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Ultra Efficient Removal of Basic Blue 41 from Textile Industry's Wastewaters by Sodium Dodecyl Sulphate Coated Magnetite Nanoparticles: Removal, Kinetic and Isotherm Study

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This research presents adsorption potential of surfactant (sodium dodecyl sulphate; SDS) coated magnetite nanoparticles as a suitable and efficient adsorbent for the removal of Basic Blue 41 (BB 41), a cationic dye from textile's company wastewater. The effects of various experimental parameters (e.g., initial pH of the solution, SDS amount, and ionic strength) on the adsorption efficiency of BB 41 were investigated and optimized. The result showed that kinetic of adsorption process is very fast, and equilibrium for dye adsorption could be obtained after 30 min. Equilibrium isotherm data were fitted to Langmuir and Freundlich models. The characteristic parameters for each model were also determined. The Langmuir isotherm gave the best correlation for the adsorption of BB 41 on the surface of adsorbent. A superior maximum adsorption capacity (2000.6 mg g^{-1}) was obtained for the adsorbent from the Langmuir model. Also, reuse study showed that Fe_3O_4 NPs could be regenerated and reused at least for six times.

Keywords: Magnetite nanoparticles, Removal, Basic dyes, Basic Blue 41

INTRODUCTION

One of the most serious sources of pollution in water is dye pollution, commonly from wastewater discharged from textile, leather and paper industries [1]. Textile industries are the major consumers of water and mainly responsible for the discharge of large quantities of dyes into natural waterways. Among the different classes of textile dyes, the basic ones have high brilliance and intensity of colors and are highly visible even in a very low concentration [2]. Since most of the dyes are highly toxic, carcinogenic, and stable against light and oxidation, thus finding an effective and suitable way to eliminate dye contaminations from water has become an urgent issue.

Various physicochemical methods have been proposed to eliminate dyes from textile-derived wastewaters such as adsorption, precipitation, chemical degradation, photodegradation, biodegradation, chemical coagulation and electrocoagulation [3]. Among all reported treatment methods, adsorption is the most attracting method for removal of wide range of dyes from environmental samples with high efficiency and low cost [4]. Moreover, among all conventional adsorbents, activated carbon is the most popular and efficient adsorbent, but it is expensive and the costs of regeneration are high [5]. In this case, various low-cost and efficient adsorbents have been recently used for removal of basic dyes from aqueous samples as alternatives to activated carbons such as silkworm pupa [6], boron waste [7], phosphoric acid modified rice straw [8], cyclodextrin polymer [9], coconut fiber particles [10], natural zeolitic

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tuff [11] and brick waste [12].

In recent years, magnetic nanomaterials have also attracted substantial interest in removal of dyes from environmental water samples as the result of their unique properties such as easy separation, large surface area and less mass transfer resistance [13,14]. Magnetic nanoadsorbents based on hemimicelles and admicelles (mixed hemimicelles) are new type adsorbents which have been extensively used in recent years as the result of easy preparation and high efficiency. These adsorbents could be produced by the adsorption of ionic surfactants such as sodium dodecyl sulfate (SDS) [15-18], cetyltrimethylammonium bromide (CTAB) [19] or ionic liquid on the surface of magnetic nanoparticles [20,21]. They could simultaneously provide hydrophobic and electrostatic interactions; two types of mechanisms for retention of analytes [22]. Also, high removal yield, easy elution of pollutant and modification engineering based on nature of analytes are other advantages associated with such adsorbents [23].

The aim of the present work is to investigate the removal of a common basic dye, Basic Blue 41 (BB 41), a cationic dye, from colored wastewaters by its adsorption on the sodium dodecyl sulphate coated magnetite nanoparticles (SDS-Fe₃O₄ NPs). Effect of pH, contact time, adsorbent dosage, and initial dye concentration on the adsorption capacity are investigated. Adsorption isotherms and kinetics for removal of BB 41 using SDS-coated MNPs are also studied.

EXPERIMENTAL

Chemical and Apparatus

BB 41 (Fig. 1a) was obtained from Dystar (Frankfurt, Germany). Ferric chloride (FeCl₃.6H₂O), ferrous chloride (FeCl₂.4H₂O), sodium hydroxide, sodium dodecyl sulphate (SDS), methanol, acetone, acetic acid, sodium acetate, sodium sulfate, and hydrochloric acid were obtained from Merck (Darmstadt, Germany). Double distilled water was used throughout the experiments. Stock solution of the dye (1000 mg l⁻¹) was prepared in the double distilled water. The working standard solutions were prepared by suitable dilution of the stock solution with double distilled water.

