Journal of Petroleum Research and Studies

Open Access
Vol. 15, No. 1, March 2025, pp. 167-179

P- ISSN: 2220-5381
E- ISSN: 2710-1096

DOI: http://doi.org/10.52716/jprs.v15i1.840

Kinetics of Oxidation Desulfurization by Non-Thermal Plasma for Heavy Naphtha

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Received 17/12/2023, Revised 21/02/2024, Accepted 25/02/2024, Published 21/03/2025

Abstract

This study optimized the condition of a model fuel containing organo-sulfur compounds (benzothiophene and dibenzothiophene) that were oxidized by non-thermal plasma. The process involved ozone generation through dielectric barrier discharge, followed by extraction using acetonitrile. The results demonstrated the efficient oxidation and removal of dibenzothiophene and benzothiophene by non-thermal plasma. The desulfurization efficiency reached 93.78% under the optimum conditions, that involving a voltage of 11Kv, temperature of 50ºC, duration of 4 hours, and a flow rate of 75 ml/min. When heavy naphtha was oxidized under optimal conditions, it was found that sulfur removal was 91. 082%. Furthermore, the kinetics of oxidation for this system were investigated, it Supposed mechanism and kinetics studies on ozone revealed that the oxidative desulfurization of organosulfur compounds could present a pseudo-first-order kinetic. The reaction rate constant for heavy naphtha was d 0.4759 h-1.

Keywords: desulfurization, non-thermal, plasma, heavy naphtha, oxidation.

حركية إزالة الكبريت بالأكسدة بالبلازما غير الحرارية للنفثا الثقيلة

الخلاصة:

قامت هذه الدراسة باختيار الظروف الامثل لمعالجة نموذج الوقود الذي يحتوي على مركبات الكبريت العضوي (البنزوثيوفين وثنائي بنزوثيوفين) التي تم أكسدتها بواسطة البلازما غير الحرارية. تضمنت العملية توليد الأوزون من خلال عملية التفريغ الكهربائي، يليه الاستخلاص باستخدام الأسيتونيتريل. أظهرت النتائج كفاءة أكسدة وإزالة ثنائي بنزوثيوفين وبنزوثيوفين بواسطة البلازما غير الحرارية. وصلت كفاءة إزالة الكبريت إلى 93.78% في ظل الظروف المثالية، والتي تتضمن جهد 11 كيلو فولت، ودرجة حرارة 50 درجة مئوية، ومدة 4 ساعات، ومعدل تدفق 75 مل / دقيقة. عندما تم أكسدة النفتا الثقيلة في ظل الظروف المثلى، وجد أن إزالة الكبريت كانت 91.082٪. علاوة على ذلك، تم دراسة حركية الأكسدة لهذا النظام، حيث كشفت الدراسات الآلية والحركية المفترضة للأوزون أن إزالة الكبريت التأكسدي لمركبات الكبريت العضوي يمكن أن تقدم حركية زائفة من الدرجة الأولى. وكان ثابت معدل التفاعل للنفتا الثقيلة هو 0.4759 h-1.

1. Introduction:

Deep desulfurization of middle distillates has been a hot topic for many years in petroleum refining due to the strict international regulations of the sulfur content in burned fuel. These rigid environmental regulations have required the sulfur content in diesel fuels to be reduced to an ultra-low sulfur level (<15 ppm) to mitigate environmental pollution in recent years [1]

According to the Iraqi government agency responsible for Iraqi petroleum, Iraq's oil refining capacity is looking forward to increasing. The rise in capacity can be attributed to the completion and operationalization of the Karbala refinery. An important goal of the Iraqi oil industry is developing and constructing new planned refineries equipped with cutting-edge technology that improve goods to satisfy low sulfur emissions standards, as the growing demand for oil products strains the current system. Desulfurization equipment at the Iraqi refinery is having trouble keeping up with the International Maritime Organizer IMO's new sulfur reduction rules. Also, not every item is included in the coverage [2].

