

Adsorptive desulfurization of model fuel by activated carbon prepared from *Bienertia sinuspersici* biomass waste

Adsorpcyjne odsiarczanie modelowego paliwa za pomocą węgla aktywnego przygotowanego z biomasy odpadowej rośliny *Bienertia sinuspersici*

Sama S. Shakir¹, Nawras J. Jassim¹, Saadoon F. Dakhil²

¹ Basra Engineering Technical College, Southern Technical University, Iraq

² Multaqa Al-Nahrein University College, Iraq

ABSTRACT: Sulfur-containing compounds lead to engine wear, corrosion, excessive emissions, and reduced performance. Efficient desulfurization enhances engine reliability and reduces its environmental impact. Adsorption provides an economic, sustainable, and efficient solution for fuel desulfurization. High-surface-area activated carbon with nanopores, prepared from *Bienertia sinuspersici* (BSAC), a promising biomass precursor, was examined for its performance in adsorbing sulfur compounds, specifically dibenzothio-phene (DBT), from a model fuel using a batch experiment setup. The prepared activated carbon had a surface area and pore volume of 1661.9 m²/g and 0.9411 cm³/g, respectively. The effects of key adsorption process parameters, such as contact time (30–150 minutes), initial DBT concentration (1000–5000 ppm), and adsorbent dose (100–500 mg), were evaluated to optimize sulfur adsorption capacity and removal efficiency. All adsorption experiments were conducted at a constant temperature of 25°C and an agitation speed of 150 rpm. The desulfurization isotherms and kinetics of DBT in the model fuel were examined in this study. The results illustrated that the adsorbent dose and initial concentration were key parameters strongly impacting both adsorption capacity and removal efficiency. The adsorption capacity of DBT reached a maximum of 36.12 mg/g at 5000 ppm with a 200 mg adsorbent dose and a contact time of 90 minutes. The highest desulfurization efficiency of 88.8% was achieved at an adsorbent dose of 400 mg, with an initial concentration of 5000 ppm and a contact time of 90 minutes. The equilibrium data were well fitted to the Langmuir isotherm ($R^2 = 0.95$), compared with the Freundlich, Temkin, and Dubinin-Radushkevich isotherms, indicating monolayer adsorption on a uniform surface. The kinetic data showed that DBT adsorption was best fitted with the pseudo-second-order kinetics model, yielding a higher value of the correlation coefficient ($R^2 = 0$).

Keywords: activated carbon, sulfur adsorption capacity, adsorption desulfurization, *Bienertia sinuspersici* biomass.

STRESZCZENIE: Związki zawierające siarkę powodują zużycie silnika, korozję, nadmierną emisję spalin i obniżenie osiągnięć. Skuteczne odsiarczanie zwiększa niezawodność silnika i zmniejsza jego wpływ na środowisko. Adsorpcja stanowi ekonomiczne, zrównoważone i skuteczne rozwiązanie w zakresie odsiarczania paliw. Wysokopowierzchniowy węgiel aktywny z nanoporami, przygotowany z rośliny *Bienertia sinuspersici*, obiecującego prekursora biomasy, został zbadany pod kątem jego wydajności w adsorpcji związków siarki, w szczególności dibenzotiofenu (DBT), z modelowego paliwa w serii eksperymentów. Przygotowany węgiel aktywny miał powierzchnię i objętość porów odpowiednio 1661,9 m²/g i 0,9411 cm³/g. W celu optymalizacji zdolności adsorpcji siarki i wydajności usuwania siarki oceniono wpływ kluczowych parametrów procesu adsorpcji, takich jak czas kontaktu (30–150 minut), początkowe stężenie DBT (1000–5000 ppm) i dawka adsorbentu (100–500 mg). Wszystkie eksperymenty adsorpcyjne przeprowadzono w stałej temperaturze 25°C i przy prędkości mieszania 150 obr./min. W niniejszej pracy zbadano izotermy odsiarczania i kinetykę DBT w paliwie modelowym. Wyniki wykazały, że dawka adsorbentu i stężenie początkowe były kluczowymi parametrami mającymi silny wpływ zarówno na zdolność adsorpcyjną, jak i skuteczność usuwania. Zdolność adsorpcyjna DBT osiąga maksimum 36,12 mg/g przy stężeniu 5000 ppm, dawce adsorbentu 200 mg i czasie kontaktu 90 minut. Najwyższą skuteczność odsiarczania wynoszącą 88,8% osiągnięto przy dawce adsorbentu 400 mg, stężeniu początkowym 5000 ppm i czasie kontaktu 90 minut. Dane równowagowe dobrze pasowały do izotermy Langmuira ($R^2 = 0,95$) w porównaniu z izotermami Freundlicha, Temkina i Dubinina-Radushkevicha, co wskazuje na adsorpcję jednowarstwową na jednolitej powierzchni. Dane kinetyczne wykazały, że adsorpcja DBT najlepiej pasowała do modelu kinetycznego pseudodrugiego rzędu, dając wyższą wartość współczynnika korelacji ($R^2 = 0$).

