Research Article

Fixed Bed Adsorption and Modelling for Desulphurization of Diesel Using Coconut Shell Activated Carbon

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Abstract: Removal of sulphur from diesel was carried out by using by using coconut shell activated carbons. Fixed bed adsorption experiments was performed for determination of break through curve at room temperature. Experiments were carried out with different flow rates, initial concentration, and bed heights. The experiments of three different bed height, 100 mm, 200 mm, and 300 mm with three different flowrate 1.6 ml/min, 3.6 ml/min and 5.6 ml/min. Also experiments with three different sulphur concentration 345.3 ppm, 167.25 ppm, and 86.625 ppm. Adsorption of sulphur from diesel is presented in the form of breakthrough curves. In almost all the experiments, the exit conc. at 0 time was more than 30% (C/C0). So breakthrough point as such can not be found. The breakthrough curve was different than typically observed in packed bed experiment so exaution time was considered and analysed for variation in various parameter. The performance of fixed bed in the form of breakthrough curves were obtained and tested using different kinetic models like Thomas, Adam-Bohart, Yoon-Nelson. Adsorption capacity (mg/g) was found from the models. The constants obtained from the models can be utilized for designing the industrial scale fixed bed. The performance of the regenerated sorbent bed was tested and found similar to that of the fresh adsorbent bed indicating the usefulness of the developed regeneration process.

1. INTRODUCTION

Dat T et al. adsorptive desulfurization of JP-8 fuel using 1- and 2-D mesoporous silica as supporting materials for silver ions, with silver loading up to 33 wt.%. Packed-bed adsorption experiments were carried out in a column reactor at room temperature, using a liquid pump to pump the JP-8 fuel through the reactor for higher desulfurization. Authors were observed that the initial performance of sulfur removal from JP-8 fuel is nearly 100% but gradually drops down and plateaus at around 68%–70% sulfur removal before dropping below 50% after 40 ml of JP-8 fuel processed [1].

Penelope Baltzopoulou et al. diesel fuel desulfurization via adsorption with the aid of activated carbon laboratory and pilot-scale studies. Investigators were carried out both laboratory and pilot scale experiments performed in dedicated fixed bed setups. Under laboratory scale conditions, maximum sulfur removal measured was exceeded 90%. The continuous tests were performed in a vertical fixed bed adsorption reactor and were employed for the generation of dynamic total sulfur breakthrough curves [2].

Alain Favre Re et al. were studied deep desulfurization of diesel feedstock by selective adsorption of refractory sulfur compounds. Investigators were studied adsorptive desulfurization using straight-run gas oil with various sulfur contents like 390 and 60 ppm of sulphur. They were carried out column breakthrough experiments were carried out in jacketed glass column which was packed with resin to avoid entrapped air bubbles within the column at 50 °C. Authors were studied fixed bed adsorption studies on SRGO with different sulfur concentrations which was effective under very mild process conditions [3].

Huang Tang et al. were investigated improvement in diesel desulfurization capacity by equilibrium isotherms analysis. Investigators were carried out adsorption experiments with both model oils and diesel fuels on fixed bed using mesoporous aluminosilicates as adsorbent. Adsorbents were loaded on
to the fixed bed and activated at 400 °C in N2 atmosphere for 2 h before adsorption process and flow rate was 12 mL/h. Authors were concluded that complexation adsorbents were more efficient to remove sulfur compounds at higher temperature[4].

Weiwei Li et al. were investigated solvent desulfurization regeneration process and analysis of activated carbon for low sulfur real diesel. Investigators were carried out dynamic adsorption experiments by flowing diesel through a fixed bed at room temperature to determine the adsorption capacity of activated carbon. The adsorption experiment was carried out with a LHSV of 5.97 h. The total sulfur content varies of real diesel in the outlet of fixed bed with different time was measured. Authors were observed that The sulphur content in diesel was reduced to less than 10 ppm from an initial sulphur content of 34.83 ppm[5].

Mykola Seredych et al. were studied Textural and chemical factors affecting adsorption capacity of activated carbon in highly efficient desulfurization of diesel fuel. Authors were carried out adsorption process at ambient temperature and pressure. They were investigated two synthetic, polymer-derived carbons, and two commercial carbons as adsorbents of dibenzothiophene and 4,6-dimethyl dibenzothiophene from simulated diesel having sulphur content 20 ppm. Model fuel was passed into the column with adsorbent from the top by peristaltic pump with a flow rate of 8.3 mL/h. Authors were observed that amount adsorbed was mainly governed by the volume of micropores, where dispersive interactions were predominant[6].

