Heterogeneous Catalyst of Oxidative Desulfurization for Reducing Sulfur Content in Indonesia Biosolar

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Abstract: Sulfur content in Indonesian diesel fuel is still very high, so it needs to be reduced to meet international regulations and improve the efficiency of diesel engines. This paper aims to reduce sulfur content on the fuel using Oxidative Desulfurization (ODS) method. Hydrogen peroxide was used as an oxidant with various heterogeneous catalysts in the ODS process. There are 3 heterogeneous catalysts used in this work, namely activated carbon-formic acid, Co-Fe/γ-Al$_2$O$_3$ and MoO$_3$/γ-Al$_2$O$_3$. These three catalysts have been used in other studies and succeeded in significantly reducing sulfur content in various diesel models. The ODS reaction was carried out using a batch stirring reactor under several reaction conditions and followed by centrifugation to separate the diesel and the oxidated sulfur compounds. As the results, Co-Fe/γ-Al$_2$O$_3$ catalysts gave the highest percentage of 9.8% desulfurization with reaction conditions of 5 g catalyst, the molar ratio of H$_2$O$_2$ to sulfur = 120, and 25 mL of Biosolar.

1 INTRODUCTION

Diesel fuel in Indonesia is still far from international regulatory standards because it has a high sulfur content. Pertamina DEX has the lowest sulfur content of 300 ppm, Dexlite has a sulfur content of 1,200 ppm, and Biosolar. Meanwhile, based on the international standard EURO VI, the sulfur content in diesel fuel is 0.001% by mass (10 ppm).

Due to this high sulfur content, the sulfur oxide content can be oxidized to sulfuric/sulfuric acid which causes corrosion and wear and tear on vehicle engine parts. In addition, sulfur oxides can affect the efficiency of the catalyst system in the exhaust gas pipeline. Therefore, desulfurization technology is needed to reduce sulfur content in diesel fuel.

One alternative process to reduce sulfur content is the Oxidative Desulfurization (ODS) method. Many researchers have reduced the sulfur content by oxidizing dibenzothiophene to sulfoxide and sulfone, because dibenzothiophene is the sulfur compound with the most content (Joskić et al., 2014). Compared with HDS, ODS has several advantages, such as using atmospheric pressure operating conditions, relatively low temperature up to 100°C, low cost, high selectivity, no use of expensive hydrogen, and potential for desulfurization of sterically hindered sulfides such as 4,6-dimethyldibenzothiophene (DMDBT) (Murata et al., 2004).

Oxidative Desulfurization (ODS) process was used in this paper, which has been extensively studied in reducing sulfur content but has not yet been applied to Indonesia Biosolar fuel (Nikolas et al., 2021). Thus, further research is needed regarding the use of ODS technology in Indonesian Biodiesel (B-30) fuel to reduce its sulfur content.

In the ODS process, hydrogen peroxide (H$_2$O$_2$) is the most used oxidant because of its affordable cost, availability, and producing oxygen and water by-products that are not harmful to the environment (Shang et al., 2003). Therefore, this paper used an oxidant in the form of hydrogen peroxide (H2O2). For the use of solvents, according to Jia et al., (2011), the solvent in the ODS process can cause problems in the separation between the biodiesel oil and the solvent phase with the loss of some amount of the oil phase. So, it is recommended that in the ODS process using a solid catalyst, the use of solvents should be avoided. And separation can be carried out using centrifugation.

Based on the phase, catalyst in the desulfurization process is divided into two types, which are homogeneous catalysts and heterogeneous catalysts. However, the homogeneous catalyst is difficult to separate from the reaction because they have the same

phase. This gives the heterogeneous catalyst an advantage in the ODS process because separating the catalyst from the reaction is easier. In addition, heterogeneous catalysts have a large surface area which can increase the interaction of the material with the catalyst (Haghighi and Gooneh-Farahani, 2020). In this paper, heterogeneous catalysts are used in ODS process with three different types of catalysts.

