

# Removal of crystal violet dye by a novel adsorbent derived from waste active sludge used in wastewater treatment

Deniz Akin Sahbaz, Sahra Dandil and Caglayan Acikgoz

## ABSTRACT

This study dealt with preparation of the activated carbon derived from active sludge as an adsorbent for the adsorption of crystal violet (CV) from aqueous solution. The waste active sludge was activated chemically with KOH and carbonized to get activated carbon with a large surface area and a high porosity. The activated carbon was characterized by Fourier transform infrared (FTIR) spectroscopy, Brunauer–Emmett–Teller (BET) instrument, and scanning electron microscopy (SEM). Adsorption of CV from aqueous solution onto the activated carbon was investigated under varying conditions, such as adsorbent dosage (1–6 g/L), solution pH (4–9), contact time period (0–150 min), initial dye concentration (20–100 mg/L), and temperature (25–55 °C). 4.0 g/L of adsorbent dosage was chosen as the optimum level due to having a high removal rate (96.2%) (initial CV concentration 60 mg/L; 150 rpm; pH 6; 25 °C). The adsorption kinetic and adsorption isotherms were well described by the pseudo second order kinetic and the Freundlich isotherm model, respectively. The thermodynamic parameters indicated that the adsorption is a spontaneous process and favored at higher temperatures. The results show that the activated carbon derived from active sludge could be employed as a low-cost material for the removal of CV dye.

**Key words** | activated carbon, active sludge, adsorption, carbonization, crystal violet

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## INTRODUCTION

High amounts of waste active sludge are produced by wastewater treatment plants. The disposal of sludge becomes a problem because of the significant amount and hazardous content. Nowadays, it has great potential for the production of activated carbon. Activated carbon is obtained from waste active sludge in order to remove different impurities, such as dyes (Goswami & Phukan 2017), metals (Hadi *et al.* 2015), pesticides (Vukčević *et al.* 2015), and drugs (Acosta *et al.* 2016). In this way, the amount of waste in an excessive amount can be reduced and waste management can be provided, and a new and useful material can be obtained by evaluating the waste. Recently, waste active sludge has been produced as a result of wastewater treatment activities and has emerged as an interesting option for the production of activated carbon. Activated carbon is characterized by a very large surface area and porosity

obtained by treating carbonaceous materials with physical or chemical processes. The carbonization of carbonaceous materials oxidized by an acid, base, or salt are carried out by pyrolyzing process at 400–900 °C. After all treatments, the product has active sites, a porous structure, and a large surface area so it is widely used as an adsorbent for the removal of organic chemicals and metal ions from wastewater (Al-Qodah & Shawabkah 2009).

Effluents from industrial plants which use dyes cause various environmental problems. In addition, carcinogenic and mutagenic dyes such as crystal violet (CV) present great danger for human health (Shoukat *et al.* 2017). For this reason, CV dye, even at the level of 1 ppb, it is not desirable in the environment and should be removed (Fabryanty *et al.* 2017). CV is widely used in textile dyeing, printing, dermatology, and veterinary fields (Aljeboree 2016). It is in the

cationic triphenylmethane dye group and is positively charged (Wu et al. 2015). CV dye removal has been rarely studied by activated carbon produced by carbonization and activation of waste active sludge. Also, high CV dye removal rate has been obtained by using carbonization of chemically activated waste active sludge in this study.

In this study, waste active sludge which is used in a membrane bioreactor system in our laboratory to remove textile dyes from simultaneous textile wastewater was activated chemically. The chemical activation process was carried out with potassium hydroxide (KOH). After the chemical activation, the material was carbonized. The resulting material was used to adsorb CV from textile wastewater. Important parameters for adsorption, such as the pH, the activated carbon dosage, the initial CV concentration, and the temperature were investigated.

## MATERIALS AND METHODS

### Materials

In this study, the active sludge containing *Trametes versicolor* fungus culture was taken from a membrane bioreactor system. The active sludge had been used to treat textile industry wastewater to remove textile dyes in the membrane bioreactor system. CV dye was used as an adsorbate. It was obtained from Fluka. KOH was used as a chemical reagent and purchased from Carlo Erba.