Vinay M. Bhandari et al. were studied desulfurization of diesel using ion exchanged zeolites. Studies were carried out on a model diesel comprising all the major refractory sulfur compounds such as benzothiophene, dibenzothiophene and 4,6-dimethyl dibenzothiophene and also on commercial diesel. All dynamic adsorption experiments or breakthrough tests were carried out in quartz column using upflow conditions in all the experiments. Authors were carried out experiments with both solvent wash using sulfur-free n-octane and without solvent wash. Authors were observed that various ion exchanged zeolite adsorbents was confirmed high capacity for sulfur removal [7]. Jie Bu et al. were investigated desulfurization of diesel fuels by selective adsorption on activated carbons competitive adsorption of polycyclic aromatic sulfur hetero cycles and polycyclic aromatic hydrocarbons. Both batch and fixed bed continuous adsorption experiments were carried out of real and model diesel fuels containing aromatics and sulfur compounds over several activated carbons. In order to study the adsorption performance of activated carbons, four experiments were carried out using MD in a fixed bed column packed with AC7 at 25 °C and employing a liquid hourly space velocity of 0.5 h−1. Authors were concluded that adsorption capacity of PASHs decreases significantly in the presence of PAHs as result of the adsorption competition due to similar structure, molecular diameter and adsorption mechanisms[8].

Soumen Dasgupta et al. were investigated adsorptive desulfurization of diesel by regenerable nickel based adsorbents. Investigators were investigated NiY and NiMCM 41 for adsorptive sulphur removal from refinery diesel. Adsorption breakthrough experiments were carried out in an automated adsorber system. Authors were concluded that NiY and NiMCM–41 could reduce the sulphur level from 450 ppm in an actual refinery diesel to less than 50 ppm[9]. Anne Kerolaine O. Rodrigues et al. were studied Pd loaded mesoporous silica as a robust adsorbent in adsorption/desorption desulfurization cycles. Investigators were carried out fixed bed adsorption/breakthrough experiments in a fixed bed setup by introducing benzothiophene solutions. The adsorption column studies using SBA-15 and PdCl2/SBA-15 as packing were carried out at 25 °C and with flow rates of 0.50 and 0.35 mL/min. Authors were investigated that PdCl2/SBA-15 had superior sulfur adsorption capacity, as compared to the metal halide alone and the silica support. It was also observed that the adsorbent could be nearly 100% regenerated, upon successive adsorption/desorption cycles[10].

Marko Muzic et al. were investigated the removal of organic sulfur compounds from diesel fuel by activated carbon was investigated in a fixed bed column. The experiments were conducted to study the effects of process parameters such as flow rate, bed depth and temperature. They were developed mathematical model of the fixed bed adsorber. Fixed bed adsorptive desulfurization was carried out by simulation of an industrial adsorption column. Investigators achieved sulfur content of less than 0.7 mg/kg for the lowest flow rate of 1 mL/min[11]. Marko Muzic et al. were studied kinetic and statistical studies of adsorptive desulfurization of diesel fuel on commercial activated carbons. Adsorptive desulfurization experiments were carried out using a semiautomatic laboratory apparatus.
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Investigators were studied statistical process two-level one-half fractional factorial experimental design with five process parameters. Experiments were carried out to determine the sulfur adsorption dependency on time, were used to perform kinetic characterization and to screen the best performing activated carbon[12].

M. Muzic et.al. desulfurization of diesel fuel in a fixed bed adsorption column experimental study and simulation. Investigators were investigated desulfurization of diesel in a fixed bed adsorption column using activated carbon as an adsorbent. The experiments were carried out at different feed flow rates, bed depths, and temperatures, and each experiment resulted in at least one sample containing less than 10 mg/kg of sulfur. The adsorptive desulfurization experiments were carried out in a fixed bed adsorption column were used to investigate the influence of feed flow, working temperature, and bed depth on output sulfur concentration and breakthrough time[13]. Authors were carried out desulphurization experiments using p-complexation based sorbents for diesel, gasoline, and jet fuels. They were investigated that the best sorbent, Cu(I)-Y(VPIE), has breakthrough adsorption capacities of 0.395 and 0.278 mmol S/g of sorbent for commercial jetfuel (364.1 ppmw S) and diesel (297.2 ppmw S), respectively. Based on fixed-bed adsorption data, it was found that the sorbent capacities for total sulfur removal decrease[14].