Due to their high ability in oxidation reactions, various transition metals have been investigated as a catalyst in the desulfurization process (Rajendran et al., 2020). Nazmi et al. (2020) conducted a paper to reduce sulfur content using Co-Fe/γ-Al$_2$O$_3$ catalyst with the ODS method in n-octane diesel. The research was conducted with various compositions of catalyst, oxidant, and oxidation time which succeeded to reduced 93% sulfur content in 30 min. Therefore, in this paper, Co-Fe/γ-Al$_2$O$_3$ was used as a catalyst with hydrogen peroxide as an oxidant to reduce sulfur content in Biosolar (B-30). Jia et al. (2011) have investigated transition metals in reducing sulfur content with ODS using a MoO$_3$/γ-Al$_2$O$_3$ catalyst in n-octane model diesel. The sulfur content of diesel fuel can be reduced up to 97.2% with an oxidation time of 10 min. Therefore, in the present investigation, MoO$_3$/γ-Al$_2$O$_3$ is used as a catalyst in the ODS process to reduce sulfur content in Biosolar. One paper of the ODS process using activated carbon-formic acid (AC-HCOOH) catalyst and oxidizing H$_2$O$_2$ resulted in a desulfurization percentage of 98% in the n-octane model diesel (Yu et al., 2005). Therefore, in this paper, the ODS process was carried out to determine the catalyst with the best desulfurization results, and the sulfur content was determined by ASTM-FTIR absorbance correlation.

2 EXPERIMENTAL SECTION

2.1 Materials

Biosolar (B-30) was obtained from PT. Pertamina with 360.9 ppm. Activated carbon Jacobi AquaSorb® 2000, is granular coal-based, and technical-grade formic acid were obtained commercially. Ammonium heptamolybdate tetrahydrate as a precursor for MoO$_3$/γ-Al$_2$O$_3$ catalyst was purchased commercially. Iron (III) nitrate nanohydrate and cobalt (II) hexahydrate as precursors for Co-Fe/γ-Al$_2$O$_3$ catalyst was purchased commercially. γ-Al$_2$O$_3$ as the support catalyst precursor of MoO$_3$/γ-Al$_2$O$_3$ and Co-Fe/γ-Al$_2$O$_3$ was also purchased commercially. Hydrogen peroxide (30 wt %, technical-grade reagent) was purchased commercially.

2.2 Catalyst Preparation

Co-Fe/γ-Al$_2$O$_3$ and MoO$_3$/γ-Al$_2$O$_3$ catalysts were prepared by the incipient wetness impregnation method. According to the loading of Co-Fe and MoO$_3$, an appropriate amount of cobalt (II) nitrate hexahydrate, iron (III) nitrate nonahydrate, and ammonium heptamolybdate tetrahydrate were dissolved in distilled water and then slowly added to γ-Al$_2$O$_3$ at ambient temperature. The mixture was dried in an open vessel with stirring at 373 K for 2 h to evaporate the excess water. The precursor of Co-Fe/γ-Al$_2$O$_3$ was calcined at 773 K for 5 h, while the precursor of MoO$_3$/γ-Al$_2$O$_3$ was calcined for 6 h to obtain a catalyst.

Activated carbon Jacobi AquaSorb® 2000 was prepared using 10g of activated carbon soaked and washed repeatedly in deionized water. This process aims to neutralize and clean the activated carbon sample until the water looks clean and not cloudy. Then, the sample was filtered to separate the solids from water and dried in an oven at 120°C for 6 hours to remove the water content.