### Methods

#### Activation

Waste active sludge used in this study was collected from the membrane bioreactor system in our laboratory. The active sludge (containing *Trametes versicolor*) was used for the decolorization of simulated textile wastewater in the membrane bioreactor system. Waste active sludge was dried in ambient conditions at room temperature and then sieved to obtain a 0.60 mm particle size and kept in a plastic bag until further use. In order to increase the surface area for obtaining more efficiency in chemical activation, it was treated with KOH as a chemical agent. In a fume hood, the

sludge and KOH were mixed by stirring with a magnetic stirrer in a beaker at room temperature. The impregnation ratio was chosen as 1:1 (waste active sludge:KOH, wt/wt). The mixture was stirred until brown and water-free material was obtained.

Chemically activated sludge was pyrolyzed in a tubular oven under nitrogen atmosphere up to 500 °C for 1 h and then cooled down to room temperature. The obtained material was washed with HCl and distilled water. Then, it was dried in an oven at 110 °C for 24 h. The product was stored in a glass vial for use in adsorption experiments.

### Batch adsorption experiments

The activated carbon derived from active sludge was used as an adsorbent for adsorption of CV from aqueous solutions in batch mode. The adsorption systems were studied as a function of adsorbent dosage (1–6 g/L), solution pH (4–9), contact time period (0–150 min), initial dye concentration (20–100 mg/L), and temperature (25–55 °C). The optimal parameters for adsorption were determined following one factor at a time (OFAT) and the adsorbent dosage, solution pH with the highest adsorption capacity and efficiency were used for kinetic, isotherm, and thermodynamic analysis. In the adsorption process, the activated carbon was separated from the liquid phase by centrifugation (Centurion 4000) at 3,000 rpm for 10 min. The dye concentration in the supernatant was determined by spectrophotometric analysis at 590 nm on a UV-Vis spectrophotometer (Agilent Cary 60 UV-Vis). All the experiments were repeated twice for consistent results and the mean values used for the adsorption studies.

### Characterization of the obtained activated carbon

N<sub>2</sub> adsorption isotherm was used to determine textural characterization of the activated carbon. The surface area of the sample was detected with Brunauer–Emmett–Teller (BET) method. The surface chemistry was proved by Fourier transform infrared (FTIR) spectroscopy. The surface morphology and composition of the activated carbon were studied by using scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS).

## THEORETICAL

The percentage of CV dye removal onto activated carbon from aqueous solution and the amount of dye adsorbed per weight of adsorbent,  $q_t$  (mg/g) can be calculated by using the following equations:

$$\text{Removal\%} = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (1)$$

$$q_t = \frac{(C_0 - C_t)}{w} \times V \quad (2)$$

where  $C_0$  (mg/L) is the initial CV concentration (mg/L),  $C_t$  (mg/L) is the CV concentration at time  $t$  (min),  $V$  (L) is the volume of CV solution, and  $w$  (g) is the weight of activated carbon used.

## RESULTS AND DISCUSSION

### Characterization of AC

Surface area is an important property because it affects the performance of material in various applications. It proves the porosity of the material. Surface areas of porous materials were determined by BET analysis under an inert gas atmosphere. In this study,  $N_2$  adsorption isotherm was used to determine the BET surface area ( $S_{\text{BET}}$ ) of the activated carbon at 77 K.

The most widely used commercial active carbons have a specific surface area of the order of 600–1,200  $\text{m}^2/\text{g}$ . The pore volume limits the size of molecules that can be adsorbed while the surface area limits the amount of material which can be adsorbed assuming a suitable molecular size. The adsorptive capacity of adsorbent is related to its internal and external surface area and pore volume.  $S_{\text{BET}}$  of the activated carbon obtained from waste active sludge was determined as 14.9860  $\text{m}^2/\text{g}$  and pore volume is 0.055533  $\text{cm}^3/\text{g}$ . In the literature, the surface area results of activated carbon produced with the same method using KOH from other resources were reported as 5.25  $\text{m}^2/\text{g}$  ( $V_t = 0.015 \text{ cm}^3/\text{g}$ ), 27.15  $\text{m}^2/\text{g}$  ( $V_t = 0.05 \text{ cm}^3/\text{g}$ ), 63.53  $\text{m}^2/\text{g}$  ( $V_t = 0.06 \text{ cm}^3/\text{g}$ ) for *Acacia mangium* wood, desiccated coconut residue activated carbon at 400 °C (AC/KOH:1/3), desiccated coconut

residue activated carbon (AC/KOH:1/1), respectively (Yahya *et al.* 2015a, 2015b, 2016).

The morphology of the activated carbon was determined by SEM. In addition, for the composition analysis, an energy dispersive X-ray spectroscopy was used. Figure 1 shows a SEM image of the activated carbon. As can be seen from the figure, the obtained activated carbon has some spherical forms. EDS spectrum of the activated carbon shows that the obtained activated carbon has C, O, Fe, and K elements with a high content of C (approximately 75%) (Figure 2).

