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Removal of brilliant cresyl blue from aqueous solutions using modified zirconia nanoparticles as an adsorbent under ultrasonic action

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ABSTRACT

Zirconia nanoparticles were modified by sodium dodecyl sulfate (SDS) under ultrasonic action and used as a new adsorbent for the removal of brilliant cresyl blue dye from aqueous solutions. The effect of different parameters such as pH, salt concentration, and amount of adsorbent on the removal procedure was studied and optimum conditions were established. The adsorption data were fitted to Langmuir, Freundlich, and Temkin isotherms and the equilibrium adsorption was best described by the Langmuir isotherm model. The kinetic studies indicated that the experimental data were fitted well by the pseudo-second-order model. The maximum adsorption capacity was found to be 34.4 mg g^{-1} of adsorbent for brilliant cresyl blue. The removal procedure was applied to the removal of brilliant cresyl blue form water samples.

Keywords: Brilliant cresyl blue; Nano-ZrO_2; Sodium dodecyl sulfate; Removal

1. Introduction

Synthetic dyes have been widely used as coloring agents in paper, textile, wool, foodstuffs, and cosmetics. However, some of these dyes pose a potential risk to human health and are even carcinogenic [1]. Comparing to natural dyes, synthetic dyes have several advantages such as high stability to light, oxygen, and pH, color uniformity, and relatively lower production costs [2]. Many industries use dyes or pigments to color their products. Some of these dyes or pigments are inevitably left in the industrial waste, which are hazardous to the environment. Therefore, the removal of dyes from textile wastewaters is one of the major environmental problems because of the difficulty of treating such water samples by conventional treatment methods [3].

Brilliant cresyl blue is a cationic dye (Fig. 1) which is extensively used for nuclear counterstaining in biological research and textiles. Most of the dyes, including brilliant cresyl blue are toxic and must be removed before entering the environment. Different physical and chemical techniques including adsorption, coagulation, and cloud point extraction [4,5] have been employed for the removal of dyes from textile effluents.

Activated carbon is the most popular adsorbent and has been used with great success because of its large surface area, high adsorption capacity, and high degree of surface reactivity. Unfortunately, it is considered as an expensive material [6,7]. Thus, there has been a great interest to introduce new adsorbents for dye removal. Different materials such as zeolites [8],

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natural clay [9], sand [10], biosorbents [11,12], saw dust [13], acorn [14], Diaion HP 2MG [15], oxidized multiwalled carbon nanotube [16], peanut hull [17], and modified magnetic iron oxide nanoparticles [18,19] have also been applied for the removal of color dyes. In recent years, considerable attention has been devoted to study the removal of the dyes from wastewater by adsorption using agricultural and natural products and by-products [11]. Nano materials have shown to be the most promising adsorbents for removing different types of dyes [18], and also for preconcentration and removal of metal ions [20–22]. One of the specific properties of nanomaterials is the presence of a high percent of the atoms on their surface. The surface atoms are unsaturated and can therefore bind with other atoms and possess high chemical activity. Nanoparticulate metal oxides ZrO_2, TiO_2, and CeO_2 exhibit intrinsic surface reactivity and high surface areas and can strongly chemisorb many substances. These materials have a very high adsorption capacity toward organic compounds such as dyes and metal ions [23]. Zirconia is stable over a wide pH range and has high thermal stability. The surface modification of zirconia nanoparticles may increase the options in the development of analytical procedures [24]. Zirconia has mainly been used for photocatalytic degradation of dyes [25]. However, there are a limited number of researches on the use of zirconia as an adsorbent. Recently, Sandoval et al. [26] applied granular activated carbon media impregnated with zirconium dioxide nanoparticles for the removal of arsenic and methylene blue from water.

Therefore, this work focuses on the evaluation of zirconia nanoparticles modified by sodium dodecyl sulfate (nano-ZrO_2-SDS) as a new adsorbent for the removal of brilliant cresyl blue dye. The effects of different parameters such as pH, adsorbent dose, and initial dye concentration on the adsorption of brilliant cresyl blue dye onto nano-ZrO_2-SDS were investigated.