Słowa kluczowe: węgiel aktywny, zdolność adsorpcji siarki, odsiarczanie adsorpcyjne, biomasa *Bienertia sinuspersici*.

Corresponding author: N.J. Jassim, e-mail: nawres.jameel@stu.edu.iq

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Introduction

Crude oil consists of various forms of organic sulfur compounds, including sulfides, mercaptans, benzothiophene (BT), disulfides, dibenzothiophene (DBT), and sulfur alkyl derivatives, which are undesirable contaminants in crude oil. When released into the environment, these compounds lead to various environmental and health problems (Kolmetz, 2017). The demand for fuel has been rising in many countries over the past two decades. However, these organic sulfur compounds adversely affect oil quality. The combustion of sulfur compounds in the fuel leads to the emission of sulfur oxides, which are the primary sources of air pollution and acid rain. Sulfur compounds can cause numerous corrosion problems in refining equipment, pipelines, pumping systems, and combustion engines when used as transportation fuels. Furthermore, sulfur emissions cause many harmful diseases in the human respiratory system, such as lung cancer, aggravate cardiovascular disease, and trigger asthma (Corro, 2002). Therefore, the removal of sulfur compounds from crude oil is important for producing clean-burning fuels.

Desulfurization is a crucial process in modern refineries and is closely tied to environmental considerations, economic factors, engineering design, and the quality of the final product. Most refineries employ a hydro-desulfurization process to capture sulfur compounds from crude oil, making it suitable for further processing. In hydro-desulfurization, severe operating conditions involving elevated temperatures, high pressure, and large amounts of hydrogen gas are required. Therefore, the recent research has focused on employing alternative methods to HDS techniques, such as oxidative desulfurization (ODS), bio-desulfurization (BDS), and adsorption desulfurization (ADS).

Among these methods, adsorptive desulfurization (ADS) appears to be a more promising treatment method due to its high removal and selectivity for sulfur compounds from fuel, the low cost of adsorbents, and low energy consumption compared with other desulfurization methods. Efficient sulfur separation by adsorptive desulfurization can achieve sulfur levels of less than 10 mg/l from an initial sulfur content of 500 mg/l in diesel fuel (Saleh et al., 2016).

Different types of adsorbent materials have been investigated for the efficient removal of sulfur compounds from transportation fuels, including bio-agricultural waste, metal-supported zeolites, industrial waste, and activated carbon (Amirza et al., 2017; Flayih, 2020; Kutluay and Temel, 2021).

Among numerous adsorbents, activated carbon (AC) has been considered an excellent adsorbent due to its porosity, high surface area, large pore volume, high affinity toward sulfur compounds, cost-effectiveness, and the ability to be prepared from different precursors (Alhamed and Bamuffeh, 2009; Yu and Han, 2015).

In this study, high-surface-area AC previously prepared from BSAC was used as a promising adsorbent material to remove DBT from simulated diesel fuel (Shakir et al., 2021). The effects of adsorption process factors, such as adsorption time, initial DBT concentration, and adsorbent dose, on sulfur removal and adsorption capacity were investigated. Additionally, the adsorption isotherm and adsorption kinetics were studied in the present work.

