60 °C. In a separate place, 50 ml of methanol was heated to a temperature of 60 °C. Furthermore, methanol was added to the oil mixture, and the catalyst was stirred for 1 hour at a constant temperature of 60 °C. Furthermore, the mixture was put into a separating funnel and left for 5 hours until two layers were formed. The upper layer is a mixture of biodiesel, triglycerides, and residual methanol, while the lower layer is a mixture of water and sulfuric acid catalyst. The upper layer is used for the transesterification reaction stage.

2.2.5 Transesterification reaction of waste cooking oil

The upper layer of the esterification reaction result was added with methanol with variations in the ratio of methanol: an upper layer of 3:1, 6:1, 9:1, and 12:1. Furthermore, Fe₃O₄-PSH catalyst was added with variations in the mass of 0.25 and 0.5% (w/w upper layer). The mixture was stirred for variations in time of 30, 45, 60, 75, and 90 minutes, and variations in temperature of 50, 60, 70, 80, and 90 °C to produce biodiesel. After the reaction ended, the Fe₃O₄-PSH magnetic catalyst could be separated with an external magnetic field. After that, it was put into a separating funnel and left for 5 hours until two layers were formed. The upper layer is a mixture of biodiesel and methanol, while the lower layer is glycerol.

2.2.6 Testing the quantity and quality of biodiesel

The obtained biodiesel was weighed, and its yield was calculated. Furthermore, the chemical composition was analyzed using GC-MS, and its quality was analyzed based on density, water content, acid number, flash point, and calorific value parameters.

3. Results

3.1 Characterization of Fe_3O_4 -PSH Catalyst

Figure 1 shows the FTIR spectra of the Fe_3O_4 -PSH compound. The appearance of absorption at wave number 500 cm^{-1} indicates the presence of typical absorption from Fe-O bonds of magnetite. The absorption peak at wave number 3418 cm^{-1} indicates an O-H bond of phenolic compounds from PSH peel extract. It can also indicate hydrogen bonds between H atoms from phenolic -OH groups and O atoms from the magnetite surface [3,10]. The absorption peaks of 1617 and 1369 cm^{-1} indicate absorption originating from the C=C bond of the aromatic ring of phenolic compounds. The FTIR spectra show magnetite has been successfully modified using PSH peel extracts.

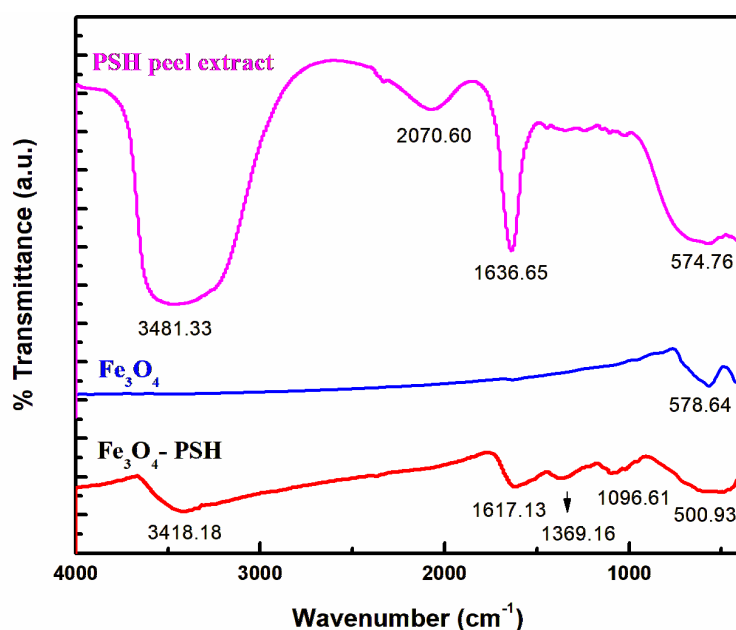


Fig. 1. FTIR spectra of Fe_3O_4 -PSH

This statement is supported by the XRD diffractogram presented in Figure 2. The diffractogram of the synthesized Fe_3O_4 -PSH shows five diffraction peaks (2θ) at angles of 30.5° , 35.8° , 43.5° , 57.5° , and 63° with the Miller index planes of the Fe_3O_4 -PSH crystal in sequence (220), (311), (400), (511), and (440). Comparison of the diffractogram peaks of the synthesis of magnetic nanoparticles to the standard data of Fe_3O_4 material JCPDS (Joint Committee on Powder Diffraction Standards) number 19-0629 [21]. Based on the Debye-Scherrer equation, the crystal size of Fe_3O_4 -PSH is 14.5 nm.

