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Multi-walled Carbon Nanotube-CO-NH(CH₂)₂NH-SO₃H: A New Adsorbent for Removal of Methylene Blue from Aqueous Media

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In this study, multi-walled carbon nanotube-CO-NH(CH₂)₂NH-SO₃H was prepared through a three-step functionalization of commercial multi-walled carbon nanotubes and then characterized using Fourier transform infrared spectroscopy (FTIR) and transmission electron microscopy (TEM). In addition, the adsorption of methylene blue was investigated by using these nanotubes. In order to remove dye, the effects of different parameters including stirring time, pH value, initial concentration of dye, and amount of carbon nanotubes were studied. More than 99.5% of dye was removed at neutral pH within a short time. Pseudo-second-order equation was predicted by adsorption kinetic. Different isotherms were studied to define adsorption mechanism, and Langmuir model showed the best results compared to the other isotherms. This project offers a facile and efficient adsorption method for removing methylene blue from aqueous solution. Short stirring time, neutral pH and high q_{max} (the maximum adsorption capacity in saturated single layer) are the significant characteristics of this research.

Keywords: Multi-walled carbon nanotubes, Methylene Blue, Aqueous solution, Adsorption

INTRODUCTION

Methylene blue (MB) is one of the most widely applied dyes. This dye is often used as a biological stain, a cyanide antidote, an oxidation indicator and a chemical analysis reagent [1]. In addition, it is used for printing, dyeing, and monitoring [2]. Carcinogenesis, mutagenicity, and toxic effects on aquatic organisms are harmful effects of this dye [3,4]. So, removing this dye from the corresponding effluent is necessary. Adsorption [5], membrane processes [6], photo catalytic degradation [7] and biological treatment are the appropriate methods for the removal of dyes. Among these methods, adsorption is the most important and popular technique due to the ease of procedure and the low cost application in the decolonization process. Recently, several adsorbents have been introduced to remove dyes from aqueous solutions, such as magnetic palygorskite nanoparticles [8], modified pyrophyllite powders [9],

graphene-modified magnetic polypyrrole nanocomposite [10], waste metal hydroxide sludge [11], oil palm trunk fiber [12], broad bean skin [13], chitosan [14], rice hull [15], clay [16], activated carbon [17], MCM41 (Mobil Composition of Matter No. 41) and MCA (Magnetic Carbonaceous Adsorbent) [18], graphene oxide [19], mesoporous SiO₂ [20] and synthesizing polymer-clay composite [21]. The first piece of work on carbon nanotubes was reported in 1991 [22]. Carbon nanotubes are divided into two categories: multi-walled carbon nanotubes (MWNTs) and single-walled carbon nanotubes (SWNTs). MWNTs consist of several coaxial cylinders, each is made of a single graphene sheet surrounding a hollow core [23]. Currently, carbon nanotubes find applications in nanotechnology, electronics, optics, biotechnology and biomedicine and other fields of material science and technology [24-26]. These compounds are also very important in chemistry and are used as adsorbents for the removal of dyes and metal ions in waste [27-31] and the synthesis of 3,4-dihydropyrimidin-2(1H)-ones/thiones. [32].

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The main purpose of this research is to investigate the removal of MB from dyeing effluents by using modified multi-walled carbon nanotube. In this study, commercial multi-walled carbon nanotube is modified by using ethylenediamine and chlorosulfonic acid and characterized through Fourier transform infrared spectroscopy (FT-IR) and transmission electron microscopy (TEM). Optimal conditions including stirring time, pH, initial concentration of dye, and modified carbon nanotube dosage are also investigated.

EXPRIMENTAL

Chemicals and Reagents

Multi-walled carbon nanotube was supplied by Industrial Research Institute of Tehran with the following characteristics: inner diameter, 3.8 nm; outer diameter, 10-30 nm; length, 10 μm , purity 99%; special surface, 270 $\text{m}^2 \text{g}^{-1}$ and thermal conductivity 1500 $\text{W m}^{-1} \text{K}^{-1}$. The materials, including methylene blue, dicyclohexylcarbodiimide (DCC), ethylenediamine, chlorosulfonic acid and *n*-hexane were supplied from Merck Chemical Company (Hohenbrunn, Germany). In addition, double distilled water was applied in all processes. The characteristics of the dye are: (a) color index number: 14, 6060, (b) molecular weight: 319.86 g mol^{-1} , (c) empirical formula: $\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCl}$ and (d) λ_{max} : 664 nm.

