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Reactive red 198 dye removal from textile wastewater by SBA-15 modified with cationic surfactant

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ABSTRACT

Highly sensitive, accurate and relatively fast technique has been utilized to remove reactive red 198 (R.R. 198) dye in textile wastewater, using synthesised nanocomposites. Determination of R.R. 198 dye was carried out by UV-Vis spectrophotometer at λmax= 515 nm. A SBA-15 mesoporous silica was synthesized then was functionalised using dodecyl trimethyl ammonium chloride (DOTAC) cationic surfactant. TEM, elemental analysis CHNS, TGA, FT-IR, and BET techniques were used to characterize the nanocomposites features. This research studies the impact of various parameters in removal of R.R. 198 dye by nanocomposite, like pH, quantity of nanocomposite, contact time, type of eluting solvent, and concentration of R.R. 198 dye was investigated. Experiments show that the new nanocomposite (DOTAC/SBA-15) is an excellent and powerful nanocomposite for removing the R.R. 198 dye from textile wastewater such as, Mazandaran textile factory, Qaemshahr sangtab textile factory, Amol babakan textile factory and Behshahr Acryltab factory with suitable fallouts.

1. Introduction

Generally, scientists has encountered the challenge of contamination of natural waters caused by dyes and some noxious elements currently [1]. These harmful and venomous materials cause several serious problems in animals, human, and plants. Hence, industrial factories and chemical waste water has been emphasized in this regard [2, 3]. For example, they become unsuitable for public consumption owing to the discharged from textile dyeing mills in the natural waters [4]. There is a huge deal of highly concentrated dyes in waste water of dyeing units of a textile plant [5]. Dyes have high usage in different industries like dyestuffs, textiles, cosmetics, and paper [2, 3]. Azoic dyes as complex aromatic structures have higher resistance to biodegradation [6]. The reactive red 198 as a water-soluble azo dye is difficult to remove from wastewater. Several approaches are used to remove dyes and other color pollutants such as chemical coagulation, aerobic/anaerobic biological degradation, membrane filtration, flocculation, chemical oxidation, and photochemical degradation. Such approaches are expensive and occasionally can not remove dyes from waste water totally [7–10]. Adsorption has been recently popular as a result of its low running costs and easy operations [11,12]. Comparing the stated method with other removal approaches, the main factors included in the higher efficiency of the adsorption method were economic justification, cost-effectiveness, and simplicity of operation. An adsorbent is involved in the adsorption method, which must be at least high effective and include proper functional groups. Thus, pollutants are removed by researchers through various adsorbents with these two features. Several adsorbents have been developed to purify wastewater including SBA-15 [13-18], CdO nanoparticles [19], multi-walled carbon nanotube [20-22], fullerene [23, 24], SDS-coated alumina [25], glass foam [26], cellulose nanofibers [27-30], copper nanowire/Carbon paste electrode [31], MgO nanoparticles [32], nickel oxide nanostructures [33], Fe3O4 magnetic nanocomposite [34-37] and graphene oxide [38, 39]. Presently, a huge deal of attention has been attracted toward mesoporous materials because of their great

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practical potentials and scientific importance in catalysis, adsorbents, host materials, and sensors [40-43]. Moreover, compared to the other silica materials, the maximum hydrothermal stability is displayed by SBA-15 [44,45] that is suitable for aqueous media applications. SBA-15 functionalized with various function groups are highly utilized as adsorbent because of their higher selectivity for ions adsorption [46]. Carboxylic acid [47], iminoacetate [48], sulfonic acid [49], amino-carboxyl [50], ionic liquid [51], dispersive liquid-liquid [52], dithizone [53], and thio and amine [54] were immobilized onto various substrates for removal of dyes. In this work, a colloidal solution was prepared for the first time, based on dodecyl trimethyl ammonium chloride (DOTAC) cationic surfactant functionalized SBA-15 nanoparticles to remove reactive red 198 dye from wastewater samples via spectrophotometer. The nanoparticles structure was characterized through various methods like transmission electron microscope (TEM), Elemental analysis CHN, Emmett, Brunauer, and Teller (BET), FT-IR, and thermogravimetric analysis (TGA). The effect of various variables was then investigated such as amount of adsorbent, pH, and removal time, on the removal of reactive red 198 dye.

