

Performance, Adsorption and Kinetic Study of AgY Zeolite for DBT Desulfurization

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Abstract

 This study focuses on preparing and evaluating AgY zeolite as an adsorbent for the desulfurization (ADS) of dibenzothiophene (DBT) using a model fuel. Kinetic models and adsorption isotherms were investigated for this process. The AgY zeolite characterization was studied using XRD, BET, and XRF. XRD and XRF techniques revealed that AgY zeolite was successfully prepared with 21.42% wt. Ag. The BET results showed that the pore volume of AgY zeolite was 0.3596 cm³/g and the surface area was 531 m²/g. The desulfurization study was done with an initial sulfur content of 100–460 ppm. With 93% sulfur removal from the initial concentration of 100 ppm, ultra-deep desulfurization was achieved. The effect of contact time on the adsorption efficiency was investigated within a range of 10-120 min, and the results showed that most sulfur removal (52%) occurred after 10 minutes, while it reached 75% after 120 min with a sulfur capacity of 57.5 mg S/g. The results indicated that the Langmuir isotherm model was the most suitable to describe the process with $R²$ of 99.29%, while the pseudo-second-order was the most fitted kinetic model to the data with R² of 98.57%.

Keywords: *AgY Zeolite; Adsorptive Desulfurization; Adsorption isotherms; kinetic models.*

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1- Introduction

 Sulfur compounds are thought to be the primary source of atmospheric sulfur emissions, an environmental hazard located in crude oil. They are responsible for the deterioration of our environment, polluting the air, declining engine performance, and causing corrosion [1]. Based on the kind and origin of the crude oil, sulfur compounds can be found in crude oil in various forms and quantities, with concentrations ranging from trace levels to more than 8% wt.[2].

 When sulfur and its derivatives in transportation fuels burn, they quickly turn into $SO₂$ and fine particles, which are airborne main toxins that damage the environment and social health by causing smog, acid rain, and dry depositions [3]. According to the World Health Organization, air pollution is the cause of three million deaths annually[4]. Environmental laws have been implemented in several nations worldwide to decrease sulfur content in fuel fractions to extremely minimal levels (10 parts per million). This has reduced the hazardous emissions from transportation fuel machines and improved air quality [5].

 Hydrodesulfurization processes (HDS) need extremely harsh operating conditions, including pressures up to 10 MPa, temperatures as high as 400°C, a large quantity of catalyst, and a high hydrogen consumption rate. This is to remove heterocyclic complexes like DBT and its alkylated replacements, such as 4, 6-DMDBT. These

heterocyclic sulfur compounds can be eliminated by the ADS process at mild conditions without expensive hydrogen [6,7]. The adsorptive desulfurization as a straightforward and ecologically friendly procedure is largely dependent on the adsorbent's textural characteristics. Pore volume, strong structural integrity, more mesoporous surface-active sites, and large surface area are the most important properties [8, 9].

 The challenge is developing an adsorbent that can adsorb organosulfur better than other competitive hydrocarbons, specifically aromatics. It also needs to have a higher capacity for adsorption and regeneration so that it can be reused again. Numerous adsorbent types have been extensively studied in the literature, including activated carbon, metal oxides, supported metals, and metal-loaded zeolites [10]. Zeolites can be loaded with different metal ions, such as Ag⁺, Zn²⁺, Pd²⁺, K⁺, Cu⁺, and Ni²⁺ using impregnation or ion-exchange methods. Modified zeolites with these metal ions increase their adsorption capacity and selectivity. Especially, Ag^+ and Pd^{2+} show a selectivity towards sulfur compounds in the presence of other compounds, such as aromatics, because of their selectivity towards polar particles and pore size [11]. The capacity of sulfur removal increased and the desulfurization efficiency was enhanced by the improved AgY [12–14].

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 In this research, NaY zeolite was modified by loading Ag ions by batch ion exchange. The characterization of the adsorbent was performed by XRD, XRF, and BET. Then the removal of DBT from simulated fuel was studied at different parameters. Also, the adsorption kinetics and isotherms models were studied.

2- Experimental work

2.1. Materials

[Table 1](#page-1-0) shows all materials used for the experimental section of the research.