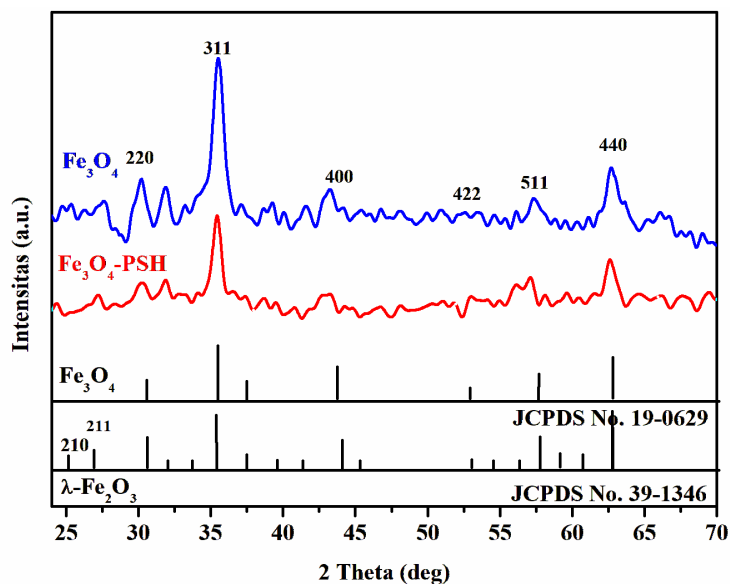


Fig. 2. Diffractogram of Fe₃O₄-PSH

Figure 3 shows the magnetization saturation value of the Fe₃O₄-PSH catalyst. The magnetization saturation value of Fe₃O₄-PSH is lower than that of Fe₃O₄. It means that the modification of magnetite compounds using PSH peel extract reduces the magnetic strength of magnetite. The decrease in the Ms value occurs due to the presence of phenolic compounds in the PSH peel extract, which are non-magnetic compounds that can reduce magnetic interactions between particles and the magnetic moment of particles [16,17].

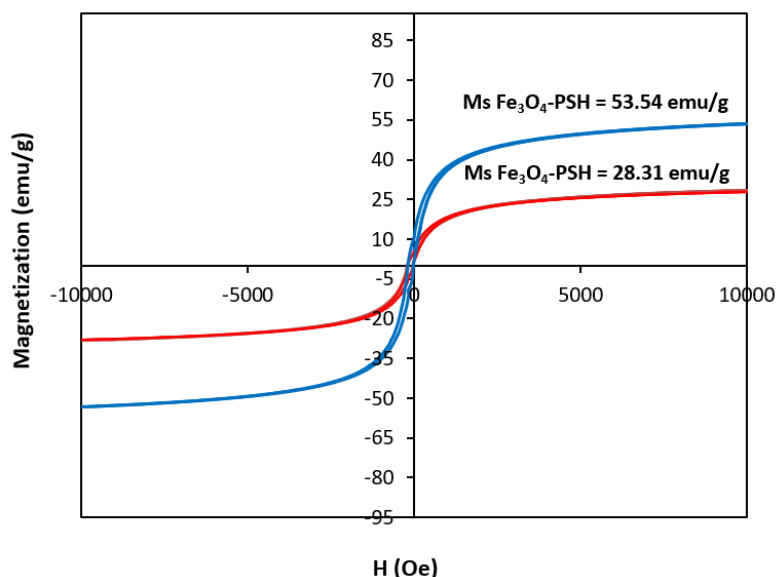


Fig. 3. Magnetization curve at room temperature for Fe₃O₄-PSH

Figure 4 shows the particle size distribution of Fe₃O₄-PSH. The particle size of Fe₃O₄-PSH is spread from 5.61 to 5901.02 with an average particle size of 2048.6 nm. A comparison of crystallite and particle size of Fe₃O₄ with several previous studies is presented in Table 1. The morphology of Fe₃O₄-PSH based on SEM photos has an irregular shape (Figure 5).