2. Experimental section

2.1. Apparatus

A GBC Model Cintra 101 UV–visible spectrophotometer (Sidney, Australia) was used for recording absorption spectra and absorbance measurements using 1 cm glass cells. A Metrohm digital pH meter Model 632 (Herisau, Switzerland) with a combined glass electrode was used to measure the pH values.

TEM images were performed by a Zeiss- EM10C-80 kV transmission electron microscope (Oberkochen, Germany).

A Universal Ultrasonic model DSA100-SK2–4/OL (Korean) was used.

2.2. Reagents

All chemicals were of analytical grade and doubled distilled water was used throughout the experiments. A stock solution of 100 mg L^{-1} of brilliant cresyl blue (Merck, Darmstadt, Germany) was prepared by dissolving 0.1 g of the reagent in water and diluting to 1,000 mL in a volumetric flask. Acetate buffer pH 4 was prepared by adding 1.0 M of HCl (Merck) to 50 mL of 0.1 M sodium acetate (Merck) and adjusting the pH to 4.0 using a pH meter. Sodium dodecyl sulfate (SDS) (Merck) was prepared by dissolving 0.1–0.7 g SDS in water and diluting to the mark in 25 mL volumetric flasks.

ZrO_2 nanoparticles (average size 20 nm) were purchased from Nanobond Technology (Tehran, Iran).

2.3. Preparation of the nano-ZrO_2-SDS adsorbent

One gram of nano-ZrO_2 was suspended in 25 mL of water containing 0.5 g of SDS in a beaker and placed under ultrasonic action to reduce the aggregation of nanoparticles for 30 min. The obtained suspension was centrifuged, washed with water, and placed in a desiccator to dry. This adsorbent was stored in a closed bottle for subsequent use.

2.4. General removal procedure

The adsorption of brilliant cresyl blue from aqueous solution was performed by batch method. 0.15 g of nano-ZrO_2-SDS adsorbent was added to 50 mL solution containing 10 mg L^{-1} of brilliant cresyl blue and 2.5 mL of acetate buffer pH 4.0. This solution was placed under ultrasonic action to reduce the aggregation of nanoparticles for 30 min. The suspension was centrifuged and the concentration of the remaining dye was determined by measuring the absorbance of the supernatant solution at 632 nm and using a

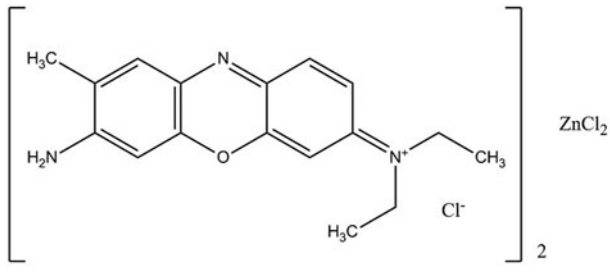

Fig. 1. The structure of brilliant cresyl blue.

calibration graph constructed for brilliant cresyl blue with the equation of $A = 0.0936C + 0.0140$ ($r = 0.9992$). The percentage of dye removal was calculated using the Eq. (1):

$$\text{Removal}\,\% = (1 - C_d/C_o) \times 100 \quad (1)$$

In which C_o and C_d are the initial and after removal concentration of brilliant cresyl blue, respectively. All experiments were carried out at room temperature (25 ± 1°C).

3. Results and discussions

Our preliminary investigation showed that nano-ZrO_2 adsorbs brilliant cresyl blue very slightly (about 0.5%). However, when ZrO_2 nanoparticles are modified by SDS the surface becomes negatively charged and its tendency toward adsorption of brilliant cresyl blue (cationic dye) is increased. Fig. 2 shows the fabrication and adsorption of brilliant cresyl blue by modified ZrO_2 nanoparticles. The TEM images of both unmodified and modified nano-ZrO_2 shown in Fig. 3 indicate that the morphology of the particles has not been changed and only the average particle size has been slightly improved by surface modification. Thus, modified ZrO_2 nanoparticles by SDS were chosen for adsorption of brilliant cresyl blue. In order to achieve highest adsorption capacity, the effect of different variables on the adsorption process was investigated.

