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Carbon Nanotubes for Removal of Fast Green Dye

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Authors' contributions

This work was carried out in collaboration among all authors. Author HMS designed the study, performed the statistical analysis, wrote the protocol supervised and reviewed all drafts of the manuscript. Authors SAMS and MMM carried out the research. All authors read and approved the final manuscript.

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ABSTRACT

The adsorption of Fast Green (FG) dye on pristine multi-walled carbon nanotubes (CNTs) was investigated in this study. The adsorption was carried out under different operating conditions. The operating conditions were contact time, adsorbent dosage, initial dye concentration, the pH of the solution and temperature. The analysis of results found that the removal percentage of FG dye on CNTs decreases with increase in initial FG concentration. It was found that the increasing of CNTs dosage enhanced the efficiency of dye removal. It was also found that the increasing of temperature significantly enhanced the removal percent of FG dye and it is indicated that the adsorption of FG dye on CNTs was in endothermic nature. The study reports that the best pH of solution for the adsorption of FG on CNTs were 4. The adsorption data have been analyzed using Langmuir and Freundlich. Fitting the equilibrium adsorption data by Langmuir and Freundlich models shows that experimental data well explained by the Langmuir equation.

Keywords: Carbon nanotubes; adsorption isotherms; dye removal; organic pollutants; wastewater treatment.

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1. INTRODUCTION

The discharge of organic pollutants from industries has become an important problem of environmental concern because of importance of this natural resource in the energetic matrix. Polycyclic aromatic hydrocarbons, e.g. fertilizers, dyes, plasticizer, pesticides, phenols and oils, are one of the main groups of these organic pollutants. These pollutants can remain in the environment for long period of time because of their high degree of aromaticity and conjugation [1]. Dyes are extensively discharged by industries and considered the main type of pollutants and posing hazard to living organisms [2,3]. They have many applications in different fields such as dyeing and printing on fibers and fabrics of all kinds, food coloring, as well as for medicinal and cosmetic uses [4].

Various methods in wastewater treatment technology have been performed to remove or degrade organics from aqueous solutions including ozonation, electrochemical treatment, photodegradation, evaporative recovery, adsorption, ion exchange [5-14]. Adsorption has been proved to be an effective and one of the most extensively used approaches for the removal of organic and inorganic pollutant from wastewater [15-17]. Many adsorbents have been developed and modified for this purpose. Among these absorbents, activated carbon is the most commonly due to its excellent capacity of adsorption for organic pollutants [18]. More recently, nano-structured materials have been widely used to remove toxic substances. CNTs can served as excellent adsorbents and widely studied carbon nanomaterials due to their large specific surface area and hollow and layered structure [19,20].

Recently, CNTs has received great interest in water treatment due to its small size, high aspect ratio, large accessible specific surface area, well-developed mesopores, as well as easily modified surfaces. Large surface area and high porosity provide enough adsorption sites for harmful contaminations present in wastewater. High aspect ratio of CNTs makes it a possible candidate for water purification. In recent years, great efforts have been made to remove various organic pollutants and metal ions in wastewater by CNTs [21]. To further improve their adsorption performance, various CNTs composites have been synthesized and widely used to remove dye pollutions from wastewater. However, the adsorption capabilities are not entirely satisfactory and it is still a challenge to explore novel CNTs composites adsorbents with high adsorption capacity, short adsorption time as well as low cost for practical utilization.

The FG dye has been found to have tumorigenic effects in experimental animals, as well as mutagenic effects in both experimental animals and humans. It furthermore risks irritation of eyes, skin, digestive tract, and respiratory tract in its undiluted form [22]. Since CNTs have emerged as a good adsorbent for dye removal from wastewater, therefore, the main aim of this study is to use pristine CNTs for FG dye removal and investigate the ability of this CNTs as adsorbent. The effect of FG concentration, CNTs dose, pH and temperature on the adsorption process were also studied. We also interested to study the two well-known isotherms, Langmuir and Freundlich.