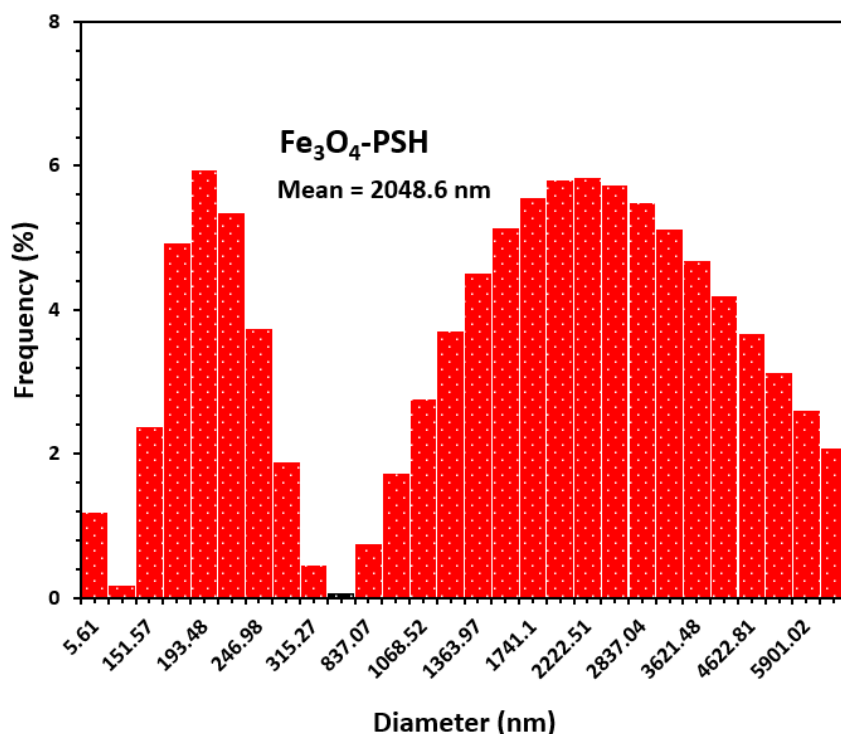


Fig. 4. Particle size distribution of Fe₃O₄-PSH

Table 1

Comparison of crystallite size and particle size of magnetite

No.	Extract source/Reaction conditions	Crystal Size (nm)	Particle Size (nm)	Morphology	Magnetization (emu/g)	References
1	<i>Archidendron Pauciflorum</i> / Sonochemical method, 60 min, 60 °C, FeSO ₄ ·7H ₂ O, FeCl ₃ ·6H ₂ O	15.00 and 17.05	2.56, 862.23, and 4,855.78	Rough surface	27.71	[10]
2	Tea dregs/ Conventional method pH 11, 60 min, 60 °C	18.92	2,670.37	-	-	[18]
3	<i>Archidendron Pauciflorum</i> / Conventional method, 60 min, 60 °C, FeSO ₄ ·7H ₂ O, FeCl ₃ ·6H ₂ O	17.6 - 20.90	318 and 294	-	25.6, and 12.9	[9]
4	<i>Parkia speciosa</i> Hass/ Conventional method, 60 min, 60 °C, FeSO ₄ ·7H ₂ O, FeCl ₃ ·6H ₂ O	14.5	2,048.6	Rough surface	28.31	This research

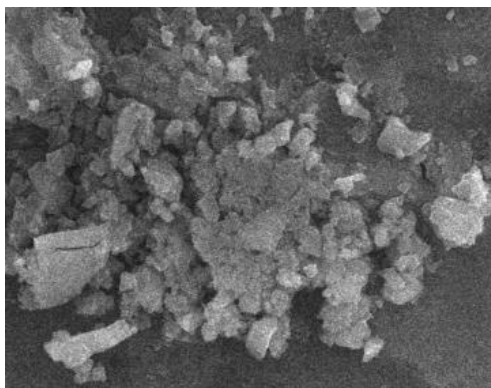


Fig. 5. SEM photo of Fe₃O₄-PSH

3.2 Optimization of Biodiesel Production Conditions with Cooking Waste Oil as Raw Material using Fe₃O₄-PSH Catalyst

3.2.1 Esterification reaction as a pre-treatment stage of waste cooking oil

The free fatty acid (FFA) content in the waste cooking oil used is relatively high because it is >1%, so the esterification reaction must be carried out as a pre-treatment. Esterification is carried out to reduce the FFA content in waste cooking oil. Before the reaction, the waste cooking oil is filtered first to remove impurities. The esterification reaction is carried out by reacting alcohol and cooking oil with the help of a sulfuric acid catalyst. The alcohol used in this study is methanol because it is more economical and easy to find. Methanol is also more soluble than ethanol and butanol because the longer the carbon chain, the lower the solubility level [13]. The pre-treatment stage is carried out to convert free fatty acids into biodiesel before the transesterification stage. The presence of free fatty acids can compete with triglycerides to react with methanol to produce biodiesel. The mechanism of biodiesel synthesis from free fatty acids with an H₂SO₄ catalyst is presented in Figure 6.

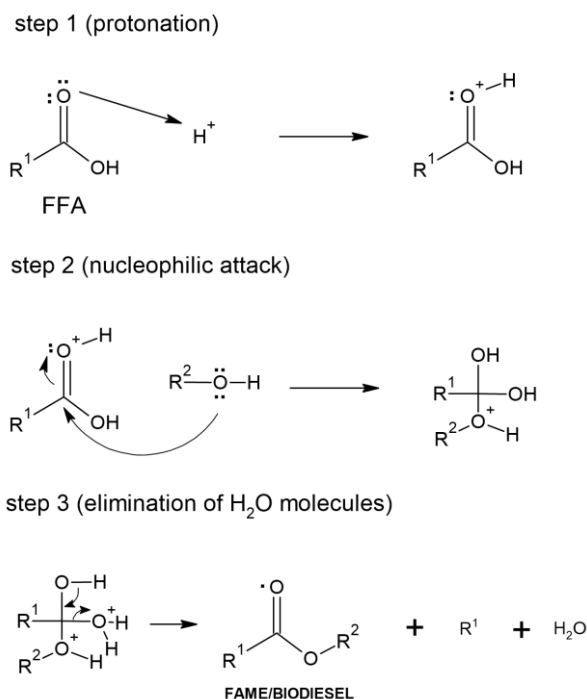


Fig. 6. Reaction mechanism of FFA esterification in used cooking oil into biodiesel