3.1. Effect of initial pH

The pH of the loading solution plays an important role in the adsorption of the dye by the adsorbent.

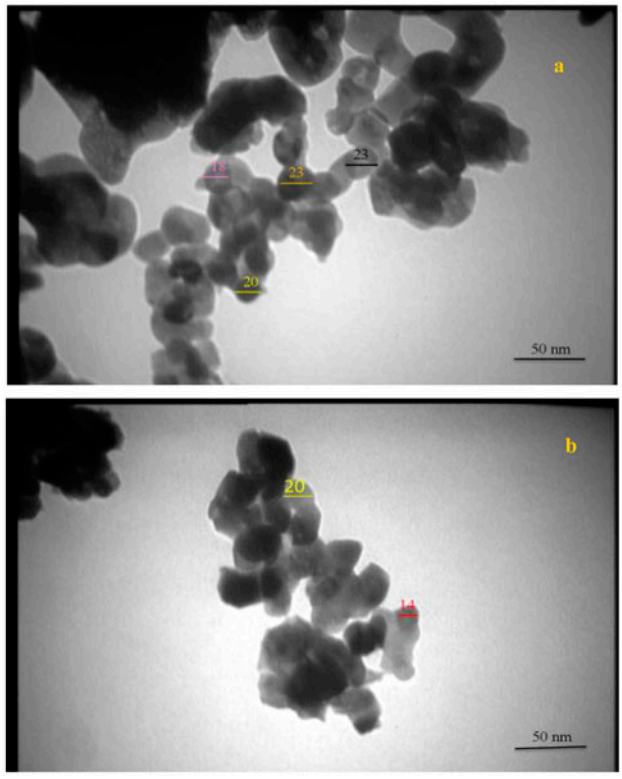

Fig. 3. TEM image (a) unmodified and (b) modified zirconia nanoparticles by SDS.

This is presumably due to the influence of the pH on both the surface properties of the adsorbent and ionization/dissociation of the dye molecules. Thus, the influence of pH of the dye solution in the range of 2.0–8.0 on the removal of 10 mg L^{-1} solution of brilliant cresyl blue was investigated. The results are shown in Fig. 4. As it is observed, highest dye

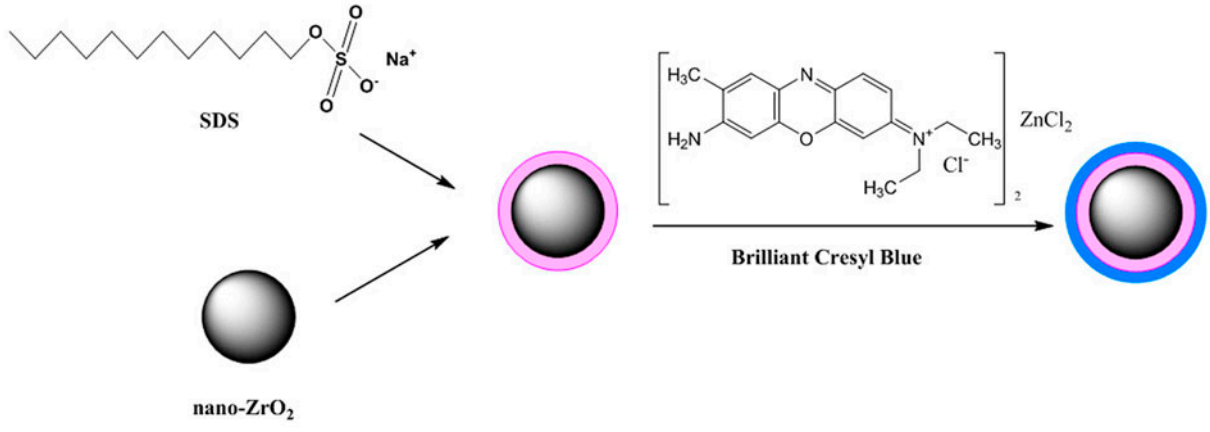

Fig. 2. Fabrication and adsorption of brilliant cresyl blue by nano-ZrO_2-SDS.

removal was obtained when pH of the solution was 4.0. At lower pH values, the surface of the adsorbent becomes more positive and the adsorption of the dye is decreased. At higher pH value the dye is neutralized and becomes less positive. Therefore, pH 4.0 was selected for further work and 2.5 mL of acetate buffer pH 4.0 was added to the solutions to maintain this pH.