2. MATERIALS AND METHODS

2.1 Materials

The FG dye was purchased from Hopkin and Williams Ltd, UK, and used as received to prepare solutions that used in this research. The characteristics of FG are shown in Table 1. The maximum absorbance wavelength (λ_{max}) of FG dye was found to be 625 nm. A stock solution of FG (100 mg/L) was prepared on a daily basis in distilled water and other concentrations (3, 5, 7 and 9 mg/L) were prepared by dilution the stock solution of FG. The stock solution of FG (100 mg/L) was covered by aluminum foil and stored in a dark place. Multi-walled pristine CNTs were purchased from Hydale Ltd., UK used as received without further treatment. Properties of the CNTs used are shown in Table 2. Sodium hydroxide (NaOH) and Nitric acid (HNO_3) were purchased from Fisher-Scientific, UK. Various molarities of HNO_3 and NaOH were used pH adjustment between 2 and 11.

2.2 Adsorption isotherm

CNTs with 10 mg was used as adsorbent and four solutions of FG with initial concentration 3, 5, 7 and 9 mg/L were used. This involved using 100 mL of FG in a reactor and placed on a stirrer for better mixing for 60 min. Samples were taken at a specific schedule, 2 mL of sample, using a glass syringe with 10 mL. A set of experiments is performed to study the effect of initial dye concentration, adsorbent dose, temperature and pH of the solution.

Table 1. Fast Green dye characteristics

Property	Value
C.I. index	C.I. 42053
Molecular Formula	$C_{37}H_{34}N_2Na_2O_{10}S_3$
Molar Mass	808.86 g/mole
IUPAC Name	Disodium2-[[4-[ethyl-[(3-sulfonatophenyl)methyl]amino]phenyl]-[4-[ethyl-[(3-sulfonatophenyl)methyl]azaniumylidene]cyclohexa-2,5-dien-1-ylidene]methyl]-5-hydroxybenzenesulfonate
Colour	Blue
λ_{max}	625 nm
pKa	3.53
Solubility	Soluble in water (20 g/100 mL)

Table 2. Properties of the CNTs used in this research work

Property	Value
Carbon Content (%)	96
Outer Diameter (nm)	~ 13-16
Length (μm)	~ 1
Aspect ratio (length/diameter)	~ 69
Bulk Density (g/cm^3)	~ 0.19
BET surface area (m^2/g)	~ 250
Ash	~ 1.5

For separating adsorbents from the taking sample before analysis, a centrifuge was used. The samples were analysed using spectrophotometer with maximum absorbance 625 nm.

The following equation was used to calculate the percentage of FG removal:

$$\%\ Removal = \frac{C_o - C_e}{C_o} * 100\ \% \quad (1)$$

Where,

C_o is the concentration of FG dye at initial and C_e is the concentration at equilibrium.

The amount of dye adsorbed per unit mass of adsorbent was calculated as follows:

$$q_e = \frac{C_o - C_e}{\mathrm{m}} * \mathrm{V} \quad (2)$$

Langmuir and Freundlich models were applied for describing the adsorption of FG on CNTs. The Langmuir model can be written in a linear form as in below equation In order to obtain Q_m and b values [23,24]:

$$\frac{C_e}{q_e} = \frac{1}{b\, Q_m} + \frac{C_e}{Q_m} \quad (3)$$

Where,

C_e is the equilibrium concentration of sorbent (mg/L), Q_m is the maximum sorption capacity (mg/g), q_e is the mass of FG adsorbed per unit mass of adsorbent at equilibrium (mg/g), b is the Langmuir adsorption constant (L/mg).

On the other hand, the Freundlich isotherm can be obtained by the equation 3. For non-ideal adsorption and when the surface is heterogeneous the Freunlidh isotherm is used [25]. Where n_f is the Freundlich coefficient and K_f is the Freundlich constant. A linear equation is used to determine K_f and n_f:

$$ln q_e = ln K_f + \frac{1}{n_f} ln C_e \quad (4)$$

3. RESULTS AND DISCUSSION

3.1 Effect of Contact Time

The effect of contact time was studied for an initial concentration of FG (7 mg/L) and 10 mg of CNTs (see Fig. 1). The influence of time on the adsorption of FG dye was examined by measuring the UV-Vis adsorption spectra for FG solution. Fig. 1 displays that the adsorption of FG was occurred rapidly from the beginning of the experiments during the first 5 min, then the adsorption is slight increased until 40 min where the maximum adsorption of FG on CNTs was located. Between 40-60 min, there was no further increase in the adsorption of FG and 60 min was chosen as an equilibrium time. The slow rate of FG adsorption at long period of time may result from the saturation of the CNTs with the FG dye.