3.2.2 Transesterification reaction of waste cooking oil using Fe_3O_4 -PSH catalyst

The transesterification reaction is carried out after the esterification stage. The transesterification reaction is assisted by the Fe_3O_4 -PSH catalyst, which acts as a base with a negative O- group on its surface. The catalyst initially reacts with methanol to form a methoxy species, which initiates the transesterification reaction. The Fe_3O_4 -PSH base catalyst is used to facilitate the formation of a methoxide solution, which is the result of the reaction with methanol.

The first stage begins with the reaction between the anion of the Fe_3O_4 -PSH catalyst and methanol to form methoxide and a protonated catalyst. The second stage is the nucleophilic attack of methoxide on the carbonyl group of triglycerides to form a tetrahedral intermediate compound. The third stage continues with the formation of alkyl esters and glyceride anions. The last stage is catalyst deprotonation, so the Fe_3O_4 -PSH catalyst regenerates into an active catalyst species that can be used in the next catalytic cycle [19]. The transesterification reaction mechanism is presented in Figure 7.

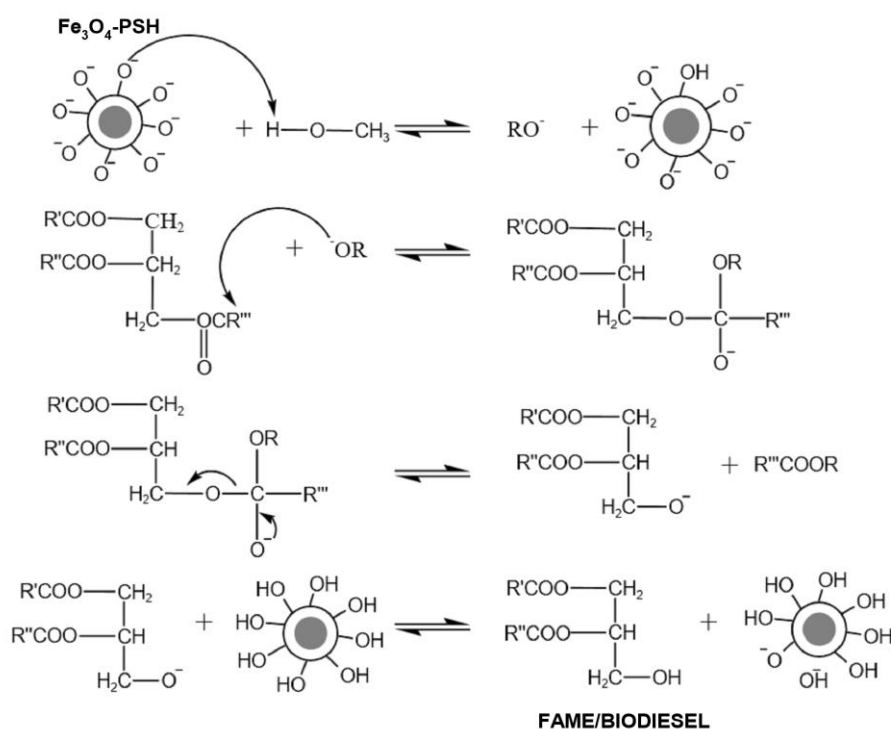


Fig. 7. Mechanism of transesterification reaction of triglycerides in used cooking oil into biodiesel.

The transesterification reaction was conducted at various temperatures, times, methanol: oil ratios, and catalyst concentrations. The transesterification reaction at various temperatures showed that the reaction temperature affected the biodiesel yield (Figure 8). The maximum biodiesel yield was obtained at a temperature of 60 °C. This optimum condition was influenced by the boiling point of methanol, which was at a temperature of 64.7 °C, so that the collision between methanol particles and triglycerides in used cooking oil took place maximally at temperatures close to the boiling point of methanol. The biodiesel yield decreased further at reaction temperatures above the boiling point of methanol because some of the methanol had evaporated and air bubbles were formed, which interfered with the reaction [14,15]. Based on statistical tests using the Statistical Package for the Social Sciences (SPSS), the significance value for the normality test was obtained in the range of 0.298

- 0.775. This significance value is > 0.05 , meaning the data is normally distributed. The homogeneity significance value obtained was 0.09 (> 0.05), meaning that the compared temperature variant data was homogeneous. So, the homogeneity assumption in the one-way ANOVA test is met. Based on the ANOVA output, the significance value is 0.001 (< 0.05). It means that temperature variations in biodiesel production with used cooking oil as raw material with $\text{Fe}_3\text{O}_4\text{-PSH}$ catalyst have a significant effect.

