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Oxidative desulfurization of light fuel oil by using hydrogen peroxide in the presence of acetic acid catalyst

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ABSTRACT

Hydrogen peroxide as an oxidant agent was used for desulfurization of a petroleum hydrocarbon fraction (C_{10} - C_{22}) with boiling range of 175-375 °C, (light fuel oil) in the presence of acetic acid as catalyst. The oxidation was performed in an ultrasonic bath. It is found that increasing the amount of hydrogen peroxide lead to increase the oxidation rate and so desulfurization efficiency, while the amount of acetic acid has low effect on the desulfurization performance. Residence time and temperature of ultrasonic bath affect significantly on oxidation process. Sulfuric component produced by oxidation process were extracted using methanol as solvent. The number of washing has a considerable effect on the extraction of sulfuric component. So that, the increasing of the number of washing from 1 to 4 times, sulfur content of the hydrocarbon decreased from about 0.554 to 0.154 wt. %. The stability of hydrocarbon during desulfurization was also studied by comparing gas chromatograms of it before and after desulfurization. It was found that the composition of hydrocarbon fraction has no considerable change due to sulfur removal.

Keywords: Oxidation; Desulfurization; Ultrasonic; Hydrogen peroxide; Acetic acid; Light fuel oil.

1. Introduction

Sulfur is the most abundant element in crude oil after carbon and hydrogen. The average content of sulfur in crude oil varies from 0.03 to 7.89 percent [1]. The main sources of sulfur in crude oil are organic compounds such as thiols, sulfides and thiophenes. Crude oil with higher viscosity (20 to 1000 cp) and density (20 °API to 10 °API) has a high content of complicated sulfuric compounds. Non-cyclic aliphatic sulfides (thioethers) and cyclic sulfides are easily removed in hydrodesulfurization process or by heat treatment [3] but sulfur in aromatic compounds such as thiophenes and benzologs such as benzo-thiophenes, dibenzothiophenes and benzo-naphto-thiophenes that have an alkyl group at 4 and/or positions, because of steric hindrance at the active sites are more resistant in front of hydro-desulfurization or heat treatment [4]. Some other methods that applied to overcome this limitation include biological desulfurization [5], extraction desulfurization, [6,7] adsorption desulfurization [8, 9] and oxidative desulfurization [10-12].

*Corresponding author. E-mail address: mhvakili@iaush.ac.ir (M.H. Vakili) Sulfur removal from crude oil is an important issue in petroleum refineries and related industries and is carried out because of some reasons such as: Sulfur removal limits corrosion along refining process, transmission and consumer equipment such as vehicles, Sulfur removal from fuels prevents air pollution and also sulfur dioxide emission in environment and sulfur removal removes odor from oil products.

Different methods are proposed to remove sulfur from crude oil and petroleum products. The most important strategies are hydro-desulfurization (HDS) [13], extractive desulfurization [6,7], oxidative desulfurization (ODS) [10-12], bio-desulfurization (BDS) [14], alkylation based desulfurization[15], chlorinolysis based desulfurization [16], supercritical water based desulfurization (SCW) [17] and adsorptive desulfurization [18-20].

Hydro-desulfurization is the most common method to decrease sulfur content of petroleum fractions which has the oldest background in refining industries, accordingly a lot of researches have been carried out on this process and its catalysts [21]. In 1903, Wilhelm Normann, a German chemist, found that catalytic hydrogenation could be used to convert unsaturated glycerides in the liquid phase into saturated compounds. In the 1950, the first noble metal catalytic reforming process was commercialized. At the same time, the catalytic hydrodesulfurization of the naphtha feed to such reformers was also commercialized.

A deep hydrodesulfurization process should be carrying out to have diesel fuels with low levels of sulfur (<< 15 ppm), that needs more active catalysts, higher operating pressure and temperature and more consumption of hydrogen. It is because of high refractory of residual sulfur compound after hydrodesulfurization, due to steric hindrance of the alkyl substituents to which the sulfur atom is bound [22]. Therefore, this inherently expensive process, become more costly [23].