FTIR analysis was used to determine the surface chemistry of the activated carbon. The existence of functional groups were detected to characterize the activated carbon. The FTIR spectra of the obtained activated carbon are shown in Figure 3. A wide band at 3,334  $\text{cm}^{-1}$  is due to the presence of -OH groups. The peak at 2,920  $\text{cm}^{-1}$  proves C-H interaction on the surface of the activated carbon. The peak at 1,615  $\text{cm}^{-1}$  indicates carbonate forms (Kacan & Kutahyalı 2012). 1,360 and 1,316  $\text{cm}^{-1}$  peaks can be attributed to bending vibration of O-H. The fingerprint region of the peaks under 820  $\text{cm}^{-1}$  indicates substitute aromatic structures. As a result, the spectra show various functional groups and these functional groups react with dye molecules in aqueous solution.

### Effect of pH

The pH value of dye solution has an important influence on the surface structure and the charge of the adsorbent along with its degree of ionization in solution. The

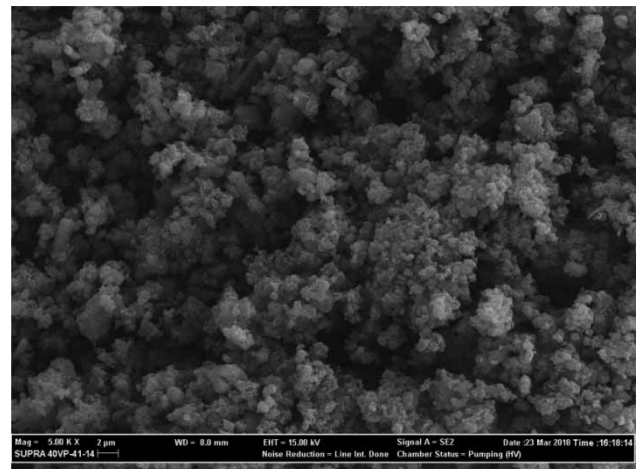
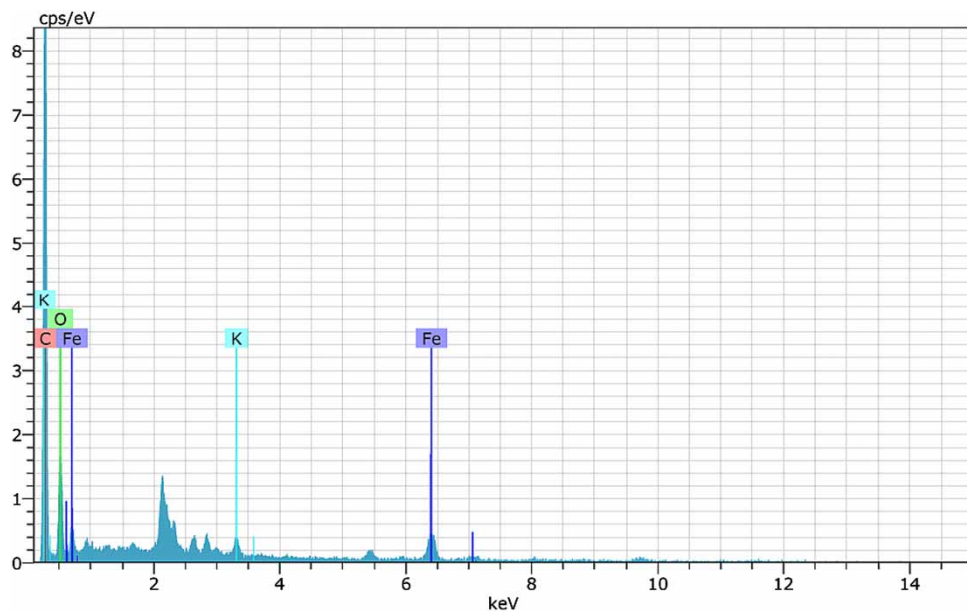
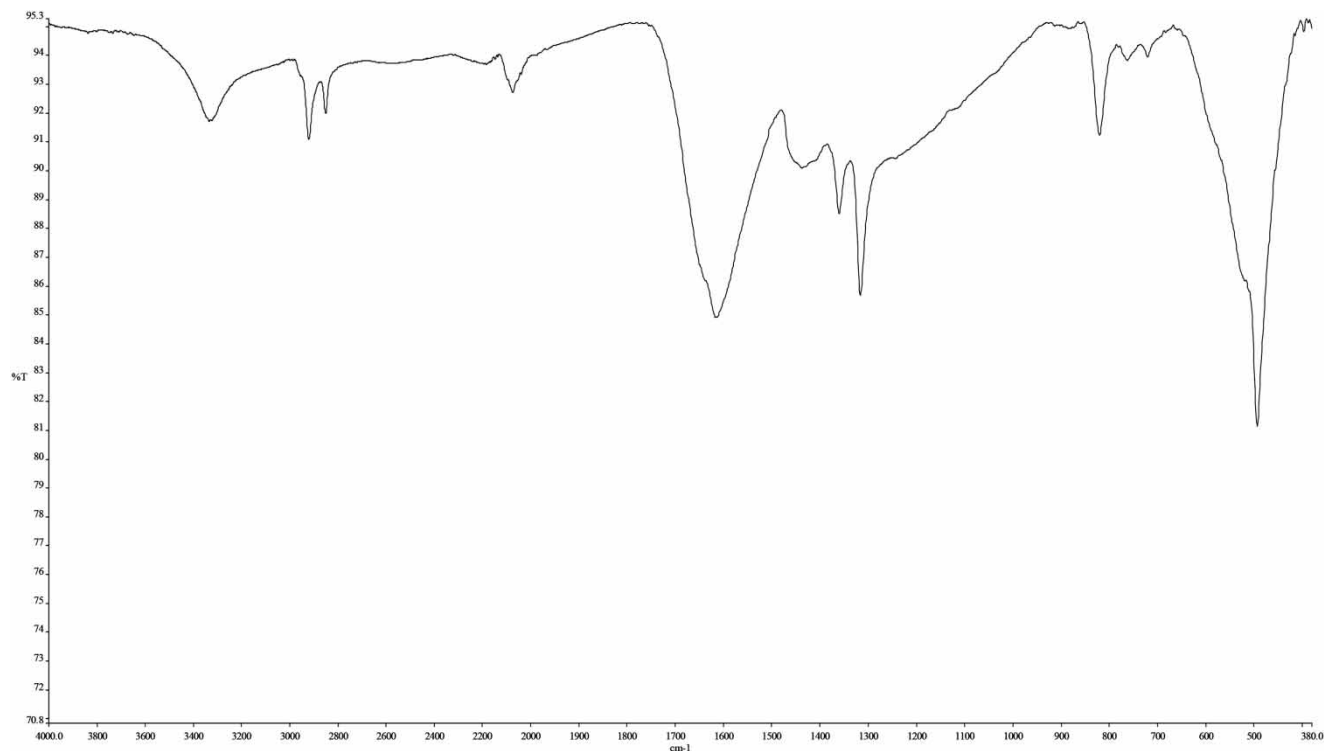


