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ORIGINAL PAPER

Removal of methylene blue from aqueous solutions by poly(2-acrylamido-2-methylpropane sulfonic acid-*co*-itaconic acid) hydrogels

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Abstract In this study, removal of methylene blue (MB) from aqueous solution by poly(AMPS-*co*-IA) hydrogels was examined by batch equilibration technique. The effects of monomer ratio, concentration of initiator and crosslinker, pH, adsorption time, initial dye concentration and adsorption temperature on the removal of MB were studied. The results show that the removal of MB was highly effected by preparation conditions of hydrogel. The maximum removal was observed at 10/90 IA/AMPS monomer ratio, 1.0% KPS, and 10.0% MBAAm concentrations. Removal of MB was strongly affected by pH. Pseudo-first-order, pseudo-second-order and intraparticle diffusion models were applied. It was concluded that adsorption of MB on hydrogel followed pseudo-second-order kinetics. It was found that the adsorption isotherm of the MB fit Langmuir-type isotherms. From the Langmuir equation, the adsorption capacity was found as 1,000 mg/g for MB dye. Thermodynamic parameters suggest that the adsorption is a typical physical process, spontaneous, and exothermic in nature. Ten adsorption—desorption cycles demonstrated that the hydrogels were suitable for repeated use without considerable change in adsorption capacity. The results revealed that this hydrogels have potential to be used as an adsorbent for the removal of MB from aqueous solution.

Keywords Hydrogel · Methylene blue dye · Removal · Adsorption–desorption · Isotherms and thermodynamics

Introduction

Industrial processes, such as dyeing of textile fibers and printing, use synthetic organic dyes that often cause environmental pollution. Dyes even in low

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concentrations are visually detected and meanwhile affect the aquatic life and food web. The removal of dye from textile wastewaters is a major environmental problem due to the difficulty of treating such waters by conventional methods. Most studies have focused on the development of a technique and a method for the treatment of dye wastewater. Recently, numerous low-cost adsorbents have been proposed for dye removal. Among them, non-conventional activated carbons from solid wastes, industrial by-products, agricultural solid wastes, clays, zeolites, peat, polysaccharides, and fungal or bacterial biomass deserve particular attention as recently summarized in a review by Crini [1]. The amounts of dyes adsorbed on the above adsorbents are not very high, some have capacities between 100 and 600 mg/g and some even lower than 50 mg/g [1]. To improve the efficiency and selectivity of the adsorption processes, it is essential to develop more effective and cheaper adsorbents with higher adsorption capacities. In recent years, polymeric adsorbents, due to their wide variations in porosity and surface chemistry, especially regenerability on site and reuse for continuous process, have been increasingly used to remove and recover organic pollutants from waste streams [2, 3]. Some groups have used various adsorbents for the removal of acidic and basic dyes from aqueous solutions [4–7]. Apart from nonconventional adsorbents, polymeric hydrogels have also showed their ability to remove toxic metal ions from the aqueous solutions [8–10]. In particular, polyacrylamide-based hydrogels have received considerable attention because of their use in many applications (as specific sorbent, etc.) [11]. Poly (2-acrylamido-2-methyl propane-1-sulfonic acid), [poly(AMPS)], is an acidic, highly hydrophilic polymer that behaves as polyelectrolyte and use in separation and enrichment technologies. Itaconic acid (IA) is a water soluble monomer having two carboxylic acid groups. Due to its double functionality, IA polymers offer interesting possibilities as functional polymers for wastewater treatment. The aim of this study was to prepare environment-responsive hydrogels of AMPS for use in dye removal.

In this study to remove the MB dye from aqueous solutions, we prepared a hydrogel by copolymerization of AMPS and IA in aqueous medium, using MBAAm as crosslinker and KPS as initiator. Prepared hydrogels were characterized by FTIR and SEM. The effects of monomer ratio, concentration of initiator and crosslinker, and time, effect of pH, initial dye concentration, and temperature on the removal of MB were investigated in aqueous solution. Moreover desorption and reusability of hydrogel was examined. Adsorption kinetic and some thermodynamic parameters were determined.

Experimental

Material and reagents

Itaconic acid (Aldrich) was used after recrystallization from distilled water, m.p. 166–167 °C. 2-acrylamido-2-methyl-1-propanesulfonic acid (AMPS) (Merck, 99%) was used without further purification. The initiator, potassium persulfate (KPS), the crosslinker, *N*,*N*′-methylenebisacrylamide (MBAAm), and cationic dye, methylene blue (MB), were obtained from Merck and used without further purification.