3.2. Effect of surfactant amounts

As it was mentioned earlier, SDS was used for surface modification of nano-ZrO_2 in order to enhance the adsorptive tendency of the oxide toward brilliant cresyl blue as a cationic dye. Thus, various adsorbents were prepared using different amount of SDS. The results of this investigation showed that the highest removal percentage is obtained when 1.0 g of nano-ZrO_2 was modified by 0.5 g of SDS in 25 mL solution.

3.3. Effect of adsorbent dose

The dependence of the dye adsorption on the dose of the modified nanoparticles at room temperature and at pH 4.0 was studied. In this regard, similar removal experiments were undertaken using various amounts of the adsorbent (0.03–0.17 g) in contact with 25 mL of 10 mg L^{-1} solution of brilliant cresyl blue. The percentage of the dye removal increased as the adsorbent dose was increased over the range of 0.03–0.15 g and it was constant at higher amounts of the adsorbent (Fig. 5). Therefore, the adsorbent dose of 0.15 g was chosen for subsequent experiments.

3.4. Effect of contact time under ultrasonic action

Adsorption is a time-dependent process and the main objective is to study the effects of contact time in order to better comprehend the adsorption kinetics of the dye on the nano-ZrO_2-SDS. The relationship between brilliant cresyl blue removal and contact time under ultrasonic action was studied at various initial brilliant cresyl blue concentrations. The results are presented in Fig. 6. It was found that more than 90% of the dye removal occurs within the first 10 min at all initial concentration which indicates that there are enough adsorption sites for the dye to be accommodated in a short time. It was observed that the equilibrium time is reached above 20 min; hence the contact time of 30 min was selected. It was also found that by applying ultrasonic action, the removal procedure is more complete and the removal percentage was higher. Applying ultrasonic action keeps the nanoparticles more dispersed and therefore more sites are available for adsorption process. The equilibrium time is much faster than some of the previously reported removal procedures for this dye and other dyes [17,18].

3.5. Adsorption isotherms

Adsorption isotherms show the relation between the amount adsorbed and the equilibrium concentration of the analyte and also give important information about the mechanism of adsorption, helping to design new adsorbing systems [27]. In this study, the adsorption data were fitted to Langmuir, Freundlich, and Temkin equations for the calculation of various adsorption parameters.

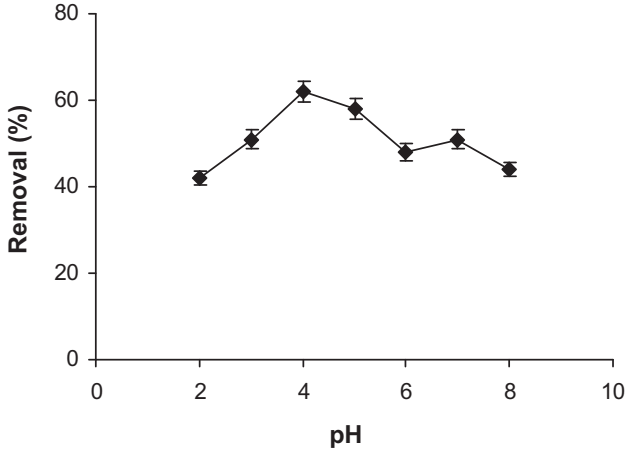

Fig. 4. Effect of pH on the removal of mg L^{-1} of brilliant cresyl blue by nano-ZrO_2-SDS. Conditions: adsorbent dose, 0.15 g; contact time, 10 min.