To achieve environmental goals, other desulfurization methods have been considered and assessed. Among these methods, oxidative desulfurization, as a promising method is significantly taken into consideration [21]. Oxidative desulfurization in comparison with hydrodesulfurization has major differences. This method doesn't need to hydrogen and also uses oxidants like hydrogen peroxide aqueous solution [24]. Oxidative desulfurization operates in low temperature and pressure while hydro-desulfurization needs higher operating temperature and pressure and as a result, needs higher investment and operating costs. Other advantages of the oxidation desulfurization process versus the hydro-desulfurization could be the reduction of catalyst costs, environmental pollution and greenhouse gas emissions. One of the main reasons for the researchers' attention to this method is its ability to remove more complex sulfur compounds, such as benzo-thiophenes and its derivatives [24, 25].

Oxidative desulfurization method contains chemical reactions between an oxidant agent and sulfur. In general, this process has two successive and separated steps. The first step is oxidation of sulfur in which nature of sulfuric compound is changed into their relating sulfoxides and sulfones and the second step is sulfur removal [24, 26]. This process is directly related to performance of these steps. In this method, sulfur removal is based on polarization of sulfuric compounds as shown in eq.1 [27,28]:

$$2R - SH + \frac{1}{2}O_2 \to R - S - S - R + H_2O$$
(1)

In recent years, many researchers have used hydrogen peroxide as oxidants [29-34]. The reason for prevalence of the use of hydrogen peroxide is due to its low cost, no contamination, no severe corrosion and its industrially availability. It is noteworthy that hydrogen peroxide is a slow oxidizer in the absence of the catalyst, therefore, use of the catalyst along with hydrogen peroxide oxidant is essential [35]. The catalysts used for this system are divided into two major categories of organic acid catalysts such as: acetic acid [36] and formic acid [37] and polyoxometalates (POMs) such as: $[PyPS]_3(NH_4)_3Mo_7O_{24}$ [38] and $[(C_8H_{17})_3NCH_3]_3PMo_{12}O_{40}$ [39].

The low rate of oxidant diffusion into the reaction mixture is a major limitation of oxidative desulfurization. To overcome this problem, ultrasonic waves can be used, because act as reaction catalyst and significantly enhances the rate of reaction kinetic and mass transfer diffusion within reaction mixture due to the cavitation effects [40]. Furthermore, ultrasonic waves can cause severe mixing within the reaction mixture and eliminate need for agitators [41].

The desulfurization by extraction method depends on the solubility of the sulfuric organic compounds in a solvent. In the mixer tank, the crude oil and solvent are mixed, and the sulfuric organic compounds are extracted in the solvent because of their higher solubility in the solvent. Then, the solvent is separated from the hydrocarbon in a distillation column and thus recycled and can be reused [42].

Extractive desulfurization has been considered due to its easy industrial application, no need to hydrogen and proper process conditions. The mixing tank operates at temperature close to the room temperature [42]. In this process, crude oil does not participate in chemical conversions and this is a completely physical extraction process. An appropriate solvent for an effective extractive desulfurization should has low solubility in oil and high solubility in sulfur compounds. The efficiency of different types of solvents, such as acetone, ethanol and polyethylene are varied from 50% to 90%, depending on the number of extractions [43, 44]. The viscosity of oil and solvent should be as low as possible in order to better mixing and extraction. In order to separate solvent and sulfur compounds by distillation, they should have a relatively high boiling point difference.