2.2. Preparation of modified AgY zeolite

 AgY zeolite with specific metal loading percentages was obtained by adjusting the batch ion exchange technique according to the published research [15]. To achieve the necessary percentage of metal loading, 1 g of AgNO₃ was dissolved in distilled water (100 mL) using a conical flask connected to a reflux glass. NaY zeolite (provided by ZR Catalyst CO., LTD) was dried in an electric oven set to 90 °C for 4 h. Then 3 g of NaY zeolite was added to the silver solution. Due to AgNO₃ sensitivity to light, the entire procedure was carried out at midnight in an airtight and dark environment. The mixture was agitated for 6 h at 70°C at 450 rpm. After filtering, the suspension was thoroughly rinsed with deionized water and left to dry overnight at 80°C.

2.3. Experiments of desulfurization

 Four model fuels containing different initial concentrations of sulfur (100-460) ppm were prepared by mixing cyclohexane with DBT. The batch experiment was run under stirring conditions at mild temperature, air pressure, and adsorbent particle size of (5-6 µm). To investigate the most fitted adsorption kinetic model isotherms, 0.3 g of the adsorbent was introduced to 50 ml of several model fuels for variable contacting time (10- 120 min) and various initial sulfur concentrations (100- 460 ppm). Also, 0.3 g of commercial NaY zeolite was introduced to 50 ml of model fuel containing 460 ppm of sulfur for 120 min to measure its sulfur saturation capacity. The adsorbent was used in powder form. The fuel was stirred at 450 rpm for a varied contact duration. After using a vacuum pump to separate the fuel from the powder, the mixture was sent for a sulfur content measuring test.

 Removal of sulfur was shown as Desulfurization efficiency (DS%) which is calculated as the ratio of sulfur removed to that initially present in the fuel.

$$
DS\% = \frac{c_{s} - c}{c_{s}} * 100\tag{1}
$$

Where: C_0 : the initial sulfur content in fuel. C: the measured sulfur content in fuel.

To determine the adsorption capacity, Eq. 2 and Eq. 3 were used:

$$
qe = \frac{Co - Ce}{m} x V \quad at \ an \ equilibrium \ time \tag{2}
$$

$$
qt = \frac{c_0 - ct}{m} x V \quad at \; different \; mixing \; time \tag{3}
$$

Where: q_e : adsorption capacity at equilibrium mg/g. q_i : adsorption capacity at sampling time mg/g. V: volume of model fuel in L. M: mass of adsorbent in g. C_0 , C_e , and C_t are sulfur content at initial, equilibrium, and intervals sampling time respectively in mg/L.

2.4. Techniques of characterization

 The crystal structure of NaY and AgY zeolites was studied using Cu-K α radiation as the X-ray source and a λ value of 1.54056 °A, a 2theta range of 5 to 80, and the Pananylatical Aeris X-ray diffraction model. The nitrogen adsorption-desorption isotherm (the Brunauer Emmett teller BET method) was employed to measure surface area and pore volume (Micrometrics, ASAP, 2020, USA). The elemental structure of NaY and AgY zeolites was determined using the X-ray fluorescence (XRF) device (Spectro Xepos, Ametek, Germany). ASTM D7039 was used to measure the total sulfur contents of the model fuel samples that were treated and the standard samples.

3- Results and discussion

3.1. Characterization of adsorbent

3.1.1. X-Ray diffraction analysis (XRD)

 Comparing the XRD patterns of a commercial NaY zeolite with modified zeolites (AgY) is illustrated in [Fig.](#page-2-0) [1.](#page-2-0) The exact peaks of the commercial Y zeolite [6.3° (1 1 1), $15.9\degree$ (3 3 1), $20.7\degree$ (4 4 0), $24.0\degree$ (5 3 3), $27.5\degree$ (6 4 2), and 31.9° (5 5 5)] appeared in the AgY XRD patterns. The modification did not affect the zeolite structure as shown by the XRD pattern and clean peaks that distinguish the pure phase of the Y zeolite. The peak at 38° indicates that there is a small quantity of Ag₂O present, and the ion-exchange process loaded silver metal onto the zeolite these findings are consistent with earlier research [15, 16].

