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Heavy Naphtha Desulfurization by Ozone Generated via the DBD Plasma Reactor

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Abstract

This work presents an investigation into the ultra-desulfurization of heavy naphtha fuel (model using BT and DBT) using oxidation and solvent extraction techniques. Ozone produced by the DBD (Dielectric barrier discharge) plasma generator was employed in the oxidation process as an oxidant, and acetonitrile was used as a solvent extraction. Box-Behnken experimental design was adopted in the current study to examine oxidation desulfurization by non-thermal plasma for the various operation conditions including time, flow rate, and temperature on sulfur removal efficiency. Acetonitrile with an oxidized oil/solvent ratio of 1:1(v/v ratio) was utilized as the extraction solvent. The results showed that the maximum sulfur removal of about 91% was obtained with a temperature of 50°C, flow rate of 75 ml/min, and time of 4 hours.

Keywords: non-thermal plasma; sulfur; oxidation; desulfurization.

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1- Introduction

The definition of naphtha is a liquid petroleum product or fraction of petroleum, often known as full-range naphtha, with a boiling point range of around 30°C (86°F) to 200°C (392°F). It comprises a complicated array of hydrocarbon molecules containing 5 to 12 carbon atoms. Usually, it makes up 15–30% of crude oil in weight. The main group of compounds in the naphtha boiling range include thiophenes, mercaptans, Benzothiophene, and sulfides. In Iraq, most petroleum treatment research aims at removing sulfur compounds from different petroleum fractions. Currently being conducted in hydrocarbon processing because sulfur is the most significant contamination present in petroleum fractions, where it occurs in amounts ranging from thousands of parts per million to almost one hundred [1] Many countries throughout the world have enacted environmental rules to get ultra-low (10 ppm) sulfur levels in distillate fuels [2, 3] Benzothiophene, dibenzothiophene, organic sulfides, disulfides, and their alkylated derivatives are all sulfur compounds present in hydrotreated petroleum [4], Fuels with sulfur-containing compounds are among the most damaging since they not only contribute to acid rain but also harmfully affect human health. In addition to problems with sulfur compounds cause the equipment, pipes, and pumps used in refining to erode, which also causes the oil processing catalysts to lose their effectiveness [5, 6], Reduced sulfur compounds are necessary for reducing their negative effects on the environment, air pollution, and the health of humans. Desulfurization plays an essential role in controlling sulfur in fuel and the issue of clean air. Because moderate to heavy fuels emit a lot of sulfur during combustion, the sulfur problem is getting increasingly problematic overall. The majority of fuel process chemistries focus on how to make clean fuels in a more effective and environmentally friendly way [7] such as adsorption desulfurization [8], absorption desulfurization, extraction desulfurization, catalytic oxidation desulfurization, oxidative desulfurization, hydrodesulfurization [9] and bio-desulfurization.

Compared to the method of hydrodesulfurization, the oxidative desulfurization (ODS) method may be used with temperatures below 80 °C, good selectivity, and no costly hydrogen, and it is also possible to carry out under moderate working conditions such as ambient temperature and air pressure [10, 11]. There are several types of oxidants, including nitrogen oxides, nitric acid, hydrogen peroxide, Ozone, organic hydroperoxides, molecular oxygen, peracids and others, which can be utilized to produce low-Sulfur fuels using oxidation desulfurization methods [12]. Oxidants are used to first convert organic sulfur compounds into their sulfoxides and subsequently into their more polar sulfones. Finally, Polar extractants, including methanol, acetonitrile, and dimethyl formamide, are used to remove these oxidized sulfur compounds from the fuel, etc. [13].

The "directed" energy consumption occurs in non-thermal plasma. In this instance, the energy provided to the discharge is mostly employed to produce extremely

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energetic electrons, with minimal energy wasted in heating a gas's volume. Therefore, technologies based on non-thermal plasma can be highly effective in enhancing oxidation and molecular dissociation or generating free radicals to stimulate plasma chemical processes that may be applied for the production of Ozone or other ecological purposes like the decomposition of contaminants in air streams [14].