Experimental Section

Materials

The modified high-surface-area activated carbon prepared from *Bienertia sinuspersici* (BSAC) was used as an adsorbent in this study (Shakir et al., 2021). The chemicals used in this study were of analytical grade and were used without further treatment. DBT was supplied by the Riedel Company, Germany, and the solvent n-octane was supplied by MERCK, Germany. These chemicals were used to prepare the simulated diesel fuel for the adsorption experiments.

Preparation of Activated Carbon (BSAC)

Carbonization and activation were achieved in a two-step process using the locally known raw material “Al-Tahma”, according to the activation method developed by (Shakir et al., 2021). The method is summarized as follows: raw BSAC was washed with distilled water, then ground and sieved until the final powder particle size was within the range of 600 μm to 1.18 mm. To activate the carbon, a novel approach combining the activation effects of organic and inorganic acids was employed. The term “organic load” refers to the treatment of activated carbon with different concentrations of organic acid, specifically citric acid. The optimum organic load used in this study was determined to be 49% citric acid. After the organic activation process, the material underwent additional treatment with phosphoric acid at a fixed impregnation ratio of 1.3 : 1 (H_3PO_4 to carbon). This concentration was derived from a prior study, which identified it as optimal, and was used as the reference value in the current research (Shakir et al., 2021).

Model Fuel Sample Preparation

A model diesel fuel sample was prepared by dissolving 1.00 g of dibenzothiophene (DBT) in 99.00 g of pure (100%) n-octane to obtain a stock solution with a DBT concentration of 10,000 ppm on a w/w basis. The total equivalent sulfur content in this sample was approximately 1720 ppmw, based on the sulfur content of DBT ($\approx 17.2\%$). Lower concentrations were prepared by further dilution with n-octane to obtain desired

concentrations within the required range (334.66–1024 ppmw sulfur) for analytical and adsorption experiments.

Adsorption Desulfurization of Model Fuel

Adsorption desulfurization (ADS) experiments of model fuel were carried out using a Poly Science-Dual Action Shaker (USA). The adsorption experiments were conducted at room temperature, with contact times ranging from 30 to 150 min. Initial DBT concentrations were 1000, 2000, 3000, 4000, and 5000 mg/l, and the adsorbent doses of BSAC were 100, 200, 300, 400, and 500 mg under ambient pressure. The volume of model fuel was fixed at 10 ml for all experiments, and the rotation speed of the shaker was maintained at 150 rpm.

The residual sulfur concentration of the simulated fuel after adsorption was measured using an RX-360 Sulfur Meter (Japan). The adsorption removal percentage and adsorption capacity of DBT absorbed by BSAC were calculated according to Equations (1) and (2), respectively (Ibrahim and Aljanabi, 2015).

$$\text{Desulfurization}\% = \frac{C_o - C_e}{C_o} \cdot 100\% \quad (1)$$

$$q_e = (C_o - C_e) \frac{V}{W} \quad (2)$$

where:

C_o – initial sulfur concentration [mg/l] of the simulated diesel fuel,

C_e – concentration of the simulated diesel fuel at equilibrium after adsorption,

q_e – adsorption capacity at equilibrium [mg sulfur/g BSAC],

V – volume of simulated diesel fuel [l],

W – mass of BSAC [g].

Adsorption Isotherms and Kinetics Study

Adsorption isotherm models were investigated to understand the adsorption mechanism of BSAC. Two adsorption isotherm models were examined: the Langmuir isotherm (Allen et al., 2004) and the Freundlich adsorption model (Voudrias et al., 2002). The Langmuir isotherms model can be expressed by Equation (3):

$$\frac{1}{q_e} = \frac{1}{K_L q_m C_e} + \frac{1}{q_m} \quad (3)$$

where:

C_e – equilibrium DBT concentration [mg/L],

q_e – adsorption capacity at equilibrium [mg/g],

q_m – maximum adsorption capacity [mg/g] or monolayer capacity,

K_L – constant related to the free energy of adsorption and the inverse of the concentration at which half-saturation of the adsorbent is reached [L/mg].