3.2 Effect of Dye Concentration

The effect of dye concentration on removal efficiency using CNTs was investigated and is shown in Fig. 2. The experimental results of FG adsorption on CNTs were obtained at various concentrations (3, 5, 7 and 9 mg/L). As observed from figure, the efficiency of FG removal decreased with increasing the initial FG concentration. However, as the initial dye concentration increased, the actual amount of FG adsorbed per unit mass of CNTs increased, as a results of increased concentration gradient between adsorbate in solution with higher concentration of dye and adsorbate in the adsorbent.

3.3 Effect of pH

The oxidation states and hence the properties of CNTs are mainly rely on the pH of the solution [26]. The effect of pH on adsorption of dye on CNTs is shown in Fig. 3. The surface charge of the adsorbents and the degree of ionization of dye are influenced by the pH of the solution due to the protonation and deprotonation of the functional groups [27]. The adsorption of other ions is affected by the pH of the solution because the hydrogen ion (H^+) and hydroxyl ion (OH^-) are strongly adsorbed to the surface of adsorbent. The effect of pH on removing of dye was studied at different pH [2, 4, 6.3, 9, 11] at fixed dye concentration 7mg/L and 0.01g of CNTs. As shown in Fig. 3, the removing percent increases in acidic medium and have maximum value at pH 4 and decreases when solution become basic have minimum value at pH 11.

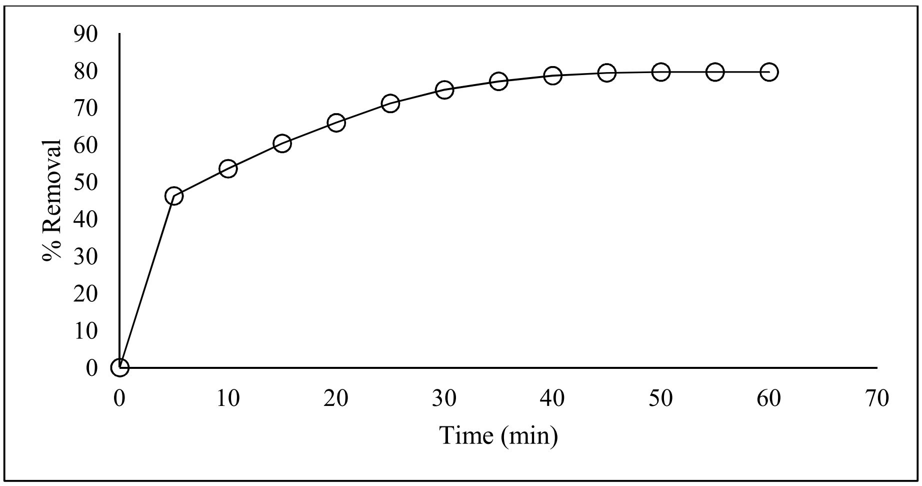

Fig. 1. Effect of contact time on adsorption of FG on CNTs (CNTs = 10 mg, T = 20 °C, V = 100 mL, pH = 6.3)