One of the NTP methods is the dielectric barrier discharge (DBD) system, and the DBD discharges are appropriate for application in high-volume plasma chemistry. When two planar or cylindrical electrodes connected to an AC power source have at least one insulating layer between them, this is known as a barrier discharge, sometimes called a quiet discharge. The fundamental benefit of this kind of electrical discharge is the cost-effective and reliable establishment of non-equilibrium plasma states in atmospheric pressure gases. This property has led to many important DBD applications, including industrial ozone production, pollution control, plasma chemical vapor deposition and surface activation, excimer lamp excitation, and, more recently, surface modification of diverse materials [15]. In addition, to excited and dissociated atoms and molecules, active radicals, including ionic and atomic species, are created by the ionization, dissociation, and excitation of the source gases caused by electron impact. These chemicals can initiate chemical reactions in plasma [16].

The creation of Ozone is the major plasma chemical synthesis application of non-equilibrium discharges, using ozone generators that use quiet discharges to convert the diatomic oxygen molecule (0_2) to the more active triatomic ozone molecule (0_3); Ozone is a strong oxidant. It is an almost colorless gas with a strong odor. Due to its strong oxidizing properties, it is employed as a potent germicide, viricide, and bleaching agent. New ozone applications for deNOx and deSOx processes (removal of nitrogen and sulfur oxides from flue gas of large-scale combustion processes) are now being investigated on a pilot plant scale, and their performance and economics seem promising [17].

In the present study, the effect of non-thermal plasma on the oxidation desulfurization of the model fuel was investigated, and the mathematical relationship between variables by response surface method. Furthermore, Study the effect of the non-thermal plasma oxidation process on non-hydrotreated Iraqi heavy naphtha to reduce the sulfur content of this oil fraction

2- Experimental Work

2.1. Material and Method

2.1.1. Materials

Benzothiophene (97%), dibenzothiophene (99%) from (mackline company, China), n-hexane (99%) from (Merck, Germany) and acetonitrile (ACN) (99.7%) from (BDH England company), and heavy naphtha from field east of Baghdad with physical properties shown in Table 1.

Table 1. Physical Properties of Iraqi Heavy Naphtha

Physical Properties	**Value**
Density at 15.6 ^{0}C	0.7359 g/cm^3
Specific gravity	0.7366
API	60.61
Viscosity at 40 ^{0}C	0.077856
Boiling point	100-150C°
Sulfur content	525 ppm

2.1.2. Model fuel

Model Fuel was prepared by dissolving BT and DBT in hexane to maintain a total sulfur content of 500ppm 50% of each BT and DBT.

2.1.3. Method

- Dielectric barrier discharge reactor

Fig. 1 shows a schematic representation of the experimental setup for O3 production. The entire system was composed of plasma power, a DBD reactor, and a mass flow controller, and O3 was produced using a self-assembly wire-cylinder reactor. A 0.1 cm thick quartz glass tube serves as the wire-cylinder reactor. A layer of Copper wire mesh Type 304 attached to the AC low electrode (LV) of plasma power has been covered outside the reactor, and a 2 cm copper rod attached to the AC high voltage (HV) electrode of plasma power is fixed in the reactor's axial center. The distance between the two electrodes is 1 cm. A mass flow controller is used to inject air into the DBD reactor at a rate of 75 mL/min when it has been created using an air compressor. When a voltage greater than the breakdown voltage of air was given to the two electrodes at a frequency of (11) kV (50 Hz), the air is discharged by gas breakdown.

Meanwhile, extremely reactive oxygen-free radicals and high-energy electrons are produced in the reactor. Then, a Rapid reaction between excess oxygen and oxygen-free radicals produces new O3. The O3 product from the reactor's bottom was added to model oil as an oxidant to study a desulfurization process; the process was conducted under normal atmospheric pressure.