2.3 Oxidative Desulfurization of Biosolar

A typical procedure was as follows. All ODS reactions were conducted in a 150 mL beaker glass, equipped with a magnetic stirrer bar and fitted with the hot plate. For the ODS process using activated carbon catalyst, a mixture of commercial diesel oil (100 mL), 30 wt% hydrogen peroxide (3.4 mL), H$_2$O (5 mL), formic acid (1 mL), and AC Jacobi (0.7 g) was stirred at 750 rpm in a beaker glass under various oxidation temperatures (30°C, 60°C, and 70°C) for 60 min. For ODS runs of MoO$_3$/γ-Al$_2$O$_3$ catalyst, 1 g catalyst, and 25 mL diesel oil were stirred until the reaction temperature reached the desired temperature (40°C, 60°C, and 70°C), and then 1.5 mL hydrogen peroxide (molar ratio of H$_2$O$_2$/s = 120/1) were added to the beaker glass and stirred for 30 min. For ODS runs of Co-Fe/γ-Al$_2$O$_3$, catalyst weight variation (1 g, 3 g, and 5 g) and 25 mL diesel oil were stirred until the reaction reached the desired temperature (30°C, 50°C, and 70°C), and then 1.5 mL hydrogen peroxide (molar ratio of H$_2$O$_2$/s = 120/1) were added to the beaker glass and stirred for 30 min.

The beaker glass was fitted with a condenser, a mechanical stirrer bar, and a thermometer. The oxidized oil and adsorbent were separated by centrifugation.

3 METHOD OF ANALYSIS

The separated oil phase was analyzed using Fourier-Transform Infrared Spectroscopy (FTIR) to determine the total sulfur content. According to Az-Zahra et al. (2022), FTIR method can identify and measure total sulfur content quantitatively and qualitatively in diesel fuel with 62% accuracy, towards ASTM D-4294 method. FTIR method as a sulfur detector does not require sophisticated sample preparation and expensive costs.

The wavenumber that shows strong absorption of sulfur is at 1169 cm^{-1}. Meanwhile, the wavenumber that shows the presence of aromatic range is at 1458 cm^{-1}, which shows the characteristic of Biosolar since 70% of Biosolar contains diesel oil that formed of the aromatic ring (Az-Zahra et al., 2022). According to Coates (2006), in the wavenumber range of 1200-1100 cm^{-1}, it states the presence of sulfone compounds in the presence of S=O strain. While the aromatic ring group C=C-C will appear at wavenumbers 1510-1450 cm^{-1} with non-polar properties and is suitable as a basis for identifying diesel oil. Bonds with a wave number of about 1458 cm^{-1} are C-H bonds with bending vibrations. The results obtained from comparing the two peaks were calibrated using a model made from the combined FTIR data and the sulfur contents from ASTM-D test results. The absorbance of 1169 cm^{-1} and 1458 cm^{-1} resulted in IR Spectrum is defined as W_{1169} and W_{1458}. So, the equation for determining the sulfur content in each sample is obtained as follows:

$$\text{Total Sulfur Content (ppm)} = \frac{\left(\frac{W_{1169}}{W_{1458}}\right)}{0,001847} \quad (1)$$

For removal rates of sulfur were calculated as follows

$$\text{Desulfurization (\%)} = \frac{TS_0 - TS_t}{TS_0} \times 100\% \quad (2)$$

Where TS_0 is the initial total sulfur content of diesel fuel and TS_t is the final total sulfur content of diesel fuel after ODS reaction.

4 RESULTS AND DISCUSSION

4.1 Catalyst Characterization

4.1.1 Characterization of Co-Fe/γ-Al$_2$O$_3$ and MoO$_3$/γ-Al$_2$O$_3$

Co-Fe/γ-Al$_2$O$_3$ and MoO$_3$/γ-Al$_2$O$_3$ catalysts were characterized by X-Ray fluorescence (XRF) using S2 PUMA Bruker to identify the elemental composition of the catalyst. In this paper, XRF analysis was conducted at Pusat Riset Kimia Maju, Puspiptek, Serpong. Table 1 shows the composition of the XRF analysis results with a comparison of the theoretical composition. XRF analysis was only carried out on one sample of each catalyst to prove the results of the catalyst preparation.

Table 1: Comparison between theoretical and actual composition with XRF analysis.

