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Performance evaluation of multi-walled carbon nanotubes for decolorization of synthetic industrial wastewater: equilibrium, kinetics, and thermodynamics

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ABSTRACT

In this study, the application of multi-walled carbon nanotubes (MWCNTs) for the decolorization of synthetic industrial wastewater was assessed. This experimental research was conducted on a laboratory scale using a discontinuous system. The effects of pH, initial dye concentration, a dosage of MWCNTs adsorbent, temperature, and reaction time on the removal of reactive yellow 15 (RY 15) and reactive yellow 42 (RY 42) were investigated. Experimental results showed that the pH value of 3 was optimum for the removal of the dyes by the MWCNTs. The maximum adsorption capacity of 147.85 and 171.6 mg/g were obtained for RY 15 and RY 42, respectively at an initial dye concentration of 50 mg/L and adsorbent concentration of 0.2 g/L. Moreover, the equilibrium data for the adsorption of RY 15 and RY 42 by the MWCNTs followed the Langmuir isotherm. Results indicated that the RY 42 and RY 15 adsorption by MWCNTs followed the pseudo-second-order kinetic model. Furthermore, the adsorption of RY 15 and RY 42 onto MWCNTs was an endothermic reaction. MWCNTs are cost-effective and readily available, therefore, these materials could be used as an efficient adsorbent for the removal of RY 15 and RY 42 dyes from an aqueous solution.

Keywords: Multi-walled carbon nanotubes; Reactive yellow dye; Kinetics; Isotherms; Thermodynamics

1. Introduction

Dyes are fragrant water-soluble aromatic compounds widely used in various industries. Among these industries, the textile and leather industries rank first in the use of dyes [1]. Although there is no accurate data concerning the volumes of dyes discharged in the environment, wastewater containing dyes are incessantly discharged into the environment by these industries, thereby contaminating the soils and water systems. There are various classes of dyes, depending on their chemical nature. Azo dyes are more diverse compared to other classes and account for half of the annual dye production [2,3]. Azo dyes can be classified based on their chemical structure (azo, anthraquinone, triphenylmethyl, sulfur, etc.) or how they attach to textile fibers (direct, acidic, reactive dyes). The largest quantities of dyes introduce to wastewater in the textile industry during the washing and drying stages [4]. Azo dyes are extensively used in textile, leather, hygiene and cosmetic products, plastic dyes industry, etc. [5]. The removal of dyes from wastewater is emphasized for three reasons:

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(1) reactive dyes constitute 20%–30% of the total quantity of dyes in the market, (2) 10%–15% of dyes in effluents can be removed during the dyeing process through management practices, and (3) the common removal methods such as biodegradation are not efficient in the total removal of dyes from wastewater [6]. The discharge of dye wastewater into waterways prevent light penetration in water, which reduces photosynthesis, and damages the beauty of the environment [7]. Moreover, some azo dyes are carcinogenic and toxic to the environment [8–10].

Nano adsorbents including carbon nanotubes (CNTs) and zeolites are very efficient in the removal of pollutants in adsorption processes. CNTs have potential applications in environmental protection and are used in combating biological and chemical pollutants [11]. CNTs are widely used in electronic transistors and biosensors due to their unique properties and because of their special physical, mechanical, and electronic properties [12,13]. Recently, scientists have reported the effectiveness of multi-walled carbon nanotubes (MWCNTs) in the removal of dioxins [14]. Smaller-sized CNTs have a high surface area, high porosity and mechanical strength, and considerable electrical conductivity [15]. Single-walled carbon nanotubes (SWCNTs) and MWCNTs are classified based on the number of their constituent layers. These nanoparticles are suitable adsorbents for removing organic and mineral pollutants (dyes, benzene, phenanthrene, nickel, etc.) from large volumes of wastewater [16,17]. There are various physical, chemical, and biological methods for the removal of organic dyes from wastewater [18–21]. Various adsorption techniques such as chemical oxidation, anaerobic treatment, adsorption, biological adsorption, membrane and photocatalytic filtration have been employed in dye removal [22]. Among these methods, adsorption techniques are high-quality processes from the economic point of view [23], activated carbon being the widely used adsorbent because of its high adsorption capacity [24,25]. The removal of dye from textile wastewater using low-cost adsorbents such as ash oil, chitosan, and natural-clay soils [26] has been reported. Therefore, the present study was intended to study the efficiency of MWCNTs in removal of reactive yellow 15 (RY 15) and reactive yellow 42 (RY 42) dyes, and to investigate the effects of various factors such as pH, initial dye concentration, temperature, adsorbent dosage and equilibrium time on RY 15 and RY 42 dyes removal from synthetic wastewater.