Figure 1 | SEM image of the activated carbon.



**Figure 2** | EDS analysis of the activated carbon.



**Figure 3** | FTIR spectra of the activated carbon obtained from waste active sludge.

effect of pH on CV adsorption was studied in the pH range 4.0–9.0. At all experimental pH values, the adsorption process exhibited similar behavior due to the low

pKa value (0.8) of the CV dye (Kumari *et al.* 2017). The adsorption capacities of the activated carbon were about  $27 \pm 1$  mg/g. The percentage of dye removal increased

from 90.0% to 92.9% when the pH of the dye solution increased from 4 to 9.

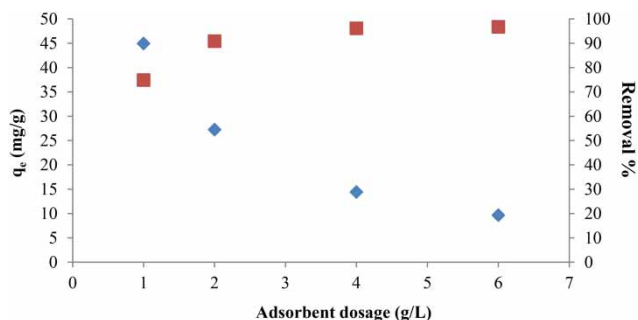
### Effect of adsorbent dosage

The dosage of the activated carbon was varied from 1 g/L to 6 g/L. As shown in Figure 4, the adsorption capacity of CV dye decreased from 44.97 mg/g to 9.69 mg/g and the removal rate increased from 74.9% to 96.8% because of the increase in the amount of available adsorption sites when the dosage of the activated carbon increased from 1 g/L to 6 g/L.