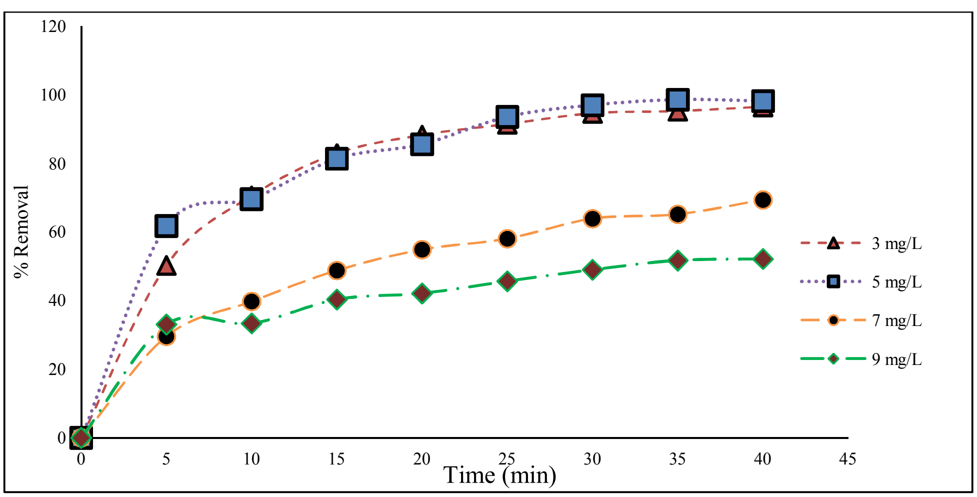

Fig. 2. Effect of initial dye concentration on adsorption of FG on CNTs (CNTs = 10 mg, T = 20°C, V = 100 mL, pH = 6.3)

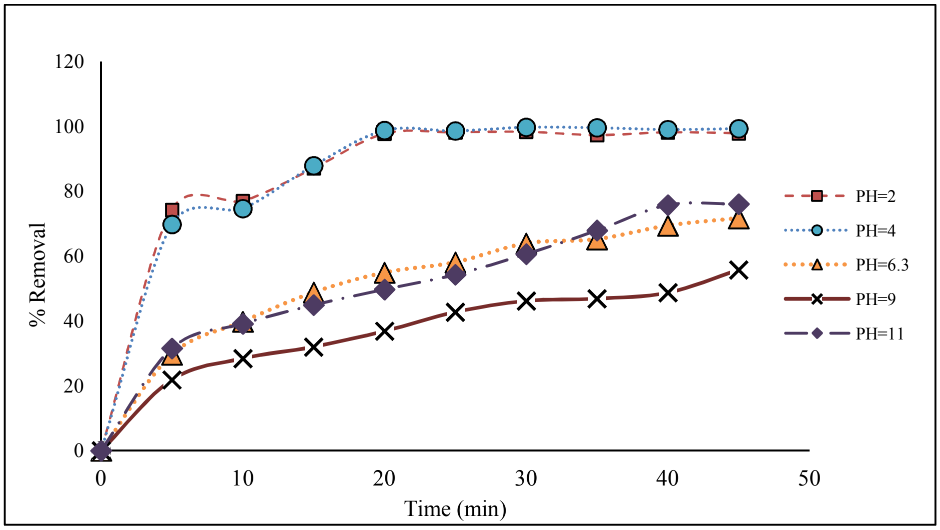

Fig. 3. Effect of pH on removal percent of FG on CNTs (C_o = 7 mg/L, CNTs = 10 mg, T = 20°C, V = 100 mL)

3.4 Effect of CNTs Dose

The effect of CNTs dose (10, 15, 20 and 25) of dye solution (7 mg/L) on dye removal percent is shown in Fig. 4. The dye removal was favored as the amount of CNTs increased. A large adsorbent dose, as expected, reduces the saturation of the adsorption sites, since dye molecules are more shared per adsorbent unit due to an increase in total adsorbent surface and adsorption sites availability. FG dye can easily access the adsorption sites and the q_e is high when the adsorbent dose is small. A similar behavior was found for the dye adsorption on different adsorbents such as pumpkin seed hull [28], jackfruit peel [29], papaya seeds [26]. In this research, the CNTs dose of 20 mg was found to be appropriate for efficient FG adsorption.

3.5 Effect of Temperature on Adsorption of FG

The adsorption of FG on CNTs was studied at temperature (20, 30, 40 and 50 ^{o}C). Fig. 5 shows the effect of temperature on adsorption of FG dye on CNTs. Temperature is considered one of the important factor affecting the dye adsorption on CNTs. Adsorption is a process which can be endothermic or exothermic for different adsorbents and dyes. If increasing temperature decreases the removal efficiency of the dye, dye adsorption on CNTs will be endothermic, and if increasing temperature decreases the removal efficiency of the dye, dye adsorption on CNTs will be exothermic [30]. The experimental result shown that the removing percent increases with increasing in the solution temperature this indicate that the adsorption of dye onto CNTs is endothermic in nature.