2. Materials and methods

2.1. Chemicals and reagents

MWCNTs were supplied by the Research Institute of Petroleum Industry (RIPI), Tehran, Iran. The scanning electron micrograph (SEM) of MWCNTs is presented in Fig. 1. The micrograph represents a nano-scale structure, the MWCNTs sizes range from 20 nm to 30 nm. RY 15 and RY 42 dyes were purchased from Alvan Sabet Co., Iran. A stock solution of 1,000 mg/L of RY 15 and RY 42 dyes were prepared by dissolving the dyes with deionized water in 1 L volumetric flask. The working standard solutions were prepared from the stock solutions by serial dilution. HCl and NaOH (0.1 M) were used for the adjustment of pH.

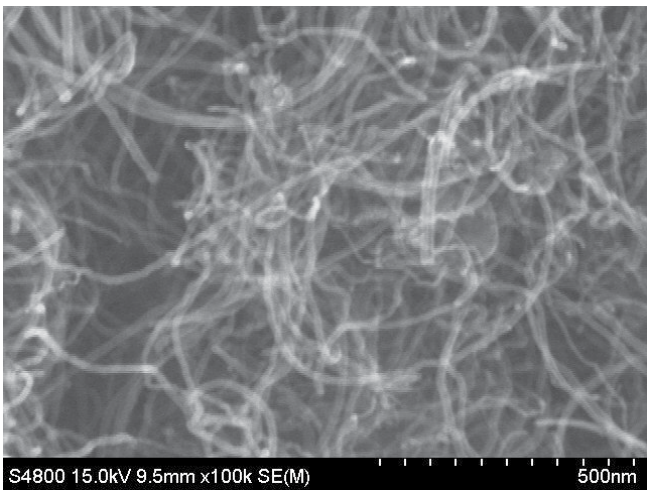

Fig. 1. Scanning electron micrograph (SEM) of MWCNTs.

2.2. Adsorption experiment

The optimum pH was considered first in the experiment. pH values of 3, 5, 7, 9, and 11 were used for this purpose. The effect of initial dye concentrations was investigated using 10, 20, 30, and 50 mg/L dye concentrations. This was used to determine the optimum initial dye concentration and to investigate the equilibrium studies. Reaction times of 5, 10, 15, 30, 45, 60, 75, and 90 were tested to determine the optimum reaction time and kinetics. Various amounts of MWCNTs (0.2, 0.3, 0.4, 0.5, and 0.75 g/L) were considered in the experiments to assess the optimum amount of the adsorbent. Temperatures of 15°C, 25°C, 35°C, 45°C, and 55°C was used to examine the optimum adsorption temperature and to report the thermodynamic parameters. In selecting this optimum temperature, both the ambient temperature and the probable temperature of the aqueous solution to be treated were taken into consideration. A shaking incubator (Model: SI-100R, Korea) was employed to carry out the adsorption at the higher temperatures.