2.2. Procedure

2.2.1. ozone generation

In the DBD reactor, there are two steps in the Ozone generating method: first, when the energetic electrons (e) produced by the electrical discharge collision with the oxygen molecule, the oxygen molecule breaks down into the oxygen atom, and then the oxygen atom and oxygen molecule collide, creating Ozone as a result. Each of the stages of the reaction is indicated by Eq. 1 and Eq. 2 [18]

$$O_2 + e \rightarrow O + O + e \qquad (1)$$

$$O_2 + O \rightarrow O_3 \qquad (2)$$

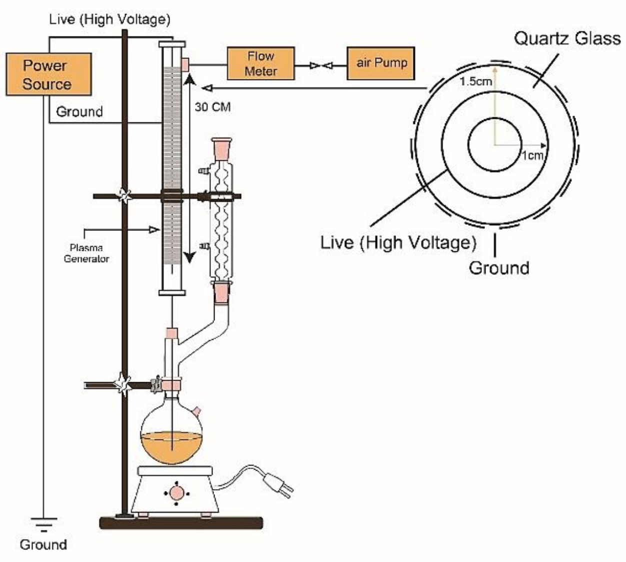

Fig. 1. Schematic Diagram of Dielectric Barrier Reactor

2.2.2. Oxidative Desulfurization Model fuel

The oxidative desulfurization ODS process was studied under various conditions, including reaction temperature, time, and airflow rate. The oxidation process begins when DBD ozone flows into a 250 ml round flask holding 50 ml model fuel that is put on a magnetic stirrer for a specified amount of time to oxidize sulfur compounds to their corresponding oxidants (benzothiophene sulfone and dibenzothiophene sulfone) as shown in Eq. 3 and Eq. 4.

$$\text{Benzothiophene} + Ozone \rightarrow \text{Benzothiophene sulfone} \tag{3}$$

$$\text{Dibenzothiophene} + Ozone \rightarrow \text{Dibenzothiophene sulfone} \tag{4}$$

After the oxidation reaction, Acetonitrile 1:1 oil/solvent (v/v) was used to extract oxidized sulfur at room temperature for 30 minutes while stirring continuously. After that, the oil phase was separated from the water phase, and an ICP-OES was used to calculate the total sulfur content of the treated samples; the effectiveness of sulfur removal was determined using Eq. 5.

$$sulfurmoval\ efficiency\ \% = \frac{(Initial\ S\ concetration-\ final\ \ S\ concentration\ )}{Initial\ S\ concentration} \times 100 \tag{5}$$

2.2.3. Design of Experiment

The Response Surface Methodology (RSM) is a combination of mathematical and statistical methods that may be used to simulate and understand problems with engineering; it may also be used as an optimization approach to analyze the relationship between observed experimental responses and controlled factors. One of the RSM approaches most frequently used for experimental design is the Box-Behnken method, which is also known as a central composite design (CCD). Comparatively, to traditional approaches, the Box-Behnken design method may examine studies with a minimum of tests and a high degree of accuracy. The Box-Behnken matrix, which is determined by Eq. 6 was used to establish the number of trials needed to cover the variables' range.

$$N = 2k(k-1) + r \tag{6}$$

Where N is the number of experiments, k is the number of variables, and r is the replicate number of central point [19]. Sixteen experiments were applied, with 3 factors (temperature, time, and flow rate) as shown in Table 2, and the minimum values for temperature, time, and flow rate are 30 C, 1hr, and 50ml/min, respectively. And the maximum values are 70 C, 8 h, and 125ml/min, respectively. The response is the sulfur removal efficiency (S%) that is calculated by Eq. 5.