The maximum adsorbed amount at equilibrium ( $q_e$ ) was 44.97 mg/g obtained from 1.0 g/L of adsorbent dosage. This dosage here was considered as the optimum level, but the time to reach equilibrium was considered as long. 4.0 g/L of adsorbent dosage was chosen as an optimum level due to having a high removal rate (96.2%) and fast equilibrium time desirable in industrial applications.

### Effect of contact time and initial CV concentration

To determine the equilibration time for the maximum uptake of the CV, the adsorption on the activated carbon was studied for five different initial concentrations as a function of contact time, and the results are shown in Figure 2. The CV adsorption rates increased dramatically in the first 15 min for various initial concentrations, and reached equilibrium at 150 min. The high adsorption rate during the initial time period was due to a number of available adsorption sites of the activated carbon (Li et al. 2011).



**Figure 4** | Effect of the adsorbent dosage on adsorption of CV dye by the activated carbon (adsorption conditions: initial CV concentration 60 mg/L; 150 rpm; pH 6; 25 °C).

The effect of initial CV concentration on adsorption capacity at optimal pH was determined by using CV solutions with a concentration in the range of 20–100 mg/L. As shown in Figure 5, at equilibrium, the amount of CV adsorbed onto the activated carbon increased from 4.62 to 22.31 mg/g as the initial concentration increased from 20 to 100 mg/L, which was attributed to the increase in the driving force of the concentration gradient.

### Adsorption kinetics

The adsorption kinetic data were tested by mathematical models, including Lagergren pseudo first order model (Lagergren 1898), pseudo second order model (Ho & McKay 1999), and intraparticle diffusion model (Weber & Morris 1963), with linearized forms as follows.

Lagergren pseudo first order model:

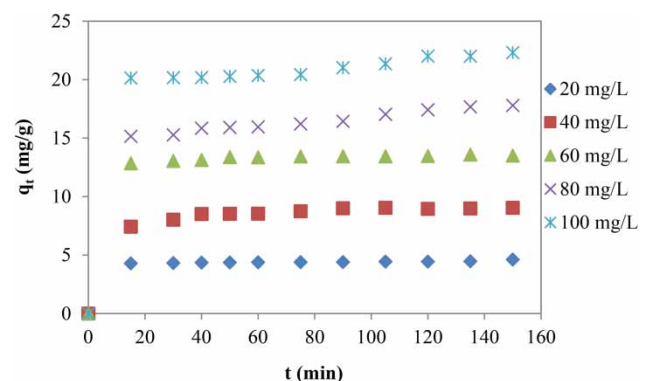
$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} \times t \quad (3)$$

Pseudo second order model:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \times t \quad (4)$$

Intraparticle diffusion model:

$$q_t = k_i \times t^{0.5} + C \quad (5)$$



**Figure 5** | The effect of contact time on the adsorption of CV onto the activated carbon at different initial CV concentrations (adsorption conditions: adsorbent dosage 4 g/L; 150 rpm; pH 6; 25 °C).

where  $q_e$  and  $q_t$  are the amounts of CV adsorbed (mg/g) at equilibrium and at any time  $t$  (min);  $k_1$  ( $\text{min}^{-1}$ ),  $k_2$  ( $\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$ ), and  $k_i$  ( $\text{mg}\cdot\text{g}^{-1}\cdot\text{min}^{-1/2}$ ) are the pseudo first order and pseudo second order and intraparticle diffusion rate constants, respectively.  $c$  is a constant ( $\text{mg}\cdot\text{g}^{-1}$ ) and usually associated with boundary layer contribution, where  $C=0$  indicates that intraparticle diffusion is the only control step in the adsorption process,  $C < 0$  indicates that the liquid film thickness hinders the intraparticle diffusion process, and  $C > 0$  indicates that there is a rapid adsorption stage in a short period at the beginning of the adsorption process.

Figure 6 shows the pseudo second order model plot at different initial concentrations. As can be seen in the figure, the  $t/q_t$  versus  $t$  is linear for each initial concentration.

The kinetic parameters obtained by linear regression for the Lagergren first order and pseudo second order kinetic models are given in Table 1. Correlation coefficient ( $R^2$ ) values for pseudo second order model are found to be closer to unity and the calculated  $q_e$  values agree well with the experimental values ( $q_{e,exp}$ ). Thus, it is determined that the pseudo second order model is suitable for the CV adsorption onto the activated sludge. The kinetic model shows that the adsorption of the CV on the activated sludge is controlled by electrostatic attraction (Qiu et al. 2009). Also, the external film diffusion may not be the rate-limiting step for the CV adsorption onto the activated sludge (Li et al. 2019).