After each step of the experiments, samples were analyzed using a UV/VIS Washington D.C., 2001 model spectrophotometer at a wavelength of 420 nm (RY 15) and 430 nm (RY 42) respectively. The adsorption capacities of the adsorbents were calculated using Eq. (1).

$$q = \left(C_0 - C_e\right)\frac{V}{M} \tag{1}$$

where C_0 and C_e (mg/L) are the initial and equilibrium concentration of the dye solution, respectively, V (mL) is the volume of the solution and M (g) is the mass of MWCNTs used.

2.3. Equilibrium studies

The Langmuir and Freundlich adsorption isotherm models were used for the mathematical modeling of RY 15 and RY 42 adsorption process.

The linear equation for the Freundlich isotherm is presented in Eq. (2):

$$\ln q = \ln k_f + \frac{1}{n}\ln C_e \quad (2)$$

For the Freundlich isotherm, a plot of ln$q$ against lnC_e will yield a straight line; the slope of this line will be 1/n, and its y-intercept equals lnk_f from where k_f and n are calculated. k_f is the Freundlich constant relating to the adsorption capacity and n is the Freundlich model constant indicating the intensity of adsorption.

The linear equation for the Langmuir isotherm is presented in Eq. (3):

$$\frac{C_e}{q_e} = \frac{1}{k_L q_m} + \frac{C_e}{q_m} \quad (3)$$

where k_L and q_m are empirical constants. If the Langmuir isotherm model fits the equilibrium data, a plot of C_e/q_e against C_e will yield a straight line according to Eq. (3); the slope of this line will be $1/q_m$, and its y-intercept equals $1/k_L\ q_m$ from where k_L and q_m would be obtained. The Langmuir and Freundlich isotherms were obtained based on the adsorption data of the equilibrium studies (effect of initial dye concentration).

2.4. Kinetic studies

The kinetic equations study the transfer of adsorbed particles of the material per unit time, it determines the reaction rates. In the present research, pseudo-first-order (Eq. (4)) and pseudo-second-order (Eq. (5)) kinetic models were investigated.

$$\log\left(q_e - q_t\right) = \log q_e - \frac{k_1 t}{2.303} \quad (4)$$

A plot of log(q_e–q_t) vs. t should give a linear relationship from which k_1 and q_e could be determined from the slope and intercept of Eq. (4), respectively.

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

If the pseudo-second-order kinetic equation is appropriate, a plot t/q against t should a linear relationship from which q_e and k_2 can be determined.

q_e is the amount of dye adsorbed at equilibrium per unit weight of the adsorbent, q_t is the amount of dye adsorbed at any time, k_1 and k_2 are the pseudo-first-order rate and pseudo-second-order rate constant, respectively.

2.5. Thermodynamic studies

For the thermodynamic studies, parameters such as enthalpy change ($\Delta H°$), entropy ($\Delta S°$), and Gibbs free energy change ($\Delta G°$) were determined based on Eqs. (6)–(8).

$$k_d = \frac{C_a}{C_e} \quad (6)$$

$$\Delta G° = -RT\ln k_c \quad (7)$$

$$\ln k_d = \frac{\Delta S°}{R} + \frac{\Delta H°}{RT} \quad (8)$$

where C_a is the amount of dye adsorbed on the adsorbent per liter of the solution at equilibrium, C_e is the equilibrium concentration of dye in the solution, R is the universal gas constant (8.314 J/mol K), T is the absolute temperature, and k_c is the thermodynamic equilibrium constant.

3. Results and discussion

3.1. Effects of pH

Among the factors that influence the effective surface charge of adsorbents are pH and pollutant ionization [27]. The effect of pH on the removal of RY 15 and RY 42 by MWCNTs is shown in Fig. 2. As shown in Fig. 2, the maximum removal of the dyes was achieved at pH = 3. Fig. 2 also presents the amounts of RY 15 and RY 42 adsorbed by the MWCNTs, approximately 35 and 32 mg/g of RY 15 and RY 42, respectively were adsorbed by MWCNTs at pH 3.