Spectrophotometric measurements of BB 41 were done

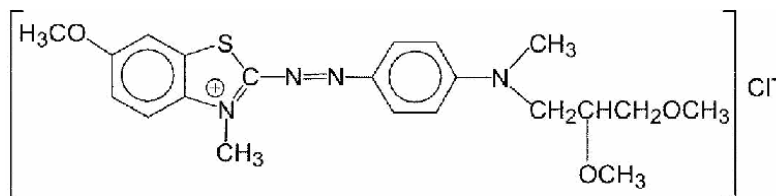
at λ_{\max} (610 nm) by a Cesil CE-7200 UV-Vis spectrophotometer (Cambridge, England). Magnetic separation was done by a super magnet with 1.4 Tesla magnetic field (10 × 5 × 4 cm). A Heidolph motor-stirrer (Schwabach, Germany) was applied for stirring of the dye solutions with a glassware stirrer. Also, a shaker with a temperature controller was applied for shaking the dye solutions in the isotherm studies at a constant temperature.

Synthesis of Fe₃O₄ NPs

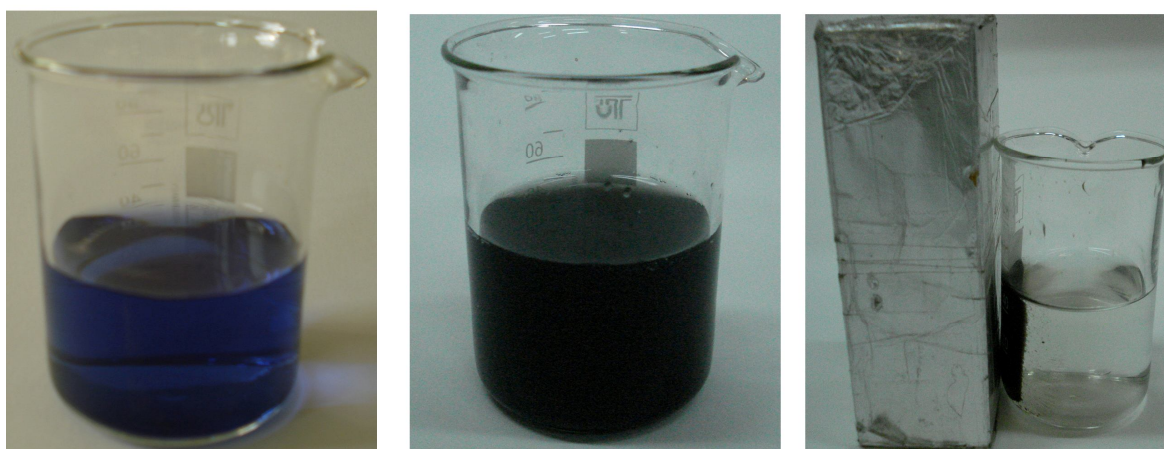
Fe₃O₄ NPs were prepared by chemical co-precipitation method [19]. Briefly, 10.4 g of FeCl₃.6H₂O, 4.0 g of FeCl₂.4H₂O and 1.7 ml of HCl (12 M) were dissolved in 50 ml of deionized water in order to prepare stock solution of ferrous and ferric chloride in a beaker and it was then degassed with N₂ for 20 min. Simultaneously, 500 ml of 1.5 M NaOH solution was degassed (for 15 min) and heated to 80 °C in a reactor. Then, the stock solution was added dropwise *via* dropping funnel during 30 min under N₂ protection and vigorous stirring (1000 rpm) by a glassware stirrer. During the whole process, temperature of the solution was maintained at 80 °C and nitrogen gas was purged to prevent intrusion of oxygen. After completion of reaction, the obtained precipitate of Fe₃O₄ NPs was separated from the reaction medium by the magnet, and then washed with 500 ml deionized water four times. Finally, the obtained Fe₃O₄ NPs were resuspended in 500 ml of degassed deionized water.