The Freundlich isotherm model is expressed by Equation (4):

$$\log(q_e) = \log(k_f) + \frac{1}{n} \log C_e \quad (4)$$

where:

q_e and C_e are defined in Equation (3),

k_f – Freundlich constant or distribution coefficient,

$1/n$ – adsorption intensity.

The Freundlich constants k_f and $1/n$ were determined from the slope and intercept of the plot of $\log q_e$ versus $\log C_e$, respectively. When $1/n$ is more than zero, that means the adsorption process is favorable under the given conditions. When $1/n$ is greater than 1, the adsorption process is unfavorable, and when $1/n = 1$, it is irreversible.

Adsorption kinetics models were studied using the pseudo-first-order kinetic model (Fierro et al., 2008) and the pseudo-second-order kinetic model (Unuabonah et al., 2019). Their mathematical expressions are given in Equations (5) and (6).

Pseudo-first-order kinetic model:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303} t \quad (5)$$

where:

q_e – amount of sulfur adsorbed at equilibrium [mg/g],

q_t – amount of sulfur adsorbed at time t [mg/g],

k_1 – pseudo-first-order rate constant (1/min)

A plot of $\log(q_e - q_t)$ versus (t) should yield a straight line. Pseudo-second-order kinetic model:

$$\frac{t}{q_t} = \frac{1}{K_2 \cdot q_e^2} + \frac{1}{q_e} t \quad (6)$$

where:

K_2 – pseudo-second-order rate constant [g/(mg · min)].

Results and Discussion

Effect of Adsorption Parameters

Effect of Equilibrium Contact Time

The effect of contact time on sulfur removal percentage at room temperature, using an adsorbent dose of 0.2 g and an initial DBT concentration of 2000 mg/l, was investigated, as shown in Figure 1. The results indicate that the rate of sulfur removal was rapid at the beginning and then increased slightly with time. A contact time of 90 minutes resulted in a constant desulfurization value of approximately 65%, after which equilibrium was achieved. The equilibrium adsorption capacity was 18.07 mg/g at 90 min. The equilibrium time obtained in this study is shorter than the equilibrium time of 150 minutes observed by (Ibrahim and Aljanabi, 2015).

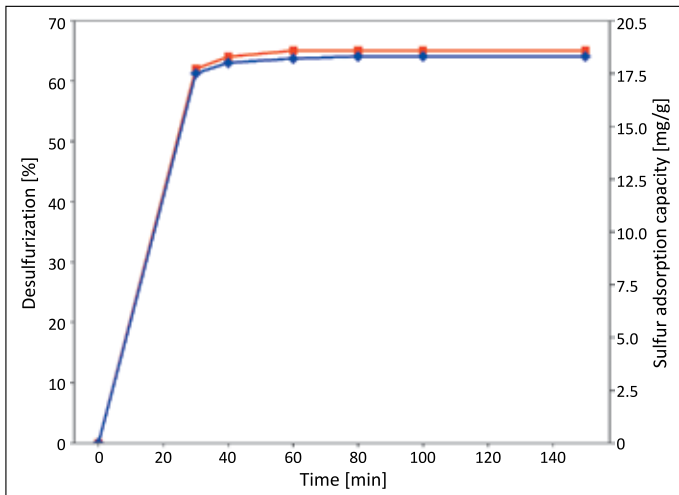


Figure 1. Effect of contact time on model fuel desulfurization (%) and sulfur adsorption capacity at ($T = 25^{\circ}\text{C}$, 150 rpm, initial DBT concentration 2000 mg/l, and adsorbent dose = 200 mg/l)

Effect of Initial DBT Concentration

The effect of the initial DBT concentration in the range of (1000 mg/l to 5000 mg/l) on the desulfurization percentage and adsorption capacity is illustrated in Figure 2. The other adsorption parameters were kept constant at 25°C , an adsorbent dose of 200 mg, a contact time of 90 min, and a mixing speed of 150 rpm. The results indicate that the desulfurization percentage decreased as the initial DBT concentration in the model fuel increased. The adsorption capacity increased with increasing initial DBT concentration. The sulfur removal percentage was 66.07% at an initial DBT concentration of 1000 mg/l and decreased to 42% at 5000 mg/l. This behavior can be attributed to the limited number of available adsorption active sites for a fixed adsorbent dosage, which become saturated when the