Table 2. Sulfur Removal Efficiency in Experiments (Observed) and Predictions under Different Conditions

Flow rate	temperature	time	S%
50	50	4	82.0833
75	50	4	93.7878
100	50	4	84.7134
125	50	4	74.3248
150	50	4	77.3000
75	25	4	60.7312
75	30	4	71.7516
75	40	4	81.7266
75	50	4	93.7878
75	60	4	92.5110
75	70	4	90.4476
75	50	1	47.3582
75	50	2	76.8588
75	50	4	93.7878
75	50	6	92.8366
75	50	8	92.9178

3- Result and discussion

3.1. Model fuel

Minitab software was used to fit the observed data into a second-order polynomial according to the Box-Behnken design Eq. 7:

$$Y = B_{\circ} + \sum_{i=1}^{3} B_i X_i + \sum_{i=1}^{3} B_{ii} X_i^2 \tag{7}$$

Where y is the predicted response, β0 is the intercept coefficient, βi is the linear effect (slope) of input variable xi, βij is the interaction effect of linear by linear between two input variables xi, and βii is the squared effect [20]. So, using the fitting results from Eq. 8, the relationship between sulfur removal effectiveness and independent factors is presented.

$$S\ removal\ \% = -62.7 + 0.10Q + 3.498T + 23.03t - 0.00228Q^2 - 0.03006T^2 - 1.962t^2 \tag{8}$$

The high R^2 (94.67%) in RSM analysis indicates that the predicted model is relatively well-fitting with real data. Very little divergence exists between the experimental and quadratic model points. (Eq. 8) is visually represented in Fig. 2 by a 45° line. This again suggests a strong match between the S removal % predicted from the proposed model and the S removal % (actual) from experiments.

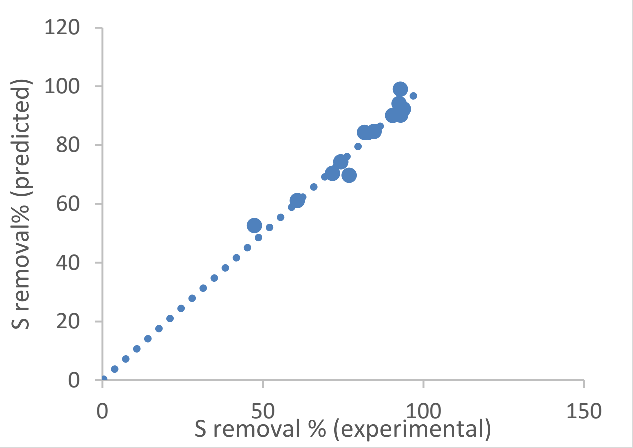

Fig. 2. Experimental and Predicted Response Plot

According to the Petro chart in Fig. 3 and based on their 0.05 -value, the airflow rate has the greatest impact on the effectiveness of sulfur removal, followed by reaction temperature and time. Generally, The process of ODS from Fig. 3 was significantly influenced by the airflow rate fed into the DBD reactor. As the airflow rate increased, desulfurization efficiency increased gradually, established a plateau, and declined. The amount of air supplied per unit of time was insufficient when the airflow rate was lower than 75 ml/min, which led to the reactor producing less Ozone and other energetic oxidants.