Dye Adsorption Optimization

Optimization studies were carried out according to the following procedure: (1) a 40 ml aqueous solution of the dye (20 mg l⁻¹) were prepared in a 100 ml beakers by the addition of the appropriate amount of stock solution of the dye, (2) 1.0 ml of Fe₃O₄ NPs suspension (containing 10 mg of Fe₃O₄ NPs) was added to the dye solution, (3) pH of the solution was adjusted to desired value and then SDS was added into the solution, (4) the mixture solution was stirred to enhance the dye adsorption, (5) after dye adsorption; Fe₃O₄ NPs were separated quickly (1 min) from the sample solutions by using the magnet, (6) the residual dye concentration in the supernatant clear solution was determined spectrophotometrically using a calibration curve. Dye removal efficiency is shown in Fig. 1b. The



(a)



(b)

Fig. 1. Molecular structure of BB 41 (a). Images of the dye removal steps (b): (I) dye solution before addition of Fe₃O₄ NPs, (II) dye solution after addition of Fe₃O₄ NPs and SDS, and (III) dye solution after being exposed to a very strong magnet.

following equation was applied to calculate the dye removal efficiency in the treatment experiments:

$$\text{Dye removal efficiency (\%)} = \frac{(C_0 - C)}{C_0} \times 100 \quad (1)$$

where C_0 and C are the initial and residual concentrations of the dye in the solution (mg l^{-1}), respectively.

Adsorption Studies

Adsorption isotherm studies were carried out in a set of 70 ml glass bottle, where 10 mg of Fe₃O₄ NPs, 5 mg of SDS and 40 ml of the dyesolution with different initial concentrations (mg l^{-1}) were added. The dye solution was

prepared in dyeing vat composition for the basic dyes (3% CH₃COOH, 10% Na₂SO₄ and 2% (w/v) CH₃COONa). The solution was shaken at 200 rpm in an isothermal shaker for 2 h to reach the equilibrium at constant temperature (25 °C). The bottles were then taken from the shaker and after magnetic separation of SDS-Fe₃O₄ NPs, the equilibrium concentration of the dye was measured. The amount of dye uptaken by SDS-Fe₃O₄ NPs, q_e (mg g^{-1}), was obtained as follows:

$$q_e = \frac{C_0 - C_e}{m_s} \quad (2)$$

where C_0 and C_e (mg l^{-1}) are the initial and equilibrium

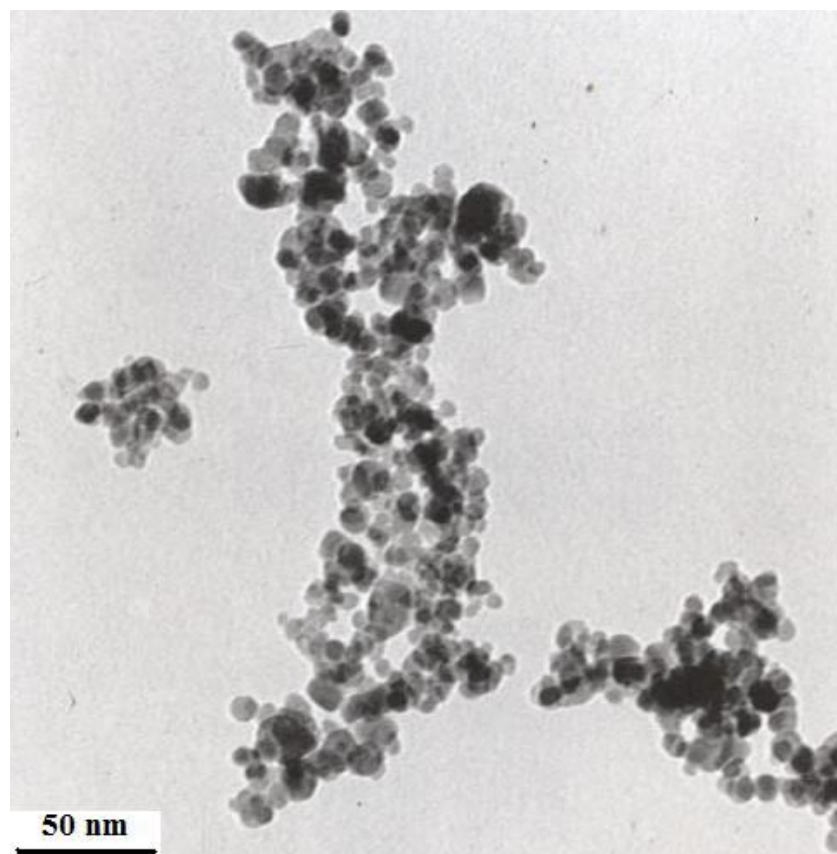


Fig. 2. TEM image of Fe₃O₄ NPs.