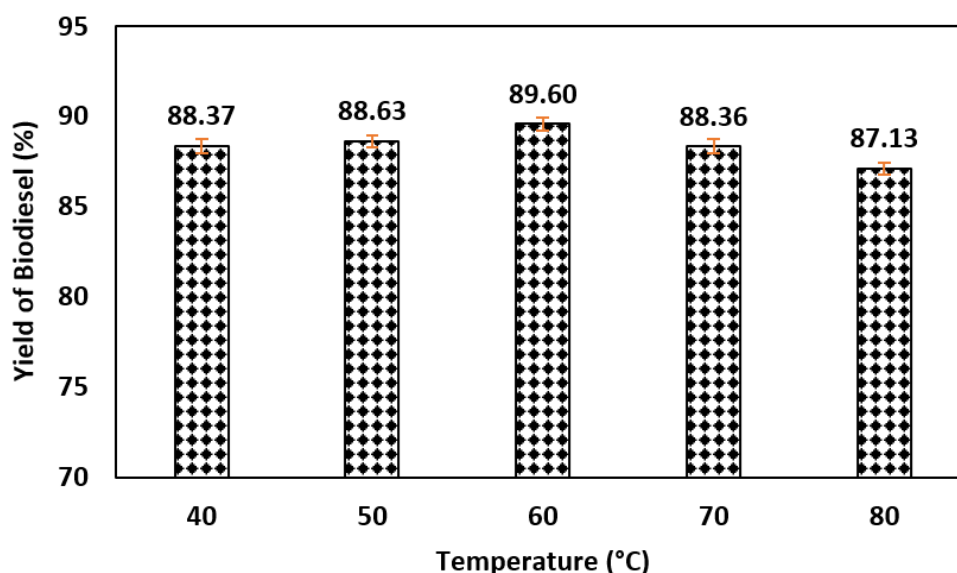


Fig. 8. Biodiesel yield at various reaction temperatures

Reaction time is essential in the transesterification reaction because it affects the kinetic energy. The greater the kinetic energy, the greater the collision between molecules and activation energy. However, a reaction carried out for too long will cause a shift in equilibrium to the left, resulting in a decrease in the yield obtained. Figure 9 shows that the longer the reaction time, the higher the yield. However, in this study, it is necessary to increase the reaction time to obtain the optimum transesterification reaction time conditions because the graph continues to increase at a reaction time of 90 minutes. The significance values for the normality test, homogeneity test, and ANOVA output were 0.662, 0.298, and 0.001, respectively. It means that the data is normally distributed and homogeneous, and temperature variations significantly affect the biodiesel yield produced.

Based on the stoichiometric reaction between triglycerides and methanol to produce biodiesel, 3 moles of methanol must react with 1 mole of triglycerides to produce 1 mole of biodiesel. It means that the ratio of moles of methanol and oil is 3:1, according to the theory. In this study, adding methanol decreased the biodiesel yield (Figure 10), allegedly because the excess amount of methanol caused competition between methanol molecules in reacting with triglycerides, thus disrupting collisions between particles in producing biodiesel. The significance values for the normality test, homogeneity test, and ANOVA output were 0.474, 0.544, and 0.01, respectively. It means that the data is normally distributed and homogeneous, and temperature variations significantly affect the biodiesel yield produced.

Based on Figure 11, it can be seen that the biodiesel yield obtained increased with the increase in the mass of the $\text{Fe}_3\text{O}_4\text{-PSH}$ catalyst. The addition of catalyst mass affected the biodiesel yield obtained, with the highest biodiesel yield at a $\text{Fe}_3\text{O}_4\text{-PSH}$ catalyst mass of 0.50% (w/w). It is in line with the function of the catalyst as a substance that can help accelerate the reaction rate by reducing the activation energy. In addition, the large surface area of the $\text{Fe}_3\text{O}_4\text{-PSH}$ magnetic catalyst is one of

the supporting factors for the amount of biodiesel produced. The large catalyst surface can increase collisions or interactions with particles so that catalytic activity increases and yield increases [20]. The yield of biodiesel produced using the Fe₃O₄-PSH catalyst is higher than the homogeneous KOH catalyst, so the hypothesis stating that heterogeneous catalysts can increase biodiesel yield has been proven. The significance values for the normality test, homogeneity test and ANOVA output were 0.421, 0.641, and 0.01, respectively. It means that the data is normally distributed and homogeneous, and temperature variations significantly affect the biodiesel yield produced