The Lagergren first order model reveals that the adsorption is physisorption, which leads to a slow adsorption

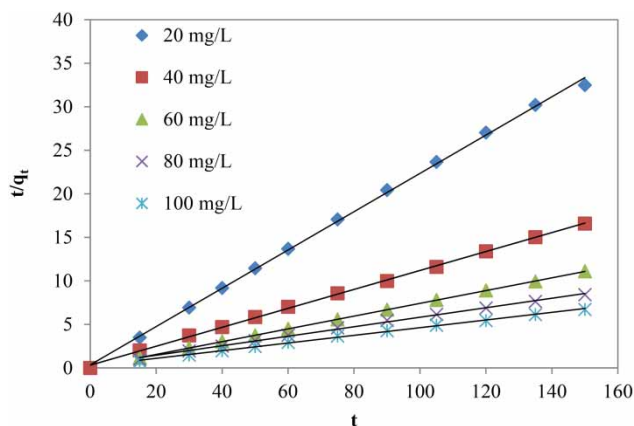


Figure 6 | Plot of the pseudo second order equation at different initial concentrations.

Table 1 | Kinetic constants of Lagergren first order and pseudo second order kinetic models for adsorption of CV onto the activated carbon

Initial CV concentration (mg/L)	$q_{e,exp}$ (mg/g)	Lagergren first order kinetic model $R^2$	Pseudo second order kinetic model $q_{e,cal}$ $k_2$ $R^2$ (mg/g) ( $\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$ )		
20	4.62	0.5550	4.55	0.1339	0.9990
40	9.05	0.7877	9.20	0.0363	0.9993
60	14.26	0.7972	14.43	0.0334	0.9999
80	17.79	0.7568	18.31	0.0080	0.9974
100	22.31	0.6639	22.73	0.0085	0.9979

process. However, the pseudo second order model shows a good fit for the adsorption of CV on the activated carbon, therefore, we proposed that both chemisorption and physisorption mechanisms might take place for the adsorption process. CV adsorption shows physicochemical characteristics (Ai et al. 2019), but the chemisorption is to be the rate-controlling step in this study. These results are in agreement with other studies in the literature (Fabryanty et al. 2017; Ai et al. 2019; Yuan et al. 2019).

Figure 7 shows the results of kinetic data linearization according to intraparticle diffusion model (Equation (5)).

The intraparticle diffusion model generally includes three steps, which are external mass transport, intra-particle diffusion, and adsorption of adsorbate on the interior surface site of adsorbents.

The overall rate of adsorption may be controlled by one of the steps or a combination of more steps. For the all concentrations (20–100 mg/L), the adsorption process tends to be divided into two stages. Multi-linear plots were observed and none of them passed through the origin. The first

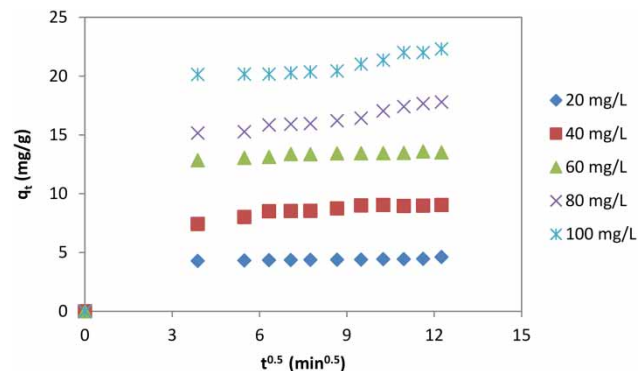


Figure 7 | Intraparticle diffusion plot for adsorption of CV onto the activated carbon.

portion and second portion could be assigned to the intraparticle diffusion steps and the final equilibrium stages, respectively (Li et al. 2019). All parameters of the intraparticle diffusion model are listed in Table 2.