2. DIESEL AND ACTIVATED CARBON

2.1. Diesel Sample

Before processing for desulfurization, diesel oil was characterized Various physicochemical properties like kinematic viscosity, density, ash contents, flash point and Cetane Index of the diesel oil was studied and shown in table no 2.1.

Table 2.1. Characterization of Diesel sample

<table>
<thead>
<tr>
<th>Properties</th>
<th>Diesel sample</th>
<th>Testing method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kinematic Viscosity(cSt) at 40°C</td>
<td>2.320</td>
<td>ISO 3104</td>
</tr>
<tr>
<td>Density (g/ml) at 15°C</td>
<td>0.8273</td>
<td>ISO 12185</td>
</tr>
<tr>
<td>Ash Content %wt</td>
<td>0.01</td>
<td>ISO 6245</td>
</tr>
<tr>
<td>Flash Point(°C)</td>
<td>36.5</td>
<td>ISO 2719</td>
</tr>
<tr>
<td>Cetane Index</td>
<td>53.0</td>
<td>ISO 4264</td>
</tr>
<tr>
<td>Sulfur content, ppm</td>
<td>334.5</td>
<td>ASTM D5453-2012</td>
</tr>
</tbody>
</table>

2.2. Coconut Shell Activated Carbon

Adsorbent characterization including surface analysis was done for the best performing sorbent coconut shell activated carbon in order to better understand the mechanism and properties that lead to the improved adsorption performance. Coconut shell activated carbon was prepared and characterized various properties like Ph, Moisture Content, Ash content, Volatile matter content, Iodine number, Bet surface area, Bulk Density Resulting estimated properties values are given in table no 2.2.

Table 2.2. Characterization of coconut shell activated carbon

<table>
<thead>
<tr>
<th>Sr No</th>
<th>Properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>i</td>
<td>P.H</td>
<td>6.9</td>
</tr>
<tr>
<td>ii</td>
<td>% Moisture Content</td>
<td>0.5</td>
</tr>
<tr>
<td>iii</td>
<td>% Ash content</td>
<td>1.88</td>
</tr>
<tr>
<td>iv</td>
<td>Volatile matter content</td>
<td>18.86</td>
</tr>
<tr>
<td>v</td>
<td>Iodine number</td>
<td>942 mg/g</td>
</tr>
<tr>
<td>vi</td>
<td>Bet surface area</td>
<td>435.1 m²/gm</td>
</tr>
<tr>
<td>vii</td>
<td>Bulk Density</td>
<td>0.590 g/cc</td>
</tr>
</tbody>
</table>

3. PACKED BED EXPERIMENTS FOR SULPHUR REMOVAL

Fixed bed adsorption experiments was performed for determination of break through curve at room temperature for coconut shell activated carbons of selected size. All adsorption/breakthrough experiments were performed in vertical column equipped with a supporting glass fits. Initially the adsorbents was loaded inside the adsorber. The adsorbent was packed in a glass column having a bed dimension of 4.5 cm i.d. and 300 mm length. Experimental run was conducted by upflow contactor. The flow rate was adjusted by using needle valve. At the bottom end there is a wire mesh filter attached. Adsorbent was filled Above the 1 inch layer of sand. The tanks was filled with diesel.
sample and outlet sample was collected at bottom. Experiments were carried out with different flow rates, initial concentration, and bed heights.

3.1. Breakthrough Curve

Breakthrough curve represents the evaluation of the solution concentration in function of adsorption parameter like contact between liquid and solid phase solvent concentration and temperature. Breakthrough curves described behavior of fixed bed adsorption columns and the break through point is reached when outlet concentration begins to rise rapidly. The breakthrough curve normally takes an S-shape. The steepness of the breakthrough curve determines the extent to which the capacity of an adsorbent bed can be utilized. Effect of various parameters like flowrate, initial concentration and bed height on breakthrough curve was studied.