The free energy (ΔG) of adsorption was calculated from bellow equation [26]:

$$\Delta G = -RT\ln b \tag{5}$$

Where,

T is the temperature (K), R is the gas constant (8.314 J/mol) and *b* is the Langmuir constant. The entropy (ΔS) and enthalpy (ΔH) of adsorption were calculated at different temperature by using Van't Hoff equation [31]:

$$\ln b = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \tag{6}$$

From the plot of In *b* versus *1/T* show Fig. 6, the ΔS and ΔH were calculated from the slope and intercept, respectively, with these parameters being given in Table 3.

3.6 Adsorption Isotherms

The adsorption capacity of CNTs for FG dye can be determined by measuring equilibrium isotherms. Adsorption isotherms, for the analysis and design of an adsorption system, plays a crucial role in the predictive modeling procedures. According to the linear form of Langmuir and Freundlich adsorption isotherms were analysed. Fig. 7 shows all of the isotherm analyses results. Table 4 shows isotherm constants for FG dye on CNTs. Freundlich constants, K_F and $1/n_f$, indicate the adsorption capacity and adsorption intensity, respectively. Higher the value of $1/n_f$, the higher will be the affinity between the adsorbate and the adsorbent, and the heterogeneity of the adsorbent sites. The $1/n_f$ value indicates the relative distribution of energy sites and depends on the nature and strength of the adsorption process. The $1/n_f$ value of 0.69 refers to the fact that more than 90% of the active adsorption sites have equal energy level. The K_f value can be taken as a relative indicator of the adsorption capacity and the magnitude of K_f also showed the higher uptake of FG dye at higher temperature and endothermic nature of adsorption process. The b value between 0 and 1 indicates favorable adsorption of FG dye onto CNTs used. The comparison of the values in Table 4 for understudy isotherm models shows that the R^2 values for Langmuir model are closer to unity that shows its applicability for explanation of FG dye adsorption on this adsorbent. The experimental results are well represented by the Langmuir model more than the Freundlich model.

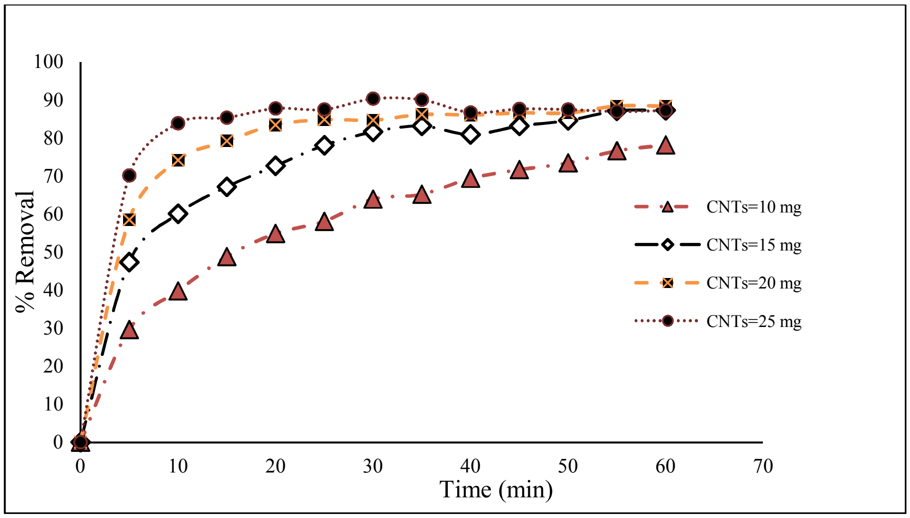

Fig. 4. Effect of CNTs dose on removal percentage of FG ([FG] =20 mg/L, V = 100 mL, pH = 6.3)