Fig. 1. XRD analysis of modified AgY zeolite

3.1.2. N_2 [adsorption-desorption](https://measurlabs.com/methods/brunauer-emmett-teller-bet-analysis/#:~:text=What%20is%20BET%20analysis%3F,solid%20surface%20of%20the%20sample.) isotherm

The results of the N_2 adsorption-desorption isotherm of AgY and Na Y zeolites are shown in [Table 2.](#page-2-1) The pore volume of AgY zeolite $(0.3596 \text{ cm}^3/\text{g})$ was higher than that of NaY zeolite $(0.278 \text{ cm}^3/\text{g})$ but the crystalline structure remained intact, and the surface area of NaY zeolite (703.04 m²/g) was larger than of the AgY zeolite $(531 \text{ m}^2/\text{g})$. This may be because of the ion exchange process's delamination and integration of Ag ions into the zeolite's structure. This behavior is consistent with zeolite delamination found in earlier research [17]. The ionic radius of silver (0.15 nm) is larger than that of Na (0.1 nm) which may cause a little expansion of the zeolite lattice and an increase in pore volume. Similar results were shown in an earlier study [18] when NaY zeolite ions exchanged with nickel ions.

3.1.3. X-ray fluorescence (XRF)

 The required Ag loading percentage on the zeolite was given by the XRF analysis conducted for the modified AgY zeolite and commercial NaY zeolites shown in [Table](#page-2-1) [2.](#page-2-1) The analysis reveals that the zeolite's Si/Al molar ratio was in the range of NaY zeolite, indicating that the adsorbent crystals were not affected by ion exchange and thermal treatment. The result was consistent with [19].

Table 2. Structure properties of adsorbents obtained by the BET and XRF

Adsorbent	Surface area (m^2/g)	Pore volume cm^3/g	$Ag\%$ wt.	Si/Al
NaY	703.04	0.2780		2.65
AgY	531	0.3596	21.42	3.053

3.2. Performance evaluation of adsorption desulfurization process

3.2.1. Effect of sulfur initial concentration

 Initial sulfur concentration was studied to determine the best adsorption isotherms to describe the adsorption experiments. For desulfurization over AgY zeolite, the removal of sulfur efficiency decreased with rising sulfur content in model fuel. As shown in [Fig. 2,](#page-2-2) deep-ultra desulfurization was achieved for 100 ppm of sulfur and the final concentration was below 10 ppm while the efficiency for 100, 200, 300, and 460 ppm concentrations of sulfur decreased as follows: 93%, 88%, 81%, and 75% respectively. AgY zeolite showed a sulfur saturation capacity of 57.5 mg S/g adsorbent while NaY zeolite exhibited only 18.4 mg S/g adsorbent. While the sulfur initial concentration was raised, the number of active sites of the adsorbent was still constant. Therefore, the sulfur atoms quantity was in excess, and further removal beyond the saturation point couldn't be applied. These results agreed with Thaligari [20].

Fig. 2. The relation between initial sulfur concentration and removal efficiency of DBT by AgY zeolite (0.3 g/50 ml, t=120 min, AS= 450 rpm, $T = 25^{\circ}C$)

3.2.2. Effect of contact time

 Sulfur removal was studied at different concentrations. It is clear from [Fig. 3](#page-3-0) that most of the removal happened after 10 min for AgY zeolite (52%). AgY zeolite sulfur saturation capacity was raised until equilibrium reached 60 min and no additional reasonable desulfurization was seen. According to Song [19], the adsorbent active sites are gradually engaged with the sulfur compound for a predetermined contact duration until the saturation capacity is attained, and the maximum adsorption occurs.

Regardless of increasing the contact duration, no noticeable desulfurization happened.

Fig. 3. The effect of contact time on desulfurization of DBT by AgY zeolite (m= $0.3g/50$ ml, T= $25 °C$, AS= 450 rpm, Sulfur Content= 460 ppm)

3.3. Adsorption isotherms models

The relation between the adsorbent's quantity (q_e) and the remaining concentration of the adsorbate (C_e) at equilibrium state and constant temperature was described by the adsorption isotherms [3]. The advantage of using adsorption isotherm is to fully understand the relation of the contacting mechanism between the dissolved compounds and the adsorbent in the solution[21]. Furthermore, adsorption isotherms deliver a demonstration of how molecules are distributed in a liquid medium and solid phase when an equilibrium state is achieved during the adsorption process [22].