Light fuel oil is a petroleum product that is produced from combination of kerosene and gas oil and is used for heating purposes. Desulfurization of this hydrocarbon fraction has received less attention in the literatures. Molaei Dehkordi et al. investigated the oxidative desulfurization of a simulated light fuel oil (dibenzothiophene and benzothiophene in toluene). They found that oxidation is an effective method for desulfurization of simulated solution [45]

The goal of the present paper is studying the combination of several inexpensive and simple methods which are oxidation, extraction, ultrasonic waves and ultraviolet ray on the susceptibility of reducing the sulfur content in an industrial light fuel oil $(C_{10}-C_{22})$ sample. Moreover, determine the optimum values of various used operating conditions including amount of hydrogen peroxide, acetic acid, residence time for sample in ultrasonic bath and its temperature, the number of washing times with methanol and effect of ultraviolet ray.

2. Experimental

2.1. Material

Hydrogen peroxide 30%, acetic acid (glacial) and methanol (>>99.5%) were purchased from Merck Co., Germany. A hydrocarbon fraction, light fuel oil, was supplied from Isfahan Refinery Co., Iran. The specifications of this fraction were determined and are shown in Table 1S.

2.2. Experimental method for sulfur removal

An experimental method for sulfur removal from hydrocarbon fraction was conducted in two steps as fallowing:

In the first step (oxidation) desired values of acetic acid(1, 3, 5, 7 ml) and hydrogen peroxide (1, 3, 5, 7 ml) were added to 30 ml of light fuel oil with 1.0100 wt% sulfur content and mixed by shaking at room temperature for about 2 min. Then, in order to carry out oxidation reaction, it was placed in the ultrasonic bath. Different values of bath temperature (30, 50, 70 and 90 °C) and residence time (10, 20, 30, 40 and 50 min) were studied. Some experiments were also performed under ultraviolet ray to investigate the effect of it on the oxidation.

In the second step (extraction) oxidation products were poured into a decanter and 30 ml methanol added to it. It was shaken vigorously for about 5 min. it was then allowed to separate into two phases. Hydrocarbon phase was separated, and its sulfur content measured with the sulfur analyzer set (Rigaku model NEX QC, USA) according to ASTM D4294. All tests were carried out at atmospheric pressure.

The hydrocarbon mixture was washed with methanol for 1 to 4 times. In the first washing, contrary to expectation, the methanol phase is placed under the hydrocarbon phase, because the methanol phase is combined with the remained hydrogen peroxide and the acetic acid which results in increasing of phase density. This phenomenon doesn't repeat in the next washings and the methanol phase is placed above the sample. A schematic of experimental steps is shown in Fig. 1S.

Hydrocarbon stability against the desulfurization was also studied. For this purpose, 2 ml acid acetic and 5 ml hydrogen peroxide were added to 30 ml light fuel oil and desulfurized in accordance with the above-mentioned procedure. In this experiment, residence time and temperature of ultrasonic bath were set equal to 40 min and 50 °C respectively. Gas chromatography were performed on the hydrocarbon before and after desulfurization process by Agilent 7890B GC System.

3. Results and Discussion

3.1. Effect of hydrogen peroxide

The effect of hydrogen peroxide as an oxidant agent on the performance of oxidation reaction and sulfur removal was investigated by conducting a series of experiments with 2 ml of acetic acid and 1, 3, 5 and 7 ml of hydrogen peroxide, where in, the ultrasonic time and temperature were 10 minutes and 40 °C, respectively. The results are shown in Fig. 1.

As shown in Fig. 1, by adding 1, 3, 5 and 7 ml of hydrogen peroxide, sulfur content of the sample was reduced by 11.42%, 16.65%, 38.11% and 25.71% respectively. It shows that by increasing the amount of hydrogen peroxide, the amount of sulfur removal would increase due to higher oxidation rate. During the oxidation reaction two species is formed, hydroxyl radical and peracetic acid, which have the most effect on oxidation process. Increasing the hydrogen peroxide (molar ratio of H₂O₂ to sulfur species) in the reaction media, lead to increase the formation of these two species. Therefore, more sulfuric compounds convert to their corresponding sulfoxides and sulfones. Haghighat Mamaghani et. al. found the similar result in desulfurization of dibenzothiophene with hydrogen peroxide and formic acid [46].