The desulfurization process is a chemical process to eliminate sulfur from a given substance. This process includes the removal of sulfur from a specific molecule or the elimination of sulfur compounds from a mixture, such as streams prevalent in oil refineries. There are different technologies such as Hydrodesulfurization, extraction, and oxidation. Every technology has its specificity, that Hydrodesulfurization (HDS) is a chemical process that requires a catalyst and safer Reduction in catalyst life and fuel quality due to severe conditions. High operational cost and requirements of excess amounts of hydrogen gas. Difficulty of removing benzothiophenes and their alkylated derivatives, although Highly effective in removing sulfur from light petroleum fractions such as gasoline and naphtha [3].

The process of oxidative desulfurization (ODS) is gaining popularity as an alternative to conventional methods for the deep desulfurization of light oil. Reducing the sulfur content of fuels with contemporary hydrodesulfurization (HDS) technology requires either the development of the reactor capacity, reaction pressure, reaction temperature, and activity of the catalysts or a combination of these elements [4]. Converting heterocyclic sulfur-containing compounds exhibits challenges for HDS. The DBT-methylated derivatives 4-methyl dibenzothiophene and 4,6-dimethyl dibenzothiophene (4, 6DMDBT) belong to these chemicals. Atmospheric pressure and temperatures below 100 oC are used for ODS operations. The reaction conditions are moderate, and the unique operating requirements are low. ODS may also remove nitrogen, and its selectivity is fairly excellent. Oxidation is a simple method for removing BT, DBT, and other sulfur compounds [5].

Sulfur-containing molecules can be oxidized with the use of selective oxidant chemicals. Due to their enhanced relative polarity, they could be extracted from light oil. Hydroperoxides, peroxy salts, peroxy organic acids, Polyoxometalate [6], ozone, nitrogen oxides, etc., are all examples of oxidants. These substances generate sulfones or sulfoxides by adding oxygen to sulfur in sulfides, disulfides, mercaptans (thiols), and thiophene. Under appropriate conditions, light oil reacts with an oxidant to produce oxidized sulfur compounds. The sulfones that have developed are carried away into the liquid phase. In addition, they leave a solid residue in light oil. Under particular conditions, light oil reacts with an oxidant to generate oxidized sulfur compounds [7].

The Non-thermal plasma has a wide range of applications in different areas that have been investigated on their oxidative desulfurization. Non-thermal plasma was very promising for the desulfurization of benzothiophene and dibenzothiophene in the model oil and heavy naphtha using ozone as the oxidant. However, relatively fewer studies in ODS kinetics of the individual sulfur compounds existing in real feedstock by using ozone oxidants have been reported in the literature. An innovative method for deep sulfur removal from model fuel containing 4,6-dimethyldibenzodithiophene, dibenzodithiophene, benzothiophene, and thiophene was developed by [8] using ozone oxidation produced by a dielectric barrier discharge reactor in conjunction with ionic liquid [BMIM]CH3COO ([BMIM]Ac) extraction. They found that without any catalyst, the desulfurization efficiency of thiophene and benzothiophene reached 99.9%. However, when TiO2/MCM-41 was utilized as a catalyst, dibenzothiophene and 4,6-DMDBT had 98.6% and 95.2% efficiencies, respectively under ideal conditions (time 30 min, air flow rate 150 mL min-1 and discharge voltage of 17.2–18.6 kV). Regarding sulfur removal efficiency, TS was the most effective, followed by BT, then DBT, and finally 4,6-DMDBT.

Ionic liquid [BMIM]FeCl4 (1-butyl-3-methylimidazolium tetrachlorof(errata) was used in an extraction point following the application of dielectric barrier discharge (DBD) plasma and a catalyst for oxidation of sulfur compounds in model oil [9]. Diesel oil's sulfur content of DBT was reduced by as much as 97.5% under ideal conditions (air flow rate of 60 mL/min, applied voltage amplitude of 16 kV to the DBD, input frequency of 79 kHz, catalyst quantity of 1.25 wt%, reaction duration of 10 minutes and temperature 25oC). The oxidation of benzothiophene (BT) and 4,6-DMDBT (4,6-dimethyl-dibenzothiophene) at these conditions also resulted in a significant desulfurization rate. DBT, 4,6-DMDBT, and BT were shown to have progressively lower oxidation reactivities than other S compounds.