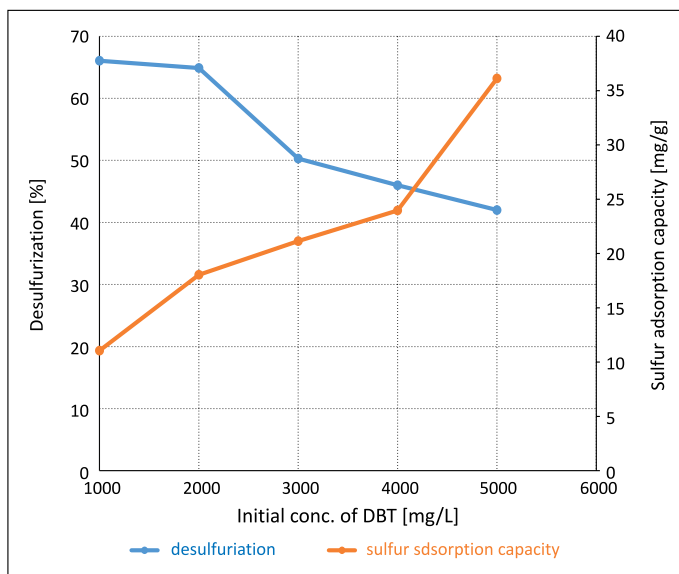


Figure 2. Effect of initial concentration of DBT on model fuel desulfurization (%) and sulfur adsorption capacity at ($T = 25^{\circ}\text{C}$, 150 rpm, and adsorbent dose = 200 mg/l)

initial concentration reaches a specific value. These results are consistent with those reported by Ibrahim and Aljanabi (2015).

Increasing the initial DBT concentration provides a concentration driving force to overcome the mass transfer barrier between the BSAC surface and the sulfur compound in the model fuel; therefore, higher initial DBT concentrations lead to increased adsorption capacity of sulfur compounds (Wang et al., 2020). However, at high concentrations, the active sites become saturated, which limits the adsorption of sulfur compounds. This behavior confirms that the adsorption capacity depends on the concentration gradient and availability of free active sites.

Effect of Adsorbent Dose

The effect of adsorbent dose on the desulfurization percentage and sulfur adsorption capacity is shown in Figure 3. The adsorbent dose was varied from 100 to 500 mg using a constant volume of model fuel of 10 ml. The initial concentration, contact time, temperature, and stirring speed were kept constant at (1000 mg/l, 90 minutes, room temperature, and 150 rpm), respectively. The desulfurization percentage increased from 56.17% at an adsorbent dose of 100 mg to 88.8% at 400 mg. Therefore, 400 mg was considered the optimum adsorbent dose for sulfur removal at an initial DBT concentration of 1000 mg/l. An adsorbent dose greater than 400 mg resulted in a slight decrease in desulfurization efficiency, likely due to the formation of a sludgy system (BSAC/model fuel) that was difficult to treat and mix at a fixed fuel volume of 10 ml. As shown in Figure 3, the desulfurization of the model fuel increased proportionally with increasing adsorbent dose due to the greater number of available BSAC surface active sites for sulfur adsorption. Similar behavior was reported by Ibrahim and Aljanabi (2015). Conversely, the adsorption capacity decreased

