

Adsorption of Crystal Violet Dye onto Anionic Polyacrylamide-Modified Graphite: Equilibrium, Kinetics, and Mechanism

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Abstract. The textile industry is a major source of wastewater with a high concentration of organic and inorganic chemicals, including dyes. In this study, exfoliated graphite was modified using surfactants sodium dodecyl sulfate (SDS) and N-cetyl-N,N,N-trimethyl ammonium bromide (CTAB) as well as coagulant anionic polyacrylamide (APAM) to improve the adsorption capacity of pure exfoliated graphite to treat simulated crystal violet (CV) dye. This work also studied the effect of contact time, dye concentration, pH and adsorbent loading. Furthermore, adsorption kinetics were investigated by varying contact time and dye concentration. It was observed that modification with surfactants SDS and CTAB did not show appreciable results. However, modification of graphite flakes with APAM resulted in 89.27% removal efficiency compared to unmodified graphite flakes. The graphite flakes were characterized using XRF analysis, BET surface area, and FTIR analysis. UV/Vis-Spectrometer analyzed the concentration of dye solution at a wavelength of 592nm. The availability of active sites and its ability to regenerate the exfoliated graphite made it a potential candidate for the adsorption of CV dye. Moreover, Langmuir and Freundlich adsorption models were applied to investigate dye adsorption behavior. It was observed that the Langmuir model provides the best fit for this system.

Keywords: Adsorption Capacity, Anionic Polyacrylamide (APAM), N-Cetyl-N,N,N-Trimethyl Ammonium Bromide (CTAB), Removal Efficiency, Sodium Dodecyl Sulfate (SDS)

INTRODUCTION

Climate change has induced the threat of water scarcity worldwide. Climate change has changed precipitation patterns that brought prolonged droughts. It has far-reaching and interconnected effects on the whole world (Chan et al., 2024; Gilli et al., 2024). Water is becoming a rare commodity due to the wastage at different

process stages and its careless use in industry. The industrial sector is a major consumer of water, and at the same time a major contributor to wastewater. Pulp and paper, fertilizer, printing, and textile sector are major contributors to wastewater (Mittal et al., 2010). Particularly the textile industry consists of permanent coloring dyes discharged in water channels. Most of the dyes used in textile industry are

carcinogenic in nature as well as harmful to aquatic life (Liu et al., 2013; Li et al., 2015). Wastewater in the textile industry mainly consists of coloring agents, including permanent dyes. These dyes can be removed by various physicochemical methods like coagulation and flocculation, adsorption, membrane filtration, and chemical methods like ozonation (Khaled et al., 2009). One effective and suitable process for removing dye from wastewater is adsorption.

Graphite flakes are unique in nature due to their adsorption capability as well as regeneration with cost-effectiveness (Hoang et al., 2019). Hence, in the future, an independent and thorough investigation of the re-usability and regeneration potential of this adsorbent would be of greater interest for further studies. Moreover, many developed processes include modification of adsorbent to increase its adsorption capacity. Such modification is accomplished with chemicals, e.g. acids, alkali, anions, cations, anionic coagulants, and surfactants (Rossner et al. 2009).

Sodium dodecyl sulfate, commonly known as SDS, is an organic origin compound with the chemical formula $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$. It is a surfactant with anionic nature that is used as an emulsifier, wetting agent, pharmaceutical, and detergent (Cheruiyot et al., 2019). SDS is a surfactant that decreases the interfacial tensions and stabilizes the interface. It helps the particles to adhere to their surface. Using surfactants modifies the surface of the adsorbent and help to attach the impurities with weak Van Der Waals forces (Nandi et al., 2008). Therefore, using surfactant to modify adsorbent enhances the adsorption efficiency. However, one of the major obstacles to using sodium dodecyl sulfate is