As indicated in Fig. 2, the quantity of the dyes adsorbed was higher at lower pH values, with pH = 3 being the optimum value for their removal. Sadani et al [28] also noticed in their study that the optimum pH value was in the acidic range. Oxygen functional groups on the surface of nanotubes are of special importance; this is because they strengthen the bonds between OH^- groups on the adsorbent surface at pH values higher than 4 and give the surface a negative charge. However, at pH values lower than 4, the number of H^+ ions on the adsorbent surface increases and this gives the surface a positive charge [29]. In this case, the surface of the MWCNTs is positively charged at lower pH, and consequently, the surface has an affinity towards the negatively charged species of the RY 42 and RY 15 dyes. However, as pH increases, the surface of the MWCNTs becomes negatively charged, thereby repelling the RY 42 and RY 15 negatively charged species. Dutta et al [30] studied reactive red 198 removals by titanium dioxide nanoparticles and reported that the percentage removal decreases with increasing pH value. The results of this study conform to those of the present one.

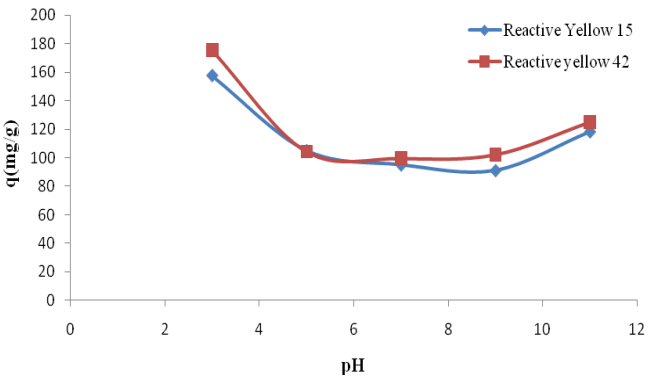

Fig. 2. Effects of pH on the removal of RY 15 and RY 42 by multi-walled carbon nanotubes.

3.2. Effects of initial dye concentration

The effect of initial dye concentration on removal of the RY 42 and RY 15 by MWCNTs are presented in Figs. 3 and 4, respectively. As revealed from the Figs, the maximum removal of the dyes was observed at the initial concentration of 50 mg/L for the two dyes, and the optimum reaction times for the dyes were 45 min for RY 42 and 90 min for RY 15.

Figs. 3 and 4 also showed that the maximum adsorption capacity of the MWCNTs for RY 42 at its initial concentration of 50 mg/L was 164.6 mg/g after 45 min and was 155.55 mg/g for RY 15 at the same initial concentration after 90 min. The amounts of RY 42 adsorption at concentrations of 10, 20, 30, and 50 mg/L by the adsorbent after 45 min were 31.65, 33.45, 78.95, and 164.6 mg/g, respectively, while the corresponding values for RY 15 at 10, 20, 30, and 50 mg/L after 90 min were 14.2, 59.55, 94.9, and 155.55 mg/g, respectively. Therefore, the increase in the initial concentrations of the dyes improved the adsorption capacity of the MWCNTs which could be caused by the increase in the driving force due to the concentration gradient that developed between the bulk solution of the dye and the surface of the MWCNTs. Ghaneian et al. [31] studied dye removal from aqueous solutions and stated that the amount of adsorption changed with the passage of time because most adsorption sites were vacant at the start of the adsorption process and the pollutant concentration in the solution was higher. However, as time went by, dye concentration declined and, hence, the adsorption rate also decreased. The results of the present research are somewhat similar to those they reported. Aksu and Akin [32] stated that the incoming dye concentration was a driving force for overcoming the total resistance resulting from the transfer of dye mass between the solid and liquid phases.

3.3. Effects of adsorbent dose

The effect of adsorbent doses on the removal of the dyes is presented in Fig. 5. Fig. 5 demonstrates that the maximum removal of the dyes was observed when the adsorbent used was 0.2 g/L.