Apparatus

A Jenway model 3510 pH-meter was used for the determination of pH. A UV-Vis spectrophotometer (model: PerkinElmer Lambda 45) was also used for the determination of dye concentration in the solution. IR spectra were obtained by using a 470-Shimadzu IR spectrophotometer. TEM images were taken by using High Resolution Transmission Electron Microscopy EM10C-100 KV and Tecnai 20.

Extraction Procedure

Synthesis of multi-walled carbon nanotube-CO-NH(CH₂)₂NH-SO₃H (MWCNTs-EDA-SO₃H). First, multi-walled carbon nanotube-CO-NH(CH₂)₂NH₂ (MWCNTs-EDA) was synthesized [33]. 1.0 g of MWCNTs-EDA was transferred into a round-bottomed

flask. 50 ml of *n*-hexane was added to the flask. The flask was immediately equipped with a mechanical stirrer, a dropping funnel and an ice bath. 50 ml of chloro sulfonic acid (diluted by 50 ml of *n*-hexane) was added to dropping funnel and poured into the flask for 12 h. Finally, the reaction mixture was stirred for 24 h at room temperature. The appropriate product was produced through the distillation of solvent and then dried under vacuum at 80 °C for 8 h. The synthesis route of MWCNTs-EDA-SO₃H is illustrated in Fig. 1.

Removal of MB from Aqueous Solution

To study the adsorption, 30 mg of the modified carbon nanotube was added to 50 ml of aqueous solution that contained 50 mg l^{-1} of MB at room temperature. Then, in the next step, pH value was optimized by 0.10 M of HCl or 0.10 M of NaOH. The best pH was obtained in neutral conditions. Following that, 99.5% of dye was removed from solution for 10 min. Finally, the percentage of adsorbed MB and the amount of dye sorbed at time *t* (q_t , mg g^{-1}) and equilibrium time (q_e , mg g^{-1}) were calculated.

RESULTS AND DISCUSSION

Modified multi-walled carbon nanotubes were recognized by using FT-IR and TEM. The IR spectra of MWCNTs-COOH, MWCNTs-EDA and MWCNTs-EDA-SO₃H were compared (Fig. 2). The formation of -COOH group in nanotube structure was confirmed through observing the new peaks appeared in 1702 cm^{-1} (C=O stretching vibration) and 1385 cm^{-1} (C-O stretching vibration) (Fig. 2a). The disappearance of the strong and sharp peak in 1385 cm^{-1} , the shift of stretching vibration peak of C=O from 1702 to 1655 cm^{-1} , the appearance of peak in 1561 cm^{-1} (C-N stretching vibration and N-H bending vibration) and also the formation of a shoulder in 3500 cm^{-1} (NH stretching vibration) were indicative of conversion of MWCNTs-COOH into MWCNTs-EDA (Fig. 2b). The formation of MWCNTs-EDA-SO₃H was confirmed through observing several new peaks appeared in the spectrum (Fig. 2c). The most important peaks were in 1150 cm^{-1} (S=O stretching vibration) and 1008 cm^{-1} (S-O stretching vibration) regions. The TEM was employed due to its ability to measure the nanotube diameter in the bundle