its emulsifying properties. Graphite is not soluble in polar and non-polar solvents but can be suspended by emulsifiers. Similarly, N-cetyl-N,N,N-trimethyl ammonium bromide (CTAB) is a quaternary ammonium surfactant with molecular formula $\text{C}_{19}\text{H}_{42}\text{BrN}$. It is a solid odorless powder with a white colour with cationic nature and the ability to be used as an emulsifying agent. However, the problem encountered with the usage of CTAB is its emulsifying property (Wu et al., 2013). Moreover, anionic polyacrylamide (APAM) is a water-soluble polymer with high affinity, the ability to be polymerized, high solubility in water, and is soluble in inorganic solvents. APAM is also preferred due to its excellent coagulation properties, easy handling, and it is cost-effectiveness. It also enhances the adsorption properties of graphite to be used as an adsorbent for dye extraction (Cheruiyot et al., 2019). In this study, APAM is used as modifying agent for increasing the adsorption capacity of graphite. Crystal violet (CV) is also known as methyl violet or hexamethyl pararosaniline chloride. Crystal violet dye is naturally carcinogenic and classified as an in subordinate molecule (Mittal et al., 2010; Nandi et al., 2008). The removal of methylene blue and malachite green dyes is also possible, along with crystal violet dye, by using APAM-modified graphite as an adsorbent (Mittal et al., 2010; Nandi et al., 2008). Therefore, it is compulsory to remove this dye to protect human health and also for environmental protection. Adsorption isotherms have proved extremely useful in studies involving adsorption strategies. The Freundlich and Langmuir isotherms are the two basic approaches for the estimation of adsorption capacity, understanding the adsorption mechanism, and optimizing

adsorption conditions across various applications.

MATERIALS AND METHODS

Chemicals and Reagents

Crystal Violet (CV) dye was obtained from Spectrum Chemical MFG CORP with CAS No. 548-62-9. The molecular Formula and Molecular weight of CV are $(C_{25}H_{30}N_3Cl)$ and 407.9 g mol^{-1} , respectively. Similarly, sodium dodecyl sulfate (SDS) was used to modify graphite. It was obtained from BDH Laboratory Supplies Poole, England, with CAS No. 121-51-3. N-cetyl- N,N,N-trimethyl ammonium bromide (CTAB) having CAS No. 57-09-0 was also used to modify graphite and obtained from UNI-CHEM Chemical Reagents. Polyacrylamide obtained from Lan Yao Chemicals China with CAS No. 9003-05-8 was used to treat graphite to enhance its removal efficiency.

Characterizations

Graphite and APAM modified graphite characterization consist of some common analysis i.e. pH_{pzc}, X-Ray Fluorescence (XRF), Fourier Transform Infra-Red Spectroscopy (FTIR), and N₂-sorption analysis to obtain Brunauer-Emmet-Teller (BET) specific surface area.

Adsorption Studies

The adsorption performance of graphite was determined by calculating removal efficiency while changing contact time and keeping initial concentration constant and changing the initial concentration of dye while keeping graphite dosage constant (Hoang et al., 2019). Similarly, adsorption performance of SDS and CTAB modified graphite was studied. Finally, APAM modified graphite adsorption performance was studied

using a Perkin Elmer Lambda 25 UV/Vis-Spectrometer.

Graphite Modification

A stock solution of crystal violet of 100 ppm was prepared. Similarly, ten dilutions were made from the stock solution. The surfactant of sodium dodecyl sulfate (SDS) was taken 1 gram to prepare a 200 ml solution by using deionized water. The solution was kept on a magnetic hot plate and stirrer it for 2 hours and then sonicated for 30 minutes. About 20 g of graphite was measured and added to the solution of SDS. The solution was stirred using a magnetic hot plate for 2 hours and then sonicated for 30 minutes. The solution was filtered and then dried in vacuum oven. The procedure was repeated, but using different surfactants of 1 gram N-cetyl-N,N,N-trimethyl ammonium bromide (CTAB) and 0.5 g of coagulant anionic polyacrylamide (APAM).

Adsorption Measurement

The absorbance of each sample was obtained from a Perkin Elmer Lambda 25 UV/Vis-Spectrometer. X-Ray Fluorescence (XRF) analysis was performed to determine the elemental composition of graphite flakes and APAM-modified graphite by an XRF Spectrometer. Fourier Transform Infrared Spectroscopy (FTIR) was used to determine chemical properties, including functional groups. Moreover, a magnetic stirrer hot plate was used to mix the solution thoroughly.