3.2. Effect of Feed Flow Rate

The desulphurization performance of adsorbent strongly depends on the feed flow of diesel. The effect of feed flow rate on the adsorption of sulphur was investigated by varying the feed flow from 1.6 ml/min, 3.6 ml/min, and 5.6 ml/min. with constant adsorbent bed height at constant feed concentration.

It was found that as flow rate increases from 1.6 ml/min to 5.6 ml/min, adsorptive capacity decreases significantly. The desulphurization performance of adsorbent strongly depends on the feed flow of diesel. Breakthrough curves obtained at different feed flow rates of diesel are shown in fig 3.2.1, fig 3.2.2, and fig 3.2.3.

Breakthrough occurred faster for increasing flow rates as for high feed flow rate, the adsorbate phase did not have enough time to be in contact with adsorbent particles and resulted in lower removal of sulphur. It was observed that lowering of feed flow was causing the lowering of initial values of output sulfur concentration and the extension of breakthrough time. Adsorptive desulphurization experiments were carried out at different flow rate: 1.6 ml/min, 3.6 ml/min, 5.6 ml/min. with constant adsorbent bed height and sulphur concentration.

It was found that all flow rates 1.6 ml/min the exhaustion time was 720 minutes for bed height 100 mm, 200 mm and 300 mm respectively. At flowrate 3.6 ml/min for bed height 100 mm, 200 mm and 300 mm, exhaustion time was 540 min, 570 min respectively. The exhaustion time was 720 min for bed height 100 mm and 300 mm, and 750 min for 200 mm bed height.

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**Fig. 3.2.1. Effect of Flow rate on Breakthrough Curve (Bed Height: 100 mm)**

**Fig. 3.2.2. Effect of Flow rate on Breakthrough Curve (Bed Height: 200 mm)**
3.3. Effect of Bed Height

For study the effect of bed height, keeping the same volume of adsorbent, the height of the packed bed was varied. To investigate the effect of the packed bed dimensions on the desulfurization capacity of the adsorbent, height of the packed bed were chosen and filled with selected adsorbent particles. Sulphur adsorption by coconut shell activated carbon in fixed bed column mainly depends upon the height of adsorbent bed in the column. The experiments was carried out at three different bed heights namely 100 mm, 200 mm, and 300 mm keeping other parameters like conc. feed flow rate constant. To investigate the effect of the packed bed dimensions on the desulfurization capacity of the adsorbent, height of the packed bed were chosen and filled with selected adsorbent particles. The breakthrough curves for sulphur removal from diesel at different bed heights representing adsorbent mass loadings are shown in fig 3.3.1fig 3.3.2 and fig 3.3.3. Exhaustion time was seen increasing with increase in adsorbent bed height, which may be due to the availability of more time for contact between adsorbate and adsorbent.

For all the cases, the exhaustion time increases with bed height. For the bed height of 100 mm, exhaustion time was 750,660 and 690 minutes respectively. The corresponding exhaustion time was 720, at flow rate 1.6 ml/min and 630 min for 3.6 ml/min and 5.6 ml/min respectively for bed height 200 mm. It was found that increase in bed height leads to decrease in the output sulfur concentration. 200 mm bed height gives better results for flow rate 5.6 ml/min with sulphur removal 77.93%. Maximum sulphur removal for 200 mm and 300 mm bed was found 74.11 % and 75.68 % respectively for flow rate 5.6ml/min.
3.4. Effect of Feed Concentration

Adsorption experiments were carried out for the effect of adsorbate concentration on the column performance was studied by varying the feed concentration. The experiments was carried out at three different bed heights namely 100 mm, 200 mm, 300 mm keeping other parameters like concentration of feed and feed flow rate constant. Adsorption experiments were carried out for the effect of adsorbate concentration such as 334.5 ppm, 167.25ppm and 86.625 ppm respectively on the column performance at optimum bed height and feed flow. Sulphur removal for optimum bed height 300mm with feed flowrate 3.6 ml/min for varied conc. 334.5 ppm, 167.25ppm and 86.625 ppm was found to be 60.83 %, 59.94 %, and 68.83 % respectively.