2. Materials and Methods

2-1. Reagents and Instruments

All chemicals were in analytical grade reagents. The solutions were made utilizing double distilled water. The anionic dye (Reactive red 198) was bought from Dystar (Fig. 1), and utilized with no further purification. A stock reactive red solution (1000 mg L–1) was made and diluted suitably to the considered initial concentration. The dye represents a strong absorption peak at 515 nm in the visible region. This wavelength is equivalent to the highest absorption peak of the R.R. 198 (λmax=515). DOTAC cationic surfactant and Plaronic P-123 bought from Sigma Aldrich. To measure the pH, a Metrohm pH meter (model 744) was used with a mixed pH glass electrode calibrated against two standard buffer solutions (4.0 and 7.0). Other instruments included Trans-mission electron microscope (TEM) with specification (HitachiHigh-Technologies Europe GmbH, HF2000, Germany, 100 kV volt-age), Brunauer-Emmett-Teller technique (BET) (Quanta chrome, Chem BET 300 TPR/TPD), analysis CHNS (Costech ECS4010), thermogravimetric analysis (TGA) (Bahr thermo analyse), Fourier transform infrared (FT-IR) (Agilent Resolutions pro), and UV-Vis spectrophotometer (Jenway model 6505).

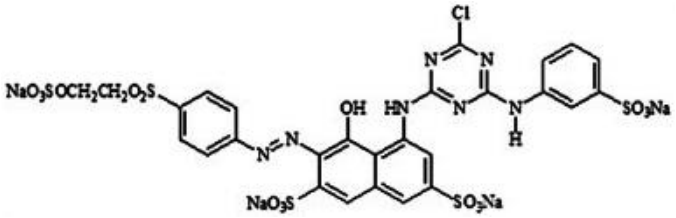


Fig. 1. The reactive red 198 chemical structure

2-2. Synthesis of SBA-15 mesoporous silica

SBA-15 was synthesized via a known process using a combination of triblock copolymer, Pluronic P123, HCl, TEOS, H2O, and KCl. Approximately P123 was dissolved in a combination of distilled water and HCl by adding KCl. Then, TEOS was inserted into the solution. For 24 h, the mixture was statically. The solid was recovered by filtration and rinsed with water. Then, by refluxing the surfactant was extracted via a Soxhlet extractor. The gained SBA-15 was dried at 100°C overnight[39].

2-3. Modification of SBA-15 nanoparticles

2.0 g of SBA-15 was inserted to NaHCO3 0.16 M (25 mL) and in into a beaker (100 mL). Then the solution was stirred for 30 min via a shaker to mix the SBA-15 with NaHCO3. The solution was centrifuged for 10 min at 5000 rpm. The SBA-15 was washed using distilled water two times by upper solution overflow at the bottom. The mixture was placed into centrifuge tube adding DOTAC cationic surfactant (1.0 g) to 50 mL acetone in a container, while stirring for 180 min via a shaker. It was centrifuged for 20 min. The centrifuging tube was placed in an oven at 60 ˚C based on tube overflow upper solution for drying and making powder (Fig. 2).

