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Yun Xing[a]; Dehua Deng[a]
[a] College of Chemistry and Chemical Engineering, Anyang Normal University, Anyang, China

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Enhanced Adsorption of Malachite Green by EDTAD-modified Sugarcane Bagasse

Yun Xing and Dehua Deng
College of Chemistry and Chemical Engineering, Anyang Normal
University, Anyang, China

Abstract: Ethylenediaminetetraacetic dianhydride (EDTAD) modified sugarcane bagasse (SB) was prepared and characterized by Fourier transform infrared spectroscopy (FTIR). Due to the presence of a large number of carboxyl groups, the adsorption capacity of the EDTAD modified SB (EDTAD-SB) for malachite green (MG) showed a significant increase compared with SB. Increase in ion strength of solution-induced decline of MG sorption. The maximum adsorption capacities were $157.2\,\mathrm{mg\,g^{-1}}$ for MG, according to the Langmuir equation. Kinetic studies showed better correlation coefficients for a pseudo-second-order kinetic model, confirming that the sorption rate was controlled by a chemisorption process.

Keywords: adsorption, Ethylenediaminetetraacetic dianhydride, malachite green, sugarcane bagasse

INTRODUCTION

The removal of dye from wastewater is currently one of the major problems faced by the textile dyeing industry. The most widely used methods for removing dyes from wastewater systems include physicochemical, chemical, and biological methods, such as flocculation, coagulation, precipitation, membrane filtration, electrochemical techniques, ozonation, and fungal decolorization. However, all these processes are costly and cannot be used by small industries to treat the wide range

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Address correspondence to Yun Xing, College of Chemistry and Chemical Engineering, Anyang Normal University, Anyang 455002, China. Tel.: +86 03722900040. E-mail: xy7627@yahoo.cn

of dye wastewater. The adsorption process provides an attractive alternative treatment, especially if the adsorbent is inexpensive and readily available. Adsorption techniques employing solid sorbents are widely used to remove certain classes of chemical pollutants from waters, especially those that are not easily biodegradable. Granular activated carbon is the most popular adsorbent that has been used with great success. However, adsorbent-grade activated carbon is cost-prohibitive and both the regeneration and the disposal of the used carbon are often very difficult. Consequently, many investigators have studied the feasibility of using low-cost substances. Safarikova et al. had used magnetically modified *Chlorella vulgaris* cells to the adsorption of six dyes (aniline blue, Bismarck brown, congo red, crystal violet, safranin O and Saturn blue LBRR) (1). Du et al. had used chitosan nanoparticles for the adsorption of eosin Y (2). Lata et al. had studied the adsorption of rhodamine-B from aqueous solution on formaldehyde treated parthenium biomass (3). Nandi et al. had reported the studies on the adsorption of crystal violet dye using kaolin as an adsorbent (4). Ardejani et al. had reported the adsorption of direct Red 80 dye from aqueous solution onto almond shells (5). Shi et al. had done the batch study of methylene blue removal from aqueous solutions by adsorption on NaOH-treated firry sawdust (6).

China is the world's leading producer of sugarcane for both the alcohol and the sugar industries. These industries produce a large amount of sugarcane bagasse and although it is burned to produce energy for sugar mills, leftovers are still significant. Thus, on account of the importance of bagasse sugar as an industrial waste, there is a great interest in developing chemical methods for recycling it. Sugarcane bagasse (SB) has around 50% cellulose, 27% polyoses, and 23% lignin. These three biological polymers have many hydroxyl and/or phenolic functions that can be chemically reacted to produce materials with new properties (7). Conversion of SB to the adsorbent has the potential to address these issues and it represents an opportunity to add value to this by-product.

Thus the objective of this study was to establish the potential of SB as a precursor for preparing adsorbent for the removal of the dyes. In this paper, SB was modified by EDTAD to increase the adsorption capacity for dyes. Fourier transform infrared (FTIR) and Scanning electron microscopy (SEM) were used to characterize the modified SB. Cationic dye of MG was selected as adsorbate in this study. The adsorption performances, including adsorption kinetics and equilibrium, were investigated in detail. To the best of our knowledge, it is the first time this method is used in the field of the MG's adsorption.