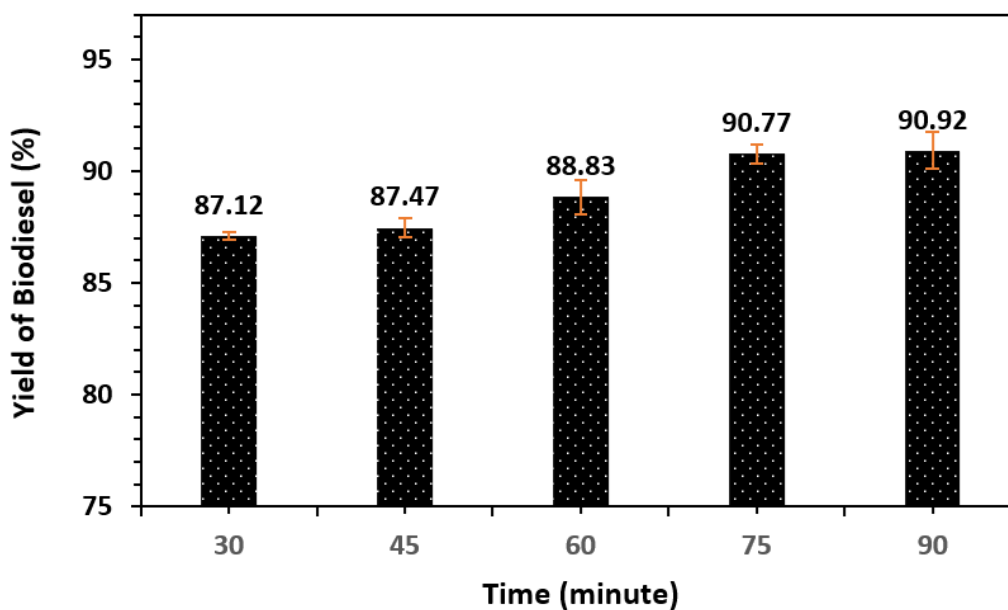


Fig. 9. Biodiesel yield at various reaction times

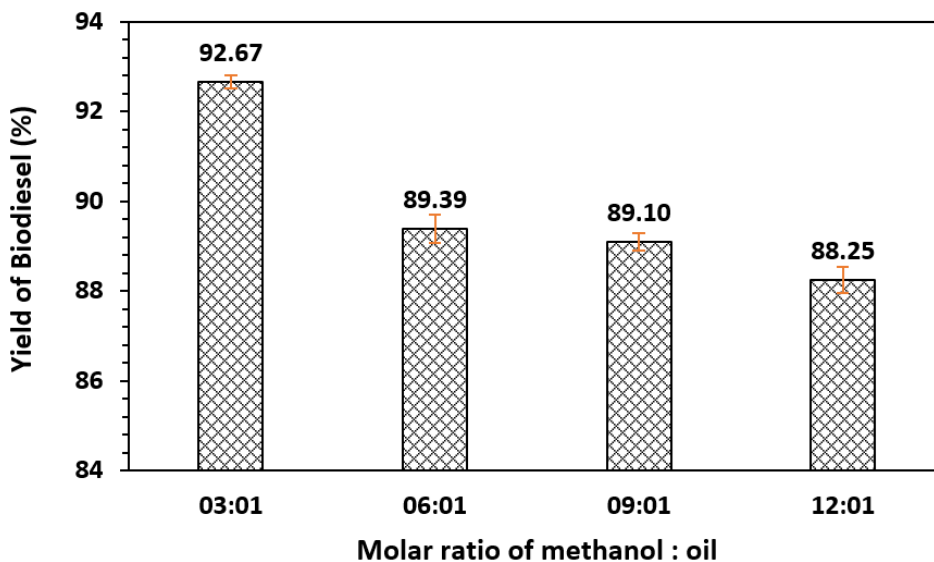


Fig. 10. Biodiesel yield at various molar ratios of methanol: oil

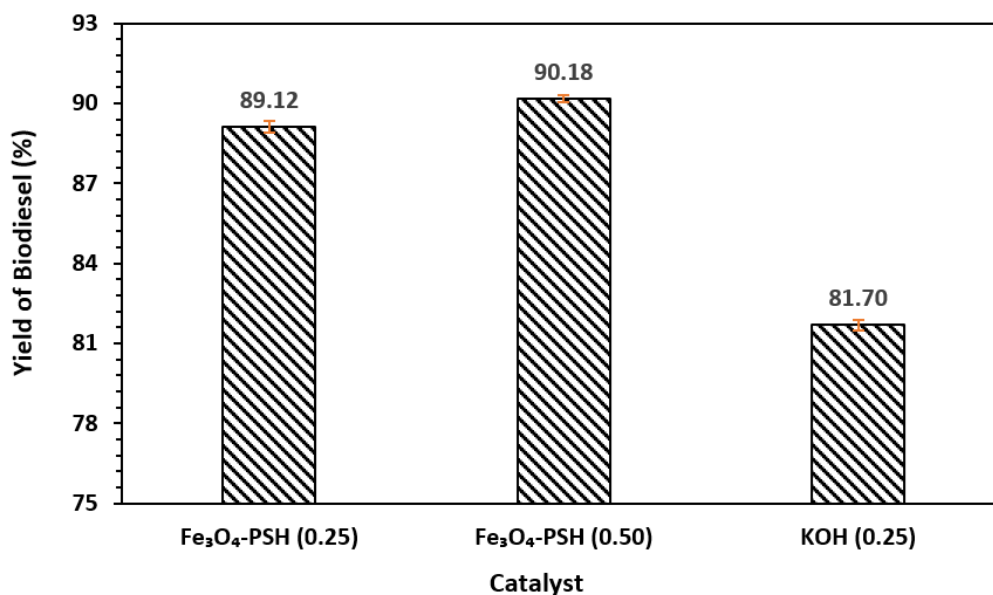


Fig. 11. Biodiesel yield at various catalyst concentrations

3.2.3 Chemical composition of biodiesel

The chemical composition of biodiesel produced using Fe₃O₄-PSH was analyzed using GC-MS. Figure 12 shows that biodiesel contains various chemical compounds. However, the six most significant compositions are methyl ester stearate (42.31%), methyl ester palmitate (39.70%), linoleic acid methyl ester (10.74%), methyl ester stearate (4.12%) and myristate methyl ester (1.00%).

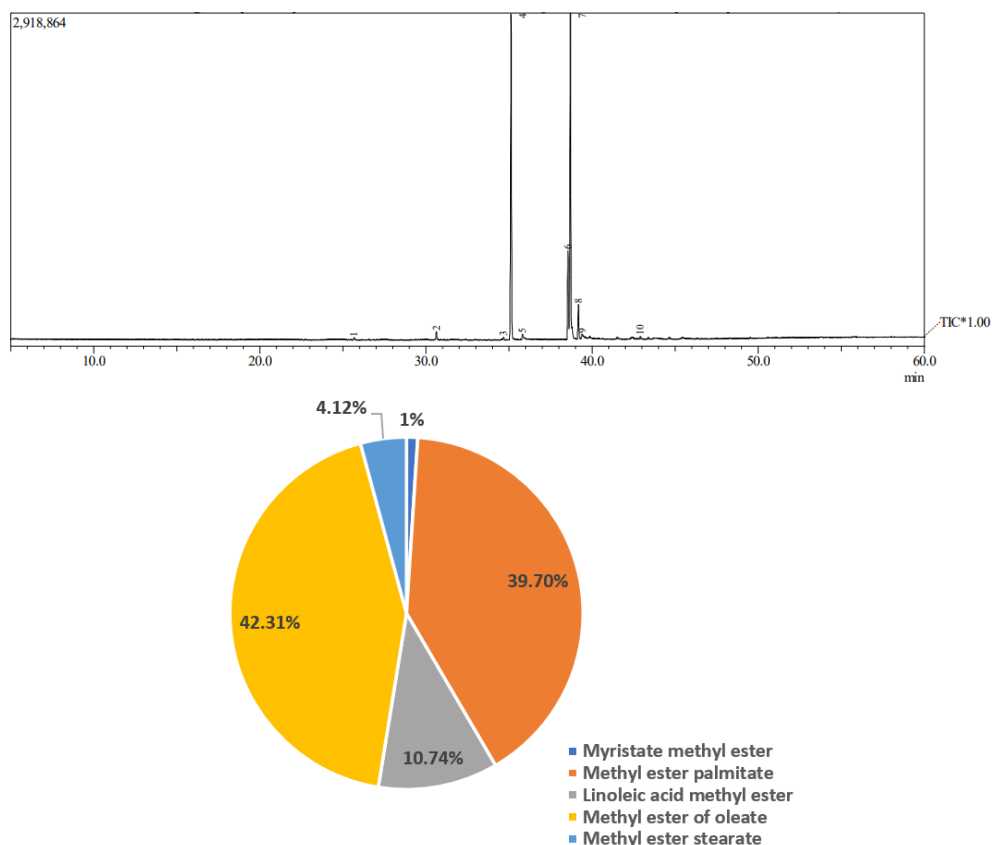


Fig. 12. Chemical composition analysis of biodiesel using GC-MS

3.2.4 Biodiesel Quality Test

The quality of biodiesel produced using the Fe₃O₄-PSH catalyst analyzed in this study includes the parameters of density, water content, acid number, flash point, and calorific value. Quality tests were carried out at various temperatures, times, methanol: water ratios, and catalyst concentrations. The results of the quality test are presented in Table 2. The quality test results indicate that the biodiesel produced has met SNI 7182:2015 and ASTM D7467. From Table 2, it can be seen that the water content for biodiesel produced using a KOH catalyst does not meet SNI 7182:2015. The catalyst performance is quite good compared to other catalysts, as presented in Table 3.