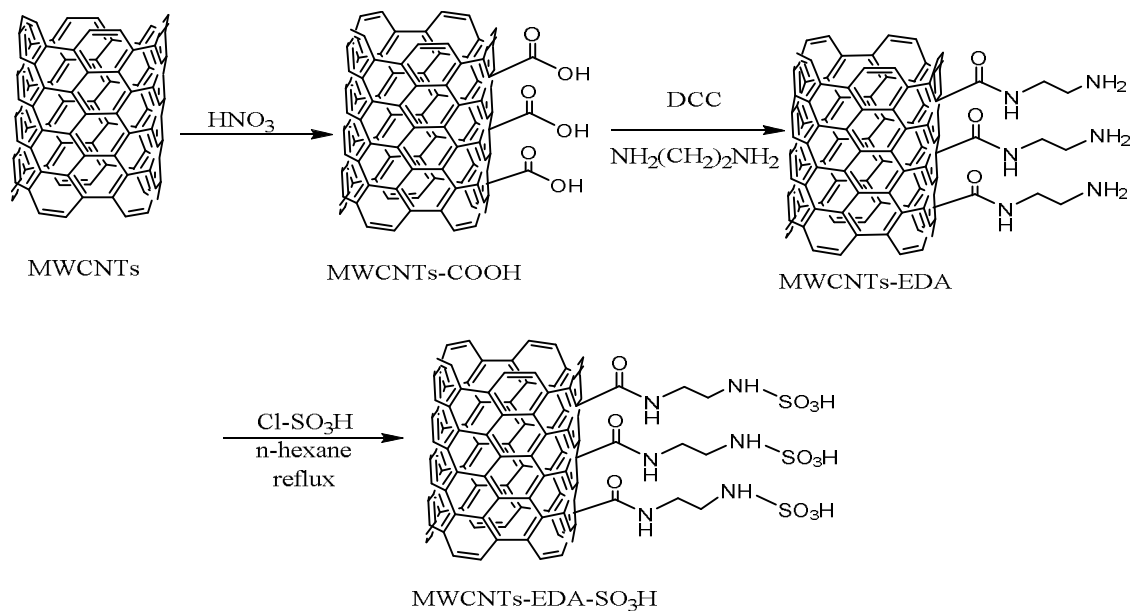


Fig. 1. Synthesis route of MWCNTs-EDA-SO₃H.

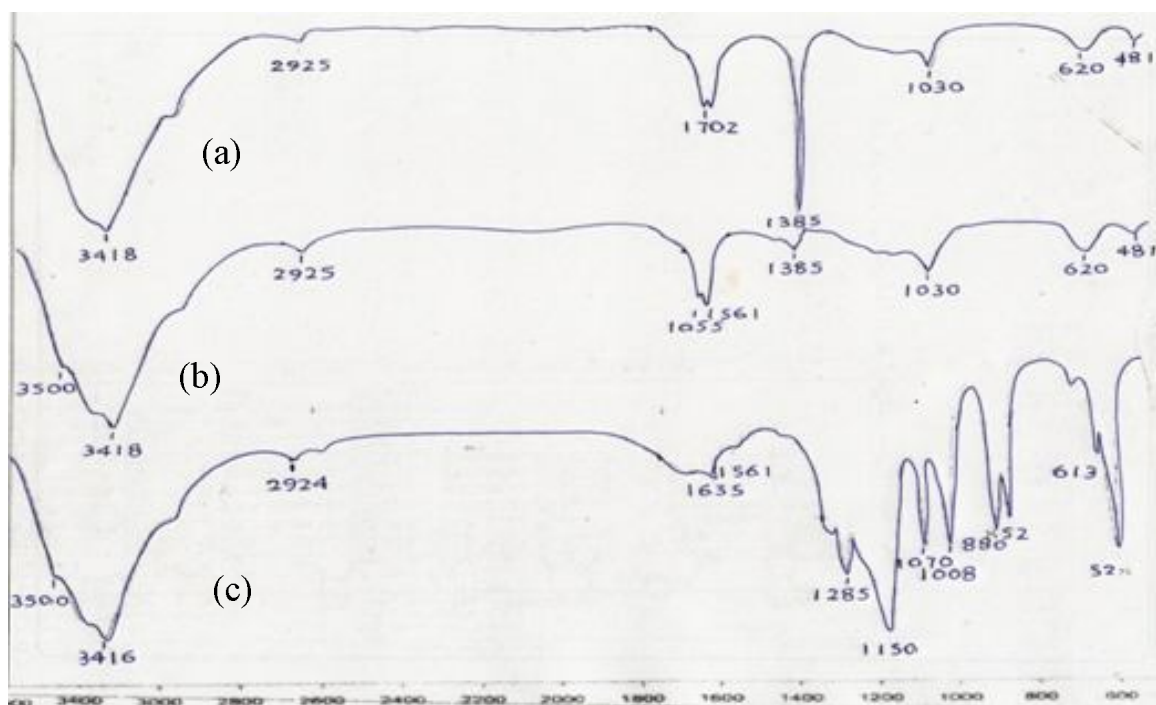


Fig. 2. FT-IR spectra of MWCNTs-COOH (a), MWCNTs-EDA (b), MWCNTs-EDA-SO₃H (c).

