

Residue Oil Desulfurization Using Oxidation and Extraction Method

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ABSTRACT

This study successfully improved the performance of oxidative desulfurization method to reduce sulfur content from residue oil (condensate) with modifications of oxidation and extraction steps repeated for several stages. Residue oil used in this study contained 386.2 ppm of initial sulfur content. In oxidation process, H₂O₂ as oxidizer and acid as catalyst were used within temperature range of 30–60 °C and for interval time from 30 to 120 min. In extraction process, various alcohol solvents (methanol, ethanol, and propanol) were used at the temperature of 30 °C for 30 min toward every ratio of residue oil to solvent (v/v). The best reducing sulfur result was 35.9 ppm or 90.7% desulfurization, and it was achieved after 4 recursively extractions using ethanol as the solvent. This study successfully reduced sulfur content in residue oil to meet the international standard (< 50 ppm).

Keywords: extraction; oxidation; oxidative desulfurization; residue oil

ABSTRAK

Studi ini telah berhasil meningkatkan performa dari metode desulfurisasi oksidatif untuk menurunkan kadar sulfur pada minyak residu (kondensat) dengan modifikasi pada proses oksidasi dan ekstraksi yang dilakukan beberapa tahap. Minyak residu yang digunakan pada studi ini mengandung sulfur dengan konsentrasi awal sebesar 386,6 ppm. Pada proses oksidasi, H₂O₂ sebagai pengoksidasi dan katalis asam digunakan pada rentang suhu dari 30–60 °C dan rentang waktu dari 30 sampai 120 menit. Pada proses ekstraksi, berbagai macam alkohol (metanol, etanol dan propanol) digunakan sebagai pelarut pada suhu 30 °C dan waktu 30 menit untuk setiap rasio minyak residu terhadap solvent (v/v). Hasil terbaik pada penurunan kadar sulfur yang didapatkan adalah sebesar 35,9 ppm atau 90,7% desulfurisasi. Hasil tersebut dicapai setelah melakukan 4 tahapan ekstraksi secara berulang dengan menggunakan etanol sebagai pelarut. Studi ini telah berhasil mencapai target untuk menurunkan kadar sulfur dalam minyak residu untuk memenuhi standar internasional (< 50 ppm).

Kata Kunci: ekstraksi; oksidasi; desulfurisasi oksidatif; minyak residu

INTRODUCTION

In general, natural gas is hydrocarbon gas mixture which mostly consist of light component (C₁-C₄), heavy component (C₅⁺) and small amount of carbon dioxide, nitrogen, hydrogen sulfide, or helium. Natural gas is one of Indonesia's natural resources that can be processed into various products of energy such as liquefied natural gas (LNG). Residue oil (condensate) is byproduct which obtained from LNG production. The residue oil has quality similar to crude oil. In the daily life, residue oil is widely used as a solvent in industrial processing, raw material in petrochemical industry and fuel for vehicles. Because of its advantages, residue oil is an important commodity in Indonesia.

The major component of residue oil is Addi. In addition to major component, residue oil contains contaminant such as carbon dioxide and sulfur. Because

sulfur is an acid gas, it can harm the environment and convert to SO_x compound in combustion process. SO_x is the main source of acid rain and air pollution [1-3]. High sulfur content is not only affecting the environment, but also cause adverse effects on the quality petroleum and natural gas products as it may reduce API gravity, decrease octane rating and release toxic hydrogen sulfide [4-5].

Sulfur removal in liquid fuels such as kerosene, gasoline, diesel fuel, and residue oil is relatively more difficult than that in natural gas, therefore the proper method of removing sulfur content in liquid fuels is important. Conventional method to remove sulfur usually involving hydrogen gas at high operating condition and using expensive catalyst [6-7]. The process of sulfur removal in liquid fuels is generally done by Hydrodesulfurization (HDS) process. Hydrodesulfurization is a standard catalytic process for

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removing sulfur from petroleum products that have been developed since 1930. In this process, the crude oil is heated and then mixed with hydrogen and catalyst to convert sulfur content into hydrogen sulfide. To meet the standard sulfur content of less than 50 ppm, the hydrodesulfurization method should be operate at high temperature (300–400 °C) and pressure (30–130 atm) and require highly active catalysts. The capital and operating cost is high to be operate in the industry [8]. Therefore, various desulfurization techniques have been developed for the removal of sulfur in liquid fuels including microwaves, bacteria, electricity, adsorption, and extraction [3,9]. The most prospective technique in terms of both economic and technical process is Oxidative Desulfurization [10].