The figure presents the amounts of RY 15 and RY 42 adsorbed when the adsorbent dosages were 0.2, 0.4, 0.5, and 0.75 g/L. The quantity of RY 42 adsorbed at adsorbent dose of 0.2 g/L, retention time of 45 min, and initial concentration of 50 mg/L was 202 mg/g, while the amount of RY 15 adsorbed at adsorbent dose of 0.2 g/L, retention time of 90 min, and initial dye concentration of 50 mg/L was 156.25 mg/g. Therefore, the amount of the dyes adsorbed per unit weight of the MWCNTs decreased with increase in the adsorbent dose. The decrease might be as a result of saturation of the adsorbent active site [31]. In research carried out by Ehrampoush et al. [33], it was concluded that the value of q_e (mg/g) decreased from 0.82 to 0.4 mg/g when the amount of the adsorbent increased from 0.5 to 5 g.

3.4. Adsorption isotherms

Isotherm models express equilibrium relationships between the material adsorbed on the surface of the adsorbent and the concentration of the adsorbate [34]. The results relating to two of the most widely used isotherms (Freundlich and Langmuir isotherms) are presented in Figs. 6 and 7, and are summarized in Tables 1 and 2.

Considering the results obtained at equilibrium time using Langmuir and Freundlich isotherms, and the highest correlation coefficient, the adsorption of RY 15 and RY 42 onto MWCNTs followed the Langmuir isotherm. The Langmuir monolayer adsorption constant, q_{max} (mg/g) and Langmuir energy of adsorption constant, k_L are presented in Tables 1 and 2. The results obtained had greater conformity to the results of the study conducted by Ehrampoush et al. [33] and Ghaneian et al. [35]. Moussavi et al. [36] carried out

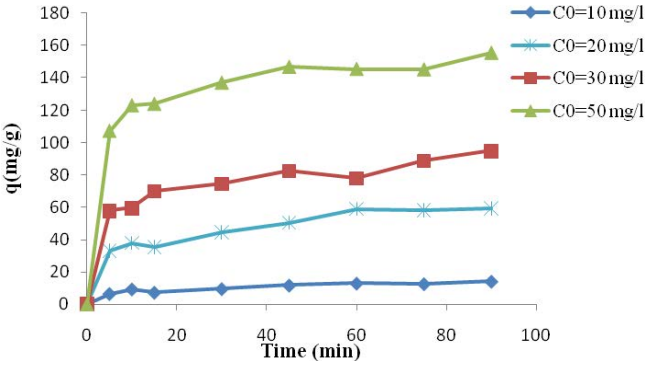

Fig. 4. Effects of the initial concentration of RY 15 on its removal by multi-walled carbon nanotubes at various reaction times.

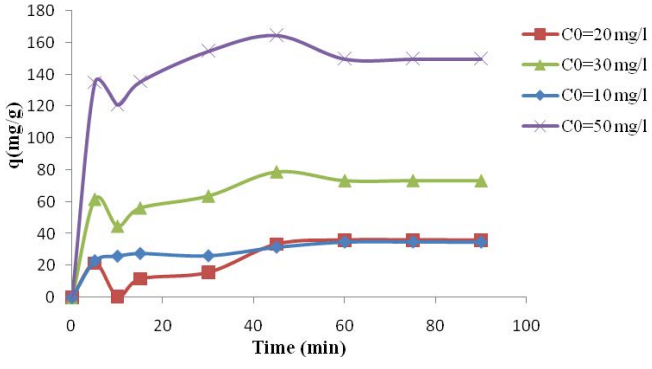

Fig. 3. Effects of the initial concentration of RY 42 on its removal by multi-walled carbon nanotubes at various reaction times.