Non-catalytic oxidative desulfurization of diesel oil with ozone in an oil/acetonitrile biphasic system is studied [10]. These organosulfur compounds exhibited a rise in oxidation reactivities from lowest to highest, DBT<4,6-DMDBT<BT. The biphasic oxidation system was about 97% efficient in reducing the sulfur level of actual diesel from 1450 to 48 g/g within 1 h. This method gives new illumination on the topic of diesel fuel ultra-deep desulfurization. This study aims to investigate the ODS kinetics for oil fraction organosulfur compounds by using ozone oxidants. it is crucial for the understanding of their ODS behaviour and benefits the design and development of a practical ODS process.

2. Material and Method:

2.1 Materials

Benzothiophene (97%), dibenzothiophene (99%) from (mackline company, China), n-hexane (99%) from (Merck, Germany) and acetonitrile (ACN) (99.7%) from (BDH England company), Heavy naphtha was obtained from distillation of crude oil (API 38) in the Petroleum Research and Development Center, that supplied from (East Baghdad field). The physical properties are 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	0.077856cp
Boiling point	100-150C°
Sulfur content	525 ppm

A model fuel was prepared by dissolving benzothiophene (BT) and dibenzothiophene (DBT) in hexane to achieve a total sulfur content of 500ppm, with 50% of each BT and DBT.

2.2 Method

2.2.1 Dielectric barrier discharge reactor

Figure (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 centre. 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.

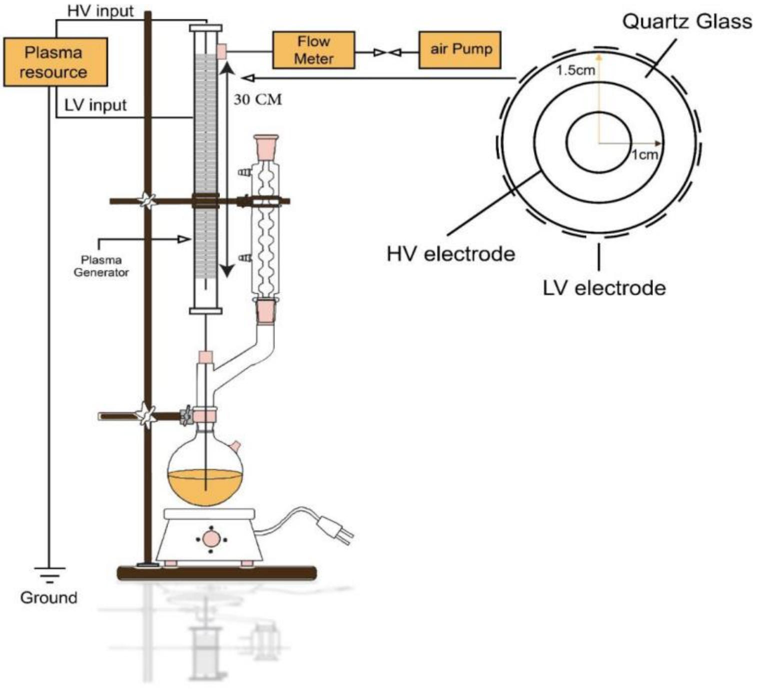


Fig. (1): schematic diagram of dielectric barrier reactor

2.2.2 Procedure

The process occurs in three steps, including ozone generation, oxidation, and extraction, as follows:

1. Ozone generation:

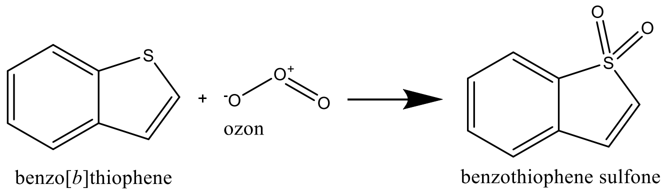
In the DBD reactor, there are two steps in the ozone generating method: first When the energetic electrons (e) produced by the electrical discharge collision with the oxygen molecule, the oxygen molecule breaks down into the oxygen atom, and then oxygen atom and oxygen molecule collide, creating ozone as a result. Each of the stages of the reaction are indicated by Eqs (1) and (2) [11].