Table 2
Biodiesel Quality Test

Reaction Conditions				Biodiesel Quality Test Results				
Temperature (°C)	Time (minute)	Molar ratio of methanol : oil	Catalyst (% b/b oil)	Density (kg/m ³) 850-890 (SNI 7182:2015)	Water content (%) < 0.05 (SNI 7182:2015)	Acid number (mg KOH/g) < 0.80 (SNI 7182:2015)	Flash point (°C) min 100 (SNI 7182:2015)	Calorific value (MJ/kg) 37 (ASTM D7467)
40	60	6:1	Fe ₃ O ₄ -PSH (0.25)	875,1	0,33	0,39	148	37,86
50	60	6:1	Fe ₃ O ₄ -PSC (0.25)	875,1	0,33	0,39	148	37,86
60	60	6:1	Fe ₃ O ₄ -PSH (0.25)	870,74	0,26	0,39	150	37,86
70	60	6:1	Fe ₃ O ₄ -PSH (0.25)	870,5	0,26	0,39	150	37,86
80	60	6:1	Fe ₃ O ₄ -PSH (0.25)	870,5	0,23	0,59	154	37,86
60	30	6:1	Fe ₃ O ₄ -PSH (0.25)	870,5	0,15	0,78	148	37,86
60	45	6:1	Fe ₃ O ₄ -PSH (0.25)	870,5	0,15	0,59	148	37,86
60	60	6:1	Fe ₃ O ₄ -PSH (0.25)	870,74	0,26	0,59	150	37,86
60	75	6:1	Fe ₃ O ₄ -PSH (0.25)	872,7	0,23	0,59	150	37,86
60	90	6:1	Fe ₃ O ₄ -PSH (0.25)	872,7	0,23	0,78	138	37,86
60	60	3:1	Fe ₃ O ₄ -PSH (0.25)	878,95	0,33	0,23	148	37,86
60	60	6:1	Fe ₃ O ₄ -PSH (0.25)	870,74	0,26	0,59	150	37,86
60	60	9:1	Fe ₃ O ₄ -PSH (0.25)	870,35	0,41	0,70	144	37,86
60	60	12:1	Fe ₃ O ₄ -PSH (0.25)	871,15	0,47	0,78	140	37,59
60	60	3:1	Fe ₃ O ₄ -PSH (0.25)	878,95	0,41	0,23	150	37,86
60	60	3:1	KOH (0.25)	862,8	2,1	0,55	150	37,86
60	60	3:1	Fe ₃ O ₄ -PSH (0.50)	870,74	0,26	0,39	150	37,86

Table 3

The comparison of the quantity and quality of biodiesel with different types of catalysts

Feedstock	Catalyst	Optimum conditions (Temperature; Time; Methanol: Oil; Catalyst wt.%;	Yield (%)	FAME	Reference
Chicken fat	CaO/ CuFe ₂ O ₄	30; 70 °C; 15:1; 3 %	94.52%	-	[22]
Chicken fat	CaO	120, 65 °C, 10:1; 1.5%	93 %.	Oleate acid (43.31 %); Stearate acid (23.48 %)	[1]
Waste cooking oil	Amberlite 62i	180; 60 °C; 6:1; 20 %	82 %	-	[23]
Waste cooking oil	Cat.TS-7	120; 60 °C; 9:1; 1.5%	93%	-	[24]
Waste cooking oil	CaSO ₄ /Fe ₂ O ₃	45; 70 °C; 5:1; 4%	89.94	-	[25]
Waste cooking oil	Fe ₃ O ₄ -PSH	60; 60 °C; 3:1; 0.5%	92.67 %	methyl ester stearate (42.31%), methyl ester palmitate (39.70%), linoleic acid methyl ester (10.74%), methyl ester stearate (4.12%) and myristate methyl ester (1.00%).	This research

4. Conclusions

This study successfully synthesized Fe₃O₄-PSH catalyst using PSH peel extract. Fe₃O₄-PSH catalyst was used to produce biodiesel under various conditions to obtain optimum production conditions. The best optimum conditions for biodiesel production with Fe₃O₄-PSH catalyst occurred at a reaction temperature of 60 °C, a mole ratio of methanol: oil = 3: 1, and a concentration of Fe₃O₄-PSH catalyst of 0.5 M. The quality test results showed that the synthesized biodiesel had met SNI 7182: 2015 and ASTM D7467. Emission testing of the biodiesel produced is needed to evaluate the environmental impact. In addition, a life cycle assessment is also needed to provide a more comprehensive view of the proposed method's sustainability.

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