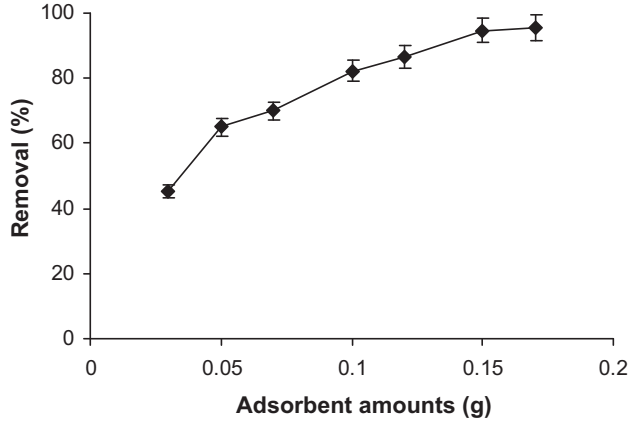

Fig. 5. Effect of adsorbent dose on the removal of 10 mg L^{-1} of brilliant cresyl blue by nano-ZrO_2-SDS. Conditions: 2.5 mL of acetate buffer pH 4.0; contact time, 10 min.

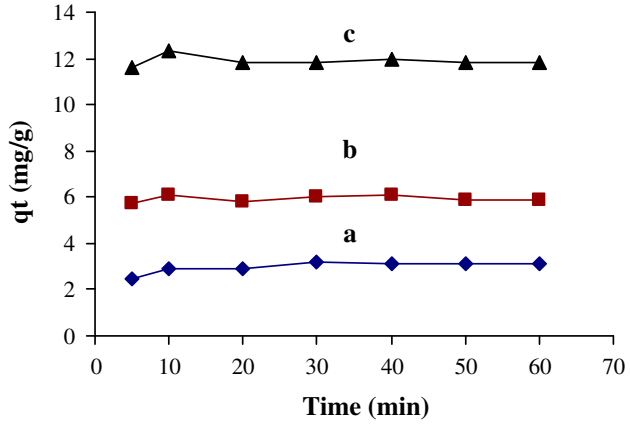

Fig. 6. Effect of time on the adsorption of different concentration of brilliant cresyl blue on nano-ZrO_2-SDS (a) 10 mg L^{-1}, (b) 20 mg L^{-1} and (c) 40 mg L^{-1}. Conditions: adsorbent dose 0.15 g; 2.5 mL of acetate buffer pH 4.0.

3.5.1. Langmuir isotherm

The adsorption phenomena at the solid–liquid interface are commonly demonstrated by Langmuir isotherm which is described by the Eq. (2):

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (2)$$

where q_e is the amount of dye adsorbed per unit weight of the adsorbent (mg g^{-1}) at equilibrium, C_e the final concentration in the solution (mg L^{-1}), q_m the maximum adsorption in monolayered adsorption systems (mg g^{-1}), and K_L is the adsorption equilibrium constant related to adsorption energy (L mg^{-1}). A plot of C_e/q_e vs. C_e shows a linear relationship, and Langmuir constants q_m and K_L can be calculated from the slope and the intercept of the plot. The values of Langmuir model were used to construct a curve C_e/q_e vs. C_e (Fig. 7(a)). The values for q_m and K_L were determined from this plot and are given in Table 1.

The Langmuir isotherm can also be expressed in terms of a dimensionless constant called equilibrium parameter, R_L which is defined as the Eq. (3):

$$R_L = \frac{1}{1 + K_L C_0} \quad (3)$$

where C_0 is the initial metal concentration (mg mL^{-1}). The value of R_L indicates the type of isotherm to be favorable ($0 < R_L < 1$), unfavorable ($R_L > 1$), linear ($R_L = 1$), or irreversible ($R_L = 0$). R_L values calculated from the results obtained above are in the range of 0.0695–0.6418 for the adsorption of brilliant cresyl blue onto nano-ZrO_2-SDS which indicates that the adsorption process is considered favorable.