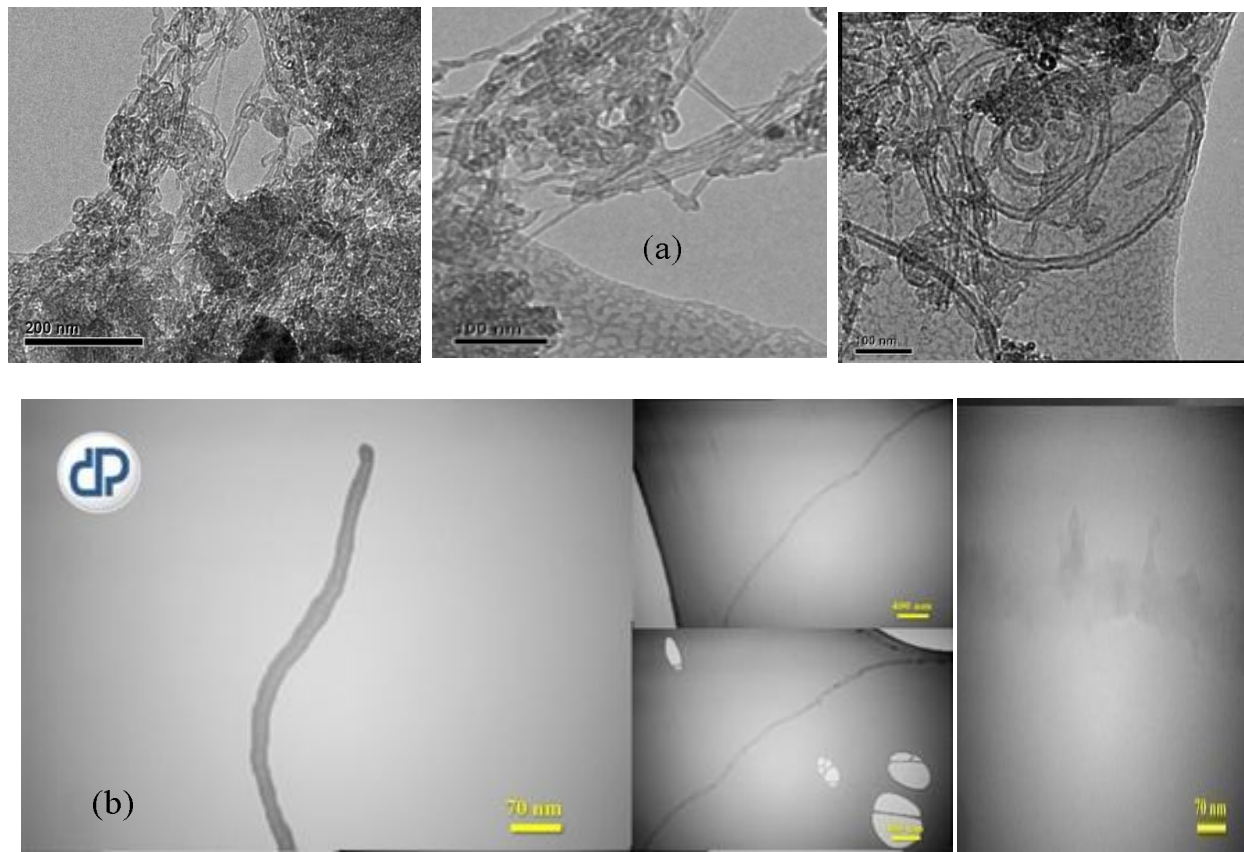


Fig. 3. TEM images of MWCNTs-EDA (a) and MWCNTs-EDA-SO₃H (b).

Table 1. Comparison between MWCNTs and Modified MWCNTs in Removal of MB

Sample	Removal of dye (%)	Sample	Removal of dye (%)
MWCNT	95.7	MWCNTS-COOH	80.9
MWCNTS-EDA	96.9	MWCNTS-EDA-SO ₃ H	99.2

(Fig. 3).

The Effect of Solution pH

Initially, the pH effect on removing MB was investigated in the range of 3.0-11.0. In this study, stirring time, adsorbent mass and temperature were constant. As shown in Fig. 4a, changes of pH did not affect adsorption quantity of MB. So, pH 7.0 was chosen as the optimum pH

for other studies.

The Effect of Stirring Time

Stirring time is one of the most important factors in determining the power of adsorbent for removing the dye. In this research, the effect of different stirring time (1 to 70 min) was investigated for determination the adsorption percentage of MB through application of the modified