Catalyst	Compound	Theoretical composition (wt.%)	Actual composition (wt.%)*
Co-Fe/γ-Al$_2$O$_3$	CoO	3.32	2.1
	Fe$_2$O$_3$	24.63	10.4
	Al$_2$O$_3$	72.05	83.1
MoO$_3$/γ-Al$_2$O$_3$	MoO$_3$	20	20.26
	Al$_2$O$_3$	80	75.76

*Based on XRF analysis.

The actual composition in Table 1 shows that the catalyst preparation succeeded in obtaining the desired compound, but the results of the percentage composition in XRF analysis slightly differed from the theoretical composition in catalyst preparation.

4.1.2 Characterization of Activated Carbon

The characterization of activated carbon carried out in this paper aims to determine the surface area and total pore volume using the Brunauer Emmett-Teller (BET) Quantachrome Quadrasorb-Evo Surface Area and Pore Size Analyzer method. This characterization was conducted at ILRC UI Laboratory. The results of the characterization are shown in Table 2. In theory, the higher the surface area of activated carbon, the greater the ability of activated carbon to adsorb polar compounds (Jamilatun and Setyawan, 2014). And with the increased surface area and a decrease in the average pore radius on activated carbon increased the total pore volume of activated carbon (Irma, Wahyuni, and Zahara, 2015).

Table 2: Structural parameters of the activated carbons.

Catalyst	BET surface area (m^2/g)	Total pore volume (cm^3/g)
Activated carbon Jacobi AquaSorb® 2000	775.3	0.52

* Based on BET analysis.

4.2 Evaluation of Various Heterogeneous Catalyst Systems

4.2.1 Comparison of Heterogeneous Catalysts

The results of the ODS process in this paper were compared to determine the catalyst's performance. The results can be seen in Table 3 below using the same solar model and oxidant. The percentage of desulfurization produced in this paper uses the Indonesian Biosolar (B-30), which has a total sulfur content that is too complex and not specific.

Table 3: Desulfurization results in ods process with various heterogeneous catalysts.

Catalyst	T (°C)	t (min)	Desulfurization (%)
Co-Fe/γ-Al$_2$O$_3$	50	30	9.8
MoO$_3$/γ-Al$_2$O$_3$	60	30	7.7
AC-HCOOH	30	60	7.6

Table 3 shows that using a catalyst can reduce sulfur content in the ODS process, but there are differences in the desulfurization percentage of the heterogeneous catalyst used. The results exhibit that the highest removal of sulfur is 9.8% using Co-Fe/γ-Al$_2$O$_3$ catalyst until 325.6 ppm. Co-Fe/γ-Al$_2$O$_3$ catalyst has three components, namely Fe$_2$O$_3$ as an active core, CoO as the promoter, and γ-Al$_2$O$_3$ as a support. The combination of Fe$_2$O$_3$ and CoO can increase the reaction activity, meanwhile γ-Al$_2$O$_3$ has a large surface area and high pores so that it can increases the performance of catalytic reactions. A screening of several transition metal-oxide catalysts showed that alumina-supported Fe-Co catalyst performed the highest oxidative desulfurization (Nazmi et al, 2020).

The percentage of sulfur removal using MoO$_3$/γ-Al$_2$O$_3$ catalyst reached 7.7% with sulfur content from 360.9 ppm to 333.3 ppm. The MoO$_3$/γ-Al$_2$O$_3$ catalyst only has two catalyst components, MoO$_3$ as an active core and γ-Al$_2$O$_3$ as a support. Molybdenum metal is used as an active core which can increase the activity and selectivity of the reaction, while γ-Al$_2$O$_3$ is used as a support because it has a high surface area and pore volume so that it can increase catalytic activity (Argyle and Bartholomew, 2015).