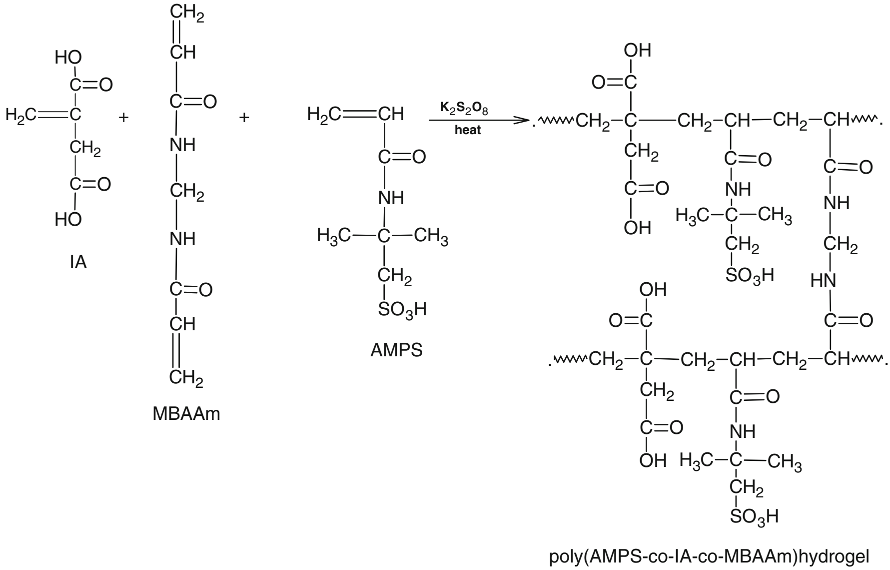
pH values were adjusted with CH_3COOH/CH_3COONa buffer for pH 3–5, Na_2HPO_4/NaH_2PO_4 buffer for pH 7, and NH_3/NH_4Cl buffer for pH 9–11.

Measurements

FT-IR spectra of the hydrogels were recorded with Jasco 460 Plus Fourier Transform IR spectrometer using KBr disc in the range of 4000–400 cm^{-1}. The surface morphology of the hydrogel was examined by a scanning electron microscopy (SEM), LEO 440 imaging mode. The concentration of the dye was measured with a Schimadzu UV-1208 model UV–Vis spectrophotometer. The pH measurements were made with Consort C931 model digital pH-meter.

Synthesis of hydrogels

The poly(2-acrylamido-2-methylpropane sulfonic acid-*co*-itaconic acid) (poly (AMPS-*co*-IA), hydrogels were synthesised by copolymerization of AMPS and IA in aqueous medium, using MBAAm as crosslinker and KPS as initiator (see Scheme 1). In brief, 9.0 mmol of AMPS, 1.0 mmol of IA, and 1.0 mmol of crosslinker were dissolved in distilled water to give a total volume of 5.0 mL. After adding 0.15 mmol of initiator KPS, the resulting solution was placed in poly(vinylchloride) straws of 0.40 cm diameters and about 20 cm long. The poly(vinylchloride) straws were sealed and immersed in a thermostated water bath at 50 °C. Polymerization was conducted for 24 h. After the reaction, the hydrogels were cut into specimens of ~10 mm in length and dried in an oven at 55 °C to constant weight and subjected to Soxhlet extraction with water to remove the



Scheme 1 The structure of poly(AMPS-*co*-IA) hydrogel

uncross-linked polymer from copolymers. The extracted gels were dried again in oven at 55 °C to constant weight.

Adsorption study

Dried samples (0.1 g each) of hydrogels were added in 100 mL Erlenmeyer including volumes of 30 cm^3 of the MB dye solution (25 ppm) adjusted to desired pH. The mixture was stirred through the experiment at 25 °C. After filtration of the solution, the dye concentration of the filtrates was determined at wavelength 665 nm by UV–Vis spectrophotometers. The removal percentage ($R\%$) and the adsorption amount (Q, mg/g) were calculated as follows:

$$R\% = \frac{(C_1 - C_2) \times 100}{C_1} \tag{1}$$

$$Q = \frac{V(C_1 - C_2)}{W} \tag{2}$$

where C_1 and C_2 are the concentrations (ppm) of dye before and after adsorption, W is the weight of the hydrogel (g), and V is the volume of solution.