METHODS

Materials

SB was collected from the market of Wuhan, Hubei Province (China). SB was washed in flowing tap water, sun-dried, and ground to pass a 45-mesh sieve. The residual sucrose in ground SB was removed by soaking and rinsing in 10 volume of boiling water twice for 30 min, the residue was dried in an oven after filtration by 200 nylon cloth, and reacted in 6 volume of ethanol (w/v) by reversed flow at 80°C for 3 h to remove sugarcane wax and other ethanol extracts, residue air-dried. Cationic dye MG was purchased from Sinopharm Group Chemical Reagent Co. Ltd. (Shanghai, China) and used without further purification. The chemical structure of MG was shown in Fig. 1. The stock solution was prepared by dissolving accurately weighted dye in distilled water to the concentration of 1000 mg L^{-1}. The experimental solutions were obtained by diluting the dye stock solutions in accurate proportions to different initial concentrations. All other chemicals were of analytical grade and used without further purification.

Synthesis of EDTAD-Modified Sugarcane Bagasse (EDTAD-SB)

0.5 g of EDTAD was added to a 40 mL of N,N-dimethylacetamide (DMAc) containing 0.5 g of SB in a three neck round bottom flask equipped with a condenser. The mixture was stirred at 60°C for 4 h. After the reaction, the product was thoroughly rinsed successively with DMAc, NaOH, and distilled water to remove unreacted EDTAD. Finally, the modified SB was freeze-dried and stored in a desiccator before use.

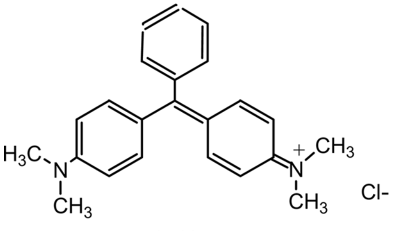


Figure 1. The chemical structure of MG.

Characterization of the Adsorbents

FTIR and SEM were used in the surface analysis of SB and EDTAD-SB. The surface morphology of the dried microspheres before and after modification were examined with a scanning electron microscopy (SEM, X-650, Hitachi, Japan): a fragment of the dried microspheres was mounted on the sample mount and sputter-coated with gold. The surface of the sample was scanned at the desired magnification. The dried microspheres were thoroughly mixed with KBr and pressed into a pellet. The IR spectra were obtained using a Fourier Transform IR spectrometer (Nicolet NEXUS-470).

Batch Adsorption Experiments

Adsorption of MG from aqueous solution was tested in batch experiments. Effects of the concentrations of the dyes, the pH of the medium, and the contact time on the adsorption and capacity were studied and the sorption kinetic was also evaluated.

The experimental procedure was as follows: 10 mg EDTAD-SB samples were put into conical flasks, into which 25 mL aqueous solutions of MG were added separately and vibrated on a rotary shaker operating at 150 rpm. When the adsorption reached equilibrium, the conical flasks were taken out and centrifuged to separate the EDTAD-SB and the solution. The concentrations of the free dyes in the filtrate were analyzed with a UV-visible spectrometer.

The amount adsorbed was calculated based on the difference of the dye concentrations and the weight of EDTAD-SB by the following equation:

$$q_e = \frac{(C_0 - C_e)V}{W} \tag{1}$$

where q_e is the amount of dyes adsorbed onto unit amount of the sorbents ($mg\,g^{-1}$), C_0 is the initial dye concentration($mg\,L^{-1}$), C_e is the equilibrium dye concentration($mg\,L^{-1}$), V is the volume of dye solution(L), and W is the dry weight of the adsorbents (g).

The adsorption temperature was at 25°C. The initial dye concentration was 100 mg L^{-1} for MG. For adsorption experiments, the concentration ranges used were 10–100 mg L^{-1} for MG.

RESULTS AND DISCUSSION

Preparation and Characterization of the Adsorbent

Figure 2 is the SEM micrographs of SB. It shows a fibrous structure that has large pore size with strands in each fiber. The process of the modification onto SB is very simple. There are many OH on the surface of SB. The OH group on the surface of SB reacts with EDTAD as shown in Scheme 1.