For the AC-HCOOH catalyst, it produces a desulfurization percentage of 7.6% from 360.9 ppm to 333.7 ppm. According to previous researchers, the oxidation of DBT with the AC-HCOOH catalytic system is better than using only the HCOOH catalyst. Activated carbon is used as a phase-transfer adsorption medium, because it is porous and has a large surface area for the reaction contact area. Large surface area for the reaction contact area. The presence of formic acid in the catalyst can increase the oxidation reaction in activated carbon by catalyzing the formation of performic acid and results in high conversion to DBT-Sulfone (Yu et al., 2004).

4.2.2 Effect of Oxidation Temperature

The oxidation of Biosolar was carried out with hydrogen peroxide catalyzed by various heterogeneous catalysts under various oxidation temperatures.

Figure 1 shows the sulfur removal at 30°C, 50°C, and 70°C using 5 g Co-Fe/γ-Al$_2$O$_3$ in 25 mL Biosolar with an oxidation time of 30 min. The removal of sulfur content increased from 30°C until it reached the highest condition at 50°C. Then when the temperature was increased to 70°C, the percentage of sulfur removal was decrease. This can happen because each catalyst has an optimum and equilibrium point in working (Pahlevi et al, 2015). In this operation, 50°C is the optimum condition.
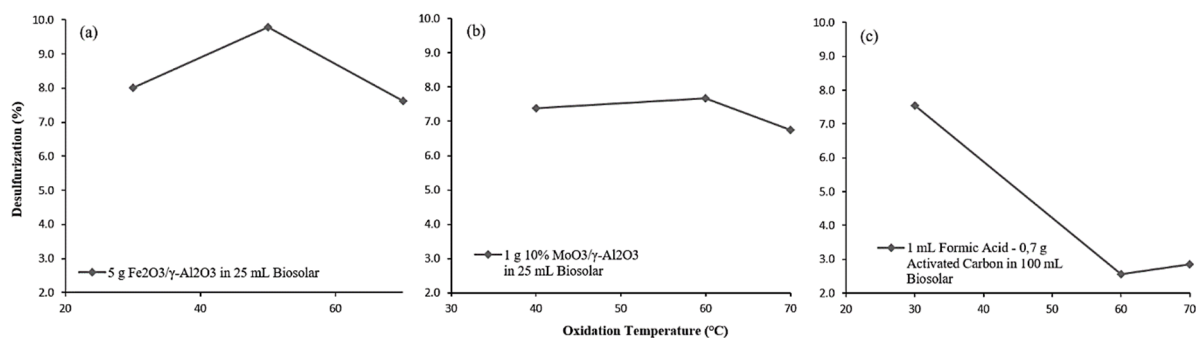

Figure 1: Effect of temperature on the ODS catalyzed by (a) Co-Fe/γ-Al$_2$O$_3$, (b) MoO$_3$/γ-Al$_2$O$_3$, (c) AC-HCOOH.

The effect of reaction temperature on sulfur removal was also carried out on MoO$_3$/γ-Al$_2$O$_3$ catalyst. Figure 1 shows the sulfur removal at 40°C, 60°C, and 70°C using 1 g 10% MoO$_3$/γ-Al$_2$O$_3$ in 25 mL Biosolar with an oxidation time of 30 min. The sulfur removal increased from 40°C until it reached the highest condition at 60°C. After that, the sulfur removal decreased at 70°C.

For the ODS process catalyzed by AC-HCOOH, as shown in Figure 1 that the percentage of desulfurization decreases as the reaction temperature increases. With an oxidation time of 60 min, the composition of the AC-HCOOH catalyst of 0.7 g-1 mL in 100 mL Biosolar will experience a decrease in the percentage of desulfurization as the oxidation temperature increases from 30°C to 60°C. However, the desulfurization percent increased again at an oxidation temperature of 60°C to 70°C. This shows that the use of high temperatures in the ODS process in this paper can reduce the performance of the oxidation results.