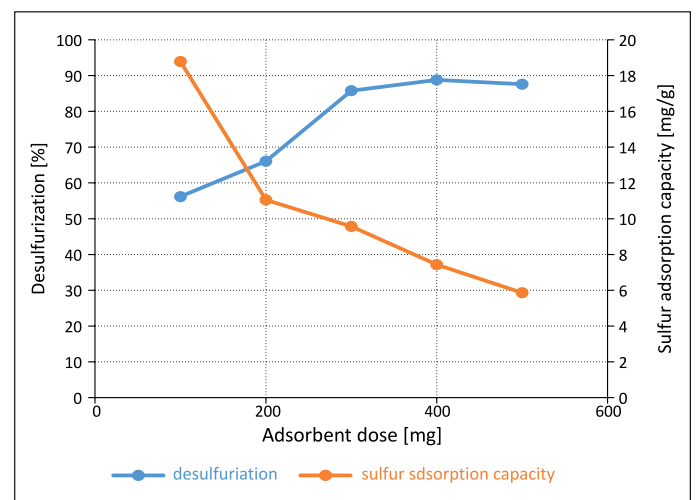


Figure 3. Effect of AC adsorbent dose on model fuel desulfurization (%) and sulfur adsorption capacity at ($T = 25^{\circ}\text{C}$, 150 rpm, and initial DBT concentration of 1000 mg/l)

with increasing adsorbent dose, which may be attributed to particle aggregation and interaction at higher BSAC dosages (Wang et al., 2020).

Adsorption Isotherm

The equilibrium data for sulfur adsorption on BSAC were fitted to the Langmuir model expressed by Equation (3). Figure 4 presents a linear plot of $(1/q_e)$ versus $(1/C_e)$. The Langmuir constants, K_L and q_m , were calculated from the linearized Langmuir equation using the slope $(1/K_L \cdot q_m)$ and the intercept $(1/q_m)$ of the plot. The isotherm data fitted the Langmuir equation well ($R^2 = 0.9512$), as shown in Table 1. K and q_m were 0.003495 L/mg and 39.841 mg/g, respectively. In addition, the value of K indicated that this model is suitable for describing equilibrium adsorption (Al-Ghouti and Da'ana, 2020).

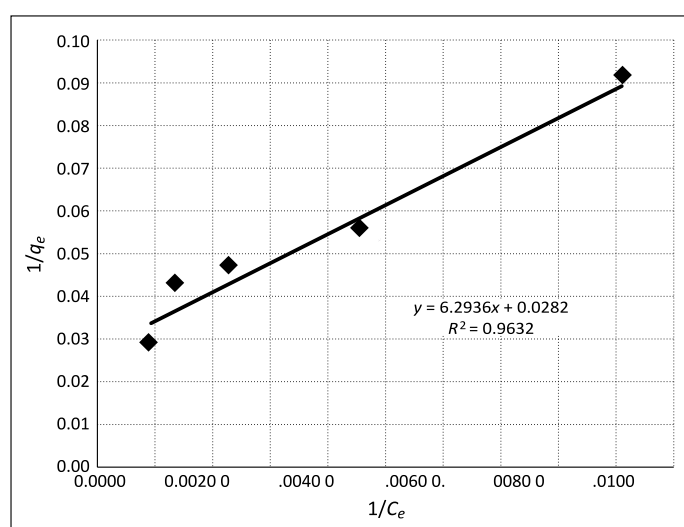


Figure 4. Langmuir isotherm model of the adsorption process at 25°C, and C_o (1000 mg/l, 2000 mg/l, 3000 mg/l, and 5000 mg/l)

The Freundlich isotherm model was also applied to analyze the adsorption isotherm data for sulfur adsorption on the BSAC. The equilibrium data were fitted to the linear form of the Freundlich equation, as shown in Equation (4). Figure 5 presents a linear plot of $\log C_e$ versus $\log q_e$, which was used to calculate the intercept value of $\log K_f$ and the slope of $1/n$, as shown in Table 1. The isotherm data fitted the Freundlich equation well, with a correlation coefficient of $R^2 = 0.9446$. The values of K_f and n were calculated from the graph and estimated as 1.1857 and 2.053, respectively. The slope $1/n$ represents the adsorption intensity, while the intercept K_f indicates the adsorption capacity of the AC surface. The calculated value of n confirms favorable adsorption conditions, with $n > 1$ (Meroufel et al., 2013).

The results indicate that the adsorption follows monolayer adsorption with uniform active sites and that the adsorbed fuel species do not interact with each other.