But reduction in the efficiency of sulfur removal in the value more than 5 ml of hydrogen peroxide may be related to reduction in acetic acid concentration due to dilution which in turn limit peracetic acid concentration as it mentioned by Jalili and Sobati [47]. This could also be due to decomposition and loss of hydrogen peroxide into oxygen molecules [46].



Fig. 1. Effect of hydrogen peroxide on the performance of oxidation reaction.

3.2. Effect of acetic acid

A series of experiments were conducted to study the effect of acid acetic amount on the sulfur removal efficiency, with 5 ml of hydrogen peroxide and 1, 3, 5 and 7 ml of acetic acid. The ultrasonic residence time and temperature were 10 minutes and 40 °C, respectively. The results are shown in Fig. 2.

It is observed from Fig. 2 that by increasing the amount of acetic acid, sulfur content of the sample was reduced with a slight slope. It may be due to the increase in peracetic acid concentration that form by the reaction of formic acid and hydrogen peroxide, which is a strong electrophilic agent and accelerates the oxidation reaction [47]. Idin and Ilkilic investigated the effect of acetic acid and formic acid on desulfurization of a fuel using hydrogen peroxide. They found that increasing the acid/H₂O₂ in oxidation media (for both formic and acetic acid) increase the efficiency of sulfur removal that in consistence with the present work [48].

Acetic acid acts as a catalyst and is used to acidify the reaction environment. It can be said that every 2 ml of acetic acid can reduce one percent of sulfur. Obviously, this increase is not economically feasible, so the optimum amount of acetic acid is 1 ml.

3.3. Effect of residence time in ultrasonic bath

In order to find an effective reaction Time, some experiments with 5 ml of hydrogen peroxide and 1 ml of acetic acid were conducted. The reaction mixture was placed in the ultrasonic bath at 40 °C for 10, 20, 30, 40 and 50 minutes. Fig. 3 shows the variation of sulfur removal percent with residence time.

As shown in Fig. 3, with 10, 20, 30, 40 and 50 min of residence time in ultrasonic bath, sulfur content of the sample was reduced by 34.54%, 37.90%, 42.30%, 48.74% and 49% respectively. It can be concluded that by increasing the residence time in the ultrasonic bath, desulfurization would be performed better because



Fig. 2. Effect of the amount of acetic acid.



Fig. 3. Effect of residence time in ultrasonic bath.

ultrasonic waves improve oxidation under mild temperature, thus there is no need to conduct the reaction at high temperatures. However, the exposure to ultrasonic waves for more than 40 minutes has a negligible effect on reaction, because the reaction has closed to the equilibrium state. Thus, the best time is 40 minutes. The effect of ultrasound time on sulfur removal efficiency depend on the type and concentration of sulfur species of hydrocarbon fractions. Duarte et. al. investigated the effect of ultrasound time on desulfurization of four types of feed. They found different results about these feeds. In some feeds, increasing in time lead to increase sulfur removal, while in some other feeds, sulfur removal first increases and for longer time begin to reduce, which is similar to the present work [49].

3.4. Effect of ultrasonic bath temperature

A series of experiments was conduct at ultrasonic bath temperatures of 30, 50, 70 and 90 °C for 40 minutes. In these cases, 5 ml of hydrogen peroxide and 1 ml of acetic were used. As it can be observed from Fig. 4, higher temperatures of ultrasonic bath cause to increase the efficiency of sulfur removal. It can be considered a pseudo-first order rate kinetic model in terms of sulfur concentration for oxidation reaction as follows [45]:

$$C_S = C_{S0} e^{-kt} \tag{2}$$



Fig. 4. Effect of ultrasonic bath temperature.

Wherein, k, C_{S0} and C_S are model constant, initial concentration and concentration at time t of sulfur, respectively. Molaei Dehkordi et. al. found that pseudo-first order rate constant is directly related to temperature [45]. Therefore, at higher temperatures, the reaction constant is higher and so based on equation 2 the sulfur concentration decreases during the oxidation reaction.