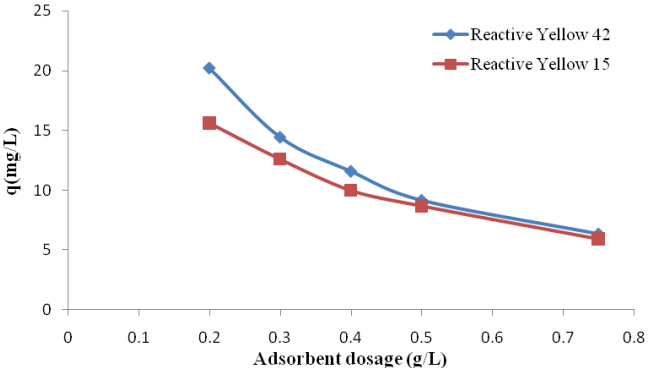

Fig. 5. Effect of adsorbent doses on the removal of the dyes.

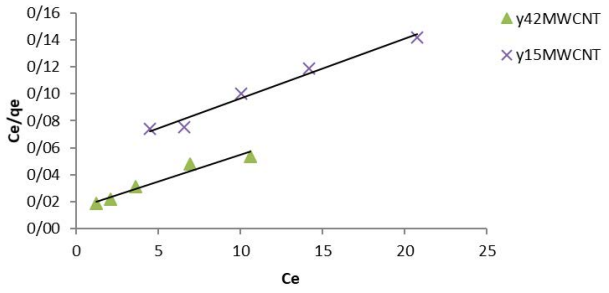

Fig. 6. The Langmuir isotherm model and adsorption of the dyes by multi-walled carbon nanotubes.

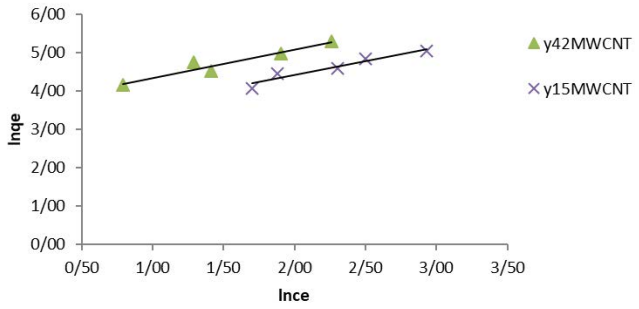

Fig. 7. The Freundlich isotherm model and adsorption of the dyes by multi-walled carbon nanotubes.

Table 1
Equilibrium constants for RY 15 adsorption onto MWCNTs

Isotherm	Constants	Values
Freundlich	R^2	0.92
	k_f (mg/g)	19.46
	n	1.38
Langmuir	R^2	0.97
	k_L (L/mg)	4,301.69
	q_{max} (mg/g)	225.73

Table 2
Equilibrium constants for RY 42 adsorption onto MWCNTs

Isotherm	Constants	Values
Freundlich	R^2	0.92
	k_f (mg/g)	36.97
	n	1.35
Langmuir	R^2	0.94
	k_L (L/mg)	16,447
	q_{max} (mg/g)	252.68

a study of the removal of acid orange 7 from synthetic textile wastewater and stated that the adsorption of this dye on SWCNTs followed the Langmuir isotherm, their conclusion also agrees with results of the present research. In Langmuir's theory, it is assumed that adsorption of the RY 15 and RY 42 dyes takes place on a series of special homogeneous sites on the MWCNTs. Khorramfar et al. [37] removed dyes from textile wastewater using extracts of Punica granatum peel and, by studying Langmuir and Freundlich isotherm dye removal models, noticed that the correlation coefficient of Langmuir isotherm was higher than that of the Freundlich isotherm.

3.5. Effects of temperature

Fig. 8 and Table 3 show the effect of temperature on the process of adsorption by MWCNTs. According to Table 3, the adsorption of RY 15 and RY 42 by MWCNTs is endothermic, that is, $\Delta H°$ values were positive.