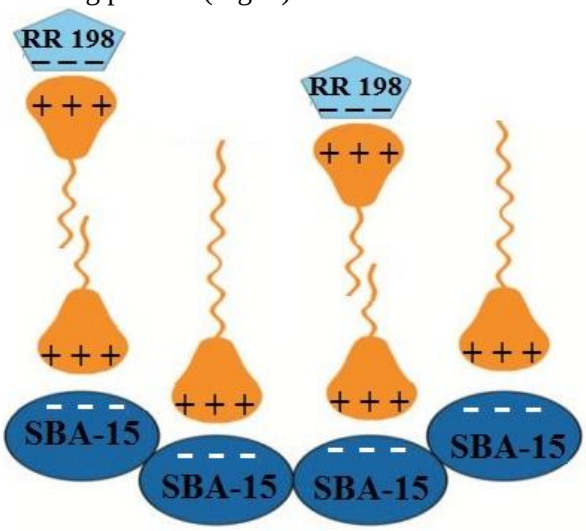


Fig. 2. A suggested binding mode of DOTAC to SBA-15 nanoparticles surface.

2-4. Procedure for removal of R.R. 198 dye

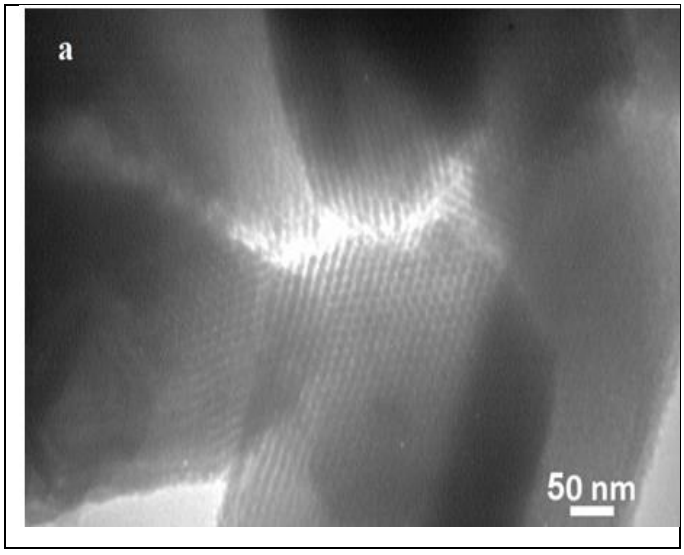
Firstly, 100 mL R.R. 198 solution (200 mg L-1) was created in pH=5 modified with CH3COOH/ CH3COONa buffer solution. Next, we inserted 150 mg nanocomposites to the solution (100 mL), and mixed for 20 min at ambient temperature. Ultimately, for phase separation, the product was centrifuged (at 5000 rpm for 5 min). Overflowing the upper solution, R.R. 198 dye adsorbed on the precipitation was diffused via the sample elution with 10.0 mL acetone. After mixing acetone, for 5 min the product was stirred and centrifuged again (at 5000 rpm for 10 min). Finally, spectrophotometry technique was utilized to determine R.R. 198 concentration. To optimize the best circumstances for R.R. 198 pre-concentration, various parameters were investigated such as type of eluting solvent, the nanocomposite dosage, the shaking time, and pH, so that a satisfactory removal can be achieved. In all optimization processes, R.R. 198 concentration were set as 200 mg L-1.

3. Results and Discussion

3.1. Characterizing the SBA-15 and nanocomposite

The synthesis of the SBA-15 and DOTAC functionalized SBA-15 were characterized by TEM, TGA, CHNS and BET.

Based on the TEM image (Fig. 3a), the SBA-15 pore size is < 20 nm with uniformly hexagonal structure. Fig. 3b shows the structure of SBA-15/DOTAC structure. It can be concluded from Fig. 3b that DOTAC coveredthe surface of SBA-15.