$$O_2 + e \rightarrow O + O + e \tag{1}$$

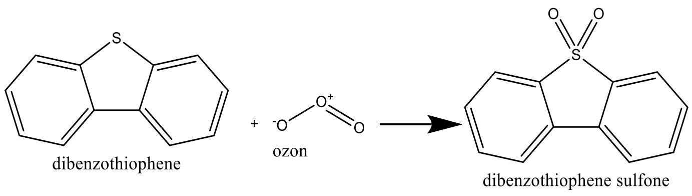
$$O_2 + O \rightarrow \mathrm{O}_3 \tag{2}$$

2. Oxidative Desulfurization Model fuel:

Freshly generated ozone from the DBD plasma was introduced into approximately 50 mL of model fuel contained in the round flask. Concurrently, magnetic stirring was applied to facilitate the oxidation of sulfur compounds for a specified duration. The oxidation process resulted in the conversion of BT and DBT (organosulfur compounds) into polar compounds, specifically benzothiophene sulfone and dibenzothiophene sulfone, as depicted in equations (3) and (4) respectively [10].



(3)



(4)

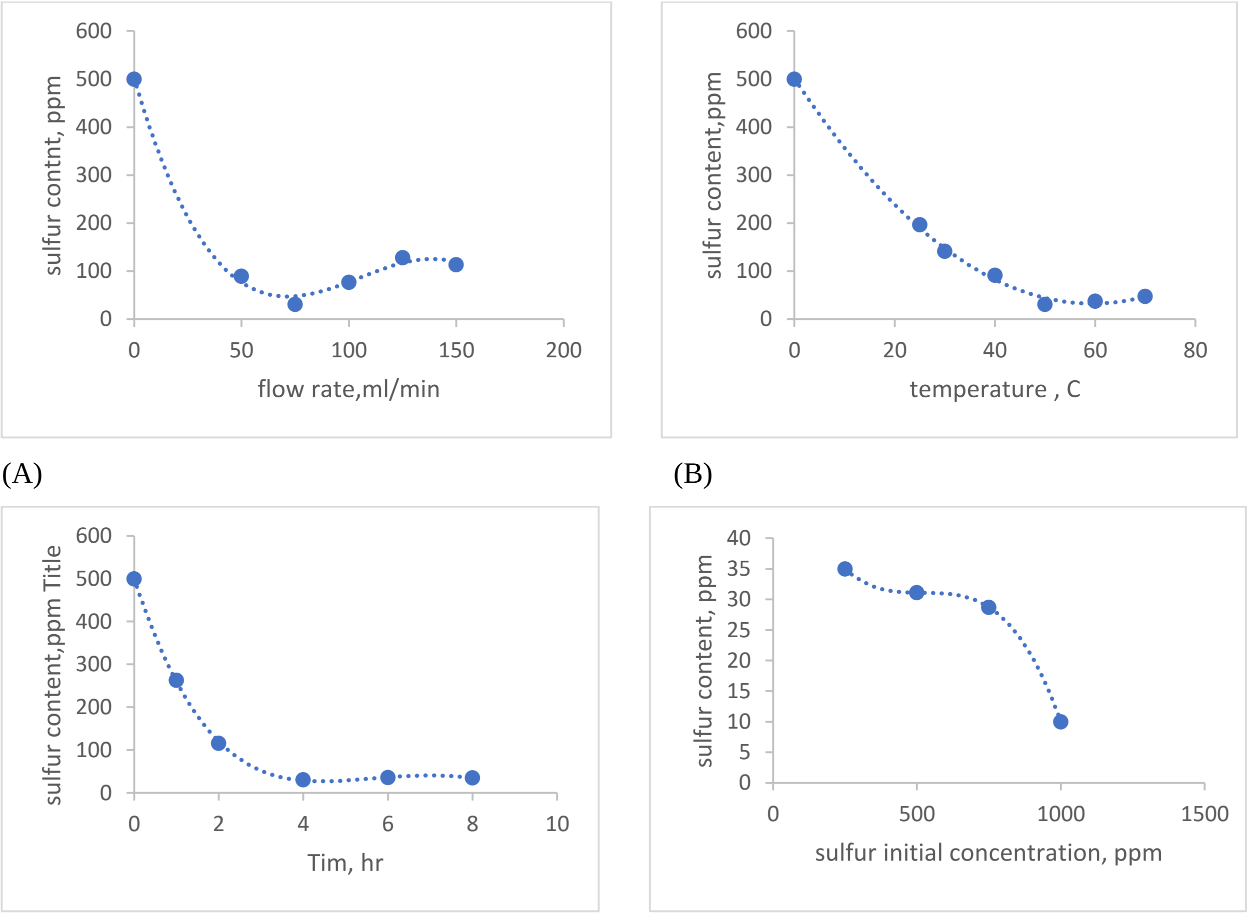
3. Extraction procedure

The liquid-liquid extraction step was then performed on the treated oil produced from the oxidation step by combining it with the solvent acetonitrile in a 1:1 oxidized oil/solvent ratio (v/v) ratio[12]. The mixture was stirred continuously and kept at room temperature for 30 minutes. After 24 hours, the oil phase was separated from the solvent phase using a separating funnel. The sulfur concentration in the oil phase was determined using the Inductive Coupled Plasma Optical Emission spectroscopy technique at the Petroleum Research and Development Center.

4. Result and Discussion

4.1 Oxidation desulfurization of model fuel

The process of ODS from Fig. (2A). was significantly influenced by the airflow rate fed into the DBD reactor. As the airflow rate increased sulfur concentration behave as polynomial function. so from figure (2) best air flow rate was 75 mL/ min which led to a sulfur removel of 93.78 %. When the airflow rate was below 75 mL/min, the amount of air introduced per unit of time was insufficient, leading to lower production of ozone and other reactive oxidants in the reactor. Similarly, when the airflow rate exceeded 75 mL/min, the levels of Ozone decreased as the airflow rate increased. The decrease in ozone levels could be attributed to Ozone decomposition through collision with impurities in the air at high flow rates, as well as thermal decomposition Figure (2B) noticed that rising reaction temperature from 25 to 50 ºC give curve down behavior for sulfur content (increases sulfur desulfurization efficiency). The reduction of sulfur decline