The oxidative desulfurization (ODS) is a promising and alternative method for sulfur removal because it has several advantages over the hydrodesulfurization method. The advantage of the this method is the sulfur removal reaction takes place at low temperature and pressure so that it does not require high energy, has high selectivity, and low investment cost. This method also does not require expensive materials such as hydrogen. Hence oxidative desulfurization method could provide sulfur removal solutions on liquid fuels effectively in technical process and economy aspect [11]. The ODS method was applied during kerosene desulfurization using H_2O_2 - CH_3COOH system with constant stirring for several hours [12-13]. The solvents used were acetonitrile, methanol, ethanol, and propanol has potential to be utilized in desulfurization. But recent study showed a new technology to take advantage of ionic liquids (ILs) as desulfurization agent [1-2,5,14-15]. It is because ILs has advantage in its properties, such as thermal stability and volatility, which can be designed with specific purposes [16]. The major drawback to utilize ILs is very high-cost materials.

In addition to hydrodesulfurization and oxidative desulfurization methods, the biodesulfurization (BDS) method can be used for sulfur removal in various fuel [3,17-18]. Biodesulfurization method is a biological sulfur removal process which the sulfur content in liquid fuel

will be consumed by microorganisms. This method required microorganisms and its media breeding to maintain the viability of these microorganisms [19].

An alternative method of sulfur removal that has the best prospects in terms of process and economy is oxidative desulfurization. In this study, the performance of the oxidative desulfurization method using technical grade solvents were investigated and applied on residual oil samples. It is expected to eliminate sulfur content in the sample to meet liquid fuel standards (< 50 ppm). This study also develope ingenious technology to be applied in industry.

EXPERIMENTAL SECTION

Materials

Residue oil used in this study contained 386.2 ppm of sulfur. The residue oil was obtained from Indonesia's oil and gas company. Hydrogen peroxide oxidizer was used with acetic acid and formic acid as catalysts in the oxidation stage. For the extraction stage, solvents methanol, ethanol, and propanol were used. The specifications of each material obtained from the supplier were shown in Table 1.

Procedure

Oxidative desulfurization is an oxidation process of residue oil with a hydrogen peroxide as oxidizer (H_2O_2) using acid catalyst to form an easily extractable compound by increasing the relative polarity. Oxidant compounds provide oxygen atoms bonding sulfur in thiols, sulfides, disulfides, and thiophene to form sulfoxides and sulfones then followed by liquid-liquid extraction to remove further sulfur content. The procedure used in this study consisted of two steps, namely the oxidation step and extraction step. The modification done in this research was performed multiple stage of extraction. The block diagram of oxidative desulfurization method in this experiment was shown in Fig.1.

Table 1. Specification of material used in the experiment

No	Material	Grade	Molecular Weight (g/mol)	Density (g/cm ³)	Normal Boiling Point (°C)	Polarity Index
1.	Hydrogen/Peroxide	35%	34.01	1.4463	150.2	-
2.	Acetic Acid	p.a	60.05	1.0490	118.1	-
3.	Formic Acid	p.a	46.03	1.2200	100.8	-
4.	Methanol	50%	32.13	0.7918	64.7	5.1
5.	Ethanol	50%	46.07	0.7890	78.4	5.2
6.	Propanol	50%	60.10	0.8034	97.1	4