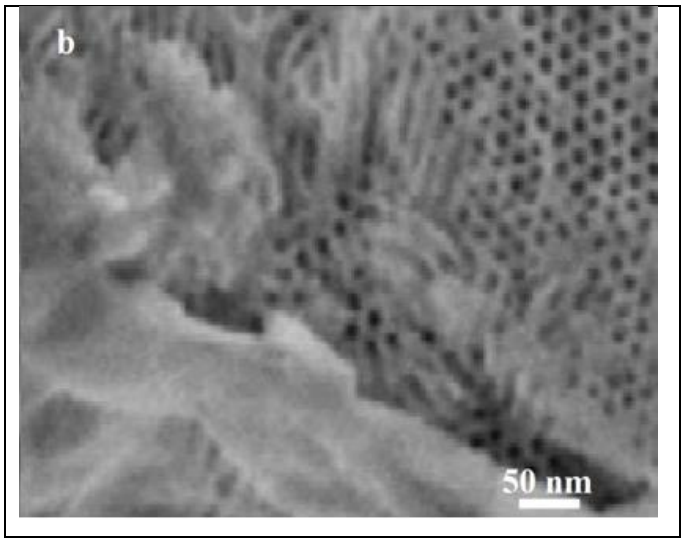


Fig. 3. (a)TEM image of the SBA-15; (b) DOTAC/SBA-15 (b)

The nanocomposites was thermogravimetrically studied to display the nanocomposite stability and the existence of organic groups in the material (Fig. 4). The weight loss 11.7% at about 120 ˚C in the SBA-15 (Fig. 4.a) and the mass loss (19.21%) within 245-340 ˚C in the nanocomposites (Fig. 4.b) are caused by the existing water and DOTAC cationic surfactant, respectively. At T > 400 ˚C, a small and continuous weight loss is found associated with the residual organic material combustion.

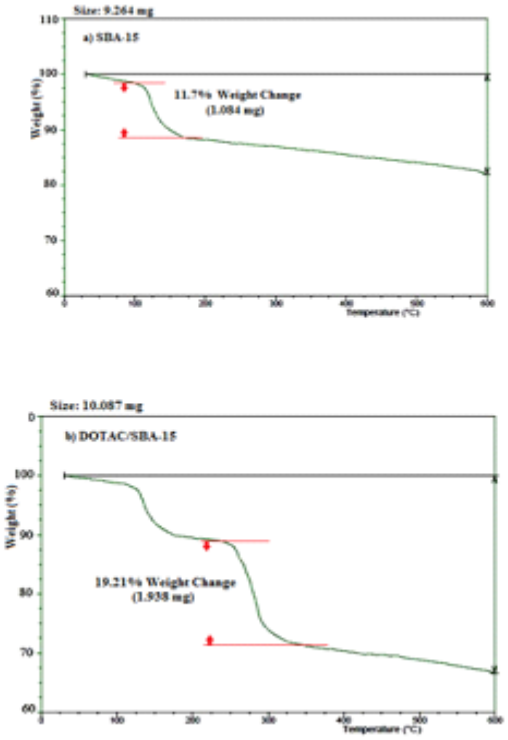


Fig. 4. TGA analysis curve for SBA-15 (a) and DOTAC/SBA-15 (b)

Through elemental analysis CHNS, the elements types were determined in synthesized nanocomposite and determination of the elements ratio in the sample. Table 1 shows the practical values of nanocomposite elements. As seen, the existence of carbon and nitrogen in CHNS analysis of nanocomposite proves the existence of DOTAC on the SBA-15 surface.

Table 1. The results of multi-elemental CHNS analysis on DOTAC/SBA-15

Reten. time (min)	Response	Weight (g)	Weight (%)	Element name	Carbon response ratio
2.319	2864.3	0.051	14.21	Carbon	1.000
1.395	210.6	0.005	1.39	Nitrogen	0.074
7.864	547.6	0.010	2.79	Hydrogen	1.191
16.375	0.04	0.000	0.00	Sulfur	0.000
Sample weight: 0.359 (g)		0.066	18.39		

Measurement of the specific surface area in SBA-15 and SBA-15/DOTAC was performed by the use of the nitrogen adsorption and desorption isotherms along with the BJH method, as observed in Table 2. The hysteresis loop became wider in SBA-15/DOTAC following the loading of DOTAC, and the curved isotherm section was transferred to lower pressure, the hysteresis loop end of which was close to P/Po = 0.4, showing the placement of DOTAC inside SBA-15 cavities (Fig. 5). Moreover, a reduction in the specific surface area of SBA-15/DOTAC cavities was indicative of the proper placement of DOTAC particles inside the SBA-15 cavities and channels.