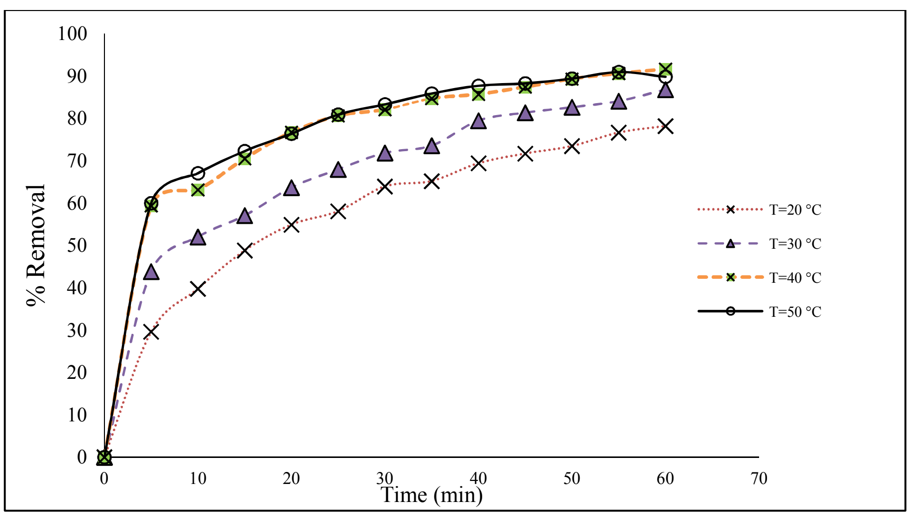

Fig. 5. Effect of Temperature on adsorption of FG on CNTs ([FG] =7 mg/L, CNTs = 10 mg, pH = 6.3)

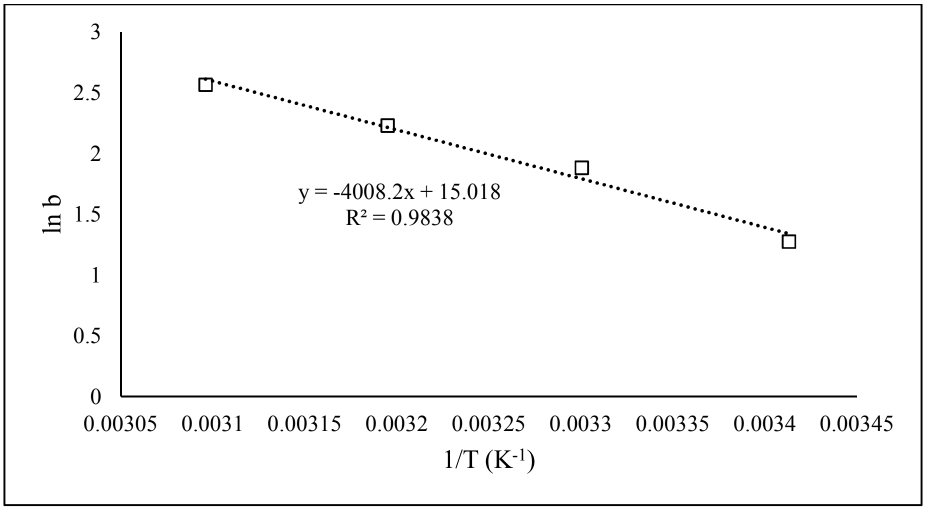

Fig. 6. Plot of *ln b* versus *1/T* for estimation of thermodynamic parameters (C_o =7 mg/L, CNTs = 10 mg, V = 100 mL, pH = 6.3)