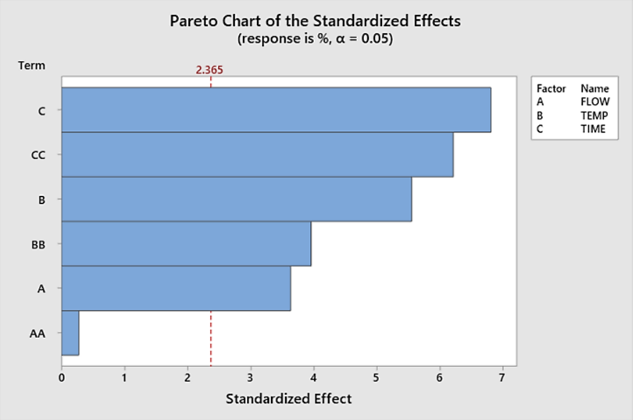

Fig. 3. Petro Chart

The interaction impact of each of the two independent factors affecting sulfur removal effectiveness was evaluated by displaying the response (sulfur removal efficiency) in three-dimensional (3D) plots, as shown in Fig. 4. Similarly, as the rate of airflow exceeded 75 mL/min, the O_3 levels decreased as the rate of airflow increased. The breakdown of the created Ozone, which might involve collision decomposition with the excess electron generated by a strong discharge voltage or collision decomposition with the excess supply impurity in the atmosphere by a high flowrate, as well as the heat was decomposing as shown in Eq. 9, where the main causes of the decline. However, the efficient air input supply was delivered within the mild airflow of 75 mL/min, providing an optimal S-removal environment. As a result, the model fuel's S-compounds were largely oxidized to their oxide of (BT and DBT). Therefore, the best air flow rate for the following experiments was 75 mL/ min.

$$O_3 \xrightarrow{e\ or\ impurity\ or\ heat} O_2 + O \qquad (9)$$

The performance of the desulfurization process may be affected by the reaction rate. Fig. 4 shows that rising reaction temperature from 25 to 50 °C increases sulfur desulfurization efficiency. The reduction of sulfur rises significantly from 60.73% at 25ºC to 93.78% at 50ºC; This rise may be explained by the reaction rate of oxidation of various sulfur-containing compounds included in model fuel because of the high dependency of the reaction rates on the reaction temperature. Although the thermal breakdown rate of the oxidizing agent O3 increases as the reaction temperature rises from 50 to 70 degrees Celsius, it has the effect of limiting the rate at which sulfur compounds oxidize. So, the most suitable reaction temperature for the process is about 50 ºC. Furthermore, the influence of response time is readily seen in that the effectiveness of sulfur removal initially increases with time until a specified time is reached; the influence of reaction time is readily seen in that sulfur removal efficiency first improves with time until a specified time before decreasing significantly. The loss of the oxidizing agent over time caused this decrease to occur.

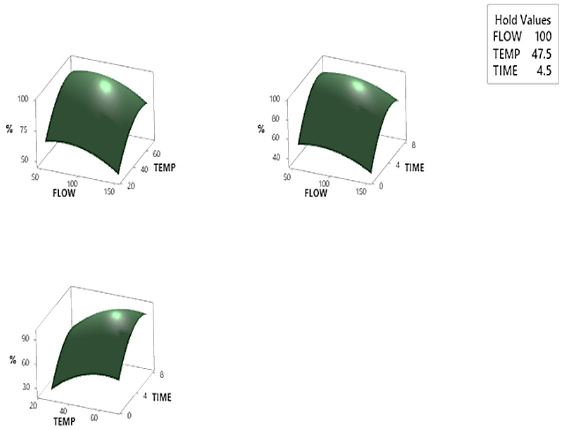

Fig. 4. Efficiency of Sulfur Removal in a 3-D Plot vs Temperature, Flow Rate, Time

3.2. Oxidation desulfurization of heavy naphtha

ODS of actual heavy naphtha was carried out on this oxidation desulfurization by non-thermal plasma technology to study the oxidation reactivity on sulfur compounds while maintaining a heavy naphtha structure. Heavy naphtha was oxidized by plasma at the optimum temperature of 50ºC, flow rate of 75ml/min, and time of 4hr; ICP_OES determined sulfur content. The effect of oxidation time was studied, as shown in Fig. 5.