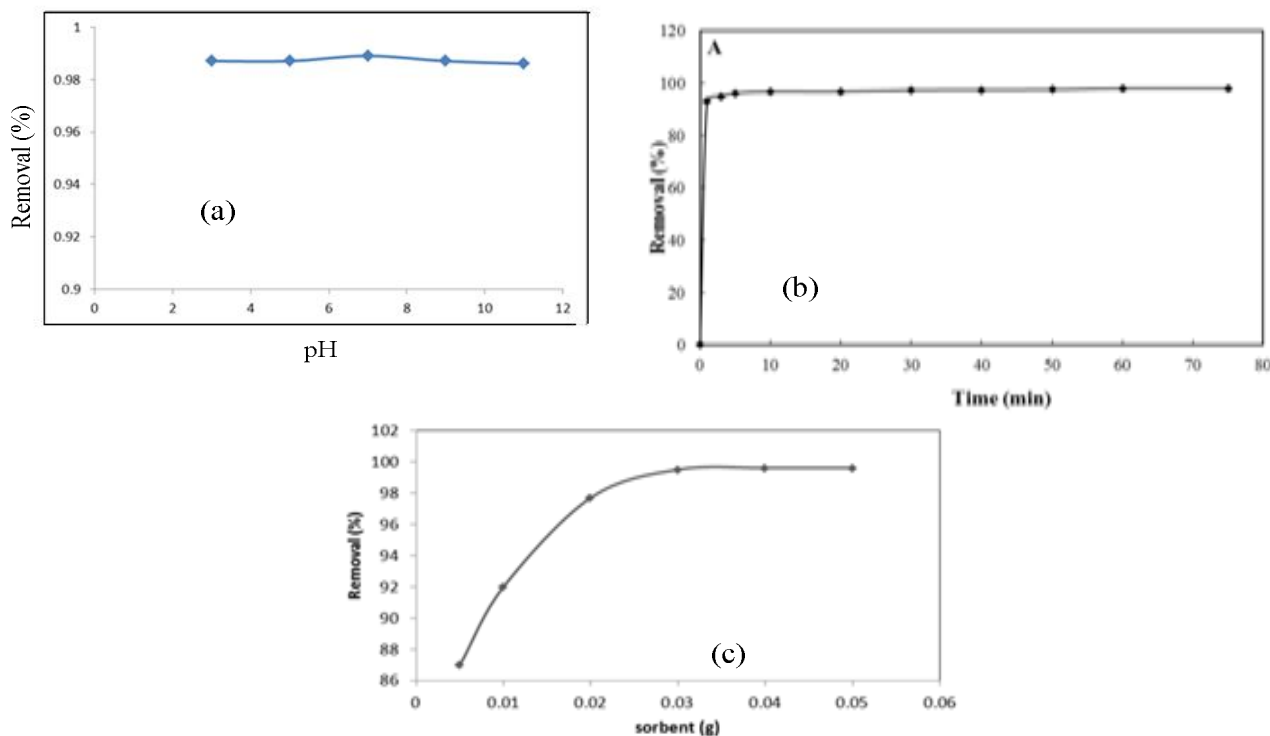


Fig. 4. (a) The effect of pH on adsorption of 50 mg l⁻¹ of MB by 0.03 g MWCNTs-EDA-SO₃H. Other conditions: stirring time 10 min, temperature 25 °C; (b) The effect of stirring time on adsorption of 50 mg l⁻¹ of MB by 0.03 g MWCNTs-EDA-SO₃H. Other conditions: pH 7.0, temperature 25 °C, stirring speed 150 rpm; (c) The effect of the adsorbent mass on adsorption of 50 mg l⁻¹ of MB. Other conditions: pH 7.0, stirring time 10 min, temperature 25 °C.

multi-walled carbon nanotubes. The optimum stirring time was found to be 10 min. The result was shown in Fig. 4b.

The Effect of the Adsorbent Mass

Different amounts of sorbent (5 to 50 mg) were chosen to investigate the effect of adsorbent mass on the removal of MB. Maximum adsorption was obtained when the mass of adsorbent was 30 mg. These results are shown in Fig. 4c.

The Effect of Initial Concentration of MB

Figure 5 shows the effect of initial concentration of dye on adsorption. According to this curve, the increase of dye concentration increases the adsorption, however in high concentrations the adsorption does not change because the places of adsorption have been saturated.

Following that, the removal of dye was compared between MWCNTs-EDA-SO₃H and other initial MWCNTs. As shown in Table 1, the removal of dye was improved by entering -SO₃H group. MB is a cationic dye and sulfonic acids are much stronger than carboxylic acids. Therefore, the electrostatic interaction between anionic sulfonate group of the sorbent and cationic dye may have a key role in adsorption process (Fig. 6).

Adsorption Kinetics

Since kinetic studies can provide essential information for the rate and mechanism of the adsorption, they were performed at 25 °C and neutral pH. The pseudo-first-order and pseudo-second-order rate equations were used to analyze the adsorption of MB by MWCNTS-EDA-SO₃H.