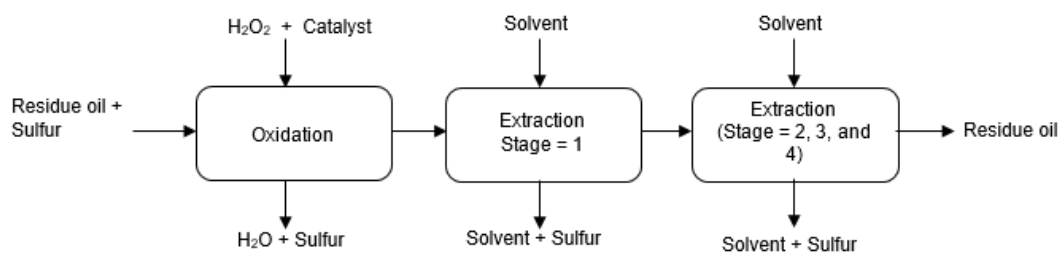


Fig 1. Oxidative desulfurization block diagram

Oxidation step

The oxidation step was carried out by mixing 100 mL of oil residue and catalysts (acetic acid or formic acid) with residue oil/catalyst (v/v) ratio of 10, 15, 25, and 30 into a flatbed flask equipped with a thermometer. The mixture was heated over a hot plate equipped with a magnetic stirrer to various temperature variables (40, 50, and 60 °C) with constant stirring. After constant temperature, hydrogen peroxide was added with the ratio of residue oil/H₂O₂ (v/v) of 10, 25, 40, and 50 to the mixture. The reaction time was set for 120 min, and after that the stirring was stopped. During the oxidation step, 5 mL of sample was taken for every 30 min; then it was analyzed using XRF (X-Ray Fluorescence) method. The two phases formed from the oxidation results were completely separated between the aqueous and the residual oil phases in the separating funnel. The residue oil phase was analyzed using XRF method to measure sulfur content of the oxidized residue oil. The oxidation experimental process was repeated 4 times to obtain precise data.

Extraction step

The residue oil obtained from oxidation step subsequently then to be continued with the liquid-liquid extraction step by mixing the oxidized residue oil with solvent (methanol, ethanol, and propanol) into the flatbed flask with the residue oil/solvent ratio (v/v) of 0.5, 1, 2, and 4. The mixture was heated to temperature of 30 °C accompanied by constant stirring for 30 min. Then the raffinate and extract phase was separated using separation funnel for 30 min. The sample was taken from both solvents to be analyzed the sulfur content by XRF method. In this study, the effect of extraction stage on the percentage of desulfurization was studied; therefore the residual oil was extracted 4 times with the similar procedure.

RESULT AND DISCUSSION

In this study, variation in the ratio of residue oil/H₂O₂ and residue oil/catalyst (v/v), variation in oxidation temperature and variation of oxidation time was conducted to determine parameters in the desulfurization oxidation method. In addition, to better

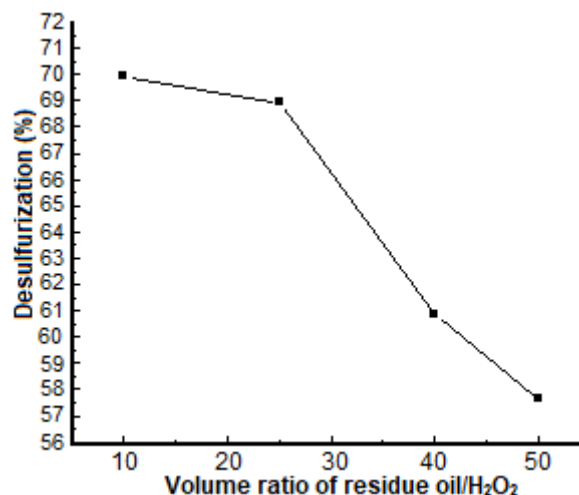
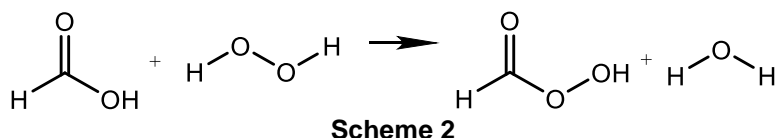
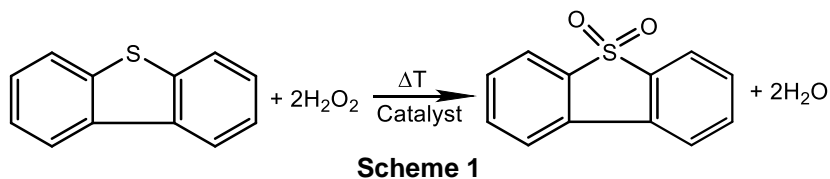


Fig 2. The effect of volume ratio residue oil/H₂O₂ ratio to percent desulfurization with operating residue oil volume 100 mL, ratio of residue oil/acetic acid 10 (v/v), oxidation temperature 50 °C, oxidation time 120 min, ratio of residue oil/H₂O₂ 10; 25; 40; 50 (v/v)]

understand the performance of solvent in the extraction stage, this study conducted variations of the ratio of residual oil/solvent (v/v) and number of extraction stage.