Reusability of the hydrogel

Methylene blue-loaded hydrogels were stirred with HNO_3 (30 cm^3, 1 M) at 25 °C for 2 h to desorb the MB dye. Thereafter, the hydrogel was washed with deionized water and dried and then again reused adsorption processes. The adsorption/desorption procedure was repeated ten times, each times with fresh solution.

Results and discussion

Synthesis and characterization of the hydrogels

The structure of poly(AMPS-*co*-IA) hydrogels is shown in Scheme 1.

A FT-IR spectrum of the poly(AMPS-*co*-IA) hydrogel is presented in Fig. 1. From the FT-IR spectrum clearly shows a broad band in (3,350–3,550) cm^{-1}, which is due to hydrogen bonded $_{V}$OH from carboxylic group and $_{V}$NH from amide group. The carbonyl stretching band for IA unit lies at 1,725 cm^{-1}. In the poly(AMPS-*co*-IA), AMPS units exhibit several characteristics band. The absorption band at 1,647 cm^{-1} is amide band from AMPS and the 1,222 cm^{-1}, 1,038 cm^{-1} bands of the sulfonic group of AMPS appear in the hydrogel spectrum.

Effects of monomer ratio on removal MB

The effect of IA/AMPS mol ratio on the adsorption capacity was studied by varying the IA/AMPS ratio from 0.5 to 1 ($n_{AMPS} + n_{IA} = 0.01$ mol) as shown in Fig. 2. Maximum removal (99.2%) was obtained at 10/90 IA/AMPS monomer ratio.

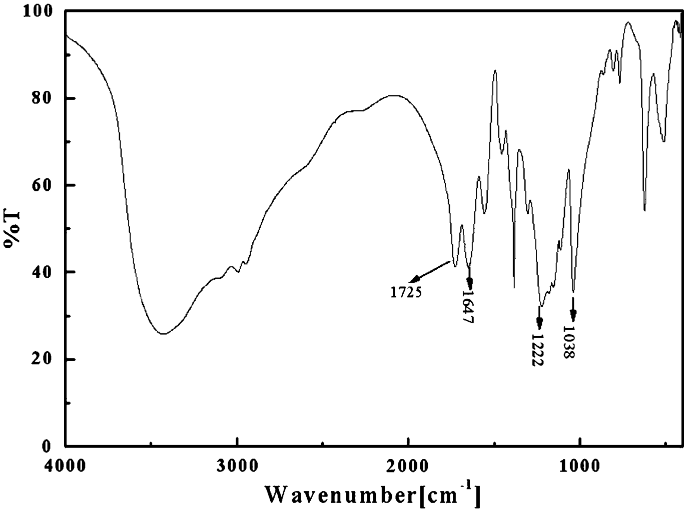
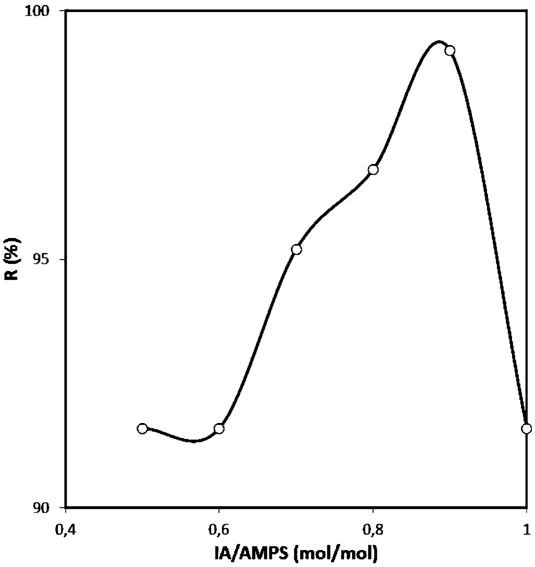


Fig. 1 FTIR spectra of poly(AMPS-*co*-IA) hydrogels

Fig. 2 Effect of monomer ratio on the adsorption amount of MB onto hydrogel [$T = 25$ °C; $t = 24$ h; $C_i = 25$ ppm; 1.0% KPS; 10% MBAAm]



According to this figure, increasing the AMPS concentration at monomer feed composition, increased the adsorption capacity, due to its strongly ionizable sulfonate group [12–15]. The surface morphology of various ratios of poly(AMPS/IA) hydrogels was detected by SEM and shown in Fig. 3. As shown in Fig 3, the copolymer composition affects the hydrogel canal structure. The canal size of the polymeric material mainly depends on copolymer composition. The poly(IA/AMPS) hydrogel of composition (10/90 mol.%) shows more and larger canals and more and