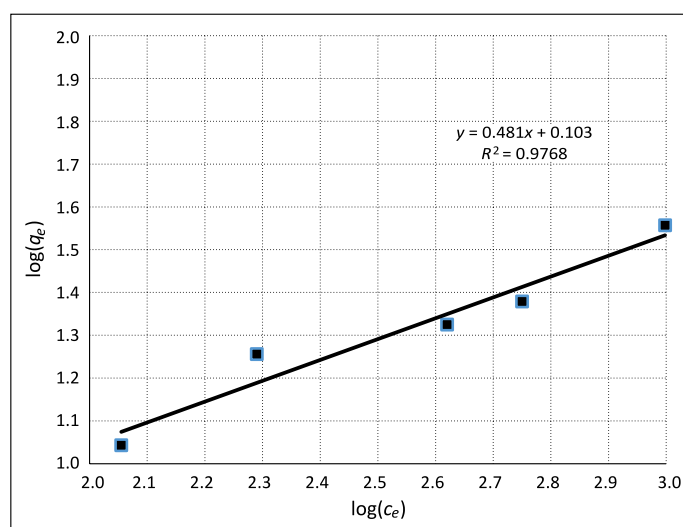


Figure 5. Freundlich isotherm model of the adsorption process at 25°C, and C_o (1000 mg/l, 2000 mg/l, 3000 mg/l, and 5000 mg/l)

Table 1. Constants and correlation coefficients of isotherm models

Langmuir isotherm data			Freundlich isotherm data		
K [l/mg]	q_m [mg/g]	R^2	K_f [(mg/g) (l/mg) ^{1/n}]	n	R^2
0.003495	39.840	0.9512	1.1857	2.053	0.9446

Adsorption Kinetics

For the pseudo-first-order kinetic model, the linear form expressed by Equation (5) was used to describe the adsorption kinetics behavior of DBT on the BSAC surface. Figure 6 shows the fitted plot of $\log(q_e - q_t)$ versus adsorption contact time [min]. The slope and intercept obtained from the fitted graph were used to obtain the amount of the DBT adsorbed at equilibrium q_e and the first-order reaction constant K_1 . A correlation coefficient R^2 of 0.9718 was obtained. Table 2 presents the values of the adsorption rate constant K_1 and equilibrium concentration q_e , which were 0.0882 min⁻¹ and 16.927 mg/g, respectively.

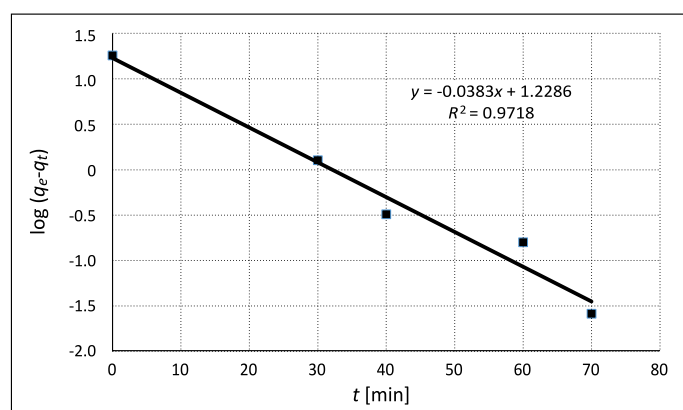


Figure 6. Pseudo first-order kinetic model for sulfur adsorption at an initial DBT concentration 1000 mg/l

For the pseudo-second-order kinetic model, the linear form expressed by Equation (6) was used to describe the adsorption kinetics of DBT. Figure 7 shows the plot of t/q_t versus t [min]. The slope and intercept of the fitted line were used to calculate q_e and K_2 . The correlation coefficient of this model was 0.9989, indicating excellent agreement with the experimental data. Moreover, the calculated equilibrium adsorbed amount of sulfur ($q_{e,cal}$) for the pseudo-second-order model was very close to the experimental value. As shown in Table 2, the adsorption rate constant K_2 and equilibrium concentration $q_{e,cal}$ were 0.06511 g/mg·min and 18.115 mg/g, respectively. Therefore, the pseudo-second-order kinetic model was found to be the most suitable for describing DBT adsorption on the BSAC surface. Similar results have been reported in the literature (Ibrahim and Aljanabi, 2015; Meroufel et al., 2013).