These parameters must be determined to demonstrate the spontaneity of the adsorption process [38]. $\Delta S°$ is

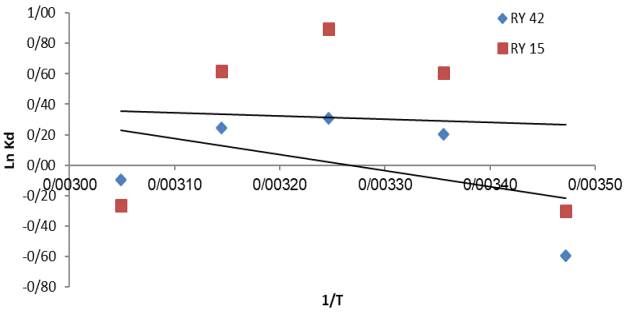

Fig. 8. Effects of temperature on RY 15 and RY 42 adsorption by the adsorbent.

Table 3
Thermodynamic parameters for RY 15 and RY 42 adsorption by MWCNT

Dyes	T (K)	q_e (mg/g)	Thermodynamics parameters		
			$\Delta G°$ (kJ/mol)	$\Delta H°$ (kJ/mol)	$\Delta S°$ (J/mol K)
RY 15	288	21.29	0.72	1.71	8.18
	298	32.39	–1.51		
	308	35.51	–2.30		
	318	32.49	–1.63		
	328	21.75	0.71		
RY 42	288	17.79	1.42	8.76	28.63
	298	27.56	–0.51		
	308	28.8	–0.78		
	318	28.02	–0.64		
	328	23.83	0.26		

positive, evidence of an increase in the degrees of freedom of the adsorbed dyes, whereas, the negative $\Delta G°$ values indicate spontaneous adsorption.

3.6. Adsorption kinetics

Kinetic figures are drawn to determine the rate and degree of dye removal, and the slope of the related line demonstrates whether the reaction rate is constant or not [39]. The pseudo-first-order and pseudo-second-order kinetics as

Table 4
Kinetic parameters for the adsorption of RY 42 and RY 15 by multi-walled carbon nanotubes

Adsorbent		Pseudo-first-order			Pseudo-second-order			
	C_0 (mg/L)	k_1 (min^{-1})	$q_{e,cal.}$ (mg/g)	R^2	k_2 (g/mg min)	$q_{e,cal.}$ (mg/g)	R^2	$q_{e,exp.}$ (mg/g)
MWCNT+RY 42	10	0.02	6.85	0.12	0.01	34.49	1.00	35.90
	20	0.01	3.81	0.14	0.03	35.67	1.00	37.00
	30	0.02	9.76	0.07	0.01	91.61	1.00	94.45
	50	0.02	5.97	0.06	0.02	156.01	1.00	171.60
MWCNT+RY 15	10	0.02	4.41	0.18	0.01	13.33	1.00	14.00
	20	0.02	13.38	0.11	0.00	59.26	1.00	60.10
	30	0.02	9.37	0.07	0.01	81.61	1.00	83.45
	50	0.03	14.03	0.19	0.00	149.40	1.00	147.85

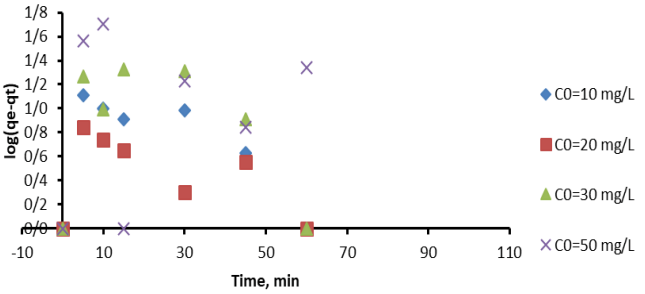

Fig. 9. Pseudo-first-order kinetic for the adsorption of RY 42 onto multi-walled carbon nanotubes.