### Adsorption isotherms

Equilibrium adsorption data were modeled by using two conventional isotherm models, namely, Freundlich and Langmuir isotherm models, that are presented in Equations (6) and (7), respectively (Freundlich 1906; Langmuir 1918; Freundlich & Heller 1939):

$$\ln q_e = \ln K_F + \frac{1}{n} \times (\ln C_e) \quad (6)$$

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{(q_m \times K_L)} \quad (7)$$

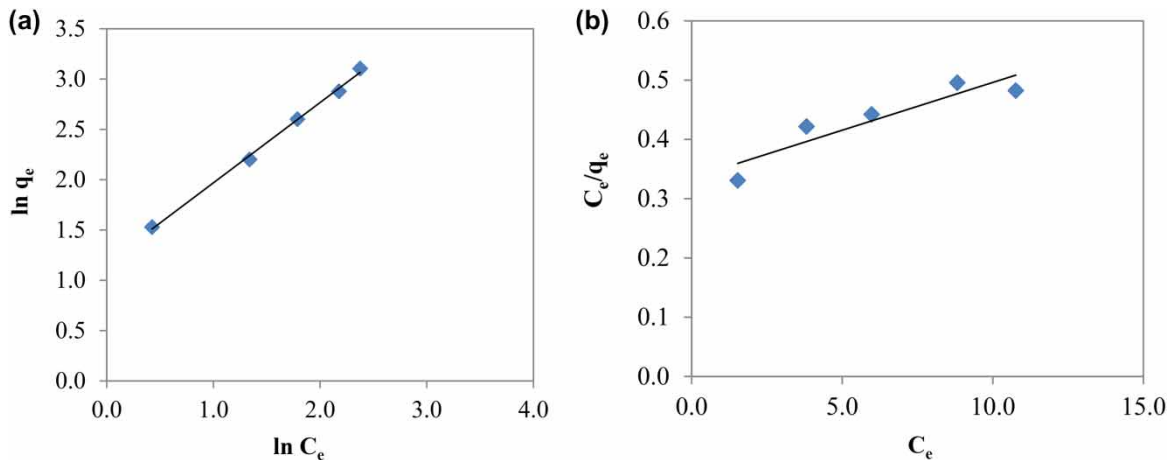
where  $C_e$  (mg/L) is the equilibrium concentration and  $q_e$  (mg/g) is the equilibrium adsorption capacity,  $q_m$  (mg/g) is the saturated adsorption capacity.  $K_L$  (L/mg) is the Langmuir adsorption constant related to the adsorption capacity.  $K_F$  is the Freundlich adsorption constant related to the adsorption capacity.

The linearized Langmuir and Freundlich isotherm plots for CV dye adsorption onto the activated carbon are shown in Figure 8(a) and 8(b).

Table 3 summarizes the Freundlich and Langmuir isotherm parameters. The Langmuir isotherm model describes the chemisorptions and monolayer coverage of adsorbates onto adsorbents. However, it is clear that the Freundlich isotherm describes better with a higher correlation coefficient ( $R^2 = 0.9973$ ) in comparison with that of Langmuir ( $R^2 = 0.8466$ ), suggesting that distribution of active sites of the adsorbents is exponential and the

**Table 2** | Parameters of the intraparticle diffusion model for adsorption of CV onto the activated carbon

Initial CV concentration (mg/L)	Intraparticle diffusion model					
	$k_{int,1}$ (mg.g <sup>-1</sup> .min <sup>-0.5</sup> )	$C_1$ (mg/g)	$R_1^2$	$k_{int,2}$ (mg.g <sup>-1</sup> .min <sup>-0.5</sup> )	$C_2$ (mg/g)	$R_2^2$
20	0.0264	4.1838	0.9786	0.0364	4.0838	0.7090
40	0.4347	5.7176	0.9863	0.1051	7.8327	0.7811
60	0.1201	12.379	0.9958	0.0351	13.111	0.7729
80	0.2561	14.090	0.7337	0.4087	12.819	0.9617
100	0.0124	20.100	0.9569	0.4343	16.987	0.9514



**Figure 8** | Linearized (a) Freundlich and (b) Langmuir isotherm plots for CV dye adsorption by the activated carbon (adsorption conditions: initial CV concentration 20–100 mg/L; adsorbent dosage 4 g/L; 150 rpm; pH 6; contact time 150 min; 25 °C).

**Table 3** | Freundlich and Langmuir isotherm constants for the adsorption of CV onto the activated carbon

Freundlich adsorption isotherm			Langmuir adsorption isotherm		
$K_F$ [mg/g (L/g) <sup>1/n</sup> ]	n	R <sup>2</sup>	$q_m$ (mg/g)	$K_L$ (L/mg)	R <sup>2</sup>
3.2258	1.2536	0.9973	62.11	0.0481	0.8466

adsorption process occurs on a heterogeneous surface. The smaller value of  $1/n$  illustrates the stronger adsorption intensity. The value of  $n$  (1.2536) is between 1 and 10, which shows a favorable process for adsorption of CV onto the activated carbon (Wong *et al.* 2018). In addition, the  $n$  parameter is larger than 1, indicating the physisorption phenomena of adsorption on the activated carbon (Jedli *et al.* 2018).