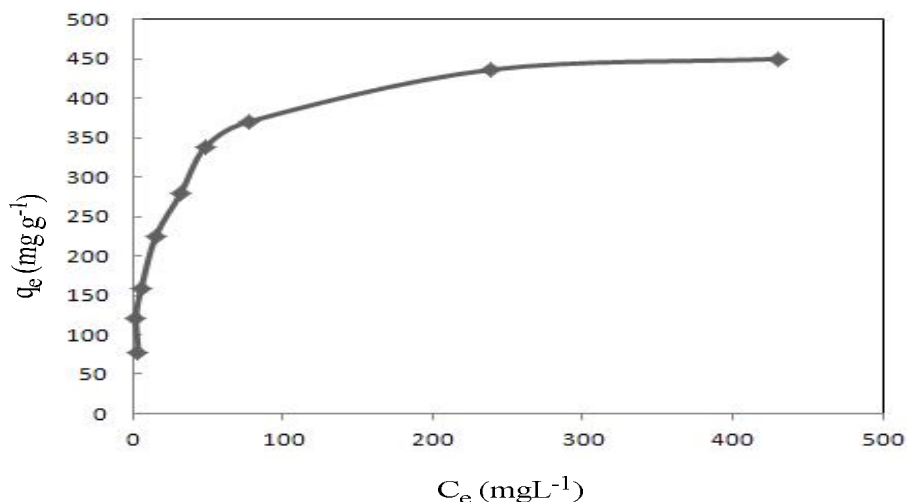


Fig. 5. Relation between equilibrium concentration and the adsorption (0.03 g adsorbent; T = 25 °C; pH = 7.0; stirring time = 45 min, sample volume = 50 ml).

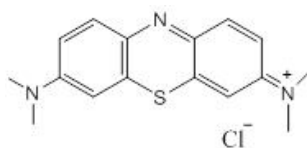


Fig. 6. Structure of methylene blue.

Table 2. Kinetic Models for MB Adsorption by MWCNTS-EDA-SO₃H

Models	Equations	Regression coefficient	Model parameters
Pseudo-first-order	$\log (q_e - q_t) = 0.783 - 0.042 t$	0.7129	$q_e = 6.06 \text{ mg g}^{-1}$ $k_1 = 0.097 \text{ min}^{-1}$
Pseudo-second-order	$\frac{t}{q_t} = 0.002 + 0.012 t$	1.000	$q_e = 83.33 \text{ mg g}^{-1}$ $k_2 = 0.072 \text{ g mg}^{-1} \text{ min}^{-1}$

These two models are shown in a linear form by Eqs. (1) and (2).

$$\log (q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (1)$$

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

Here, q_t and q_e (mg g⁻¹) are the amounts of adsorbed dye by adsorbent at t time and at equilibrium time, respectively; k_1

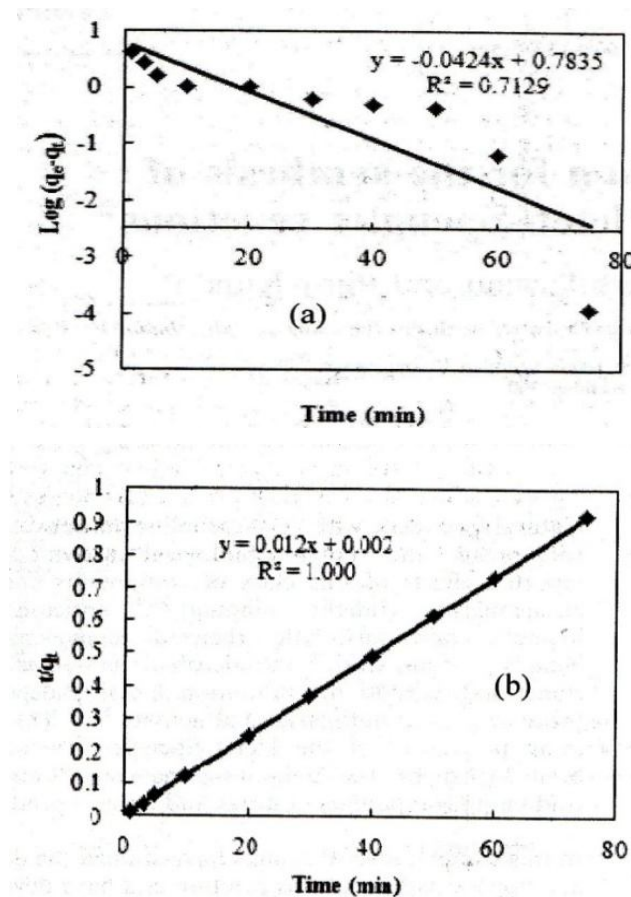


Fig. 7. Linear form of kinetic plots: (a) Pseudo-first-order kinetic model; (b) Pseudo-second-order kinetic model ($T = 25\text{ }^{\circ}\text{C}$; $\text{pH} = 7$; amount of adsorbent = 0.03 g ; $C_0 = 50\text{ mg l}^{-1}$; stirring speed = 150 rpm ; sample volume = 50 ml).