The maximum effect of temperature is from 50 to 70 °C that increase the desulfurization efficiency from 52% to 68%. Furthermore, as shown in Table 1, the flash point of light fuel oil is 77 °C, therefore, it can be concluded that the optimum temperature for this process is somewhat less than the flash point.

3.5. Effect of the number of washing

The sulfur components produced by oxidation reaction were extracted from light fuel oil by washing it for 1, 2, 3 and 4 times with methanol as solvent. As the sulfur compounds are generally polar, extraction solvent should have high polarity and be insoluble in hydrocarbon fractions. Methanol is a high polar, insoluble in hydrocarbons and easily removable solvent. Mello et. al. used methanol, acetonitrile and water as extractive solvent in an oxidative desulfurization study. They found that methanol was the most effective solvent on efficiency of sulfur removal [50]. In other work, Zannikos et. al. reported that methanol is as effective as (in some cases even more effective than) very polar dimethyl formamide and N-methyl pyrrolidone in desulfurization process [51]. As it can be seen from Fig. 5, the higher number of washings leads to the removal of more sulfur components.

By washing the mixture for 4 times, sulfur content of the sample was reduced by 84.4 %. Similar results were reported in some other researches [52, 53].

3.6. Effect of UV ray

In order to investigate the effect of UV ray on the oxidative desulfurization, 5 ml hydrogen peroxide and 1 ml acetic acid were added to 30 ml of light fuel oil and mixed by shaking for about 2 min at room temperature.



Fig. 5. Effect of the number of extraction times.

Then the mixture was placed in the ultrasonic bath at 90 °C for 40 minutes under radiation of an UV 8 Watts lamp. The sulfur products were extracted from mixture for 4 times with methanol. It was found that sulfur content reduced to 0.1027 wt% that shows 89.83 % efficiency of sulfur removal. Under similar conditions, in the absence of UV light, an efficiency of 84.45% was obtained. As a result, it can be said that oxidation under UV radiation improves the desulfurization efficiency by about 5.5%. Indeed, photon of UV light excited the electrons of hydrogen peroxide, which creates more free radicals to change sulfur species into sulfoxides and sulfones. Therefore, more sulfur disappears [54].

3.7. Hydrocarbon stability

To investigate the effects of oxidation process on properties such as distillation behavior, flash point and density of the hydrocarbon, gas chromatography tests were carried out. The results are presented in Fig. 2S for before and after desulfurization process, respectively. The data obtained from the GC were also shown in Table 2S. As shown in Fig. 2S, the methanol separation is carried out appropriately. The given peaks in both chromatograms are perfectly consistent, it was ineffective to overall composition of the sample.

4. Conclusions

An oxidative desulfurization with hydrogen peroxide as oxidant agent in the presence of acetic acid as catalyst was performed for a hydrocarbon fraction, light fuel oil, (C₁₀-C₂₂), with boiling range of 175 °C to 375 °C. Increasing of the hydrogen peroxide lead to form more hydroxyl radical and peracetic acid in the reaction media and so increase the rate of reaction. Therefore, efficiency of sulfur removal increases. The amount of the acetic acid has low effect on efficiency of desulfurization process. To enhance the rate of reaction kinetic and mass transfer diffusion in the reaction mixture, oxidation was carried out in the ultrasonic bath under different temperatures and residence times. The efficiency of desulfurization process increases with increasing of temperature and residence time of ultrasonic bath. The sulfuric oxidation products were sulfoxides and sulfones, which extracted by methanol for 1 to 4 times. It was found that the higher number of washings leads to the removal of more sulfur components. The effect of ultraviolet ray on the oxidation reaction was also investigated. The electrons of hydrogen peroxide were exited under UV radiation and created more radicals. As a result, oxidation under UV radiation improves the desulfurization efficiency by about 5.5%. To investigate the stability of hydrocarbon against the oxidation process gas chromatography tests

were carried out. It was found that desulfurization has no considerable effect on composition of light fuel oil.

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