Furthermore, the curved isotherm section was transferred to lower P/Po in SBA-15/DOTAC nanocomposite following surface modification and placement of DOTAC on the SBA-15 surface. This indicated the placement of DOTAC within the SBA-15 channels.

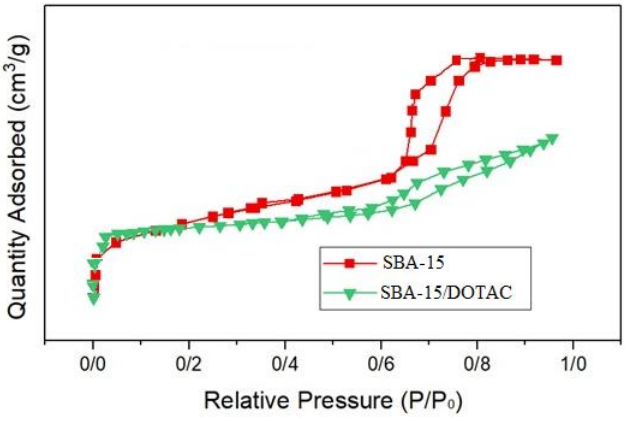


Fig. 5. N adsorption and desorption for SBA-15 and SBA-15/DOTAC samples.

The BET analysis on SBA-15 proved the SBA-15's specific surface area as 273.46 m2/g, which was decreased to 251.28 m2/g after surface modification. This surface dropping is predicted owing to the surface coverage by DOTAC cationic surfactant. Indeed, these two wide surfaces present good areas for R.R. 198 dye absorbance.

Table 2. BET analysis results from DOTAC/SBA-15

Surface area before coating (m2/g)	273.46
Surface area after coating (m2/g)	251.28
Adsorption average pore width (nm)	9.07

FT-IR was used to confirm the presence of DOTAC surfactant on SBA-15 (Figure 6). The broad absorption band at 3431 cm-1 is related to the stretching vibration of the OH groups on the surface of SBA-15. Absorption band at 2923 cm-1 is assigned to the stretching vibration of CH bond. The absorption band at 1450 cm-1 is assigned to corresponds to the vibration of the C-H bond of the CH3 group. The absorption band at 1088 cm-1 corresponds to the Si-O bond of SBA-15 and it covers the absorption band related to the C-N bond of the DOTAC surfactant, which gives a spectrum in the range of 1200 cm-1.

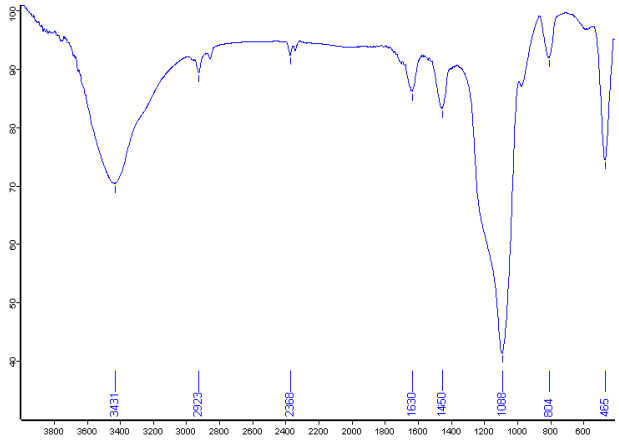


Fig. 6. FT-IR spectrum of SBA-15/DOTAC nanocomposite

3-1. Optimization of the removal stage

The effect of all the parameters affecting the removal performance should be assessed including the shaking time, type of eluting solvent, the nanocomposite dosage, pH and concentration of R.R. 198 to obtain a higher removal.