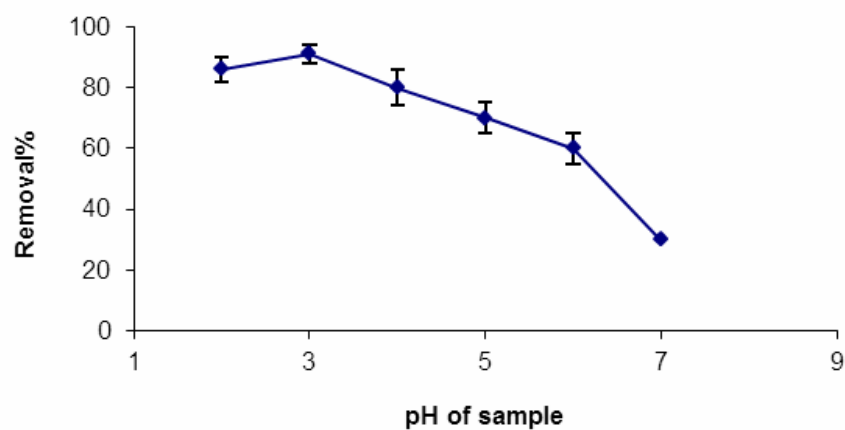


Fig. 3. Effect of sample's pH on removal efficiency of the dye. Conditions: 40 ml of dye solution (20 mg l⁻¹); 10 mg of Fe₃O₄ NPs; 2.5 mg of SDS; stirring time = 2 min; stirring speed 200 rpm; ambient temperature; double distilled matrix.

concentrations of dye in solution, respectively, and m_s is the concentration of Fe_3O_4 NPs (g l^{-1}).

Kinetic studies were performed in a 1.0 liter glass beaker, where 100 mg of Fe_3O_4 NPs and 50 mg of SDS were added into 400 ml of the dye solution with 100 mg l^{-1} at ambient temperature. The agitation speed of the motor stirrer was fixed at 200 rpm during the experiments. At the preset time intervals, the samples of 10 ml were taken from the solution and the concentration of the dye was determined by the spectrophotometer.

RESULTS AND DISCUSSION

Characterization of Fe_3O_4 NPs

Morphology of Fe_3O_4 NPs was determined by H-800 transmission electronic microscope (TEM) from Hitachi (Tokyo, Japan). TEM image of Fe_3O_4 NPs is shown in Fig. 2. As seen in this figure, most of the nanoparticles are quasi-spherical with an average diameter of 10-20 nm.

Effect of pH of Solution

Adsorption of positively charged dye groups (basic dyes) on the surface of adsorbent is primarily influenced by surface charge of adsorbent which in turn is influenced by pH of the solution. The adsorption behaviour of SDS- Fe_3O_4 NPs could be influenced by variation of the charge density on the Fe_3O_4 NPs surface. As shown in Fig. 3, maximum adsorption of BB 41 was obtained when pH was 3.0. The adsorption amount decreased when pH increased from 3.0 to 7.0. This could be attributed to the fact that the positively charged surface of Fe_3O_4 NPs was favourable for the adsorption of anionic surfactants. At higher pHs ($\text{pH} > 3$), positive charge density of Fe_3O_4 NPs surface decreased. The electrostatic attraction between negative charges of SDS and positive charges of Fe_3O_4 NPs surface was not strong enough to produce SDS- Fe_3O_4 NPs, resulting in decreasing adsorption of BB 41. On the other hand, BB 41 adsorption slightly decreased when pH was 2.0, which it might be explained by reduction of SDS adsorption on the surface of adsorbent, probably due to protonation of their sulfate groups [24]. Therefore, pH of 3.0 was selected for the

subsequent studies.

Effect of SDS Amount on Adsorption of BB 41

Figure 4 depicts the percentage of adsorbed dye as a function of concentration of added SDS. In the absence of SDS, no dyes adsorb onto the surface of Fe_3O_4 NPs. In contrast, by increasing SDS concentration, the adsorbed amount of the dye increased remarkably. The increase in adsorption could be explained by gradual formation of SDS aggregates (hemimicelles, mixed hemimicelles or admicelles) on the surface of Fe_3O_4 NPs which it prompts dye adsorption. Generally, adsorption of ionic surfactant on surface of mineral oxides could be divided into three regions (hemimicelles, mixed hemimicelles, and admicelles) (below and above the critical micelles concentration, cmc)) [25]. Based on the results (Fig. 4), maximum adsorption was obtained when SDS amounts were between 2.5 and 10 mg. Thus, 5 mg of SDS was selected for further studies.