RESULTS AND DISCUSSION

pH_{pzc}

The pH at which there was zero charge on the surface of adsorbent Anionic polyacrylamide modified graphite was

found 8.2. Details of pH variation are listed in Table 1 and based on previous research (Mohan et al., 2007). The change in pH and initial pH are plotted in Fig. 1. The difference in initial and final pH is minimum at pH 8.2. This is the pH at which the surface density of positive charges equals that of negative charges. Therefore, there is maximum adsorption at pH 8.2.

Table 1: pH_{pzc} analysis of APAM modified Graphite

Flask No.	pH _i	pH _f	Δ pH
1	3	7.1	-4.1
2	4	7.4	-3.4
3	5	7.6	-2.6
4	6	7.9	-1.9
5	7	8.1	-1.1
6	8	8.2	-0.2
7	9	8.4	0.6
8	10	8.6	1.4
9	11	8.7	2.3
10	12	8.9	3.1

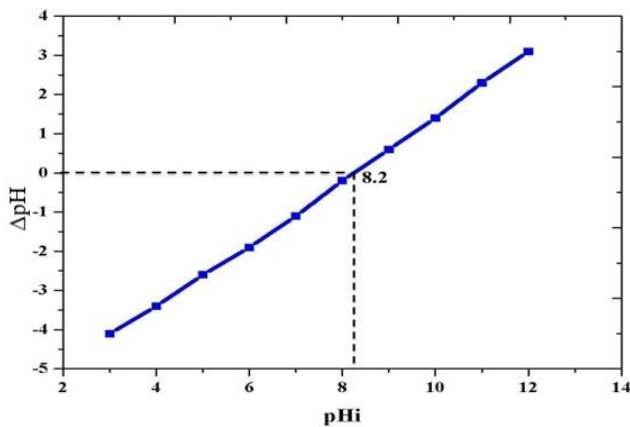


Fig.1: pH_{pzc} analysis of APAM modified graphite

XRF Analysis of Graphite Flakes and APAM modified Graphite Flakes

XRF analysis shows that the maximum quantity of SiO₂ is present in graphite flakes. XRF analysis of anionic polyacrylamide (APAM) modified graphite shows that the

quantity of SiO₂ has increased almost 3% as compared to graphite flakes. Aluminium oxide contents are also increased from 13.21% to 14.98%, as shown in Fig. 2.

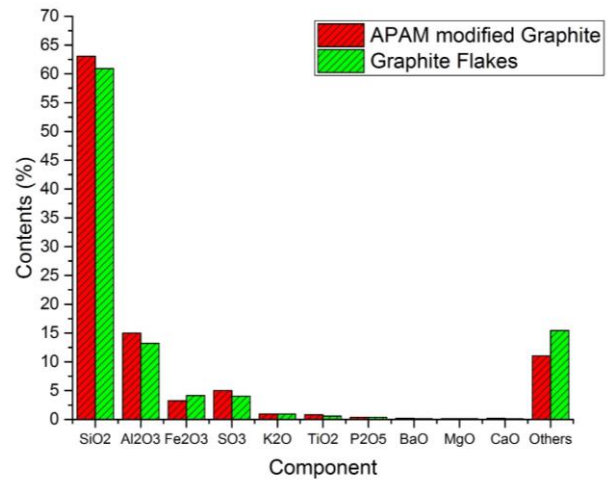


Fig. 2: XRF analysis of APAM modified Graphite

FTIR Analysis of Graphite and modified Graphite

FTIR analysis of graphite and APAM modified graphite is shown in Fig. 3. There is a slight peak at wavenumber 989.2 cm⁻¹ and 1644.7 cm⁻¹. These peaks depict the presence of double bonds in graphite. Similarly, in the case of APAM-modified graphite there is a significant peak at wavenumber 2863.4 cm⁻¹ that represents the presence of C-H bond. Furthermore, there are sharp peaks at wavenumber 3182.9 cm⁻¹, 3397.7 cm⁻¹, and 3482.6 cm⁻¹ due of presence of COOH, N- H and O-H bonds, respectively as shown in Fig. 3. These peaks are evident presence of modification of graphite with anionic polyacrylamide (APAM) (Alsohaimi et al., 2023; Zhang et al., 2023).

A comparison has been drawn that clearly shows the presence of OH, N-H, and COOH groups in modified graphite FTIR analysis. This difference is created due to the presence of these groups in Anionic polyacrylamide (APAM), as shown in Fig. 3.