Infrared spectroscopy is frequently used to investigate the structure of constituents and the chemical changes in lignocellulosic materials. FTIR spectra of SB and EDTAD-SB are shown in Fig. 3. The absorption bands and corresponding assignments are listed in Table 1. The spectrum of EDTAD-SB displayed two strong bands at 1740 and 1726 cm-1 in relation to that of SB. This demonstrated the presence of two types of carbonyl functions, one relative to carboxylic acid and another relative to the ester. The acid and ester IR bands indicate that EDTAD acylated the hydroxy group of SB to generate an ester bond with consequent release of a carboxylic acid functional group (8).

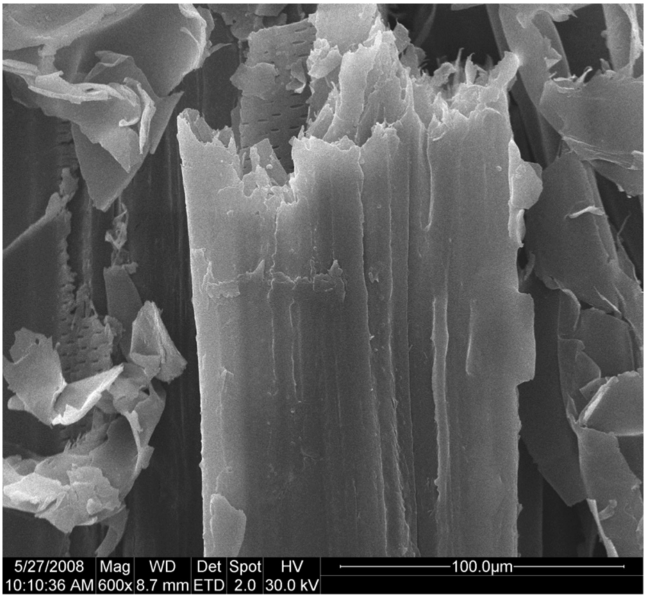
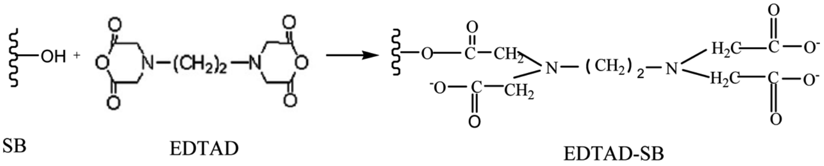


Figure 2. SEM micrographs of SB.



Scheme 1. Schematic procedure of the modification on SB surface.

Adsorption Isotherm Experiments

Studies on the adsorption isotherm are a prerequisite to understand the adsorbate–adsorbent interaction and to optimize the use of the adsorbent. Figure 4 shows the adsorption isotherms of MG at 25°C on SB and EDTAD-SB. It was observed that the adsorption capacity increased with the increase in equilibrium concentration and ultimately attained a saturated value for MG.

The adsorption curves were fitted to both the Langmuir and Freundlich equations. The Langmuir isotherm model assumes monolayer adsorption on a surface with a finite number of identical sites, that all sites are energetically equivalent, and there is no interaction between the adsorbed molecules. The Freundlich expression is an empirical equation for adsorption on a heterogeneous surface with a non-uniform distribution of heat of adsorption over the surface and multilayer sorption can

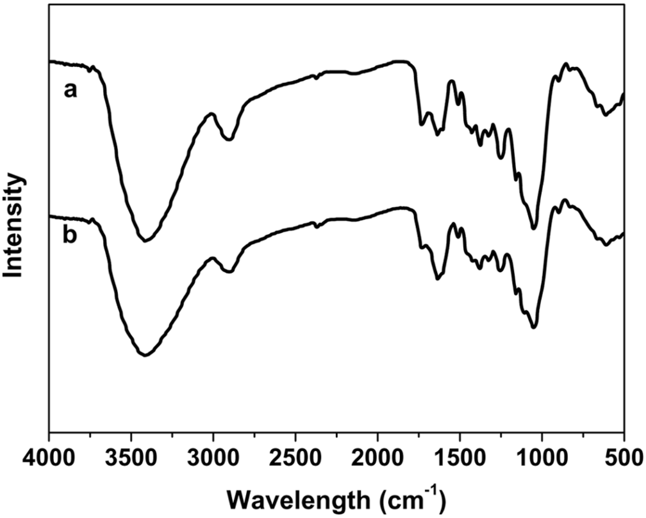


Figure 3. FTIR spectra of SB and PMAA-SB. (a) SB, (b) EDTAD-SB.