Effect of Salt Concentration

Textile industry's wastewaters contain high concentrations of various types of salts. Therefore, presence of these salts may significantly affect the performance of the adsorption process. Figure 5 shows that the variation of sodium chloride (NaCl) concentration from 0 to 5% (w/v) exhibits a major negative effect on the extent of the dye adsorption. In contrast, by increasing NaCl amount from 5 to 10% (w/v), the adsorption amount of the dye increased, may be due to salting-out effect, and after that it remained almost constant. Since the extent of adsorption is sensitive to variation of NaCl concentration, electrostatic attraction may be the main driving force of the overall BB 41 adsorption.

Kinetics of Adsorption

Figure 6a shows the adsorption kinetic of BB 41 as percentage of dye removal *versus* time at initial dye concentration of 100 mg l^{-1} . As seen, a large amount of BB 41 was removed in the first 10 min of contact time. Also, required time for equilibrium was 20-45 min. Rapid kinetics has significant practical importance as it will facilitate smaller reactor volumes ensuring efficiency and economy

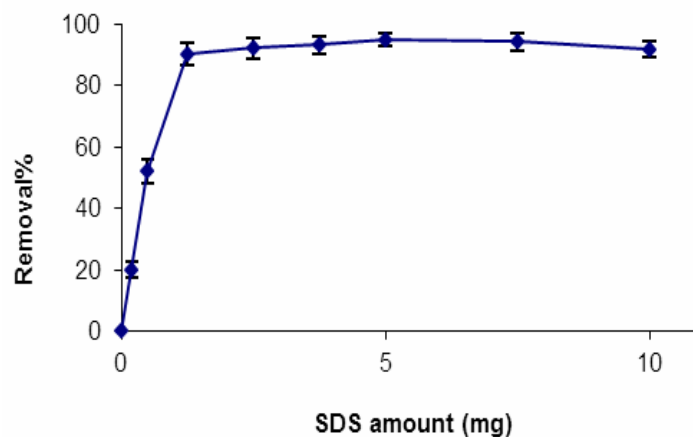


Fig. 4. Effect of SDS amounts on removal efficiency of the dye. Conditions: 40 ml of dye solution (20 mg l^{-1}); 10 mg of Fe_3O_4 NPs; sample's pH = 3.0; stirring time = 2 min; stirring speed 200 rpm; ambient temperature; double distilled matrix.

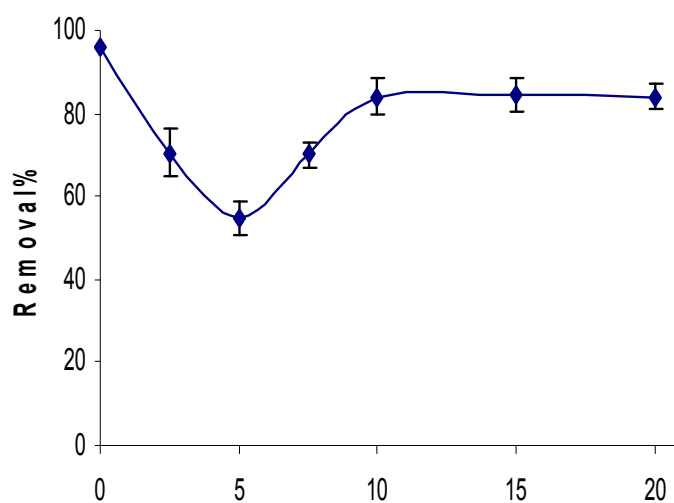


Fig. 5. Effect of NaCl amounts on removal efficiency of the dye. Conditions: 40 ml of dye solution (20 mg l^{-1}); 10 mg of Fe_3O_4 NPs; sample's pH = 3.0; 5.0 mg of SDS; stirring time = 2 min; stirring speed 200 rpm; ambient temperature; double distilled matrix.