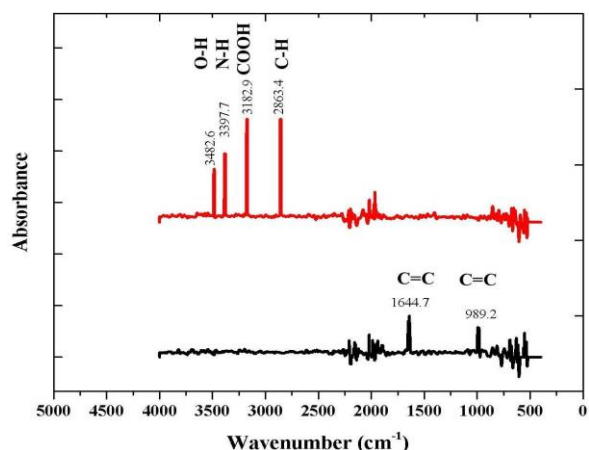


Fig. 3: FTIR analysis of Graphite and modified Graphite

Brunauer-Emmett-Teller (BET) Specific Surface Area

The BET surface area of graphite taken from another source was determined by nitrogen adsorption and found to be around $1.0 \text{ m}^2\text{g}^{-1}$ (Hussain, 2012). This indicates the presence of space for dye adsorption on graphite flakes.

Table 2. Identifying the quantity of adsorbent

Adsorbent (g)	Initial Conc. (ppm)	Final Conc. (ppm)	Initial Adsorption	Final Adsorption	Removal Efficiency (%)
5	100	39.4797	2.7328	1.0789	60.5203
10	100	36.0583	2.7328	0.9854	63.9417
15	100	33.7163	2.7328	0.9214	66.2837
20	100	32.0331	2.7328	0.8754	67.9669
25	100	32.5088	2.7328	0.8884	67.4912
30	100	32.7649	2.7328	0.8954	67.2351

Identifying the Quantity of Adsorbent

The desired quantity of adsorbent is determined by varying the quantity of adsorbent while keeping the initial concentration of dye as shown in Table 2. It can be inferred that the suitable quantity of adsorbent for maximum removal efficiency is nearly 20 grams as shown in Fig. 4.

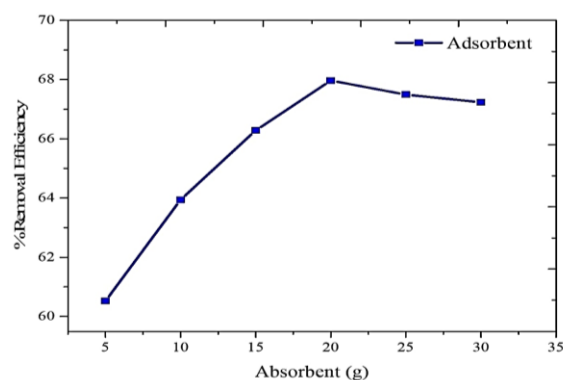


Fig. 4: Identifying the quantity of adsorbent

Adsorption Performances

Adsorption of graphite was determined by an equilibrium approach that calculates removal efficiency while changing contact time and keeping initial concentration constant (Asif et al., 2024). Crystal violet dye removal efficiency increases with increasing contact time. After the contact time of 30 minutes, dye removal efficiency approaches 67.12%, as shown in Fig. 5. There is gradual increase in removal efficiency with respect to time. However, after 25 minutes there is slight decrease in removal efficiency as shown in Fig. 5.

Adsorption performance of SDS was calculated while increasing the contact time and keeping the initial concentration of the dye constant. There is a sharp decrease in removal efficiency with respect to contact time. The reason behind this trend is the emulsifying nature of surfactants (Aref et al., 2017; Cheruiyot et al., 2019). Surfactant-made suspension of graphite and blackish-coloured solution obtained that possesses maximum absorbance. This trend is plotted in Fig. 5.