3-2. The effect of pH

Generally, pH is important in the adsorption process in surface adsorption load, and various adsorbent pollutants in solution, separation of different functional groups over active adsorbent sites, and the R.R. 198 dye molecule

structure. In the present work, the pH effect on the adsorption procedure of R.R. 198 dye was evaluated in the range of 3–11 by addition of NaOH or HCl for adsorbent particles (DOTAC/SBA-15 composite). As seen in Fig. 7, the pH of 5.0 was chosen for the removal of R.R. 198 dye. At the alkaline pH values, the removal percentage is low, because hydroxide ions compete with reactive red 198 dye to adsorption with the DOTAC cationic surfactant.

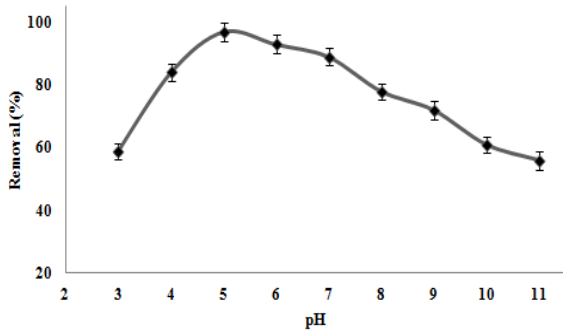


Fig. 7. The effects of pH on the removal. Removal conditions: Amount of sorbent, 150 mg; recovery solvent, 10.0 mL acetone; contact time, 20 min; R.R. 198 concentration, 200 mg L^{-1}.

3-3. Impact of the nanocomposite dosage

The effect of different quantities of sorbent (modified SBA-15) on the removal of R.R. 198 dye were assessed within 50–200 mg range. It is indicated (Fig. 8) that R.R. 198 dye was adsorbed completely on the sorbent in the 150 mg of the modified SBA-15. Thus, 150 mg was chosen as the sorbent essential for all subsequent tests.

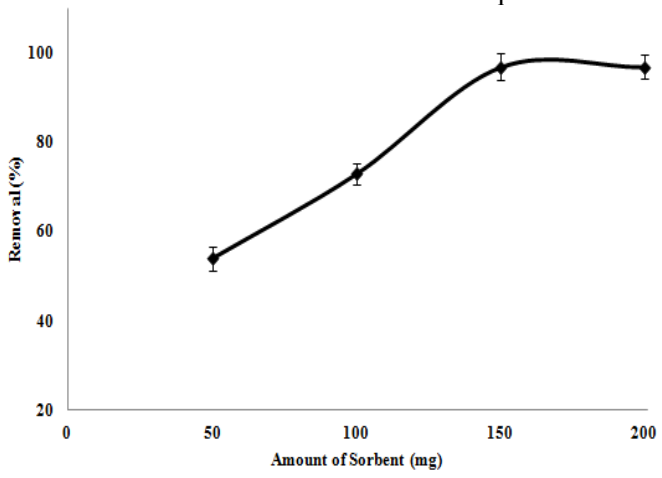


Fig. 8. Effects of nanosorbent amount on the removal. Removal circumstances: pH value, 5.0; recovery solvent, 10.0 mL acetone; contact time, 20 min; R.R. 198 concentration, 200 mg L^{-1}.

3-4. Impact of contact time

The removal of the R.R. 198 dye is based on the sample contact time with the solid phase. Therefore, repeated sets of adsorbents and dye were made to assess at different time intervals (5, 10, 15, 20, 25 and 30 min). It was revealed that the rate of dye uptake was higher (Fig. 9). At about 20 min, the of dye adsorption from the solution was over 96%.