### Adsorption thermodynamics

The thermodynamic parameters, such as standard enthalpy change ( $\Delta H^\circ$ , kJ/mol), standard entropy change ( $\Delta S^\circ$ , J/mol.K), and standard Gibbs free energy change ( $\Delta G^\circ$ , kJ/mol) were investigated by using the following equations:

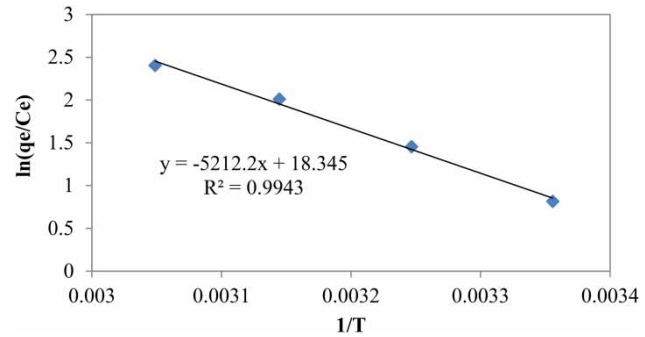
$$\ln K_c = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (8)$$

$$K_c = \frac{q_e}{C_e} \quad (9)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (10)$$

where  $R$  is the universal gas constant,  $K_C$  is the equilibrium constant, and  $T$  is the absolute temperature (K). The values of  $\Delta H^\circ$  and  $\Delta S^\circ$  were calculated from the slope and intercept of the Van't Hoff linear plot of  $\ln K_C$  versus  $1/T$  (Figure 9).

The thermodynamic parameters obtained from the Van't Hoff plot (Figure 8) and Gibbs free energy equation are given in Table 4. The positive  $\Delta H^\circ$  value of the CV adsorption shows that the process is endothermic in nature. The positive  $\Delta S^\circ$  value implies that there is an increased randomness on the activated carbon surface during the adsorption of CV. Obtained negative  $\Delta G^\circ$  values indicate that the process is spontaneous and feasible in nature.  $\Delta G^\circ$  is conversely related to temperature suggesting that at higher temperatures the adsorption

**Figure 9** | Van't Hoff plot for the adsorption of CV on the activated carbon (adsorption conditions: initial CV concentration 60 mg/L; adsorbent dosage 4 g/L; 150 rpm; pH 6; contact time 150 min; 25–55 °C).**Table 4** | Thermodynamic parameters of CV adsorption onto the activated carbon at various temperatures

T (K)	$\Delta H^\circ$ (kJ/mol)	$\Delta S^\circ$ (J/mol.K)	$\Delta G^\circ$ (kJ/mol)
298	43.33	152.5	-2.117
308			-3.642
318			-5.167
328			-6.692

process is more efficient (Rodrigues *et al.* 2019). As shown in Table 4, the decrease in  $\Delta G^\circ$  (from -2.117 kJ/mol to -6.692 kJ/mol) with the increase of temperature (from 298 K to 328 K) indicates more feasibility of the adsorption at higher temperature values. The  $\Delta G^\circ$  for physisorption and chemisorption adsorption processes are in the range of -20 to 0 kJ.mol<sup>-1</sup> and -80 to -400 kJ.mol<sup>-1</sup>, respectively (Dalle *et al.* 2018). Our  $\Delta G^\circ$  is within the range of -20 to 0 kJ.mol<sup>-1</sup>, indicating that the CV dye adsorbed onto the surface of activated carbon is through a physisorption mechanism.

### CONCLUSIONS

The removal of CV from aqueous solution by using the activated carbon derived from waste active sludge has been investigated under different experimental conditions in batch mode. The effects of solution pH, adsorbent dosage, initial CV concentration, contact time, and temperature were examined. Adsorption of CV onto the activated carbon was strongly influenced by the contact time, the



adsorbent dosage, the dye concentration, and the temperature, while variations in pH were found to have a minor effect on adsorption. The Freundlich isotherm was found to be the best fit model for the adsorption of CV. The adsorption kinetic data that followed the pseudo second order model were better than the pseudo first order model and intra-particle diffusion model and indicated by higher  $R^2$  values. The thermodynamic investigation indicated that the CV adsorption derived from the activated carbon was spontaneous and endothermic in nature.

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