This is in accordance with the research conducted by Tugrul Albayrak & Ali Gurkaynak (2012), where the ODS process with hydrogen peroxide has been carried out using a formic acid catalyst, and the desulfurization is greater at 30°C compared to 40°C. Low temperatures and low formic acid-H$_2$O$_2$ amounts are more efficient because peroxyformic acid, produced in situ by H$_2$O$_2$ and formic acid, decomposes slowly at 30 °C, thus increasing the reaction conversion. Oxidation temperatures that are too high can reduce oxidation yields due to the oxidation degradation of H$_2$O$_2$ (Houda et al., 2018). According to W. Mohammed and R. K. Almilly in 2015, the temperature is the most significant factor because it shows the interaction between temperature and the H$_2$O$_2$/diesel fuel ratio. The excess oxidant is required at high temperatures due to the loss of H$_2$O$_2$ due to thermal decomposition.

4.3 Analysis of IR Spectrum

FTIR Spectroscopy produces an infrared spectrum from the absorption of a sample for further use in identifying compounds and functional groups. The infrared spectrum produces peaks that indicate the absorbance value of the sample at various wavenumbers.

The absorbance of sulfur compounds is represented by a wavenumber of 1169 cm^{-1}, indicating the presence of sulfur compounds. Figure 2 shows the differences in absorbance levels for wavenumber 1169 cm^{-1} of Biosolar and Biosolar after ODS. The Biosolar spectrum has a peak with a higher
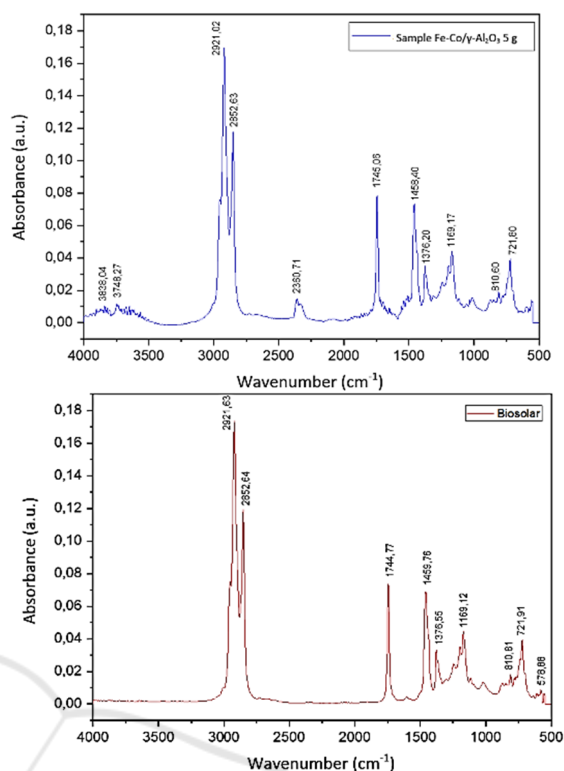

Figure 2: IR spectrum from Biosolar without ODS process and Biosolar after treated by ODS process.

absorbance value, which is 0.045 compared to 0.044 for Biosolar after ODS. The difference in absorbance levels proves that Biosolar has higher sulfur compounds. The spectrum from Biosolar also has a wavenumber of 579 cm^{-1}, which is not present in the Biosolar after ODS. The wavenumber is a disulfide shown in the wavenumber range of 705-570 cm^{-1} (Coates, 2000). This proves that the ODS process can remove disulfide compounds which are sulfur-derived compounds.

5 CONCLUSIONS

From the three heterogeneous catalysts, which are Co-Fe/γ-Al$_2$O$_3$, MoO$_3$/γ-Al$_2$O$_3$, and AC-HCOOH, catalyst Co-Fe/γ-Al$_2$O$_3$ gave the highest percentage of desulfurization in the ODS process with 9.8% with 5 g of catalyst, 1.5 mL of hydrogen peroxide (molar ratio of H2O2/s = 120/1), and 25 mL of Biosolar. The best-operating conditions for this mixture are at a temperature of 50°C with an oxidation time of 30 minutes. The results showed that many factors could affect the performance of the catalysts, including temperature.

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