Table 3. Parameters and Regression Coefficients of Langmuir, Temkin and Freundlich

Isotherms	Equations	Regression coefficient	Model parameters
Langmuir	$C_e/q_e = 0.033 + 0.002 C_e$	0.998	$q_{\text{max}} = 500\text{ mg g}^{-1}$ $K_L = 0.606\text{ l mg}^{-1}$
Freundlich	$\ln q_e = 4.454 + 307 \ln C_e$	0.868	$K_F = 85.97$ $N = 3.257$ $K_T = 1.652$
Temkin	$q_e = 36.09 + 71.94 \ln C_e$	0.964	$B = 71.94$

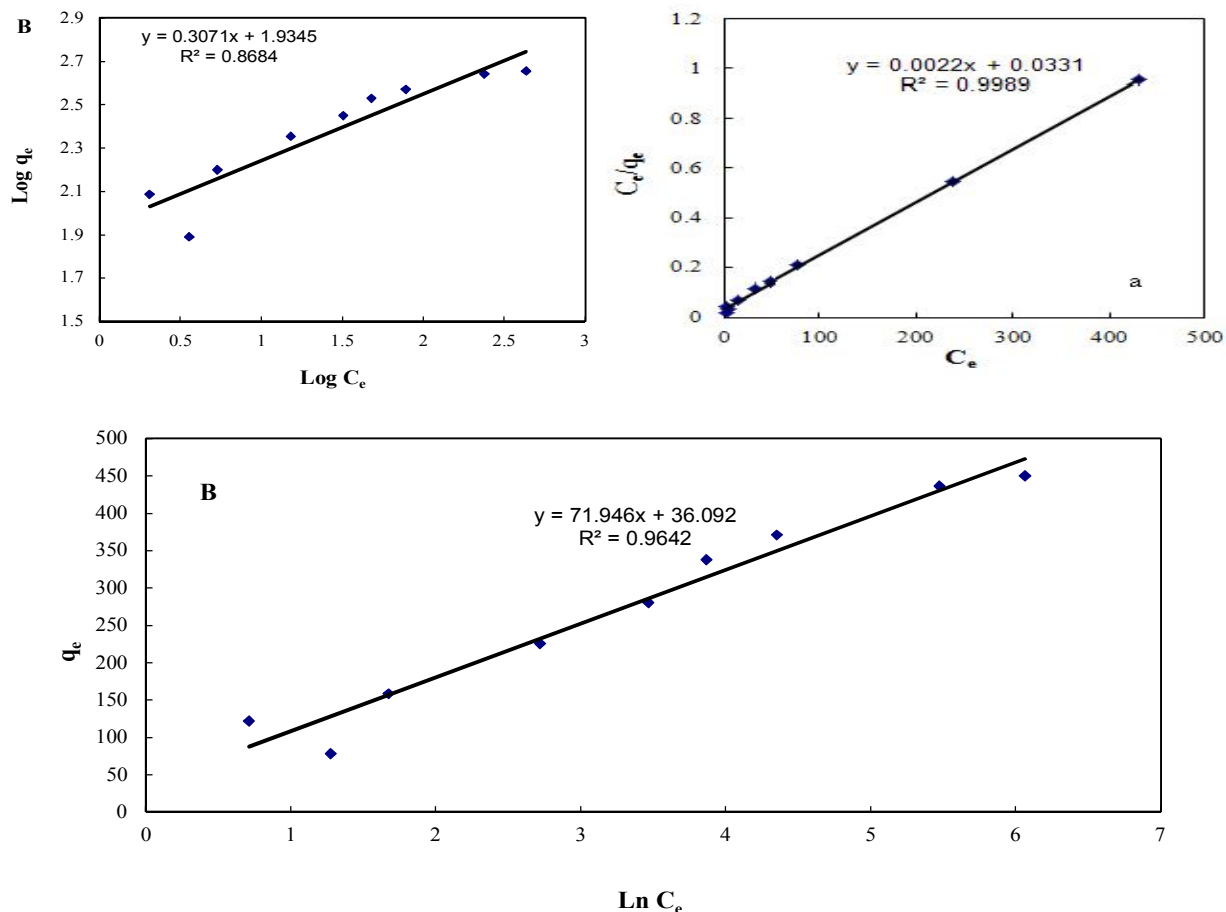


Fig. 8. Liner form of adsorption isotherm plots: Langmuir model (a); Freundlich model (b) and Temkin model (c): (T = 25 °C; pH = 7; amount of adsorbent = 0.03 g).