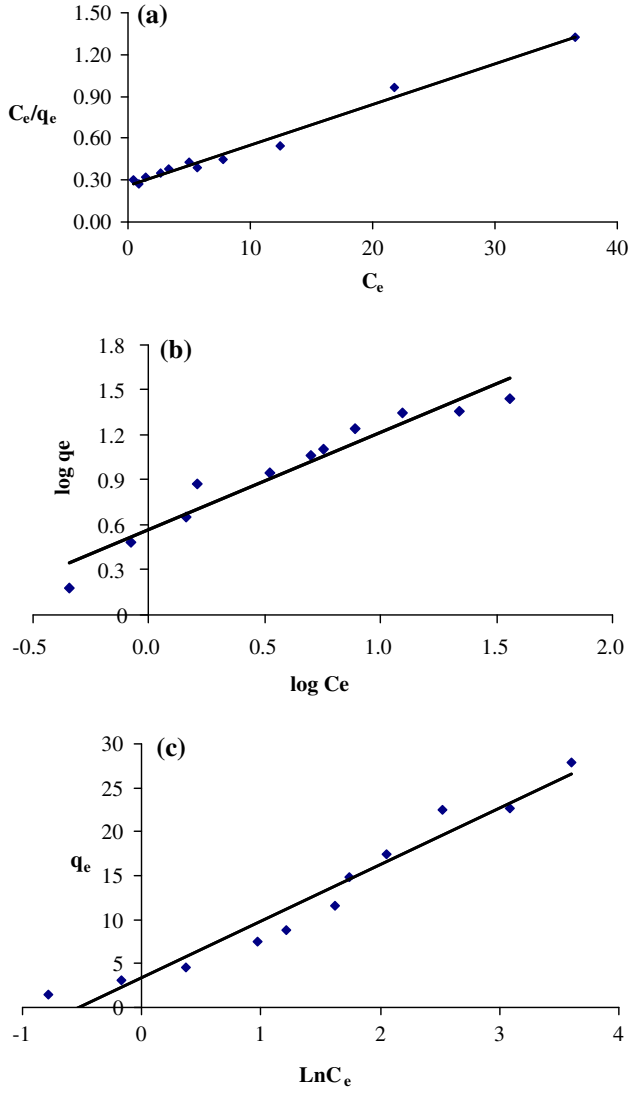

Fig. 7. Adsorption isotherms (a) Langmuir, (b) Freundlich, and (c) Temkin for brilliant cresyl blue. Conditions: adsorbent dose 0.15 g; 2.5 mL of acetate buffer pH 4.0.

3.5.2. Freundlich isotherm

The Freundlich isotherm expresses adsorption at multilayer and on energetically heterogeneous surfaces. It is an empirical equation suitable for high and middle range of solute concentration, but not for low concentrations [27]. Freundlich model in linear form is given as the Eq. (4):

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

Table 1
Comparison of the Langmuir, Freundlich, and Temkin isotherm adsorption constants for brilliant cresyl blue dye removal from aqueous solution by nano-ZrO_2-SDS

Isotherm model	Parameter	Value
Langmuir	q_m (mg/g)	34.4
	K_L (L/g)	0.1116
	R^2	0.9859
Freundlich	K_F (mg/g)	3.7136
	n	1.5396
	R^2	0.9341
Temkin	B_T (J mol^{-1})	6.423
	K_T (L g^{-1})	1.718
	R^2	0.9515

where q_e designates the amount of brilliant cresyl blue adsorbed at equilibrium in mg g^{-1}, C_e is the solute equilibrium concentration in mg L^{-1}, K_F is a constant related to the adsorption capacity, and $1/n$ is an empirical parameter related to the adsorption intensity, which varies with the heterogeneity of material. The values of Freundlich model were used to construct the graph of log q_e vs. log C_e (Fig. 7(b)) and K_F and n were determined from this plot and are given in Table 1.