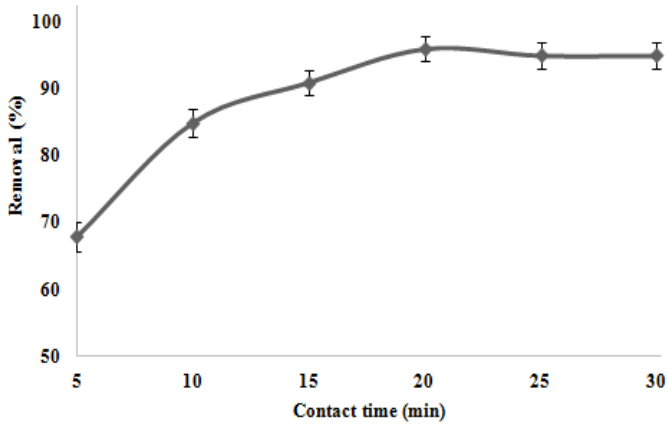


Fig. 9. The kinetics of dye adsorption on modified SBA-15. Removal conditions: Amount of sorbent, 150 mg; pH value, 5.0; recovery solvent, 10.0 mL acetone; R.R. 198 concentration, 200 mg L^{-1}.

3-5. The effect of type of eluting solvent

Different tests were conducted to achieve optimum conditions for total elution of R.R. 198 dye on DOTAC/SBA-15. We exerted 10.0 mL of different solvent types as the optimum eluent for future experiments. According to Fig. 10, acetone provides the most optimum quantitative elution of R.R. 198 dye mixed with DOTAC/SBA-15.

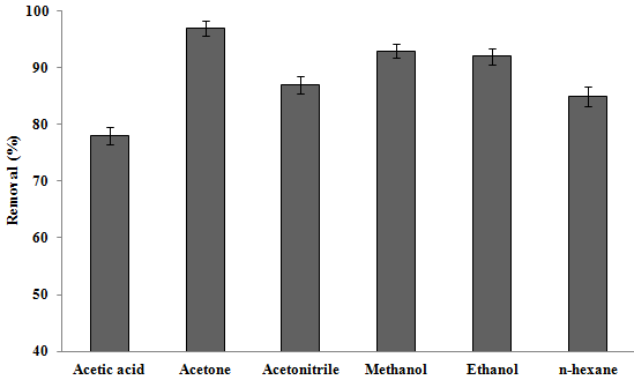


Fig. 10. The effect of type of eluent on the removal of R.R. 198 dye. Removal conditions: Amount of sorbent, 150 mg; pH value, 5.0; contact time, 20 min; R.R. 198 concentration, 200 mg L^{-1}.

3-6. Effect of R.R. 198 concentration

To investigate the possibility of our presented sorbent with respect to various R.R. 198 concentrations, the

effects of various concentrations of this dye were investigated within the range of 50 to 300 mg L^{-1}. A higher adsorption value was found at the lower concentration of R.R. 198. However, by increasing the dye dosage to 250 mg L^{-1}, the sorbent activity reduced. As seen in Fig. 11, the most suitable concentration value for R.R. 198 was 200 mg L^{-1} in terms of the removal percent as well as the loading for the sorbent is 0.135 mmol g^{-1}.

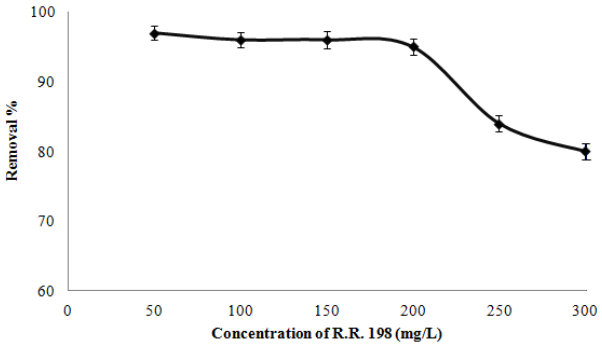


Fig. 11. The impacts of dye concentration on the removal. Removal conditions: Amount of sorbent, 150 mg; pH value, 5.0; contact time, 20 min; recovery solvent, 10.0 mL acetone.