CTAB surfactant was also used as an adsorbent for dye removal. There is a gradual decrease in the removal efficiency of CTAB due to emulsifying nature of the surfactant (Cheruiyot et al., 2019; Bello et al., 2021). This trend was shown in Fig. 5. Graphite is not soluble in polar and non-polar

solvents, but some emulsifier can suspend it. SDS and CTAB are emulsifying agents. The blackish suspension of Graphite absorbs maximum light.

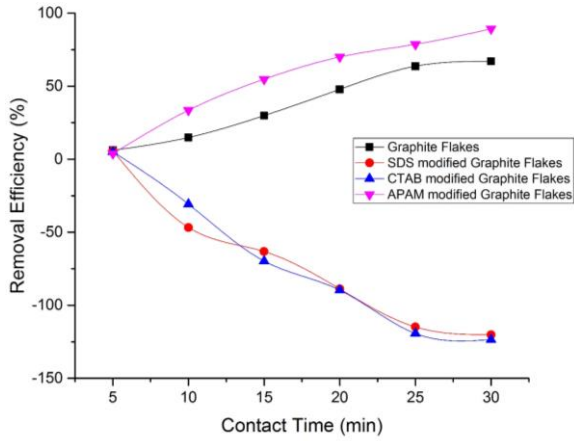


Fig. 5: Adsorption performance of graphite flakes, SDS modified graphite flakes, CTAB modified graphite flakes and APAM modified graphite

APAM modified graphite is used as an adsorbent for the removal of dye. Removal efficiency is increased compared to graphite flakes. Removal efficiency has increased to 89.27% in the case of APAM modified graphite. There is sharp increase in removal efficiency with respect to contact time, as shown in Fig. 5. Removal efficiency increases from 3.79% to 89.27% at the time span of 30 minutes. This increase in removal efficiency is the coagulant nature of APAM (Aref et al., 2017).

Adsorption Isotherm of Crystal Violet onto Graphite

The adsorption capacity of graphite flakes has been studied by keeping the adsorbent dosage constant while, decreasing the initial concentration of dye. Adsorption capacity increases by decreasing the initial concentration of dye, as shown in Table 3. Adsorption capacity increase is due to a decrease in initial concentration of dye.

The mass balance (Eq. (1)) can be used to obtain how much solute adsorbed in the solid. It employs initial concentration (C_0), final concentration (C_f), volume of solution (V), and mass of solid (m).

$$q = (C_0 - C_f) \frac{V}{m} \tag{1}$$

The equation of the Langmuir isotherm model is shown in Eq. (2).

$$q = q_{max} \left(K_L \frac{ce}{1 + K_L ce} \right) \tag{2}$$

where q (mg/g) was the amount of CV dye adsorbed onto the unit mass of graphite/modified graphite flakes; K_L was the Langmuir equilibrium constant related to the attraction of binding sites.

The main characteristics of the Langmuir equation, constants K_L and q_{max} , could be determined from a linearized form of the Langmuir equation as follows.

Table 3: Adsorption capacity of graphite flakes

Adsorbent Dos. (g)	Initial Conc. (ppm)	Final Conc. (ppm)	Initial Adsorption n Abs.	Final Adsorption Abs.	Removal Efficiency	Adsorption Capacity
20	100	93.83	2.73	2.56	6.17	0.03
20	90	76.46	2.70	2.29	15.05	0.07
20	80	57.92	2.66	1.92	27.60	0.11
20	70	45.14	2.30	1.48	35.52	0.12
20	60	30.56	1.95	0.99	49.06	0.15
20	50	16.17	1.8	0.61	67.65	0.17

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

Therefore, a straight line was obtained from the plot of C_e/q_e versus C_e with a slope of $1/q_{max}$ and an intercept of $1/(K_L q_{max})$. The sorption phenomena were predicted by the Langmuir isotherm which applies to adsorption on completely homogenous surfaces with negligible interaction between adsorbed molecules.

Langmuir Isotherm is plotted between equilibrium concentration of adsorbate and C_e/q_e as shown in Fig. 6. There is a sharp increase in C_e/q_e at equilibrium concentration of 79 mg/l.

Table 4: Langmuir isotherm for graphite flakes

Equilibrium Conc. C_e (mg/l)	Adsorption q_e (mg/g)	C_e/q_e (g/l)
93.83	0.03	3026.68
76.46	0.07	1124.35
57.92	0.11	526.55
45.14	0.13	363.99
30.56	0.15	207.92
16.17	0.17	95.70

Fig. 6 gives a graphic representation of Table 4. The Langmuir Isotherm best describes the adsorption trend of graphite flakes.