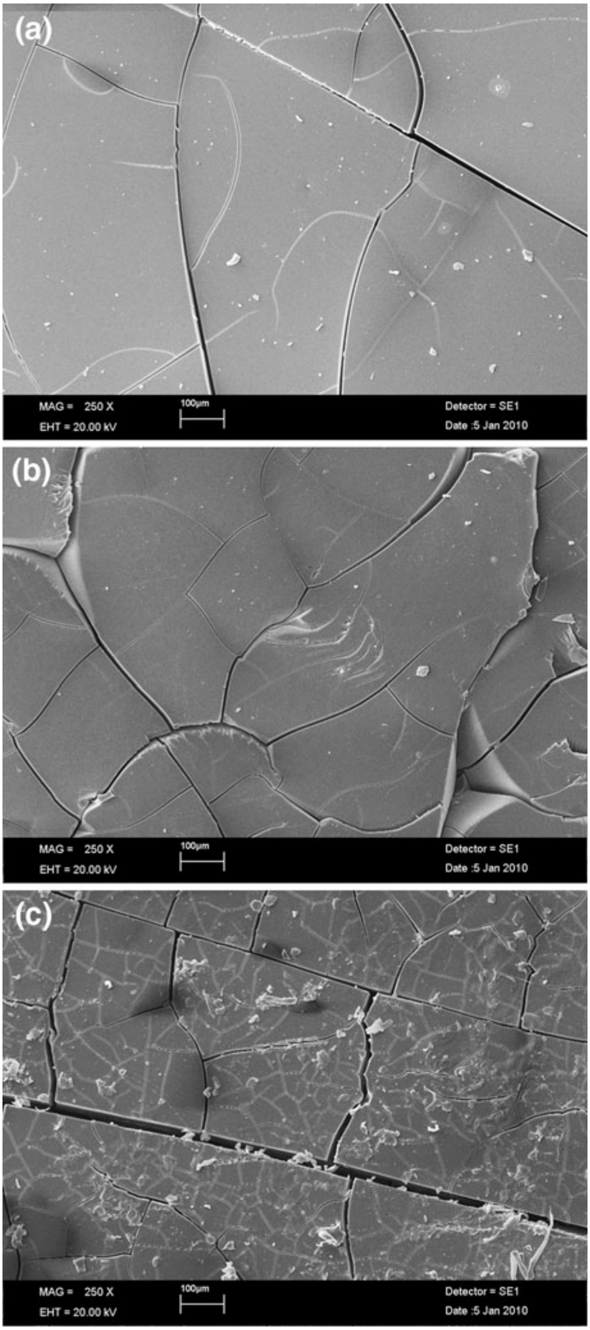


Fig. 3 **SEM** micrographs of poly(AMPS-*co*-IA) hydrogels at a magnification of 250. Monomer ratio: **a** 50/50 IA/AMPS, **b** 30/70 IA/AMPS, and **c** 10/90 IA/AMPS [$n_t = 0.1$ mol; 1.0% KPS; 10% MBAAm; $T = 55$ °C]

larger micro canal structures, which make dye easily diffuse into the hydrogel, compared with the other compositions.

Effects of initiator concentration on removal MB

The effect of variation in the amount of KPS in the feed mixture on the removal of MB was studied by varying the initiator content in the range 0.25–2.50% of the total monomer mole. Measuring the equilibrium dye uptake of the resulting hydrogels in the distilled water was performed at 25 °C. The results are depicted in the Fig. 4. As shown in Fig. 4, the hydrogels synthesised with initiator content 1.0% exhibited maximum dye uptake, while at lower and higher concentrations of KPS, the dye uptake is found to decrease. This may be attributed to the decrease in the number of radicals produced due to decrease in KPS content. In addition, further increase in KPS content above 1.0%, the optimum value, will be decreased the molecular weight in free radical polymerization. So, the network cannot be formed efficiently with small and further number of radicals in the free radical polymerization, thus causing a decrease in the dye uptake [16].