Effect of Oxidizing Agent and Catalyst

During the oxidation step of this experiment, the sulfur compound would be converted to sulfone and sulfoxide. From Fig. 2, it was shown the percentage of desulfurization on various residue oil/H₂O₂ ratio. At 10 (v/v), the sulfur removal was 69.93%. However, it was less efficient to compare to 25 (v/v) residue oil/H₂O₂ ratio, which resulting 68.92% desulfurization. It could be explained that the increase of H₂O₂ oxidizer may increase the percentage of desulfurization. Hydrogen peroxide will donate oxygen radicals and react with acetic acid catalysts to form peracid radicals (R-CH₃COO·). This radical will be offset with sulfur substances in the residue of oil so it will be more polar. Furthermore, these compounds will form two layers of residue oil and oxidized sulfur compounds. The oxidation



reaction takes place as Scheme 1. The byproduct of hydrogen peroxide decomposition is water, therefore, it more environmentally friendly.

Study conducted by Dehkordi et al., (2009) shown that the oxidation rate of H_2O_2 is strongly influenced by oxidation temperature when the oxidation temperature of $25\text{ }^\circ\text{C}$ [12]. The increase amount of H_2O_2 oxidizer were resulting decreasing of percent desulfurization while at $60\text{ }^\circ\text{C}$ the addition of H_2O_2 were resulting increasing of percentage desulfurization. Similar results were obtained in this study, at a temperature of $50\text{ }^\circ\text{C}$ the addition of oxidizing amounts led to an increase in percentage of desulfurization. Therefore, for subsequent research used the ratio of residual oil/ H_2O_2 25 (v/v).

Acetic acid and formic acid are carboxylic acids that often use for the removal of sulfuric materials in liquid fuels such as gasoline, kerosene, and diesel oil. The effect of acetic acid and formic acid catalyst are observed by the ratio of residue oil/catalyst (v/v) used (10, 15, 25, and 30 (v/v) for each acetic acid and formic acid). The results is shown in Fig. 3 and it is shown that the percentage of desulfurization is higher using acetic acid as catalyst than using formic acid. With residue oil/formic acid ratio is 10 (v/v), the desulfurization by using acetic acid is 71.9% whereas with formic acid 64.6% as shown in Fig. 3. In other words, the addition of 10 (v/v) ratio of residue oil/acetic acid reduce sulfur content from the initial value 386.2 to 108.7 ppm. If there is no addition of catalyst, it will require large number of oxidizers, long reaction time and the percent of desulfurization produced is not high, 13.7%. Therefore, for further research, the composition of residue oil/acetic acid ratio (v/v) is 10.

From Fig. 3, it can be explained that the addition of the catalyst (the ratio of the residue oil/catalyst decrease) increasing the percentage of desulfurization, the catalyst acts to accelerate the reaction by decreasing the activation energy. Acetic acid as the catalyst will react with hydrogen peroxide to form peracetic radical ($R\text{-CH}_3\text{COO}\cdot$). This compound is more reactive and has high oxidizing ability compared to performic radicals (CH_2O_3) [20]. The reaction of performic radical formation takes place as Scheme 2.

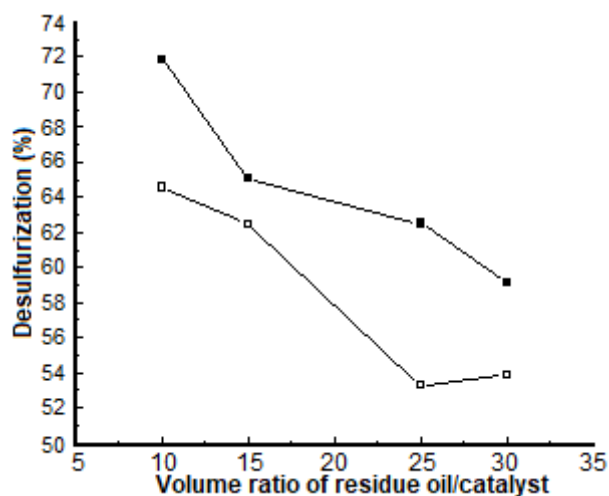


Fig 3. The effect of volume ratio residue oil/catalyst acetic acid (■) or formic acid (□) to percent desulfurization with operating condition residue oil volume 100 mL, oxidation temperature $40\text{ }^\circ\text{C}$, oxidation time 120 min, ratio of residue oil/ H_2O_2 25 (v/v), ratio of residue oil/catalyst 10; 15; 25; 30 (v/v)

However, performic radicals are more stable which is less reactive than peracid. Therefore, peracid radicals more easily oxidize the residual oil to sulfone and sulfoxide so that the percent of desulfurization of acetic acid is higher than formic acid [21].