3-7. Nanocomposite reusability

An adsorbent reusability is as a key factor in analytical approaches. As seen in Fig. 12, the reusability results for DOTAC/SBA-15, the nanocomposite can be reused for three times by rising via recovery solvent. Moreover, after three times, the adsorption level reduced to 92.5% from 96.8%. The removal reduced owing to release of the surfactant grafts, irreversible/strong interactions between surfactant and R.R. 198 dye, and nanocomposite particles loss during elution.

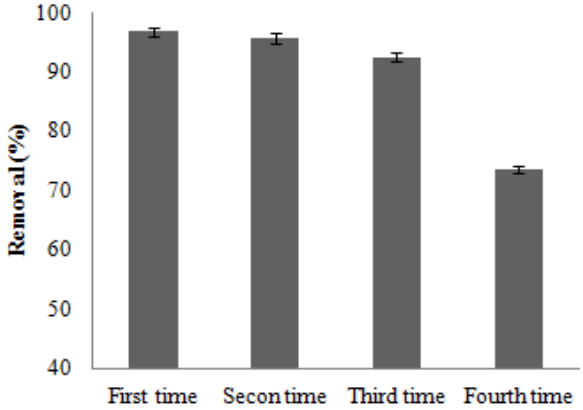


Fig. 12. The nanocomposite reusability. Removal conditions: Amount of sorbent, 150 mg; pH value, 5.0; contact time, 20 min; recovery solvent, 10.0 mL acetone; R.R. 198 concentration, 200 mg L^{-1}.

3-8. Removing of R.R. 198 dye from textile wastewater samples

First, some textile wastewater samples (100 mL) were prepared (Table 3), and filtered via a membrane filter with a pore size of 0.45-mm prior to any analysis for removal of any suspended particles. Furthermore, after filtration, the real concentration of R.R. 198 in textile wastewater specimens was determined by UV-Vis spectrophotometer. The higher removal percent is obtained (Table 3), for each real sample, indicating the successful use of the proposed method in removal of R.R. 198 dye in textile wastewater samples.

Table 3. Removal of R.R. 198 dye from textile wastewater samples

wastewater Sample	Physical-chemical information of sample	concentration of R.R. 198 (mg L^{-1})	Removal (%)
Mazandaran textile factory (Qaemshahr)	pH = 6.9, TSS = 127, TDS = 814, BOD = 240, COD = 360	21.21 ± 0.19	96.4 ± 1.9
Sangtab textile factory (Qaemshahr)	pH = 7.8, TSS = 136, TDS = 940, BOD = 260, COD = 390	17.63 ± 0.15	95.3 ± 2.1
Babakan textile factory (Amol)	pH = 7.3, TSS = 125, TDS = 850, BOD = 220, COD = 330	20.15 ± 0.17	97.9 ± 1.8
Acryltab factory (Behshahr)	pH = 6.6, TSS = 143, TDS = 920, BOD = 250, COD = 310	14.24 ± 0.11	95.8 ± 1.7

4. Conclusion

A novel operative nanosorbent was synthesized by functionalization of the SBA-15. According to the experiments, the new modified SBA-15 with DOTAC cationic surfactant is a strong sorbent for removing the R.R. 198 dye from textile wastewater. This method indicates that removal more than 95% R.R. 198 dye is possible at optimal conditions of pH = 5, R.R. 198 concentration = 200 mg L-1, and the sorbent dosage=150 mg. It was indicated that, DOTAC/SBA-15 is a superior absorbent for moderate concentrations of R.R. 198. An eco-friendly non-toxic absorbent is used in the proposed method not threatening the human health. Moreover, it is highly sensitive, simple, and relatively quick.

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