Table 1. FTIR absorption bands of SB and EDTAD-SB

Wavenumber/cm^{-1}		
SB	EDTAD-SB	Possible assignment
3415	3417	O–H stretching vibration
2902	2901	C–H stretching vibration
–	1726	C=O stretching vibration of ester
1732	–	C=O stretching vibration of hemicellulose
–	1740	C=O stretching vibration of carboxylic acid
1635	1633	C–O stretch vibration
1512	1512	Aromatic skeletal ring vibration due to lignin
1427	1428	CH_2 symmetric bending vibration
1377	1378	C–H stretching vibration
1051	1051	C–OH stretching vibration

occur. The Langmuir and Freundlich isotherms are expressed as Eqs. (2) and (3), respectively:

$$q_e = \frac{QbC_e}{1 + bC_e} \quad \text{or} \quad \frac{1}{q_e} = \frac{1}{Q} + \frac{1}{QbC_e} \tag{2}$$

$$q_e = aCe^{1/n} \quad \text{or} \quad \log q_e = \log \mathrm{a} + \frac{1}{\mathrm{n}} \log C_e \tag{3}$$

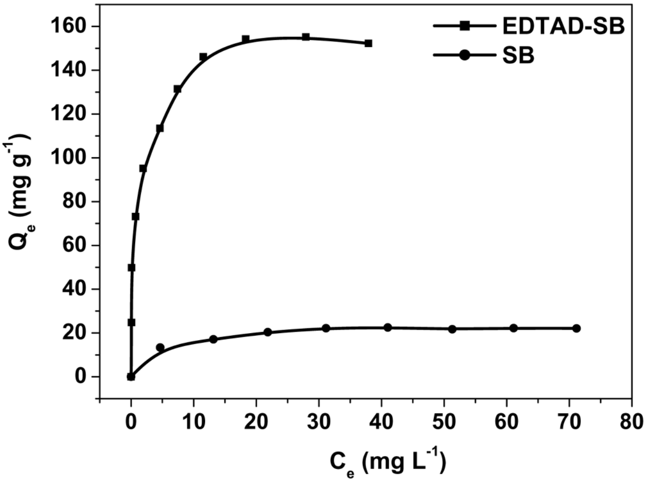


Figure 4. Adsorption isotherms of MG on SB and EDTAD-SB.

where Q is the maximum amount of adsorption ($\mathrm{mg\,g^{-1}}$), q_e is the adsorption capacity at equilibrium ($\mathrm{mg\,g^{-1}}$), b is the adsorption equilibrium constant ($\mathrm{L\,mg^{-1}}$), C_e is the equilibrium concentration of substrates in the solution ($\mathrm{mg\,L^{-1}}$), a is the Freundlich constant related to adsorption capacity of adsorbent ($\mathrm{mg^{1-1/n}\,g^{-1}\,L^{1/n}}$), and n is the Freundlich exponent related to adsorption intensity. The Langmuir and Freundlich adsorption constants evaluated from the isotherms with the correlation coefficients were listed in Table 2. It showed that the Langmuir isotherm gave better fits than the Freundlich isotherm, so it illustrated that the adsorption on the surface of EDTAD-SB was a monolayer adsorption. According to the Langmuir equation, the maximum uptake capacities (q_m) for MG were $157.2\,\mathrm{mg\,g^{-1}}$.