Effect of Oxidation Temperature

Based on the analysis results shown in Fig. 4, it can be explained that the highest percentage of desulfurization at temperature $50\text{ }^\circ\text{C}$ which is 77.6% desulfurization, so that the sulfur content decreases from 386.26 to 86.37 ppm.

The increasing of oxidation temperature causes an increase in percentage of desulfurization, where the temperature changes from 30, 40 and $50\text{ }^\circ\text{C}$ cause a significant 45, 73.5, and 77.6% desulfurization, respectively. High temperatures accelerate the oxidation reaction by increasing the rate of oxidation reaction. However, at a temperature of $60\text{ }^\circ\text{C}$, there was

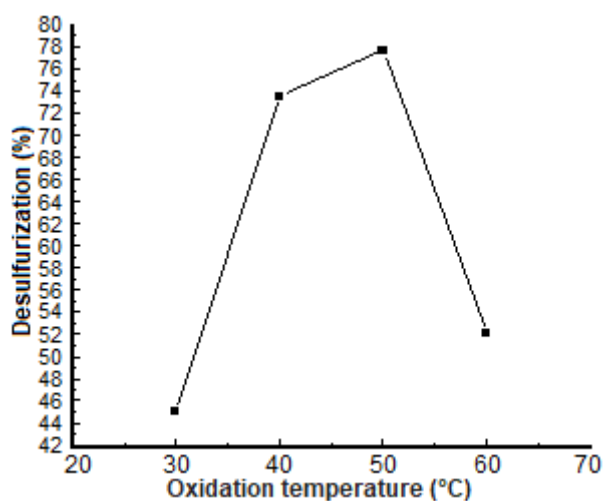


Fig 4. The effect of oxidation temperature to percent desulfurization with operating condition residue oil volume 100 mL, oxidation time 120 min, ratio of residue oil/H₂O₂ 25 (v/v), ratio of residue oil/acetic acid 10 (v/v), oxidation temperature 30, 40, 50 and 60 °C

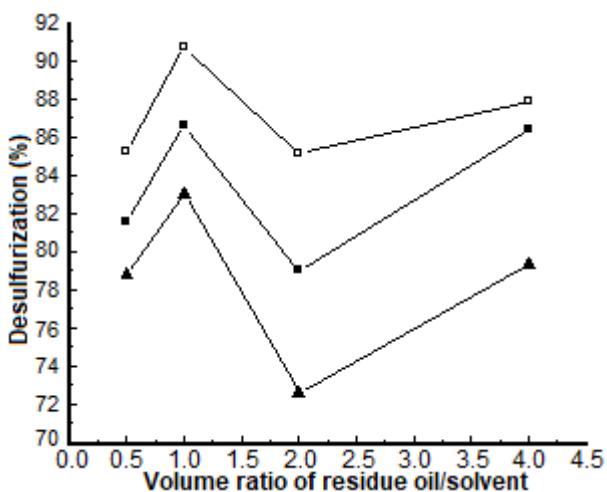


Fig 6. The effect of solvent methanol (-■-), ethanol (-□-) or propanol (-▲-) used in the extraction process to percent desulfurization with operating condition residue oil volume 100 mL, oxidation time 120 min, oxidation temperature 50 °C, ratio of residue oil/H₂O₂ 25 (v/v), ratio of residue oil/acetic acid 10 (v/v), extraction temperature 30 °C, extraction time 30 min, ratio of residue oil/solvent 0.5; 1; 2; 4 (v/v)

a decline of desulfurization to 52.1% as shown in Fig. 4. This is because the oxidation temperature approaching to 80 °C where the H₂O₂ compound start to be degraded.