Fig. 5 shows that the efficiency of oxidation sulfur compound in heavy naphtha increased directly with time at a constant air flow rate and temperature. Increased contact time between reactants is responsible for the increased desulfurization efficiency over time, enhancing the oxygenation process of sulfur compounds.

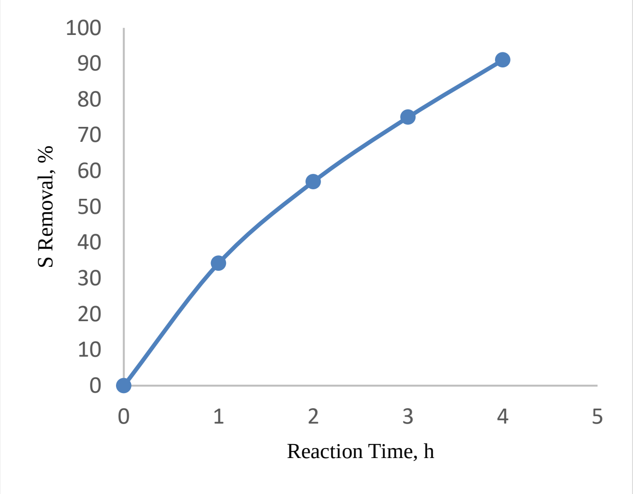

Fig. 5. Sulfur Removal (%) Versus Reaction Time at 50 ºC,75 ml/min, and 11000 Volts

Gas chromatography (GC) analysis was carried out for heavy naphtha before and after treatment, as shown in Fig. 6 and Fig. 7. To determine the composition of mixed aromatic hydrocarbons and the presence of hydrocarbon contaminants in each of the aromatic hydrocarbons, gas chromatography (GC) has become known as an essential technique. GC–FID analyses with the operating condition (injector temperature 250^{0}c, oven program 40-250 ^{0}c, detector temperature 300^{0}c, column/HP-5 capillary 30.0mx320 micro m x0.25 micro m, carrier gas (N_2) flow =1ml/min). The chromatogram traces of the naphtha before and after the oxidation are nearly identical to those of the original naphtha. (Fig. 5), neither the distribution nor the intensity of these peaks have seen any notable modifications. With molecules that have 6–12 carbons, heavy naphtha boils between 90°C and 200°C, as shown by a series of spike peaks, heavy naphtha is usually richer in paraffin and aromatics, as shown in Table 3.

It indicates that these substances were not subjected to any unfavorable consequences. However, due to extraction, the strength of aromatic chemicals is slightly reduced. It demonstrates that aromatic molecules have considerably greater polarity than saturated hydrocarbons and are intended to split into extraction solvents. Nonetheless, Solvent distillation makes it simple to recover this proportion of aromatics.

Table 3. PONA Analysis of Iraqi Heavy Naphtha

	Before oxidation	After oxidation
n-paraffin	31.43	33.05
i-paraffin	33.42	33.73
Naphthene	13.34	13.03
Aromatic	22.81	20.19