[26]. Subsequently, pseudo-first order [27] or pseudo-second order [28], and intra-particle diffusion [29] kinetic models, as commonly used models in the literature, were applied to experimental kinetic data of BB 41 in order to investigate adsorption behavior of BB 41 on SDS- Fe_3O_4

NPs.

$$\text{Pseudo-first-order kinetic model: } \log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (3)$$

Pseudo-second-order kinetic model:
$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$
 (4)

Intra-particle diffusion model:
$$q_t = k_p t^{0.5}$$
 (5)

where q_t and q_e are the amount of adsorbed dye at time t and at equilibrium time (mg g^{-1}), respectively; k_1 (min^{-1}), k_2 ($\text{g mg}^{-1} \text{min}^{-1}$), k_p ($\text{mg g}^{-1} \text{min}^{-0.5}$) are the pseudo-first order, pseudo-second order and intra-particle diffusion rate constants for adsorption process, respectively.

The validity of each model can be checked from linear plot. To find the order of kinetic, pseudo-first order kinetic equation was first tested for BB 41 kinetic data, however the straight lines could not be obtained (results not shown). For pseudo-first order kinetics plots, correlation coefficient was below 0.80 and the calculated q_e was not equal to experimental q_e , suggesting insufficiency of the model to fit kinetic data for the examined initial concentration. The same results were obtained for intra-particle diffusion model, and the correlation coefficient for this model was also below 0.80. In contrast, pseudo-second order model was well fitted to kinetic results of BB 41 (Fig. 6b). The rate parameters, k_2 and q_e were directly obtained from the intercept and slope of the plot of t/q_t versus t . Also, k_2 and q_e values which obtained by linear regression, were 9.1×10^{-3} and 370.4 mg g^{-1} , respectively. The results showed that the adsorption system followed the pseudo-second order model equation for the entire adsorption period, with regression coefficient of 0.9988. Adsorption kinetic of BB 41 on SDS- Fe_3O_4 NPs followed by pseudo-second order model, suggesting that the rate-limiting step may be chemisorption [30]. This confirmed that adsorption of BB 41 takes place probably *via* surface exchange reactions until the surface functional sites are fully occupied. In the literature, adsorption kinetics of many dye species onto various adsorbents was also fitted to pseudo-second order [15-19, 31-36].

Adsorption Isotherms

Adsorption equilibrium data which express the relationship between mass of adsorbed adsorbate per unit weight of adsorbent and liquid-phase equilibrium concentration of adsorbate are represented by adsorption

isotherms and provide important design data for adsorption system. Adsorption isotherms describe how pollutants interact with adsorbents, and so, are critical in optimizing the use of adsorbents. In order to optimize the design of an adsorption system to remove dye from solutions, it is important to establish the most appropriate correlation for the equilibrium curve. In literature, there are several isotherm equations for analyzing experimental adsorption equilibrium data [37]. In this study, the experimental data were fitted to well-known and widely used Langmuir [38] and Freundlich isotherms [39].

The fitting of Langmuir isotherm suggests that all adsorption sites are equivalent. Based on this model adsorbent is saturated after one layer of adsorbate molecules formed on the surface of adsorbent. The linearized equation of Langmuir is represented as follows:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{\max}} + \frac{1}{q_{\max}} C_e$$
 (6)

where q_e is the equilibrium dye concentration on the adsorbent (mg g^{-1}); C_e is the equilibrium dye concentration in solution (mg l^{-1}); q_{\max} is monolayer capacity of adsorbent (mg g^{-1}) and K_L is the adsorption equilibrium constant.

On the other hand, Freundlich isotherm model takes the multi-layer and heterogeneous adsorption into account. Its linearized form can be represented as:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e$$
 (7)

where q_e is the equilibrium dye concentration on the adsorbent (mg g^{-1}); C_e is equilibrium dye concentration in solution (mg l^{-1}); K_F , is Freundlich constant and n is heterogeneity factor.

Parameters related to adsorption of the BB 41 on the adsorbent for each isotherm were determined by using linear regression analysis and the square of the correlation coefficients (r^2) have been calculated (Table 1). The results showed that the equilibrium data were well explained by Langmuir isotherm model as compared to Freundlich model. On the basis of Langmuir analysis, the maximum adsorption capacity determined was 2000.6 mg g^{-1} . These