significantly from 196.35 ppm at 25ºC to 31.1ppm at 50ºC; This decline 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 O_3 increases as the reaction temperature rises from 50 to 70 degrees Celsius[13], 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 Figure (2C); As time increases, the concentration begins to decrease clearly, but after 4 hours, the sulfur concentration begins to stabilize, and thus the ideal time is 4 hours, which represents the breakthrough point. Figure (2D) show that with an initial sulfur concentration increase, the sulfur removal rate increased from 31.3 ppm to 10 ppm for 500 ppm and 1000 ppm respectively. Increasing the concentration increases reactivity in the oxidation process and removal, demonstrating that the initial concentration of sulfur has a significant impact on desulfurization effectiveness.



(A) (B)

(C) (D)

Fig. (2): Sulfur content versus different operating condition (A): air flow rate at 50 °C, time 4 hr (B): temperature at 75 ml/min, 4 hrs (C): reaction time at 50 °C, 75 ml/min, D): initial sulfur concentration at 50°C , of 75 ml/min, 4 hrs

4.2 Oxidation desulfurization of heavy naphtha

Sulfur content curve down (Desulfurization efficiency grows) as a function of contact time between reactants, enhancing the oxygenation reaction of sulfur compounds. In addition, increasing the processing time provides more contact time for the oxidant agent, O_3 and free radical, generated by DBD plasma to oxidize sulfur compounds from heavy naphtha, which decrease the sulfur content to 46.83 ppm at time 4 h (the desulfurization efficiency 91.082) as shown in Figure (3).

CO_2, SO_2, O_2, and C_3H_4 are produced when ozone is directly injected into naphtha samples, attacking the double bonds of thiophene. The Criegee mechanism is the name given to the direct action of ozone on double bonds. S-S bond scission, along with monosulfides and thiols C-S and

S-H bond cleavage, occur in disulfide compounds. This process results in the generation of additional oxidation by-products, such as alcohols, carboxylic acids, and aldehydes. When ozone and oxygen-containing air are present, the same process occurs. Sulfones and sulfoxide are produced when ozone breaks the double bond that separates the aromatic carbons of benzothiophene and dibenzothiophene [14].