Effects of crosslinker concentration on removal MB

To investigate the effect of crosslinker concentration on removal of MB, the crosslinker concentration was varied in the range 1.0–10.0% of the total monomer mole. Measuring the equilibrium dye uptake of the resulting hydrogels in the distilled water was performed at 25 °C. The experimental results are shown in Fig. 5. As seen from the figure, with increase of MBAAm concentration up to 5.0% the removal percentage rapidly increased but with increase of MBAAm concentration above 5.0% the removal percentage slightly increased. The rapid increase in dye removal can be attributed to increasing network structure, which includes more

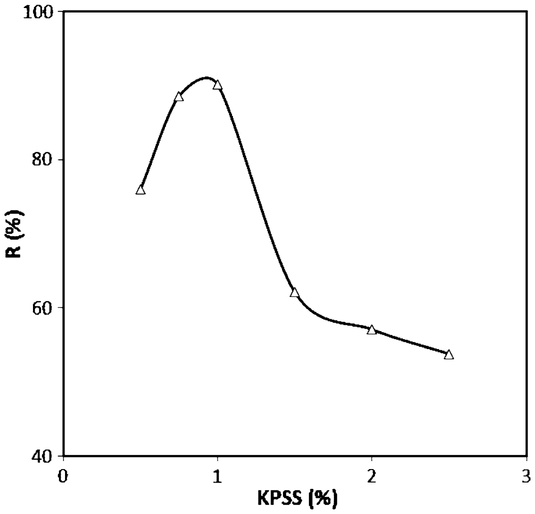
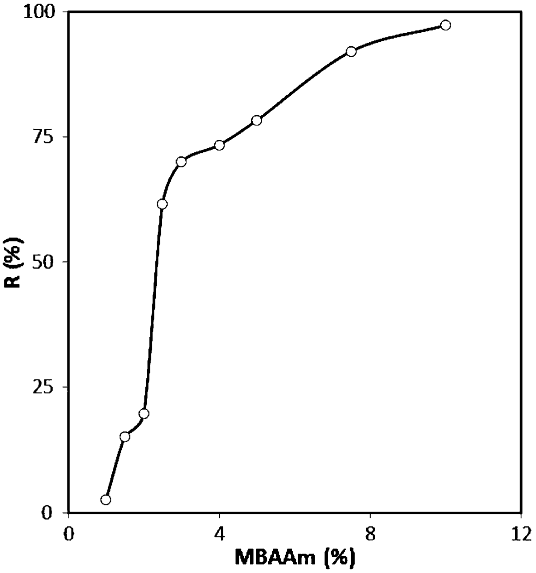


Fig. 4 Effect of initiator concentration on the adsorption amount of MB onto hydrogel [pH = 5.5; $T = 25$ °C; $t = 24$ h; $C_i = 25$ ppm; 10/90 IA/AMPS; 10% MBAAm]

Fig. 5 Effect of crosslinker concentration on the adsorption amount of MB onto hydrogel [pH = 5.5; $T = 25$ °C; $t = 24$ h; $C_i = 25$ ppm; 10/90 IA/AMPS; 1% KPS]

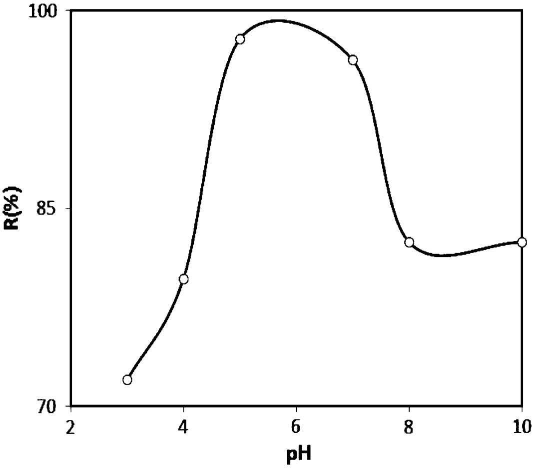


functional groups. However, extensive increase in crosslinker concentration decreases dye diffusion due to increasing crosslinker ratio of network structure.