The constants of the adsorption isotherm are q_{max} , K_L , and R_L (Table 5). These values are obtained from Langmuir's adsorption isotherm. The value of K_L indicates the extent of interaction between adsorbate and adsorbent. A large value of K_L indicates strong interaction, and vice versa. The values of parameter R_L also indicate that the adsorption process is favorable (Foroutan, 2021; Noorimotlagh et al., 2019).

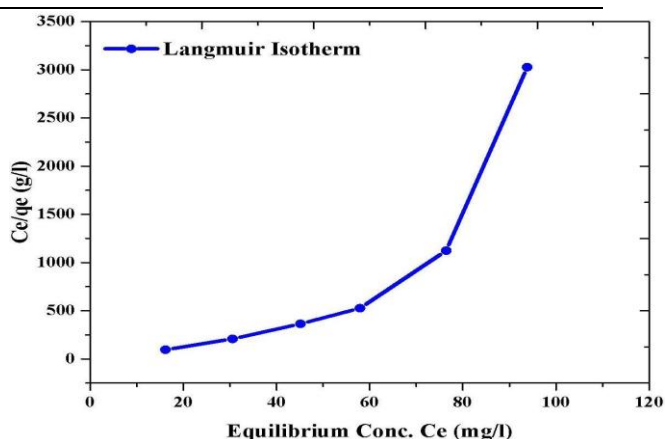


Fig. 6: Langmuir isotherm of graphite flakes

Table 5: Langmuir Constants for adsorption isotherm

Q_{max}	K_L	R_L
19.54	1.49	0.009

The Freundlich isotherm model was based on adsorption on a heterogeneous surface developed an empirical equation as shown in Eq. (4).

$$q_e = K C_e^{1/n} \quad (4)$$

where q_e is the adsorption (mg/g); C_e is the concentration of adsorbate in the solution (mg/L); K_F and n are empirical constants that are characteristics of the system, indicating the adsorption capacity and the adsorption intensity, respectively.

The above equation can be linearized in the following form, also used to confirm the applicability of the model as shown in Eq. (5).

$$\ln q_e = \ln K + \frac{1}{n} \ln C_e \quad (5)$$

where K_F was the measure of sorption capacity, and $1/n$ was the sorption intensity. The plot of $\ln q_e$ versus $\ln C_e$ had a slope with the value of $1/n$ and an intercept magnitude of $\ln K_F$.

Fig. 7 plots Freundlich adsorption isotherm data (Table 6). The Freundlich adsorption constants $1/n$ and K_f are

obtained from this isotherm. The values of the adsorption constants are listed in Table 7.

Table 6: Freundlich adsorption isotherm data for graphite flakes

Log C _e	Log q _e
1.97	-1.51
1.88	-1.18
1.76	-0.96
1.66	-0.91
1.49	-0.83
1.21	-0.77

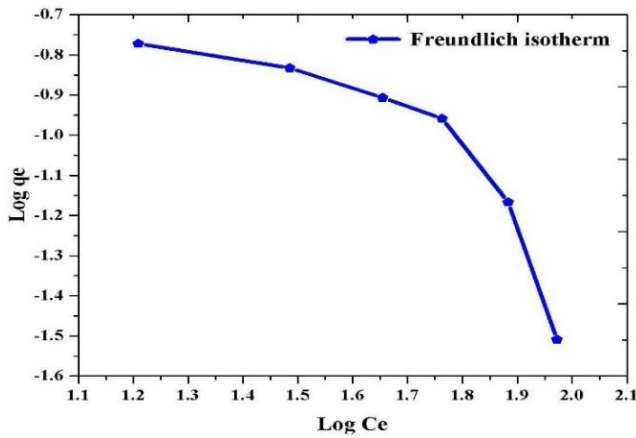


Fig. 7: Freundlich isotherm of graphite flakes

Fig. 7 shows the graphical representation of The Freundlich adsorption isotherm. In the case of hydrophobic

chemicals, non-linearity was observed. The exponent of non-linearity is 1/n. The usual range of the value of 1/n is 0.7 to 1.0. The value of K_f was found to be 2.24, which indicates a desirable bond between CV dye and the surface of graphite flakes.