The effect of initial sulphur concentration reduces with increase in the initial concentration. And the exhaustion time increased with decrease in initial concentration. Effect of sulphur concentration in feed solution on performance of breakthrough curves at optimum adsorbent bed height and flow rate is shown in fig 3.4.1. In the adsorption of sulphur a change in initial sulphur concentration affects the operating characteristics of the fixed bed adsorption column. It was observed that the adsorbent get saturated faster at higher concentrations of adsorbate due to the higher rate of adsorbent exhaustion at higher sulphur concentration. The exhaustion time for sulphur conc. 334.5 ppm, 167.25ppm and 86.625 ppm was 570 min, 570 min and 510 min respectively.
3.5. Regeneration of Activated Carbon

The regenerability of the spent adsorbent was conducted to assess its feasibility. For the improvement of life of activated carbon, regeneration of coconut shell activated carbon was carried out. Regenerability of the best performing sorbent, coconut shell activated carbon was studied to determine its feasibility for industrial applications. The regenerability consisted of solvent wash, ultrasonic treatment and heat treatment to test the regenerability of the spent sorbents. After regeneration, the adsorbent can be reused in the packed bed to absorb sulphur. Regeneration of coconut shell activated carbon was carried out by air drying and thermal treatment at high temp 800 °C. The performance of the regenerated sorbent bed is similar to that of the fresh adsorbent bed indicating the usefulness of the developed regeneration process. Fig.3.5.1. shows the removal of sulphur by using regenerated of activated carbon. The regenerability of adsorbent was checked by carrying out new adsorption cycle at optimum bed height and flow rate. It was determined that by decreasing the feed flow and increasing bed depth, the values of output sulphur concentration were decreased and the breakthrough time was extended. The results showed that the regenerated adsorbent could recovered upto 49.17%.

![Regeneration of adsorbent](image)

Fig.3.5.1. Sulphur removal by using regenerated activated carbon

4. Modeling for Packed Bed Sulphur Removal

Theoritical breakthrough curves were tested using different models with corresponding experimental values. Different experimental run were carried out for various bed height, flow rate and concentration. In the present work effect of various parameters on applicability of Thomas and Yoon Nelson model and Adams-Bohart model for packed column data is studied Fixed bed experiments were carried out for removal of sulphur using coconut shell activated carbon. The performance of fixed bed in the form of breakthrough curves were obtained and tested using different kinetic models like Thomas, Adam-Bohart, Yoon-Nelson. Adsorption capacity (mg/g) was found from the models. The constants obtained from the models can be utilized for designing the industrial scale fixed bed.

The best fit model which fitted the experimental condition were determined by the computed R^2 value.

4.1. Effect of Flow Rate

The experimental data for flow rate 1.6 ml/min to 5.6 ml/min was tested by all three models. It was observed that the data was well described by Thomas , Yoon Nelson model and Adams-Bohart model models. for all values of flow rate except flowrate 1.6 ml/min. Yoon Nelson model for different flow rate is represented in fig. no.4.1.1,fig no.4.1.2, and fig no.4.1.3, for varied bed height The values of R^2 of Yoon Nelson model for different flowrate 1.6 ml/min,3.6 ml/min and 5.6 ml/min for bed height 200 mm were greater 0.9, which fits better than other two flowrates. Value of τ decreases with flowrate for all three bed heights.
Thomas model gives better values of $R^2$ for all three flow rates for 200 mm bed height was shown in fig no.4.1.4, fig no.4.1.5 and fig no.4.1.6. For bed height 200 mm values of $R^2$ of the Thomas Model were greater 0.9, which fits better than other two bed height for all three flow rates values of adsorption capacity $q_0$ and constant $K_T$, increases with flow rates.
Adam Bohart model gives satisfactory fit for all three flow rates with different heights with value of $R^2$ near to 0.9. Value of $N_0$ increases with flowrate and value of constant $K_{AB}$ is constant for bed height 200 mm and 300 mm.
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4.2. Effect of Bed Height

It was observed that the data was well described by these three models. The experimental data for bed height 100 mm to 300 mm was tested by all three models. It was observed that the data was well described by Thomas, Yoon Nelson model and Adams-Bohart model models. It can be concluded that Adams Bohart model provided best fit with value of R² greater than 0.94.