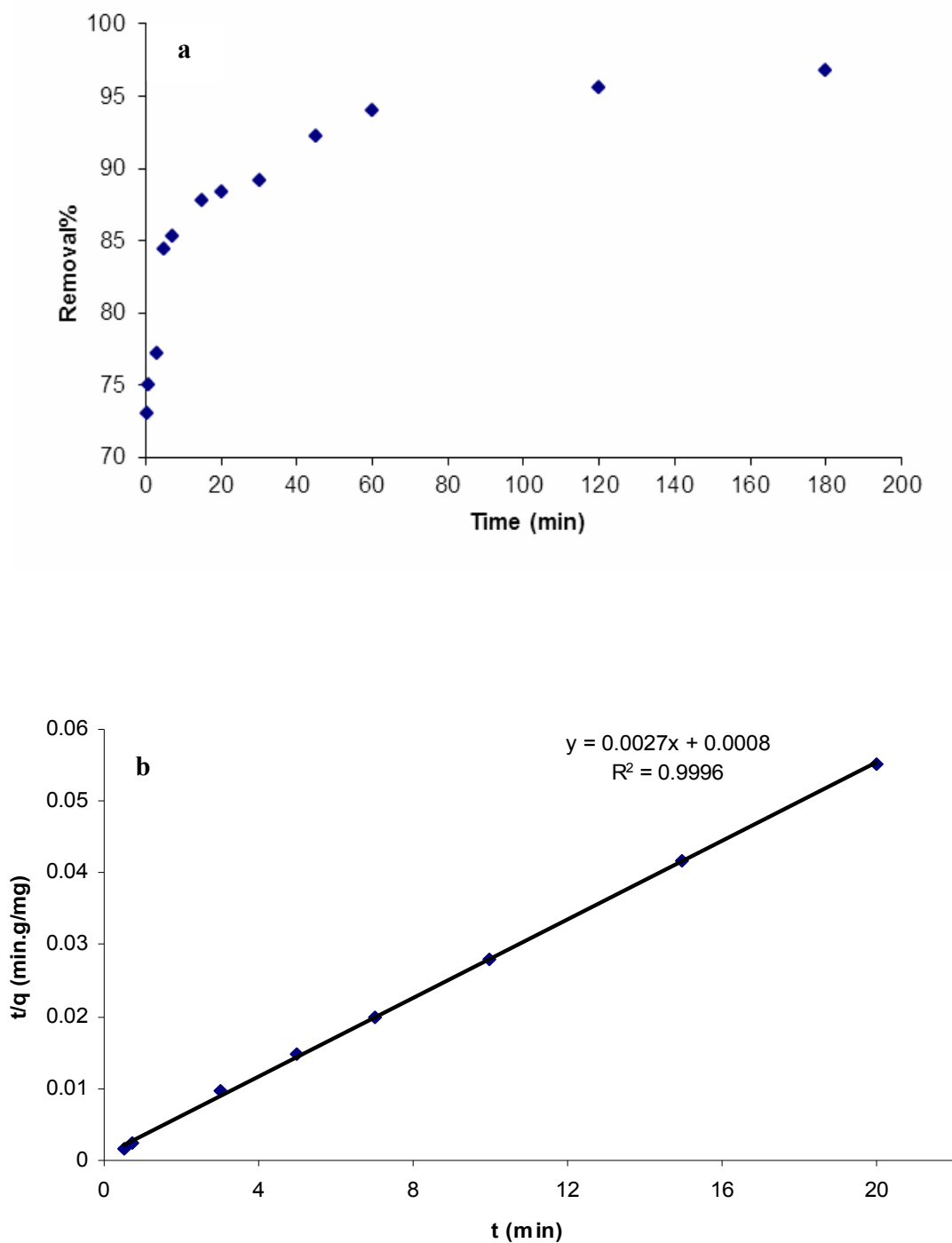


Fig. 6. (a) Adsorption kinetics of BB 41 by SDS-Fe₃O₄ NPs. Conditions: 400 ml of dye solution (100 mg l⁻¹); 100 mg of Fe₃O₄ NPs; sample's pH = 3.0; 50 mg of SDS; stirring speed 200 rpm; ambient temperature; real sample matrix. (b) Pseudo-second-order kinetic model for the adsorption of BB 41 by SDS-Fe₃O₄ NPs. Conditions: 40 ml of dye solution; 10 mg of Fe₃O₄ NPs; sample's pH = 3.0; 5.0 mg of SDS; real sample matrix.