Table 7: Constants for Freundlich adsorption isotherm

1/n	K _f
0.83	2.24

Adsorption Isotherm of Crystal Violet onto APAM modified Graphite Flakes

The adsorption capacity of APAM modified graphite increased by decreasing the initial concentration of dye. As shown in Table 8, the adsorption capacity increased from 0.19 to 0.22 by decreasing the initial concentration from 100 ppm to 50 ppm. Langmuir adsorption isotherm data were listed in Table 9. As shown in Fig. 8, there is a nearly direct relation between equilibrium concentration and C_e/q_e.

Using Eq. 2 and Eq. 3, the values of q and C_e/q_e were calculated, respectively. Fig. 8 shows the graphical representation of Table 9. Langmuir isotherm best represents the adsorption trend of APAM modified graphite.

Table 8: Adsorption capacity of APAM modified

Adsorbent Dosage (g)	Initial Conc. (ppm)	Final Conc. (ppm)	Initial Adsorption Abs.	Final Adsorption Abs.	Removal Efficiency	Adsorption Capacity
20	100	62.54	2.86	1.79	37.46	0.19
20	90	39.95	2.80	1.24	55.61	0.25
20	80	32.61	2.63	1.07	59.24	0.24
20	70	18.87	2.19	0.59	73.04	0.26
20	60	11.96	1.99	0.40	80.07	0.24
20	50	5.41	1.65	0.18	89.18	0.22

Table 9: Langmuir adsorption isotherm for APAM modified graphite

Equilibrium Conc. C_e (mg/l)	Adsorption q_e (mg/g)	C_e/q_e (g/l)
62.54	0.19	334.44
39.95	0.25	159.80
32.61	0.24	137.60
18.87	0.26	73.71
11.96	0.24	49.83
5.41	0.22	24.26

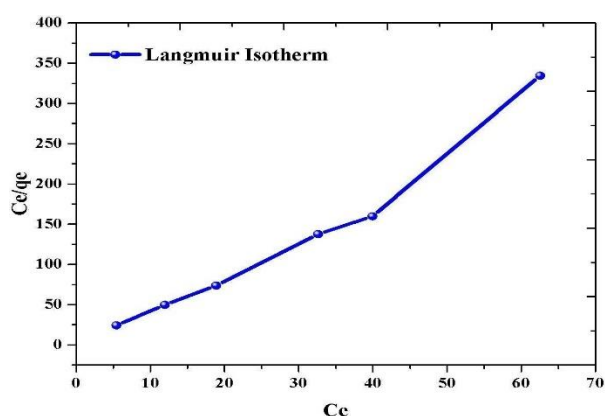


Fig. 8: Langmuir isotherm for APAM modified graphite

Table 10 lists the Langmuir constants i.e. q_{max} , K_L and R_L for APAM modified are listed in Table 10. The constants of the adsorption isotherm are Q_{max} , K_L and R_L . These values are obtained from the Langmuir adsorption isotherm. The value of K_L indicates the extent of interaction between adsorbate and adsorbent. In the case of large value of K_L , there is a strong interaction and vice versa. Hence, Langmuir best describes the adsorption trend of APAM modified graphite.

Using Eq. 4 and its linear form, Eq. 5, a Freundlich adsorption model for APAM-modified graphite was developed. The Freundlich adsorption isotherm for APAM-modified graphite is shown in Figure 9. Overall, this depicts an irregular pattern overall.

Table 10: Langmuir constants for APAM modified graphite

q_{max}	K_L	R_L
35.39	1.70	0.058

Table 11: Freundlich Adsorption Isotherm for APAM modified Graphite

Log C_e	Log q_e
1.80	-0.73
1.60	-0.60
1.51	-0.63
1.28	-0.59
1.08	-0.62
0.73	-0.65

The constants for Freundlich Isotherm were calculated from Eq. (4) and Eq. (5) as given in Table 12.