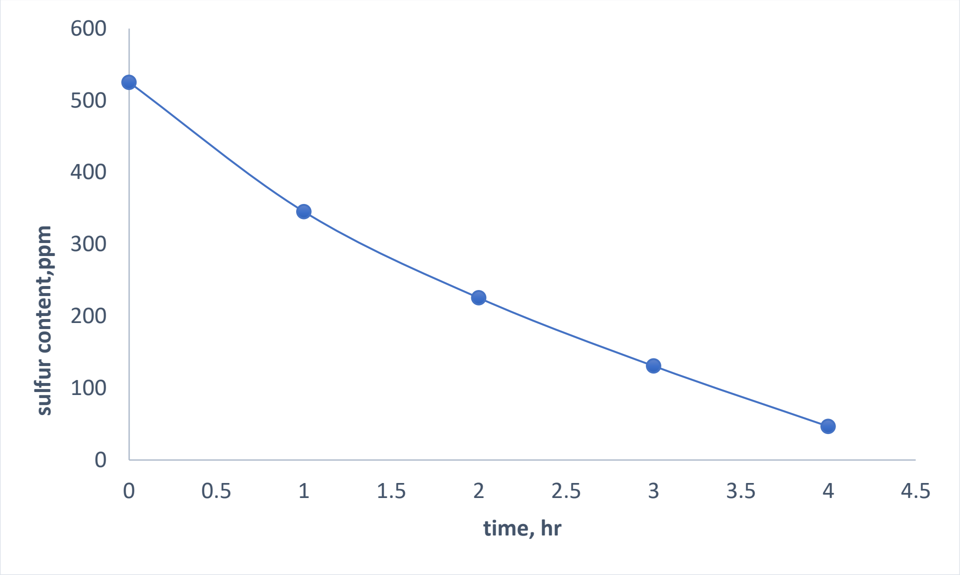


Fig. (3): Sulfur content (ppm) versus reaction time at 50 °C, and 75 ml/min

4.3 Kinetics of ODS reaction for heavy naphtha

Kinetic modelling is one way of studying response behaviour. Zero, pseudo-first, and pseudo-second-order kinetic models are the most commonly utilized [15]. The kinetics of oxidation can be determined by observing changes in sulfur concentration before and after oxidation. Oxidation kinetics for sulfur compounds are calculated as shown in Equation 5:

$$-\frac{d[Csulfur]}{dt} = K[C_{oxident\ agent}]^{a}[C_{Sulfur}]^{b} \qquad (5)$$

Where [$C_{\text{Oxidant agent}}$] and [C_{Sulfur}] denote the concentrations of oxidant agents (O_3 or OH.) and sulfur compounds (BT and DBT) exposed for oxidation reaction, respectively; a and b represent the order of reaction to the concentrations of oxidant agents and sulfur compounds, respectively.

So, that second order reaction was demonstrated in equation 6:

$$\frac{1}{[C_{sulfur}]} - \frac{1}{[C_{sulfur^{\circ}}]} = kt \qquad (6)$$

Where [C_{sulfur}^{o}] and [C_{sulfur}] denote the concentration of sulfur before and after the oxidation respactivily.

From Figure (4), the non-linearity of reaction behaviour refers to that the reaction is not considered a second-order reaction, due to one reacting material having a great excess concentration in the reaction mixture, and thus, it appears as a constant concentration compared to the concentrations of other substances. so that, this reaction behaved as a pseudo-first-order reaction which gave a linearity behaviour. this type of reaction is second-order chemical reaction that behave as first-order reactions.