Table 1. Langmuir and Freundlich Isotherms' Constants for the Adsorption of BB 41 on SDS-Fe₃O₄ NPs

Dye	Isotherm model					
	Langmuir			Freundlich		
	q _{max} (mg g ⁻¹)	K _L (l mg ⁻¹)	R _L ²	n	K _F (l mg ⁻¹)	R _F ²
BB 41	2000.6	0.13	0.9988	0.46	4.8 × 10 ⁻⁶	0.7390

Table 2. Maximum Adsorption Capacities of Different Adsorbents Used for Removal of Basic Dyes

Adsorbent	Basic dye	q _{max} (mg g ⁻¹)	Ref.
Silkworm pupa	Basic Blue 41	555.0	[6]
Boron waste	Basic Yellow 28	75.0	[7]
	Basic Red 46	74.7	
Phosphoric acid modified rice straw	Basic blue 9	208.3	[8]
	Basic red 5	188.7	
Cyclodextrin polymer	Basic Blue 3	42.4	[9]
	Basic Violet 3	35.8	
	Basic Violet 10	53.2	
Coconut fiber particles	Basic blue 41	1000	[10]
Natural zeolitic tuff	Basic blue 41	192.31	[11]
Brick waste	Basic blue 41	60-70	[12]
SDS-Fe ₃ O ₄ NPs	Methyl violet	416.7	[15]
SDS-Fe ₃ O ₄ NPs	Safranin O	769.23	[16]
SDS-Fe ₃ O ₄ NPs	Crystal violet	166.67	[17]
SDS-Spinel cobalt ferrite	Crystal violet	105	[18]
SDS-Fe ₃ O ₄ NPs	Basic blue 41	2000.6	This work

values demonstrated that the SDS-Fe₃O₄ NPs exhibited interesting adsorption properties for BB 41.

The favourable adsorption of Langmuir isotherm can be expressed in terms of a dimensionless constant separation

factor or equilibrium parameter R_L:

$$R_L = \frac{1}{1 + K_L C_0} \quad (8)$$

where C_0 is the initial concentration of the adsorbate in the solution (100 mg l^{-1}). This parameter indicates that isotherm will be shaped according to the following adsorption characteristics: $R_L > 1$ is unfavorable, $R_L = 1$ corresponds to linear, $0 < R_L < 1$ is favorable and $R_L = 0$ is irreversible. The R_L value of SDS- Fe_3O_4 NPs adsorbent was 0.07, indicating that the adsorption of BB 41 onto surface of SDS- Fe_3O_4 NPs is favorable [40].

Desorption and Regeneration Studies

Since BB 41 adsorption on the SDS- Fe_3O_4 NPs is a reversible process, therefore regeneration or activation of Fe_3O_4 NPs for reusing is possible. The primary objective of adsorbent regeneration is to restore its adsorption capacity while the secondary objective is to recover valuable components present in the adsorbed phase (*e.g.* dye). Organic solvents are known to cause disruption of surfactant aggregates [24,25]. The desorption of BB 41 from SDS- Fe_3O_4 NPs was studied using different kinds of organic solvents (acetonitrile, ethanol, methanol). Desorption ability of methanol was found higher than the other solvents. The results showed that desorption efficiency higher than 95% could be achieved using 2 mL of methanol in a short time of 1 min and also in single elution. Based on the obtained results, Fe_3O_4 NPs could be regenerated by methanol and reused at least for six successive removal processes with removal efficiency higher than 95%. As a result, in comparison with other adsorbents (Table 2), Fe_3O_4 NPs can be easily collected from sample solution and regenerated in short time due to their superparamagnetic properties. Therefore, no centrifugation or filtration needed after treatment which this property is most favourable in practical applications.

CONCLUSIONS

This study confirmed that SDS- Fe_3O_4 NPs could be an excellent adsorbent for removal of BB 41 from aqueous solution. The obtained results showed that sample pH and SDS amount significantly affect the adsorption of BB 41. It was found that in the absence and/or higher NaCl concentration ($\geq 10\%$ w/v) removal efficiencies of the dye is high but when NaCl concentration is between 0 and 10% (w/v) removal efficiencies of dyes are decreased. The

kinetics data for BB 41 followed a pseudo-second order equation, suggesting that rate-limiting step might be chemisorption. Also, the adsorption isotherm data was well fitted by Langmuir isotherm. The fitness of Langmuir model in the present system shows the formation of monolayer coverage of the adsorbate at the outer surface of the adsorbent. Also, the results showed that Fe_3O_4 NPs can be easily regenerated and reused. Compared with some recently published results about the removal of basic dyes (Table 2), the obtained results showed that SDS- Fe_3O_4 NPs could be an excellent adsorbent for removing of BB 41 with superior adsorption capacity ($q_{\text{max}} = 2000.6 \text{ mg g}^{-1}$). Finally, the data may be useful for designing and fabricating an economically cheap treatment process using batched or stirred tank flow reactors for the removal of BB 41 from industrial wastewaters.

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