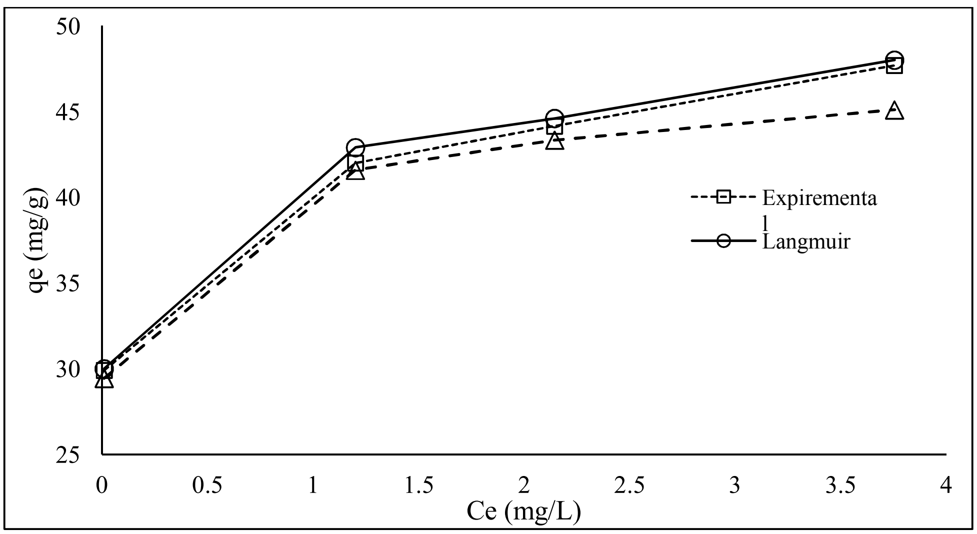

Fig. 7. Experimental data and theoretical isotherms for CNTs (CNTs = 10 mg, T = 20 °C, V = 100 mL, pH = 6.3)

Table 3. Thermodynamic parameters for FG adsorption on CNTs under different temperatures

T (K)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol K)
293	-3.10	33.32	124.86
303	-4.74		
313	-5.80		
323	-6.89		

Table 4. Isotherm constants for FG dye on CNTs

Langmuir			**Freundlich**		
Q_m (mg/g)	b (L/mg)	R^2	$1/n_f$	K_f (mg/g(mg/L)$^{(1/n)}$)	R^2
48.54	0.15	0.99	0.69	41.04	0.96

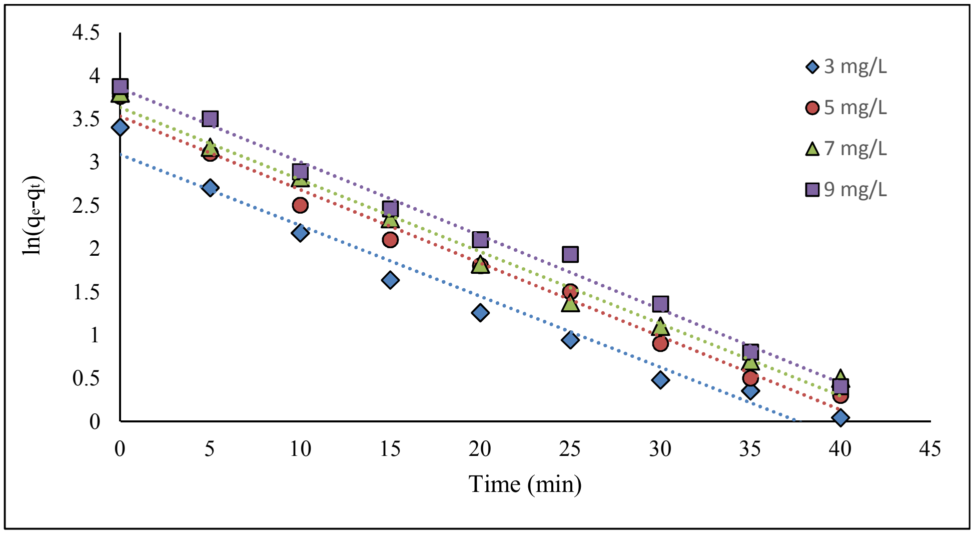

Fig. 8. Pseudo first-order kinetics for adsorption of FG by CNTs (CNTs = 10 mg, T = 20℃, V = 100 mL)