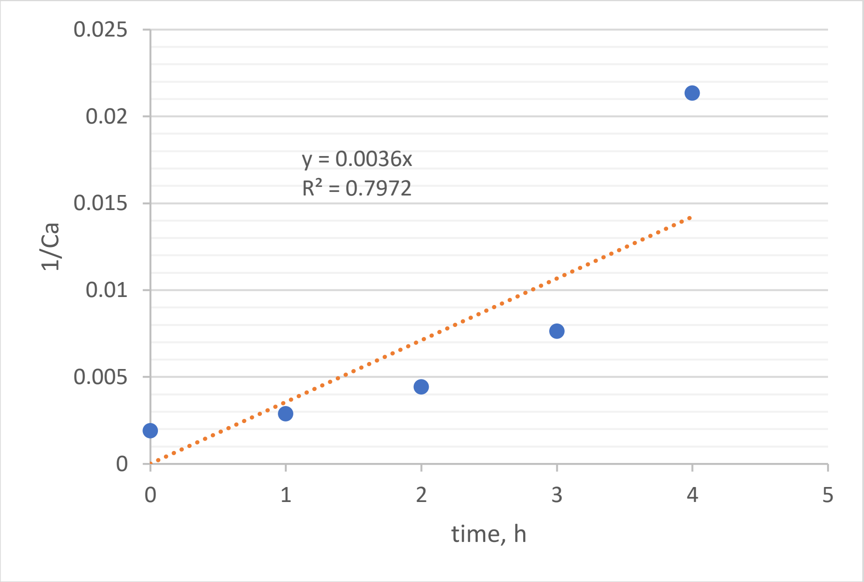


Fig. (4): Oxidation kinetics of plasma oxidation (second order reaction)

Pseudo-first order reaction kinetics governs how aromatic sulfur compounds oxidize. The concentration of the oxidant agent was thought to stay constant during the process. Therefore, Equation 7 is reduced as follows [16].

$$-\frac{d[Csulfur]}{dt} = K'[C_{Sulfur}]^{b} \qquad (7)$$

The linearized form of the pseudo-first-order oxidation reaction for BT and DBT is depicted in Equation 8.

$$ln\frac{[C_0]}{[C_t]} = K't \qquad (8)$$

The kinetic rate constant for oxidation is denoted by k', while the concentrations of the model sulfur compound at 0 and t minutes are denoted by [Co] and [Ct], respectively. [17].

From Equation 8, The plot of ln(c_0/c_t) Vs reaction time (t) showed a linear relationship as shown in Figure (5), confirming the goodness of fit of the pseudo-first-order reaction kinetics for heavy naphtha, 0.4759h^{-1}

is the kinetic rate constants. This explains how a large proportion of sulfur was converted in a short period. The faster the reaction is because of a high-rate constant value [18].

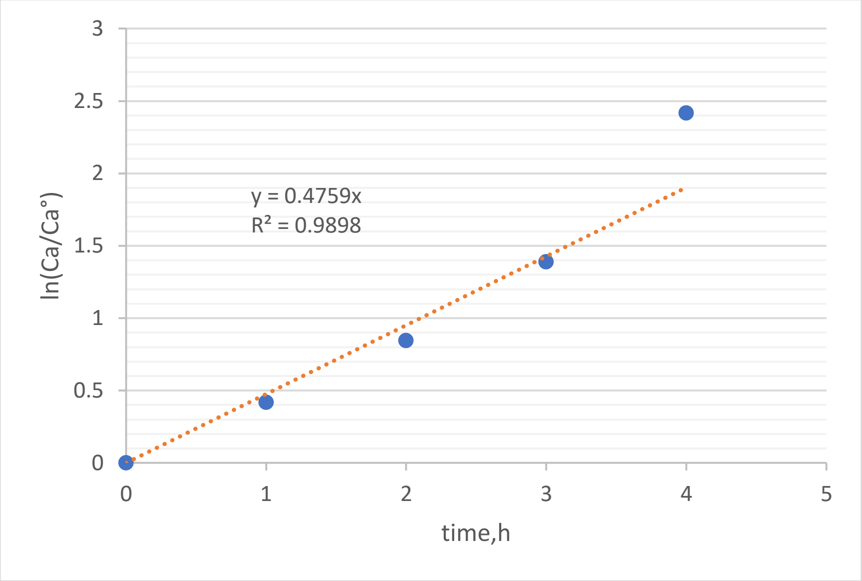


Fig. (5): Oxidation kinetics of plasma oxidation for model fuel and heavy naphtha

5. Conclusions

In this study, the performance of non-thermal plasma was investigated for the oxidation desulfurization of model fuel, with a focus on optimizing the operating conditions. The results showed that the sulfur removal efficiency reached a maximum value of 93.78% under the optimum conditions of 50°C temperature, 75 mL/min airflow rate, and 4 hours' reaction time. This non-thermal plasma system can be considered a simple, efficient, and environmentally friendly method for achieving deep desulfurization of fuel. At optimum conditions, non-thermal plasma oxidized heavy naphtha with sulfur removal efficiency reached 91.082%. in which The efficiency of oxidation sulfur compound increased directly with time the results showed that the oxidation reaction follows the pseudo-first-order reaction.

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