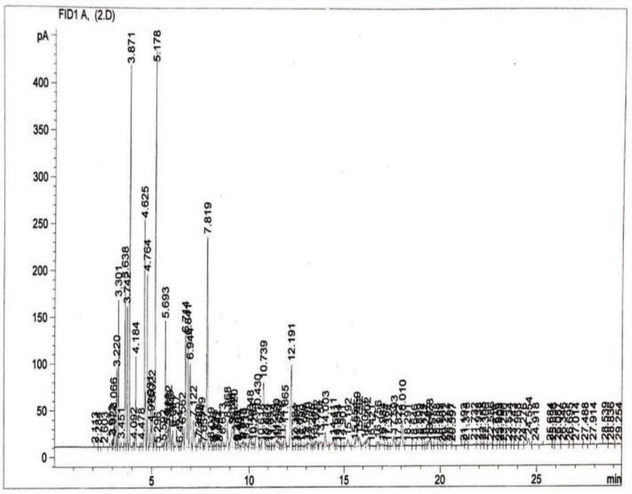

Fig. 6. GC-FID Chromatogram before Oxidation

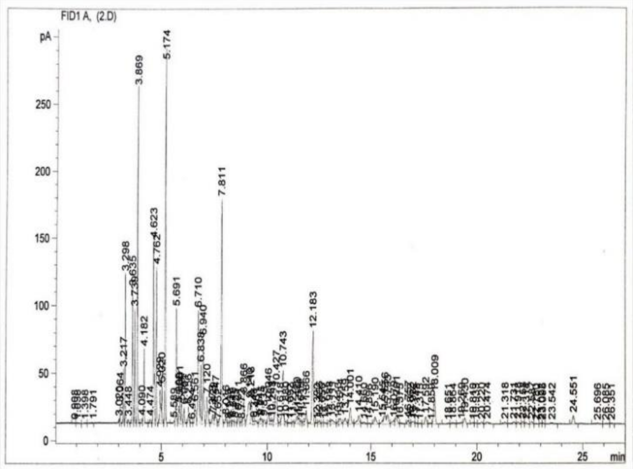

Fig. 7. GC-FID Chromatogram after Oxidation

4- Conclusion

Box–Behnken experimental design was used for oxidation desulfurization by ozone generated from non-thermal plasma of model fuel at the different operating conditions and analyzed. The suggested model represents a second-order polynomial with R^2 value (94.67%). Among the three factors examined (time, flow rate, and temperature), the flow rate has the greatest influence on the effectiveness of sulfur removal by using its value from RSM analysis.

At optimum conditions (4hr time, 75 ml/min air flow rate, and 50 ºC temperature), non-thermal plasma oxidized heavy naphtha. The sulfur removal efficiency of heavy naphtha reached 91.082%. The efficiency of oxidation sulfur compound in heavy naphtha increased directly with time at a constant air flow rate and temperature. As contact time between reactants increases, the oxygenation reaction of sulfur compounds is enhanced, leading to an increase in desulfurization efficiency over time.

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إزالة الكبريت من النفثا الثقيلة باستخدام تقنية البلازما غير الحرارية

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٢ مركز البحث والتطوير النفطي، بغداد، العراق

الخلاصة

تم دراسة إزالة الكبريت من مقطع النفثا الثقيلة و(نموذج وقود متكون من BT و DBT) باستخدام تقنيات الأكسدة واستخلاص المذيبات. تم استخدام الأوزون الناتج عن مولد البلازما DBD (تفريغ الحاجز العازل) في عملية الأكسدة كعامل مؤكسد، واستخدم acetonitrile كمستخلص مذيب. تم استخدام تصميم Box–Behnken التجريبي لفحص إزالة الكبريت من الأكسدة بواسطة البلازما غير الحرارية لظروف التشغيل المختلفة من أجل التحقيق في تأثير ثلاثة متغيرات (الوقت، ومعدل التدفق، ودرجة الحرارة) على كفاءة إزالة الكبريت. تم استخدام acetonitrile كمذيب استخلاص مع نسبة oil/solvent تبلغ ١: ١ (نسبة حجم / حجم). درجة الحرارة ٥٠ درجة مئوية، ومعدل التدفق ٧٥ مل / دقيقة ، ووقت ٤ ساعات هي العوامل التي توفر أقصى إزالة للكبريت. عندما تم أكسدة النفتا الثقيل في ظل الظروف المثلى ، وجد أن إزالة الكبريت كانت ٩١,٠٨٢٪.

الكلمات الدالة: بلازما غير حرارية، كبريت، أكسدة، نزع الكبريت.