Effects of pH on removal MB

The pH value of the solution, which affects the surface charge of the adsorbent and the degree of speciation of adsorbate, was an important controlling parameter in the adsorption process. To investigate the influence of pH value of the medium on the removal of MB, the pH range was selected from 3 to 10 in this study. As shown in Fig. 6, in the pH range from 3 to 6, the removal of MB dye onto hydrogel continuously increase with increasing pH values. The MB dye is positively charged

Fig. 6 Effect of pH on the adsorption amount of MB onto hydrogel [$T = 25$ °C; $t = 24$ h; $C_i = 25$ ppm; 10/90 IA/AMPS; 1% KPS; 10% MBAAm]



in solution and it has an affinity to materials with net negative charges. The high removal is due to the strong electrostatic interaction between the poly(AMPS-*co*-IA) hydrogel and cationic dye molecules. When pH value is increased from 3 to 6, (above the pK_a values of IA which are $pK_{a1} = 3.85$ and $pK_{a2} = 5.44$), the –COOH groups in IA ionized to $-COO^-$. As the pH of the system increases, the adsorbent surface appears negatively charged, which does favor the adsorption of cationic dye molecules due to the electrostatic attraction between the negatively charged surface and the dye cations. As a result, the removal of MB increases. At higher pH ($pH > 6$), a screening effect of the counter ions, such as NH_4^+, Na^+, shielding the charge of the carboxylate anions may prevents from an efficient repulsion, thereby causing the decrease in dye removal.

Effect of initial concentration on adsorption

Figure 7 shows the relationship between initial concentration of MB and the adsorption amount. It is clear from the Fig. 7; the adsorption amount of MB increased with increasing initial ion concentration and ultimately attains a saturated value. The experimental data have been analyzed by the Langmuir isotherms [17]. Langmuir isotherm model which monolayer coverage of the adsorption surface. This model assumes that adsorption occurs at specific homogeneous adsorption sites within the adsorbent and intermolecular forces decrease rapidly with the distance from the adsorption surface. The Langmuir adsorption model further based on the assumption that all the adsorption sites are identical and energetically equivalent. The linearized form of the Langmuir equation can be expressed as follows:

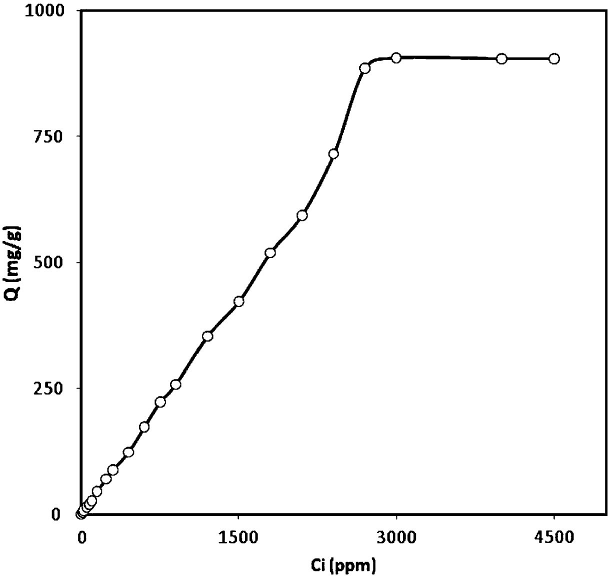


Fig. 7 Effect of initial concentration of MB on the adsorption amount of MB onto hydrogel [pH = 5.5; $T = 25$ °C; $t = 24$ h; 10/90 IA/AMPS; 1% KPS; 10% MBAAm]

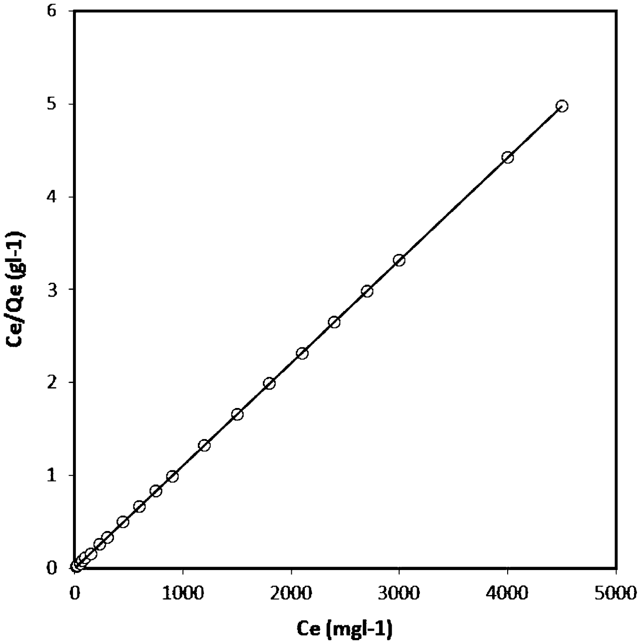


Fig. 8 Plots of C_e/Q_e versus C_e for MB

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_o} + \frac{1}{Q_o b} \tag{3}$$