The Langmuir parameters can also be used to predict affinity between the sorbate and sorbent using the dimensionless separation factor R_L, which has been defined by Hall et al. (9) as

$$R_L = \frac{1}{1 + bC_0} \tag{4}$$

where R_L is the dimensionless separation factor, C_0 is the initial concentration (mg/l) and b is the Langmuir constant (l/mg). The value of R_L can be used to predict whether a sorption system is "favorable" or "unfavorable" in accordance with the criteria shown in Table 3. The values of R_L for sorption of MG on SB and EDTAD-SB are shown in Fig. 5. The R_L values indicated that sorption was more favorable for EDTAD-SB than SB.

Many literatures have reported the use of biosorbents to remove dyes from aqueous solution. Table 4 shows the comparison between MG removal by EDTAD-SB and other adsorbents found in the literatures. Compared with these results, EDTAD-SB in this work had a higher adsorption capacity for MG.

Table 2. Adsorption isotherms and corresponding parameters for MG binding by SB and EDTAD-SB (SD, standard deviation)

	Langmuir model				Freundlich model			
Adsorbents	Q ($\mathrm{mg\ g^{-1}}$)	b ($\mathrm{L\ mg^{-1}}$)	R	SD	a ($\mathrm{mg^{1-1/n}\ g^{-1}\,L^{1/n}}$)	n	R	SD
SB	23.41	0.3021	0.9989	0.05158	10.53	5.231	0.9442	0.02861
EDTAD-SB	157.2	1.132	0.9991	0.00366	72.33	3.911	0.9502	0.08707

Table 3. Characteristics of adsorption Langmuir isotherm

Separation factor, R_L	Type of isotherms
$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$0 < R_L < 1$	Favorable
$R_L = 0$	Irreversible

Adsorption Thermodynamics

Equilibrium adsorption constant (K) was obtained following a method used by Khan and Singh (18). First, the adsorption isotherm data were plotted as $\ln(q_e/C_e)$ versus q_e and extrapolated to zero q_e. Then, a linear regression was performed on the experimental data based on least-squares analyses, and the intercept on the y-axis gives the value of ln K. Based on the $\ln K$ values, the standard free energy changes (ΔG°) for the reaction were then calculated from the relationship

$$\Delta G^\circ = -RT\ InK \tag{5}$$

where R is the universal gas constant ($8.314\,\mathrm{J\,mol^{-1}\,K^{-1}}$) and T is the temperature in Kelvin. The Gibbs-free energies of adsorption for MG

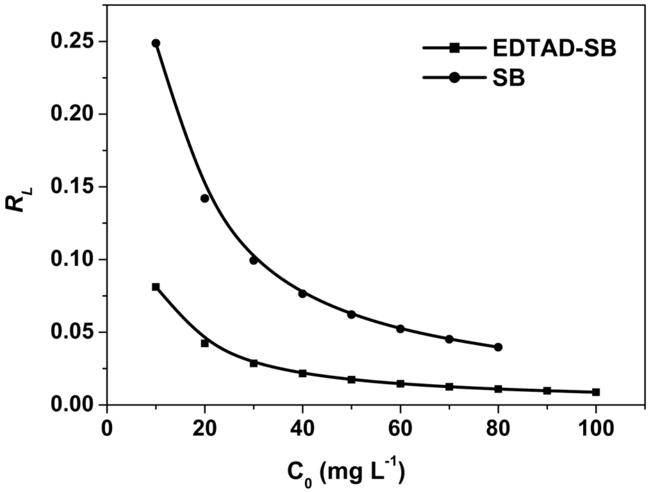


Figure 5. The values of separation factor R_L for sorption of MG on SB and EDTAD-SB.

Table 4. Comparison of MB removal between EDTAD-SB and others found in literatures

Adsorbents	Qm (mg/g)	Literatures
Activated carbon prepared from bamboo	263.58	[10]
Esterified soybean hull	178.57	[11]
EDTAD-SB	157.2	This work
Oil palm trunk fibre	149.35	[12]
Rice straw-derived char	148.74	[13]
Chitosan bead	93.55	[14]
Cyclodextrin-based adsorbent	91.9	[15]
Lemon peel	43.46	[16]
Bentonite	7.716	[17]

were calculated to be $-17.94\,\mathrm{kJ\,mol^{-1}}$. The negative value of ΔG° indicated that the adsorption of MG on EDTAD-SB was spontaneous under the experimental conditions.