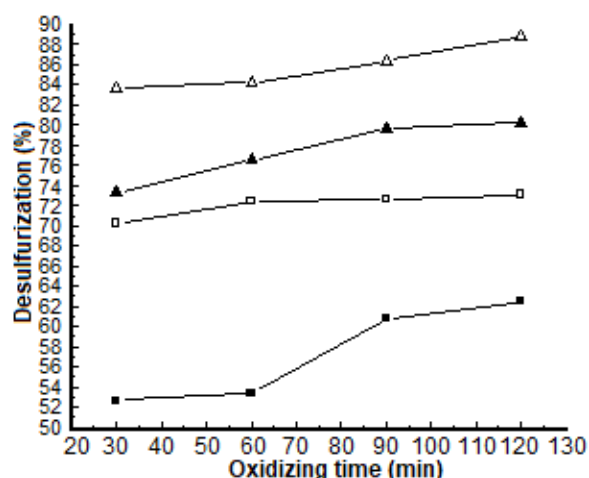


Fig 5. The effect of oxidation time to percent desulfurization at stage 1 (-■-), 2 (-□-), 3 (-▲-), and 4 (-Δ-) with operating condition residue oil volume 100 mL, oxidation temperature 50 °C, ratio of residue oil/H₂O₂ 25 (v/v), ratio of residue oil/acetic acid 10 (v/v), oxidation time 30; 60; 90; 120 min

Effect of Oxidation Time

The time of oxidation gives a subtle difference in percentage of desulfurization. Based on Fig. 5, it could be explained that the percent of desulfurization increased start from 30 to 90 min. After that, it is continuously constant until the 120 min for every oxidation stage. The highest percentage of desulfurization was obtained at the stage 4 and 120 min oxidation process, which was 88.9% desulfurization. Therefore, to increase percent desulfurization can be done with several methods such as applying oxidation stage. This is because the oxidation stage can increase the effectiveness of oxidation reactions (increased mass transfer). Although in this study oxidation process is done until the 4 stages but each stage takes 2 h hence the desulfurization process lasts longer. With economic considerations, oxidation stages are inefficient and difficult to apply in industry.

Effect of Solvent Used in the Extraction Process

Methanol, ethanol, and propanol are used in the extraction step as solvent because they are selective against sulfones, sulfoxides, and water that are polar in order to be separated by decantation. From the analysis results shown in Fig. 6, it can be explained that ethanol has the highest desulfurization among the

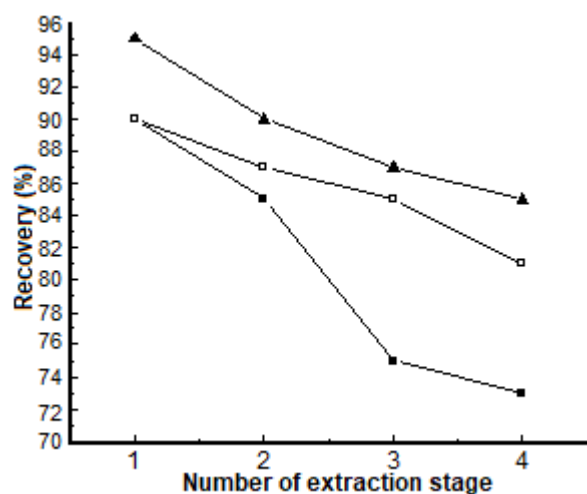


Fig 7. The effect of solvent methanol (-■-), ethanol (-□-) or propanol (-▲-) used in the extraction process to percent solvent recovery [residue oil volume 100 mL, oxidation time 120 min, oxidation temperature 50 °C, ratio of residue oil/H₂O₂ 25 (v/v), ratio of residue oil/acetic acid 10 (v/v), extraction temperature 30 °C, extraction time 30 min, ratio of residue oil/solvent 1 (v/v)]

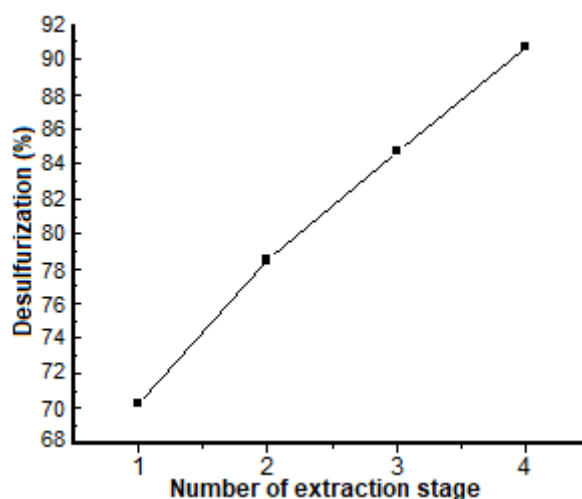


Fig 8. The effect of number of extraction stage to percent desulfurization [residue oil volume 100 mL, oxidation time 120 min, oxidation temperature 50 °C, ratio of residue oil/H₂O₂ 25 (v/v), ratio of residue oil/acetic acid 10 (v/v), extraction temperature 30 °C, extraction time 30 min, ratio of residue oil/ ethanol 1 (v/v)]