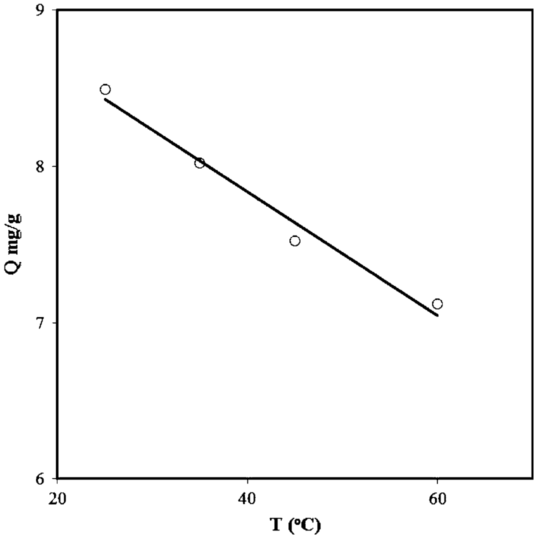
where the parameters of the equations are the following: C_e and Q_e the amount of ions remained in the solution and adsorbed onto the hydrogels at equilibrium, respectively. Q_0 is the saturated adsorption capacity, and b is an empirical parameter. The plot of C_e/Q_e versus C_e in Fig. 8 was drawn from the experimental data given in Fig. 7. The relationship between C_e/Q_e and C_e is linear, and this indicates the adsorption behavior of hydrogel fit Langmuir isotherm. From the slope of the Fig. 8, the theoretical monolayer saturation capacity of the hydrogel for MB was calculated as 1,000 mg/g against 905.91 mg/g obtained experimentally. The maximum saturation capacity Q_o obtained from the plot is higher than other types of adsorbents [18].

Effects of temperature on adsorption

Temperature is an important factor affecting the adsorption rate and dye uptake. The effect of increasing temperature on the removal of MB onto hydrogel was studied by performing adsorption experiment in a temperature range of 25–45 °C and the results are shown in Fig. 9. As shown in the figure, the adsorption amount of MB decreases with the increase in the temperature of the aqueous dye solution.

To predict the adsorption nature of MB on the poly(AMPS-*co*-IA) hydrogel was calculated thermodynamic parameters, such as enthalpy ($\Delta H°$), entropy ($\Delta S°$), and free energy ($\Delta G°$). The values of these parameters were calculated as follows:

Fig. 9 Effect of temperature on the adsorption amount of MB onto hydrogel [pH = 5.5; t = 24 h; C_i = 25 ppm; 10/90 IA/AMPS; 1% KPS; 10% MBAAm]



$$\log \frac{Q}{C_e} = -\frac{\Delta H^\circ}{2.303RT} + \frac{\Delta S^\circ}{2.303R} \tag{4}$$

$$\Delta G^\circ = \Delta H^\circ - \Delta S^\circ T \tag{5}$$

where R is the gas constant (8.314 J/mol K), C_e and Q are the amount of ions remained in the solution and adsorbed onto the reactive fibers. The relationship between $\log \frac{Q}{C_e}$ and $1/T$ for hydrogel is shown in Fig. 10. According to Eqs. 5 and the slope and intercept of linear line in Fig. 8, thermodynamic parameters of hydrogels were calculated and are shown in Table 1. The results show that the adsorption of MB on the poly (AMPS-*co*-IA) hydrogel has a decreased randomness at the

Fig. 10 Plots of $\log \frac{Q}{C_e}$ versus $1/T$

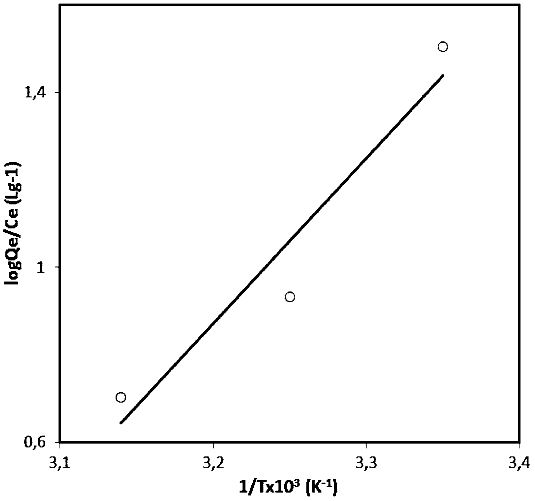


Table 1 Thermodynamic parameters for the adsorption of MB onto poly (AMPS-*co*-IA) hydrogels

[MB] (ppm)	$\Delta H°$ (kJ/mol)	$\Delta S°$ (J/mol K)	$\Delta G°$ (kJ/mol)		
			298 K	308 K	318 K
25	−72.35	−214.83	−8.32	−6.18	−4.03

solid/solution interface, and is an exothermic and spontaneous because of the negative values of $\Delta S°$, $\Delta H°$, and $\Delta G°$, respectively [19].