Adsorption Kinetics Experiments

The sorption kinetic of MG is illustrated in Fig. 6. The removal rates were very rapid during the initial stages of the sorption process. After a very rapid sorption, the uptakes increased with time and reached

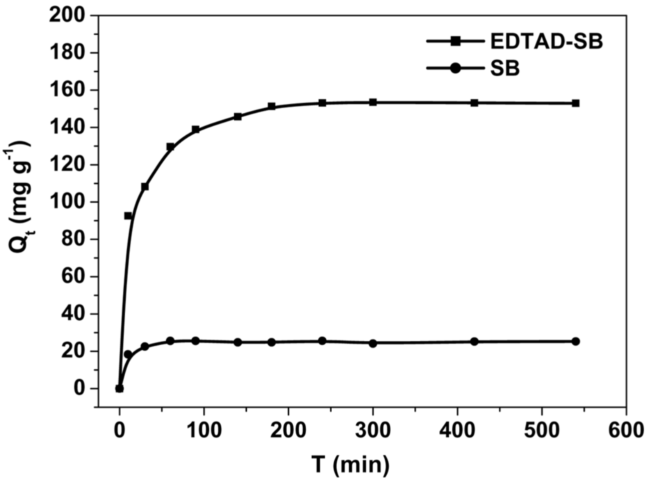


Figure 6. The sorption kinetic of MG on SB and EDTAD-SB.

equilibrium values at approximately 3 h for MG. The three phases of the dye sorptions could be attributed to boundary layer sorption, intraparticle diffusion, and sorption equilibrium respectively. Fig. 6 shows that the dye adsorption capacities of MG on EDTAD-SB was much higher than those of SB.

In order to obtain further insight into the mechanism of the adsorption of MG on EDTAD-SB, a pseudo-second-order mechanism was investigated. The pseudo-second-order equation was given by Ho (19):

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{6}$$

where k_2 (g mg^{-1} min^{-1}) is the constant of pseudo-second-order rate, q_e (mg g^{-1}) is the adsorption capacity at equilibrium, and q_t (mg g^{-1}) is the adsorption capacity at time t (min). Separating the variables in Eq. (6) and integrating gives:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} = \frac{1}{v_o} + \frac{t}{q_e} \tag{7}$$

where v_0 represents the initial adsorption rate (mg g^{-1} min^{-1}). The equilibrium adsorption capacity q_e and the pseudo-second-order rate v_0 can be experimentally determined from the slope and the intercept of the plot t/q_t against t. The corresponding parameters and regression coefficients for the model results were given in Table 5.

As shown in Table 5, the good fit ($R > 0.999$) was obtained, and its calculated equilibrium adsorption capacities $\mathrm{q_{e,cal}}$ was consistent with the experimental data. These suggested that the pseudo second-order adsorption mechanism was predominant and that the overall rate of the dye adsorption process appeared to be controlled by the chemisorption process (20,21). Moreover, from the value of v_0, it was obvious that the initial adsorption rate of EDTAD-SB was higher than that of SB. The higher rate had significant practical importance, as it would facilitate smaller reactor volumes ensuring efficiency and economy. On the other hand, the equilibrium adsorption capacity q_e of EDTAD-SB for MG

Table 5. Kinetic parameters of the pseudo-second-order equation for MG adsorption

Adsorbents	v_0 (mg g^{-1} min^{-1})	q_e (mg g^{-1})	R
SB	11.56	25.24	0.9996
EDTAD-SB	16.32	156.7	0.9999

was 156.7 mg g^{-1}. Compared with 25.24 mg g^{-1} of SB, the increases were about 6-fold for MG.