3.3.1. Langmuir model

 In 1916 Irving Langmuir proposed this isotherm which describes the adsorption as one layer and the energy distributed on the adsorbent surface. Langmuir isotherm assumes the process of adsorption is limited to only monolayer surface, and the adsorption sites are identical and restricted to a specific number. Also, the highest adsorption is reached when the primary layer is saturated with adsorbate ions. Lastly, the adsorption occurs in uniform case. The mathematical equations that describe Langmuir adsorption isotherm in the nonlinear and linear forms are listed below [23–25]:

$$
q_e = \frac{q_{max}K_L C_e}{1 + K_L C_e} \tag{4}
$$

$$
\frac{1}{q_e} = \frac{1}{q_{max} \kappa_L} \frac{1}{c_e} + \frac{1}{q_{max}} \tag{5}
$$

Where: q_e : adsorption capacity when equilibrium is achieved (mg/g) . C_e: the adsorbed concentration at equilibrium (mg/l). q_{max}: adsorption capacity at maximum (mg/g) . K_L: Langmuir constant expressed the binding sites (l/mg) .

If K_LC_e is smaller than unity, then the obtained adsorption isotherm is linear. For modest amounts of adsorption $q_e=$ q_{max} KLC_e. If KLC_e is bigger than unity, then $q_e = q_{max}$.

3.3.2. Freundlich model

 Freundlich isotherm was the first model derived for explaining adsorption isotherms. Herbert Freundlich established this model in 1909 assuming the surface of adsorbent is heterogeneous, and the active sites and energy are equivalent. In addition, it assumed the adsorbent molecules can attract each other, but the attraction is not significant enough to predicate the maximum limits of adsorption. Eq. 6 shows the nonlinear form of the isotherm. Whereas Eq. 7 shows the linear form of the isotherm model [23–25]:

$$
q_e = K_F C_e^{\frac{1}{n}} \tag{6}
$$

$$
\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{7}
$$

Where: K_F : is known as the Freundlich constant which represents the calculated capacity of adsorption [$(mg. g^{-1})$. $(mg^{-1})^{1/n}$]. n: is the intensity value for adsorption which determines the adsorption type.

3.3.3. Timken model

 This model was proposed by Russian scientist Mikhail Temkin in 1940. The Temkin model assumes that when the number of molecules presenting in the adsorption layer of a solid surface increases, the adsorption temperature decreases linearly rather than logarithmically. This model is also applicable to complex liquid-phase adsorption systems because of its remarkable ability to predict gas-phase equilibrium. Eq. 8 shows the nonlinear form of the model, and Eq. 9 shows the linear form of the Temkin isotherm model [23–25]:

$$
q_e = \frac{RT}{b} \ln K_T C_e \tag{8}
$$

$$
q_e = B \ln K_T + B \ln C_e \tag{9}
$$

$$
B = \frac{RT}{b} \tag{10}
$$

 Where: R: is the universal constant of gases (8.3144 J/mol. K). T: is the temperature in absolute value (Kelvin). K_T : is the binding constant of the Timken isotherm at equilibrium (L/g) . B: is a constant of the model

 [Fig. 4](#page-4-0) to [Fig. 6](#page-4-1) shows the application of Eqs. 5, 7, and 9 and the parameters and correlation factors of each adsorption isotherm are shown in [Table 3.](#page-4-2)

 The data shown in [Table 3](#page-4-2) indicate that the Langmuir isotherm was the most suitable for describing the experiments of DBT adsorptive desulfurization by AgY zeolite because its R² value was 99.29% which is close to unity. The Langmuir isotherm described the presence of a monolayer of the adsorbate (DBT) at the surface of the adsorbent for the given concentration range. According to Al-Ghouti [22], the Langmuir adsorption isotherm assumes there is a monolayer homogenous adsorption on the adsorbent surface. The value of n calculated from the Freundlich adsorption isotherm equation was 2.199. According to recent research [3], the value of n in the range of 2-10 indicates good physical adsorption, in the range of 1-2 indicates some adsorption difficulty, and unfavorable adsorption if n is lower than unity.

Fig. 4. Langmuir adsorption isotherm plot

Fig. 5. Freundlich adsorption isotherm plot

Fig. 6. Temkin adsorption isotherm plot

 These data showed that the experimental results fitted these models as follows: Langmuir model > Freundlich model > Temkin model.

3.4. Adsorption kinetics

 Adsorption kinetic is the rate at which pollutants travel from the liquid phase to the adsorbent surface at specific values of variables. The pseudo-first-order and pseudosecond-order are the dominant models and the most applied to study the adsorption kinetics [25].