Effect of bed height on Yoon model parameters is shown in fig no. 4.2.1, fig no.4.2.2 and fig no.4.2.3. Yoon Nelson model gives better result for all bed height for flow rate 5.6 ml/min with greater value of R² for different bed heights than flow rate 3.6 ml/min and 1.6 ml/min. Yoon Nelson time constant decreased with bed height up to 200 mm and for 300 mm it increases for all three flow rates 16 ml/min, 3.6 ml/min and 5.6 ml/min.
Thomas model gives better results with greater value of $R^2$ at flowrate 5.6 ml/min for all three bed height. Thomas model for different bed height is represented in Fig. 4.2.4, Fig. 4.2.5, and Fig. 4.2.6 for various flow rates. Thomas parameter, $K_t$, increased with bed height for flow rate 3.6 ml/min and initial concentration 334.5, and it decreases for flow rate 5.6 ml/min. While $q_0$, adsorption capacity decreased with bed height for flow rate 1.6 ml/min.
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Adams Bohart model models gives better results at flow rate 1.6 ml/min with greater value of $R^2$ for bed height 100 mm, 200 mm and 300 mm with initial sulphur concentrations 334.5ppm as shown in fig no. 4.2.7, fig no. 4.2.8 and fig no. 4.2.9. Adam Bohart model, $N_0$ increased with bed height Adam Bohart model gives poor result for bed height 200 mm and 300 mm at flow rate 5.6 ml/min.

Fig. 4.2.6: Thomas Model for Different Bed Height (5.6 ml/min flow rate)

Fig. 4.2.7: Adam Bohart for Different Bed Height (1.6 ml/min flow rate)

Fig. 4.2.8: Adam Bohart for Different Bed Height (3.6 ml/min flow rate)
Fixed bed adsorption and modelling for desulphurization of diesel using coconut shell activated carbon

4.3. Effect of Initial Concentration

It was observed that for Feed conc. 334.5 to 86.625 at optimum values of bed height and flow rate, Adam Bohart and Yoon Nelson model gives satisfactory results. Yoon Nelson model for different initial concentrations is represented in fig 4.3.1. The values of $R^2$ of Yoon Nelson model for sulphur concentrations 334.5 ppm, 167.25 ppm and 86.625 were 0.98, 0.64 and 0.68 respectively.

Thomas model is represented by fig no 6.3.2 fits the data for all initial concentration 334.5 ppm to 86.625 ppm, with value of $R^2$ greater than 0.9. Thomas model for different initial concentrations is represented in fig.4.3.2. Thomas parameter, $K_t$ increased with initial concentration while $q_0$, adsorption capacity decreased with initial concentration. With initial concentration Thomas constant decreased. The time required for 50 percent breakthrough ($\tau$) decreased with initial concentration.
Adam Bohart model gives poor result for the effect of initial concentration with value of $R^2$ less than 0.9. In case Adam Bohart model, $N_0$ increased and $K_{AB}$ decreased with initial sulphur Concentration shown in fig no 4.3.3.

Yoon & Nelson, model, Thomas model and Adam Bohart model parameters were estimated from experimental data. It was observed that the parameters like initial concentration, flow rate, bed height affect the model parameters significantly.

5. CONCLUSION

Fixed bed adsorption experiments were carried out at different feed flow rates, bed heights and feed concentration. It was found that all flowrates 1.6 ml/min the exhaustion time was 720,750 and 720 minutes for bed height 100 mm 200 mm and 300 mm respectively. At flowrate 3.6 ml/min for bed height 100 mm 200 mm and 300 mm, exhaustion time was 540 min 570 min respectively. The exhaustion time was 720 min The increase in bed height leads to decrease in the output sulfur concentration.200 mm bed height gives better results for flow rate 5.6 ml/min with sulphur removal 77.93%. Maximum sulphur removal for 200 mm and 300 mm bed was found 74.11 % and 75.68 % respectively for flow rate 5.6ml/min. It was observed that the adsorbent get saturated faster at higher concentrations of adsorbate due to the higher rate of adsorbent exhaustion at higher sulphur concentration. The exhaustion time for sulphur conc. 334.5 ppm, 167.25ppm and 86.625 ppm was 570 min 570 min and 510 min. respectively.Adsorption experiments were carried out using regenerated activated carbon. The results showed that the regenerated adsorbent could recovered upto 49.17%.

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