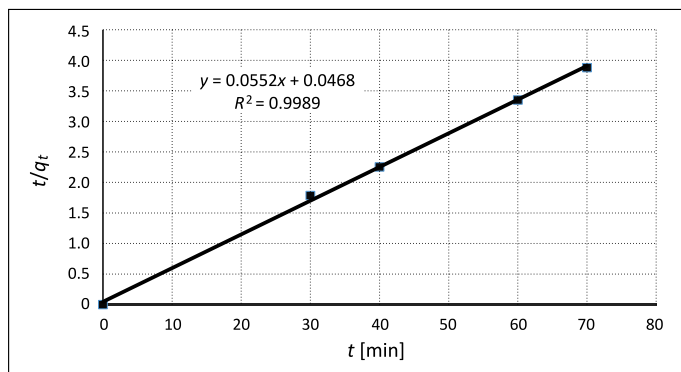


Figure 7. Pseudo-second-order kinetic model for sulfur adsorption at an initial DBT concentration 1000 mg/l

Table 2. Constants and correlation coefficients of kinetics models

Pseudo-first-order data			Pseudo-second-order data			
K_1 [min] ⁻¹	$q_{e,cal}$ [mg/g]	R^2	K_2 [g/(mg·min)]	$q_{e,exp}$ [mg/g]	$q_{e,cal}$ [mg/g]	R^2
0.0882	16.927	0.9718	0.06511	18.07	18.115	0.9989

Conclusions

This study successfully developed an optimized activated carbon adsorbent from *Bienertia sinuspersici* (BSAC) for the removal of sulfur compounds, particularly dibenzothiophene (DBT), from model fuel through an adsorption process. The adsorption performance was systematically evaluated under various operational parameters using 10 ml of model fuel.

The desulfurization efficiency was optimized under different experimental conditions. The highest sulfur removal percentages achieved were 65%, 66.07%, and 88.8% at a contact time of 90 minutes, an initial sulfur concentration of 1000 mg/l, and an adsorbent dose of 400 mg, respectively. These results demonstrate that adsorbent dosage had the most significant

impact on desulfurization efficiency, achieving nearly 90% sulfur removal under optimal conditions.

In terms of adsorption capacity, the maximum values obtained were 18.07 mg/g, 36.12 mg/g, and 18.79 mg/g, corresponding to a contact time of 90 minutes, an initial sulfur concentration of 5000 mg/l, and an adsorbent dose of 100 mg, respectively. The substantially higher adsorption capacity at elevated initial sulfur concentration (36.12 mg/g at 5000 mg/l) indicates that the BSAC adsorbent exhibits enhanced performance at a higher sulfur content, although lower adsorbent doses were more efficient per unit mass.

Equilibrium and kinetic modeling revealed that the adsorption process followed the Langmuir isotherm model, which provided a better fit to the experimental data than the other isotherm models tested. This suggests monolayer adsorption on a homogeneous surface with a finite number of adsorption sites. Furthermore, the adsorption kinetics were best described by the pseudo-second-order kinetic model, indicating that the rate-limiting step involves chemisorption through the sharing or exchange of electrons between the adsorbent and adsorbate.

An optimum activated carbon was prepared from *Bienertia sinuspersici* (BSAC) raw material as an adsorbent for the removal of sulfur compounds, such as DBT, from model fuel by the adsorption process.

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Nawras Jameel JASSIM, Ph.D.
Associate Professor, a lecturer in the Chemical and Petrochemical Techniques Engineering Department at Basra Engineering Technical College, Southern Technical University, Basra, Iraq
E-mail: nawres.jameel@stu.edu.iq



Professor Saadoon Fahad DAKHIL, Ph.D.
Lecturer in the Department of Energy Engineering and Renewable Energy, Multaqa Al-Nahreïn University College, Basra, Iraq
E-mail: saadoonalbahadili@multaqaalnahrein.com

Sama Saad SHAKIR, postgraduate student
Thermal Mechanical Techniques Engineering Department
at Basra Engineering Technical College, Southern Technical
University, Basra, Iraq
E-mail: s.shakir@fgs.stu.edu.iq