3.4.1. Pseudo-first order model

 In 1898, Sten Yangve Dennis, who is a Swedish physicist, proposed this model. Adsorption was assumed to form in one layer on the surface of adsorption between liquid and solid phases. Also, pseudo-first order was used to illustrate the first periods of adsorption phenomena. The following linear Eq. 11 represents this model [26– 29].

$$
ln (q_e - q_i) = ln q_e - k_1 t \tag{11}
$$

Where: k_1 : is the constant rate (1/min)

3.4.2. Pseudo-second order

 The pseudo-second-order kinetic model is applied to demonstrate the fully achieved process of adsorption and the total quantity of adsorption. It was assumed that the amount adsorbed on the adsorbent surface at equilibrium is proportional to the reaction speed. Also, the amount of surface active sites that are accessible has a relation to the adsorption rate [26–29]. The linearized form of the pseudo-second-order model is shown in Eq. 12.

$$
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{12}
$$

Where: K_2 : is the constant rate (1/min).

 The kinetic data of adsorption of DBT on AgY zeolite adsorbent have been calculated using two models the pseudo-first order and pseudo-second order models. [Table](#page-5-0) [4](#page-5-0) lists the correlation factors and other parameters belonging to the two applied models. From [Fig. 7](#page-5-1) and [Fig.](#page-5-2) [8,](#page-5-2) the pseudo-second-order was the best model to represent the experimental data as it had the highest correlation factor which was very close to unity (98.57%). According to previous studies [19, 31, 32], pseudosecond-order is the suitable model to describe the adsorptive desulfurization of organosulfur compounds in the presence of pi complexation chemisorption.

Fig. 7. The pseudo- first order kinetic model plot

Fig. 8. The pseudo- second order kinetic model plot

4- Conclusions

 Modification of NaY zeolite to obtain AgY zeolite was successfully achieved with an Ag content of 21.42%. The modification did not affect the structure of zeolite according to the XRD results. AgY zeolite showed an acceptable surface area of 531 $m²/g$ and a pore volume of 0.3596 cm³/g. AgY zeolite achieved a high DBT removal efficiency of 93% for a feed containing 100 ppm. The experimental results of DBT adsorption by AgY zeolite were successfully described by the Langmuir isotherm model and the pseudo-second-order model confirming the monolayer chemical adsorption.

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دراسة االداء واالمتزازية والحركية اللزالة مركب الكبريت العضوي من محاكاة و قود النقل باستخدام مادة الفضة/ زيواليت

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الخالصة

 هذه الدراسة تركز على تحضير وتقييم مادة AgY زيواليت كمادة مازة تستخدم في عملية ازالة الكبريت الامتزازية لازالة مركب الكبريت العضوي DBT من محاكاة وقود النقل. كما تم دراسة الموديلات الحركية لعملية االمتزاز. XRF ,BET ,XRD هي التقنيات التي استخدمناها خالل البحث لتشخيص خصائص الزيواليت وقد تبين من هذه التقنيات نجاح عملية تحميل ايونات الفضة على الزيواليت وبنسبة %21.24 وبحجم مسامي بلغ ٩٥٩٦.٠ سمَّ /غم ومساحة سطحية ٥٣١ م2/ غم. تم دراسة عملية ازالة الكبريت مع تركيز كبريت ابتدائي يتراوح من ppm 100-460 . تم تحقيق ازالة الكبريت العميقة عند تريكز كبريت ابتدائي ppm 100 ثم تم دراسة تأثير زمن التالمس على عملية االمتزاز بمدى يتراوح من 10 الى 120 دقيقة حيث تبين الكمية االكبر للازالة حصلت في العشر دقائق الاولى من العملية (%07) بينما وصلت نسبة الازالة الى %7% بعد ١٢٠ دقيقة ووصلت سعة االمتزاز الى 57.5 ملغم كبريت/ غم من المادة المازة. وبعد ان جمعنا البيانات المطلوبة في المختبر تم تحليل عملية االمتزاز اليجاد الموديالت االفضل وصفا للعلمية. لقد بينت النتائج ان موديل Langmuir هو الموديل االكثر تطابقا لوصف العملية 99.29%=²R وان الموديل pseudo من المرتبة الثانية هو االفضل لوصف الحركية حيث بلغ 98.57% =²R.

الكلمات الدالة: زيواليت AgY، ازالة الكبريت باالمتزاز، االيزوثرمية لالمتزاز، الموديالت الحركية.