Influence of Initial pH

The effects of the pH of the sample solutions on the adsorption of MG were evaluated by adjusting the pH by HCl or NaOH. The effect of initial pH on the biosorption percentages of dyes was examined over a range of pH values from 2 to 10 for MG. As shown in Fig. 7, the adsorption capacity for MG was minimum at the initial of pH 2. The dyes sorbed increased with the increase in the initial pH from pH 2 to 6, then, the dye adsorption was not significantly altered. For this reason, the pH 6 was selected for future experiments. It was observed that the adsorption capacities for the cationic dye increased significantly after SB had been modified with EDTAD. The solution pH affects the ionic state of functional groups (carboxyl groups) in EDTAD-SB. Carboxyl groups have a pKa value between 2 and 4 (22). At pH values lower than the pKa, carboxylate groups are predominantly positive charged, which would restrict access to adsorption sites by the cationic dye as a result of repulsive forces. At pH values higher than the pKa, more carboxyl groups are negative charged, promoting the uptake of cationic dyes on EDTAD-SB. The solution pH affected the activity of functional groups, which led to the change of the uptake of the cationic dyes.

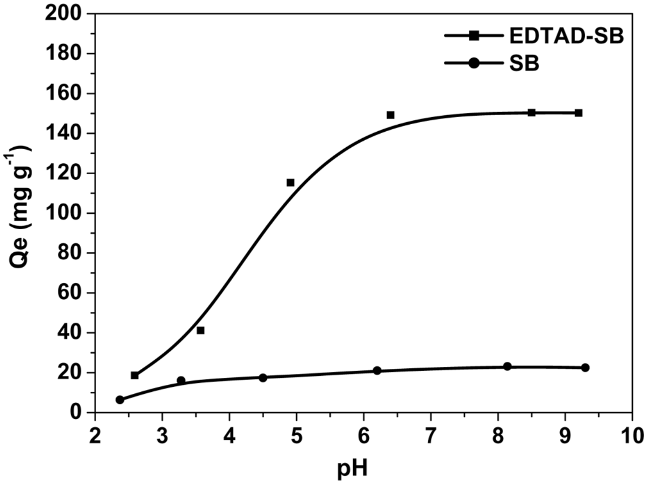


Figure 7. Effect of pH on MG sorption.

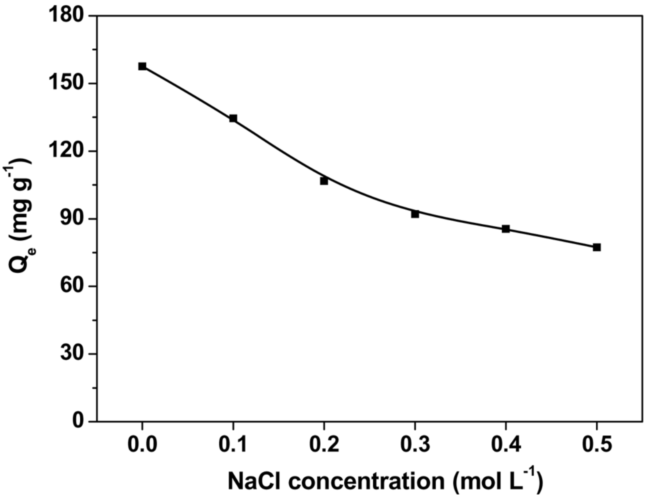


Figure 8. Effect of ion strength on sorption of MG by EDTAD-SB.

Effect of Ion Strength

The effects of ion strength on MG sorbed on EDTAD-SB were tested by the addition of sodium chloride to the dye solutions. The concentration of NaCl used ranged from 0 to 0.5 mol L^{-1}. As seen in Fig. 8, increasing the ion strength of the solution caused a sharp decrease in adsorption capacities of MG. This could be attributed to the competition of MG cation and Na$^+$ ion for the sorption sites.

CONCLUSIONS

From this work it is seen that EDTAD-SB could be an excellent adsorbent for the cationic dye. The adsorption capacity of EDTAD-SB for MG was enhanced significantly in comparison before the modification. The presence of EDTAD on the SB surface was verified by FTIR. The kinetics of adsorption followed the pseudo-second-order equation demonstrating that the overall rate of the adsorption process was controlled by a chemisorption process. The experimental data were well fitted to the Langmuir isotherm model. An increase in ion strength of the solution induced a decline of MG sorption. This study shows that grafting effective groups on SB might result in an adsorbent with high adsorption capacity for water-soluble cationic dyes.

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