Table 2. Solvent effectiveness

Solvent	Desulfurization* (%)	Residue oil Recovery* (%)	Solvent Effectiveness
Methanol	86.6	73	5.6
Ethanol	90.7	81	9
Propanol	83.3	85	5

*[residue oil volume 100 mL, oxidation time 120 min, oxidation temperature 50 °C, ratio of residue oil/H₂O₂ 25 (v/v), ratio of residue oil/acetic acid 10 (v/v), extraction temperature 30 °C, extraction time 30 min, ratio of residue oil/solvent 1 (v/v), extraction stage 4]

solvent used in this study. At the ratio of residue oil/solvent 1 (v/v), the desulfurization using ethanol, methanol, and propanol are 90.7, 86.6 and 83.3%, respectively. For every residue oil/solvent ratio, ethanol showed better percentage of desulfurization than methanol and propanol.

Based on the experiment results, the ability of ethanol to remove sulfur content in the residue oil is superior. This is because the polarity of the ethanol index of 5.2 is greater than that of methanol of 5.1 and propanol of 4. The larger the polarity index, the greater the ability to extract the oxidized sulfur content. In addition, ethanol has better capability than methanol and propanol is also due to the density. A large difference in density between the solvent and residue oil make the separation process easier to separate. The density of methanol of 0.7818 g/cm³ is similar to the residue oil of 0.7340 g/cm³ hence it is difficult to separate.

In addition to desulfurization capability, the solvents used in extraction must have a high percent of residue oil recovery so that it is not reducing the amount of

product. Based on Fig. 7, it could be explained that in the ratio of residual oil/solvent, solvent propanol has the highest recovery percentage of 85% when compared with ethanol and methanol at each extraction stage. Methanol has the lowest recovery percent because it has a density similar to the residual oil. Therefore, to determine the best solvent, this study used parameters that can represent the effect on percentage of desulfurization and percent recovery of residue oil. The parameters for selecting the best solvent are determined by the percentage of desulfurization (D) and the best percent recovery of residual oil (R) can be represented by solvent effectiveness (E). Solvent effectiveness (E) is calculated using equation 1.

$$E = \frac{R}{100 - D} \quad (1)$$

The calculated value of solvent effectiveness (E) for methanol, ethanol, and propanol is shown in Table 2. Based on Table 2, it could be concluded that ethanol has the highest effectiveness which is 9.0 compared to

methanol 5.6 and propanol 5.0. Therefore, it could be concluded that in this study the best solvent for the extraction stage is ethanol.

The number of stage used in the extraction step was observed. By using ethanol as the best solvent, and 100 mL residue oil, residue oil/H₂O₂ ratio 25 (v/v), residue oil/catalyst ratio 10 (v/v), oxidation temperature 50 °C, 2 h oxidation time, extraction temperature 30 °C, 30 min extracting time and residue oil/solvent ratio of 1 (v/v), the effect of extraction stages was presented in Fig. 8. The modifications extraction step that repeated for several stages gives positive results. The percent desulfurization is increase within the number of stage, so that the modification give opportunity to obtain more environmental friendly residue oil.

CONCLUSION

From the data results and analysis of this study, the oxidative desulfurization with extraction step modifications could be applied to remove sulfur contaminant in residue oil. This process is applicable in industrial process. The sulfur content of the oil residue (condensate) has been reduced from total sulfur 386.26 to 35.94 ppm (desulfurization 90.7%) by oxidative desulfurization method with oxidation step operating condition were residue oil/H₂O₂ ratio of 25 (v/v) and residue oil/acetic acid ratio of 30 (v/v) and oxidation temperature of 50 °C. After oxidation step, it was then continued to extraction step with residue oil/ethanol ratio of 1 (v/v) repeated 4 stage with. The use of acetic acid catalysts was better than formic acid on sulfur removal in residue oil (condensate), where percent of desulfurization for acetic acid was 71.9% while formic acid only reaches 64.6%. Ethanol showed the highest yield at the extraction stage compared to methanol and propanol, with the best percentage of desulfurization produced at 90.7% while methanol and propanol 86.6% and 83.3%, respectively.

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