Table 12: Constants for Freundlich Adsorption Isotherm

$1/n$	K_f
0.83	2.24

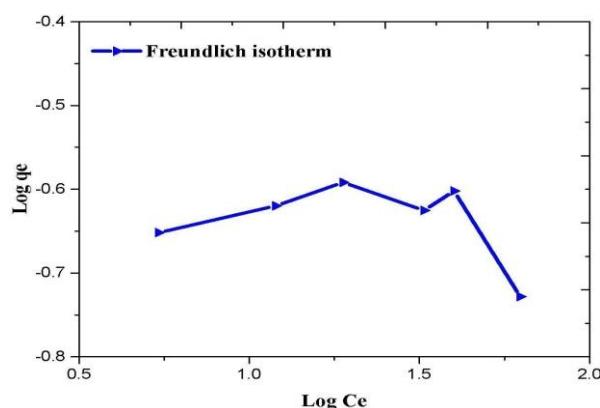


Fig. 9: Freundlich Isotherm for APAM modified Graphite

Comparison of Change in Concentration with Time (Graphite and APAM Modified Graphite)

Table 13 and Table 14 compare the change in concentration in the case of

graphite and APAM-modified graphite. For easy manner, the tables are also plotted in Fig. 10. In the case of graphite as an adsorbent, the concentration of dye decreased from 85.54 ppm to 25 ppm in the time span of 30 minutes. Similarly, in the case APAM modified graphite, the concentration dropped from 89.60 ppm to 5.46 ppm in the same time period. Hence, APAM modified graphite is a more effective adsorbent than graphite.

Table 13. Change in concentration with time in case of graphite

Time (minutes)	Absorbance	Conc. (ppm)
0	2.73	85.54
5	2.56	79.99
10	2.33	72.11
15	1.92	58.60
20	1.43	42.40
25	0.99	28.09
30	0.90	25.01

Table 14. Change in concentration with time in case of APAM modified graphite

Time (Minutes)	Absorbance	Conc. (Ppm)
0	2.86	89.61
5	2.68	83.80
10	1.90	57.97
15	1.29	37.93
20	0.87	24.10
25	0.61	15.45
30	0.31	5.47

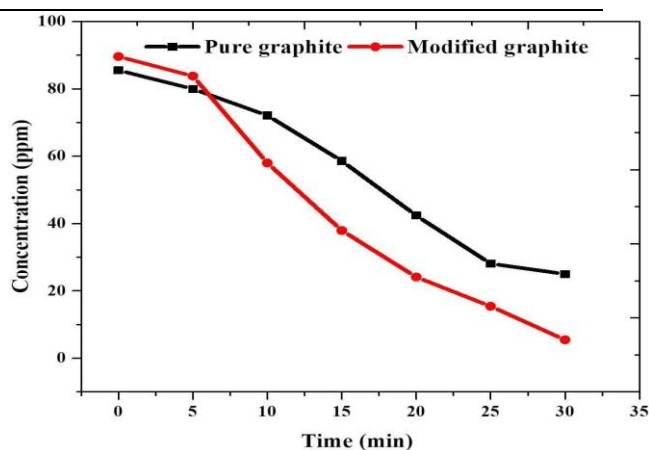


Fig. 10: Comparison of Change in Concentration with Time

CONCLUSIONS

This work is performed to sort out ways for graphite modification and its effectiveness in dye removal from wastewater during its treatment. The emulsifying property of surfactant is the main hindrance to graphite modification by surfactant. There is maximum absorption obtained in the presence of black color that is basically resulting from the suspension of graphite. Therefore, the use of surfactant for graphite modification to enhance its adsorption capacity is not suitable due to emulsifying power of surfactants. However, APAM modified graphite and dyeing wastewater has opposite Charges, which cause attraction and resulted in more dye removal from wastewater than usual graphite only. After enhancement and modification of graphite by anionic PAM, the graphite surface charge was altered from positive to negative, which pulled and attracted to the positive charges of crystal violet dye that is present in wastewater, and the crystal violet removal efficiency has improved many folds. The outer surface of the modified graphite was covered by anionic polyacrylamide through a

certain force of attraction, providing suitable attraction sites for the crystal violet dye. Kinetic model Langmuir isotherm best represents the adsorption trend of APAM modified graphite. Hence, modification of graphite by anionic polyacrylamide is one of the suitable pair for the adsorption of crystal violet dye.

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