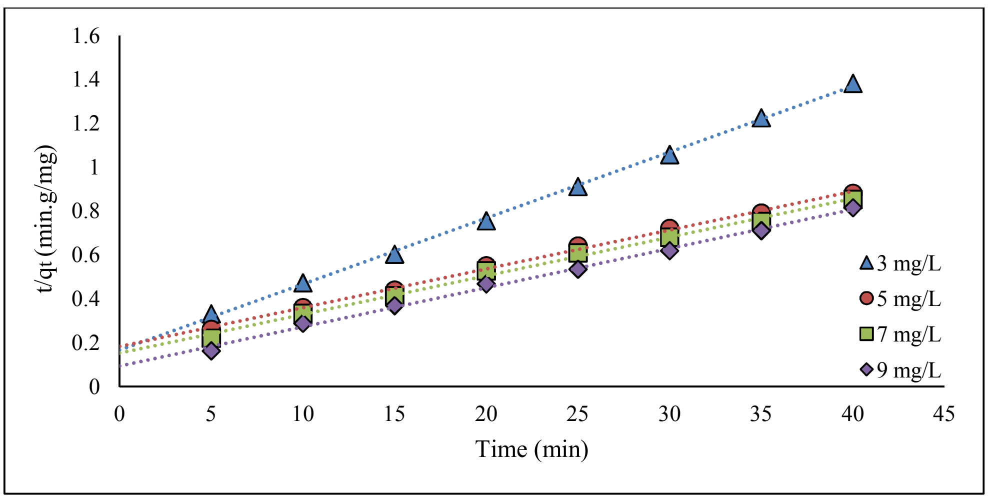

Fig. 9. Pseudo second order kinetics for adsorption of FG by CNTs (CNTs = 10 mg, T = 20℃, V = 100 mL)

Table 5. Comparison of the pseudo first and second order adsorption parameters for different dye concentrations

Initial concentration	Pseudo-First order model			Pseudo-Second order model		
(mg/L)	q_e (mg/g)	k_1 (min^{-1})	R^2	q_e (mg/g)	K_2 (g.mg^{-1}min^{-1})	R^2
3	21.76	0.0819	0.97	33.22	0.0068	0.99
5	33.78	0.0834	0.98	39.21	0.0055	0.99
7	37.34	0.0484	0.94	46.72	0.0033	0.99
9	46.99	0..0852	0.96	48.78	0.0025	0.99

3.7 Adsorption Kinetic

The pseudo-first order kinetic given by Svenska and Langergen was used to determine the rate constant of adsorption [32]:

$$\ln(q_e - q_t) = lnq_e - k_1 t \quad (7)$$

Where,

k_1 is the rate constant adsorption (min^{-1}), q_e and q_t are the amount of FG adsorbed in (mg/g) at equilibrium and at time t, respectively. For different concentrations of FG, k_1 values were calculated from the plots of $\ln(q_e - q_t)$ with t, as shown in Fig. 8.

The pseudo-second order equation, on the other hand, based on adsorption equilibrium is expressed as follows [33]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (8)$$

Where,

k_2 is the equilibrium rate constant of pseudo-second order sorption (g $mg^{-1}min^{-1}$). Equation (8) gives linear form by plotting t/q_t versus t, will give a straight line with intercept $1/kq_e^2$ and slope $1/q_e$ (as shown in Fig. 9). This figure shows a good agreement between calculated and experimental q_e values (see Table 5). For the pseudo-second order kinetic model, the correlation coefficients are higher than 0.99 indicating the applicability of this equation and the pseudo-second order nature of the adsorption process of FG on CNTs.

4. CONCLUSIONS

Carbon nanotubes was investigated for FG removal from aqueous solutions. The operating conditions used were contact time, initial concentration of FG, dose of CNTs, temperature and pH of the solution. The most effective improvements on the adsorption of FG were recorded when 5 mg/L of dye was used. It was found that the increasing of CNTs dose increased the removal percentage of FG dye. The best pH for the adsorption were recorded to be 4. The adsorption of FG onto CNTs has been found to be endothermic in nature and the increased adsorption capacity with rise in temperature confirms this observation. Langmuir and Freundlich isotherms were studied to analyze the removal of FG dye. The Langmuir adsorption model gives very satisfactory fitting to the adsorption isotherms. The kinetic studies showed that the FG dye adsorption process followed pseudo-second order kinetics models.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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