3.5.3. Temkin isotherm

The Temkin isotherm describes the adsorption behavior on heterogeneous surfaces and is expressed by a linear Eq. (5):

$$q_e = B_T \ln K_T + B_T \ln C_e \quad (5)$$

where $B_T = RT/b_T$, T is the absolute temperature (K), and R is the gas constant (8.314 J mol^{-1} K^{-1}, K_T is the equilibrium binding constant (L mg^{-1}) and B_T is related to the heat of adsorption. A curve of q_e vs. ln C_e (Fig. 7(c)) was constructed and the results are given in Table 1.

By comparing the correlation coefficient for three models Langmuir (R^2 = 0.9859), Freundlich (R^2 = 0.9341), and Temkin (R^2 = 0.9515), it is concluded that the data shows a better fit with the Langmuir model. The Langmuir data given in Table 1 shows that the adsorption capacity was 34.4 mg g^{-1} of the nano-ZrO_2-SDS.

3.6. Adsorption kinetics

The kinetic models were used to predict the variation of adsorbed brilliant cresyl blue with time using nano-ZrO_2-SDS. The rate constants of chemical adsorption were determined using the equations of the pseudo-first-order and pseudo-second-order models. Pseudo-first-order model is most widely used procedures for the adsorption of a solute from aqueous solution [27]. The Pseudo-first-order kinetic model of Lagergren is given by the Eq. (6):

$$\log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303} \quad (6)$$

where q_e and q_t are the amounts of brilliant cresyl blue adsorbed onto nano-ZrO_2-SDS (mg g^{-1}) at equilibrium and at time t, respectively, and K_1 (1/min) is first-order rate constant for adsorption. The rate constant, k_1, can be calculated from the plots of log($q_e - q_t$) vs. t.

Pseudo-second-order kinetics may be expressed as the Eq. (7):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (7)$$

where k_2 is the rate constant of the second-order adsorption (g mg^{-1} min). The straight-line plots of t/q_t against t have been tested to obtain rate parameters.

The pseudo-first-order and pseudo-second-order kinetic models for brilliant cresyl blue removal by nano-ZrO_2-SDS were investigated using above equations. The results indicate that the adsorption of brilliant cresyl blue on nano-ZrO_2-SDS is not fitted to a first-order model and correlation coefficients of about 0.1 were obtained. However, the correlation coefficients for the second-order kinetic model were higher

Table 2
Comparison of different adsorbents used for the removal of brilliant cresyl blue from aqueous solution with nano-ZrO_2-SDS

Adsorbent	Adsorption capacity (mg g^{-1})	Contact time	Ref.
Dimethyl terephatalate residue	13.0	48 (h)	[1]
Natural clay	42.0	8 (h)	[9]
Peanut hull	–	12 (h)	[17]
Magnetic multiwalled carbon nanotube	20.5	6 (h)	[18]
Nano-ZrO_2-SDS	34.4	30 (min)	This work

than 0.999 which shows the suitability of this kinetic model for the adsorption of brilliant cresyl blue by nano-ZrO_2-SDS.

3.7. Application

The removal procedure by nano-ZrO_2-SDS was successfully applied to the removal of brilliant cresyl blue spiked to different water samples. The results showed that under the optimized conditions, 92.0–94.2% of the dye can be removed from the aqueous solution.

4. Conclusions

In the present study, modified nano-ZrO_2 by SDS was investigated as a new adsorbent for the removal of brilliant cresyl blue dye from aqueous solution at room temperature. To the best of our knowledge, this is the first application of modified zirconia nanoparticles by SDS for the dye removal. The conditions of maximum adsorption of the dye such as pH, adsorbent dose, SDS concentration, and contact time were optimized. The equilibrium adsorption was best described by the Langmuir isotherm model. The experimental data fitted to Langmuir isotherm showed that the adsorption capacity is 34.4 mg of brilliant cresyl blue per gram of adsorbent which is comparable or better than some of the previously reported adsorbents for the removal of brilliant cresyl blue (Table 2). This system can be used for the successful removal of brilliant cresyl blue from water samples. Moreover, the kinetic data suggest that the pseudo-second-order model well fitted the experimental results for the removal of brilliant cresyl blue.

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