(min^{-1}) is the pseudo-first-order rate constant and k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) is the pseudo-second-order rate constant. Figures 7a and 7b show the pseudo-first-order and the pseudo-second-order kinetics, respectively, for this work. The regression coefficient (R^2) of the pseudo-first-order model is 0.7129 (mg g^{-1}) and for pseudo-second-order model is 1.000. These results suggest that the adsorption process is more accommodating with the pseudo-second-order model. The related parameters and kinetic equations are reported in Table 2.

Adsorption Isotherms

Maximum adsorption capacity determines the design of adsorption system and gives information about the

relationship between dye concentration in solution and the amount of adsorbed dye in solid phase. This parameter is obtained through the equilibrium adsorption isotherms. So, the investigation of these isotherms is very important. These isotherm plots are drawn by using experimental data and liner form of Langmuir, Freundlich and Temkin equations.

Langmuir Model

The Langmuir adsorption model is the most popular model to determine the quantity of adsorbate in an adsorbent. This is expressed in the following equation:

$$\frac{C_e}{q_e} = \frac{1}{K_f q_{max}} + \frac{C_e}{q_{max}} \tag{3}$$

Table 4. q_{max} for the Removal of MB on Various Adsorbents

Adsorbents	q_{max} (mg g ⁻¹)	Ref.
MWCNTs-EDA-SO ₃ H	500.0	This work
Fe ₃ O ₄ @PPy/RGO	270.3	[34]
Graphene nanosheet/magnetite (Fe ₃ O ₄) composite	43.82	[35]
Magnetic MWCNT nanocomposite	15.90	[36]
Magnetic loaded MWCNTs	40.06	[37]
Magnetic Ni/C nanomaterial	175.2	[38]
Acid-treated cup-stacked carbon nanotube	172.4	[39]
γ-Al ₂ O ₃	8.38	[40]
C2-symmetric cyclohexane-based hydrogels	47.1	[41]
Amphoteric wheat straw	140.3	[42]
Polydopamine microspheres	90.7	[43]
Poly(cyclotriphosphazene-co-4,4'-sulfonyldiphenol)	69.16	[44]
nanotube	170	[45]
Keratin nanofibrous membranes	158.03	[46]
Palygorskite		

q_{max} (mg g⁻¹) is the maximum adsorption capacity in saturated single layer and K_L (l mg⁻¹) is the Langmuir constant related to adsorption. The related parameters and kinetic equation that are reported in Table 3 and Fig. 8a show Langmuir isotherm for the under investigation system.

Freundlich Model

Freundlich adsorption isotherm is used for expressing the relationship between concentration of a solute on the surface of an adsorbent and the concentration of the solute in a liquid. This model is shown in Eq. (4).

$$\ln q_e = \ln K_F + 1/n \ln C_e \quad (4)$$

K_F (mg^{1-1/n} l^{1/n} g⁻¹) is adsorption capacity and n is adsorption intensification. Figure 8b and Table 3 show Freundlich

isotherm and related parameters.

Temkin Model

In Temkin isotherm model, increasing the coating of adsorbent surface causes the adsorption heat of all molecules that decrease linearly. In addition, adsorption is characterized by a uniform distribution of binding energies, up to a maximum binding energy. The Temkin isotherm can be represented by Eq. (5).

$$q_e = B \ln K_T + B \ln C_e$$

$$B = RT/b \quad (5)$$

K_T is the equilibrium binding constant (M⁻¹) corresponding to the maximum binding energy, b is related to the

adsorption heat, R is the universal gas constant ($8.314 \text{ J K}^{-1} \text{ mol}^{-1}$) and T is the temperature (K). Figure c and Table 3 show Temkin isotherm and related parameters.

Comparison between regression coefficients in these three isotherms shows that R^2 value of Langmuir model ($R^2 = 0.998$) is closer to 1.000. So, this model is more suitable than the Temkin and Freundlich models for the expression of adsorption behavior of MB onto MWCNTs-EDA-SO₃H. The MB adsorption capacity by this adsorbent and other reported adsorbents are expressed in Table 4. As shown, q_{max} for MWCNTs-EDA-SO₃H is considerably higher than that of others.

CONCLUSIONS

This project offers a facile and efficient adsorption method for removing methylene blue from aqueous solution. Short stirring time, neutral pH and high q_{max} are the significant characteristics of this work. The adsorption process is more accommodating with the pseudo-second-order model. The Langmuir model is more suitable than the Temkin and Freundlich models for the expression of adsorption behavior of MB onto adsorbent.

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