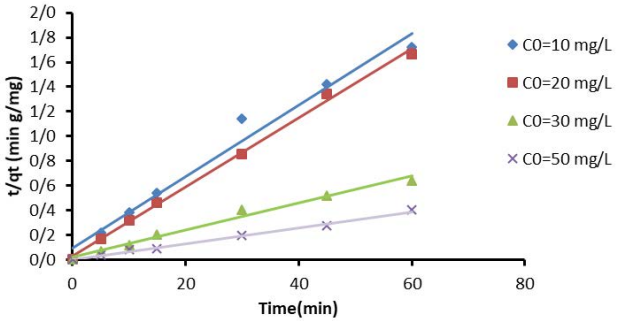

Fig. 10. Pseudo-second-order kinetic for the adsorption of RY 42 onto multi-walled carbon nanotubes.

investigated on the adsorption of RY 15 and RY 42 dyes onto MWCNTs are presented in Figs. 9–12 and the kinetic parameter reported in Table 4.

It is evident from the figures and table that the R^2 values obtained for the pseudo-second-order kinetic model were higher (R^2 = 1.00) when compared with those of the pseudo-first-order kinetic model, this showed that adsorption kinetics of RY 42 and RY 15 by MWCNTs was better described by the pseudo-second-order kinetic model. Pseudo-second-order kinetic model indicates that chemisorption controls the adsorption processes. During chemisorption, the RY 42 and RY 15 ions attach onto the surface of the MWCNTs by forming a chemical bond (usually covalent) and tend to find sites that maximize their coordination number with the surface. This result agrees with those found by Khorramfar et al. [37].

4. Conclusions

MWCNTs have been successfully utilized for the adsorption of RY 42 and RY 15 dyes. The maximum adsorption capacity of 147.85 mg/g and 171.6 were obtained for RY 15 and RY 42, respectively at an initial dye concentration of 50 mg/L and adsorbent concentration of 0.2 g/L. Increasing the contact time, initial dye concentration and the temperature increased the dye adsorption rate, whereas, the amount of dye adsorbed decreases with increasing pH and the adsorbent dose. The pseudo-second-order kinetic equation accurately described the adsorption kinetics, the equilibrium data showed a better fit to the Langmuir isotherm and the

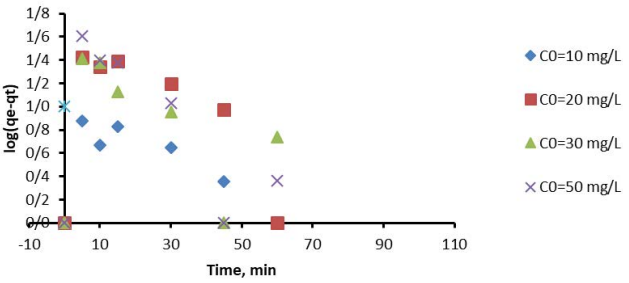

Fig. 11. Pseudo-first-order kinetic for the adsorption of RY 15 onto multi-walled carbon nanotubes.

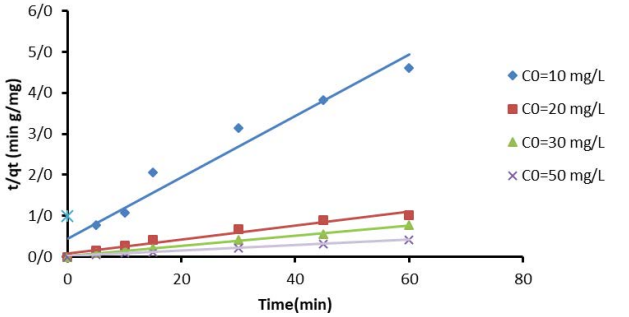

Fig. 12. Pseudo-second-order kinetic for the adsorption of RY 15 onto multi-walled carbon nanotubes.

thermodynamic analysis showed that the adsorption process is endothermic and spontaneous in nature. CNTs are readily available and cost-benefit; therefore, CNTs could be used as effective adsorbents for the removal of dyes from industrial wastewater for reuse or before their discharge into the environment.

Acknowledgment

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