Adsorption kinetic

Using various dye concentration (100, 200, 300, 400 ppm) and hydrogel (0.1 g/30 mL), the concentrations of the dye in solution were determined at regular times. The experimental data obtained are shown in Fig. 11. The MB dye adsorption of the hydrogel shows a very rapid increase with the adsorption time at first and then slows down and reaches an equilibrium value for all the four different initial concentrations of MB dye studied. The time for reaching the adsorption equilibrium is ~2 h for each initial concentration value of MB dye.

Three different kinetic models were used to adjust the experimental data of MB adsorption.

The pseudo-first-order, pseudo-second-order, and intraparticle diffusion models were tested. A pseudo-first-order kinetic model of Lagergren [20] is given as;

$$\ln(Q_e - Q_t) = \ln Q_e - k_1 t, \tag{6}$$

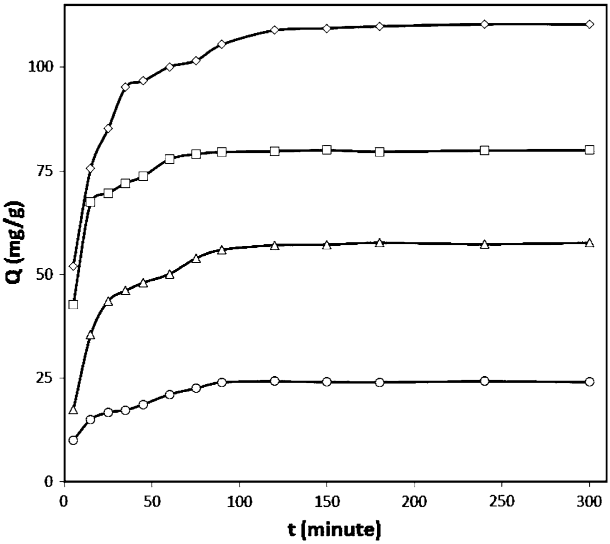


Fig. 11 Adsorption kinetic of MB on hydrogel (*circle*) 100 ppm, (*triangle*) 200 ppm, (*square*) 300 ppm, (*diamond*) 400 ppm [pH = 5.5; $T = 25$ °C; 10/90 IA/AMPS; 1% KPS; 10% MBAAm]

A pseudo-second-order kinetic model of Ho [21] is

$$\frac{t}{Q_\mathrm{t}} = \frac{1}{k_2 Q_\mathrm{e}^2} + \frac{1}{Q_\mathrm{e}} t, \tag{7}$$

Intraparticle diffusion model is expressed with the equation given by Weber and Morris [22];

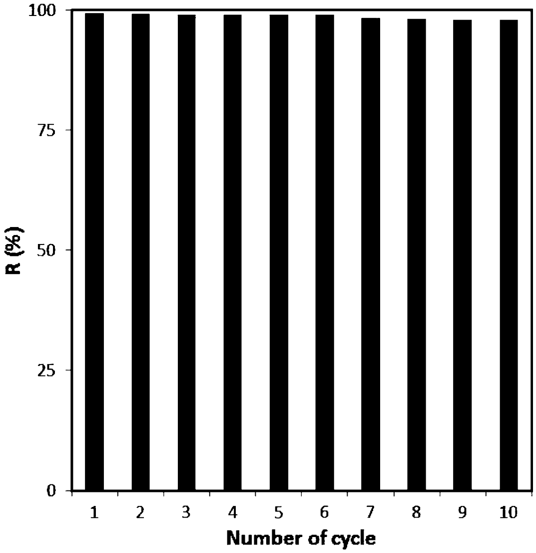
$$Q_\mathrm{t} = k_\mathrm{id} \cdot t^{1/2} \tag{8}$$

where Q_t and Q_e (mg g^{-1}) are the amount of adsorption on the hydrogel at time t and equilibrium, k_1 (min^{-1}), k_2 (gm g^{-1} min^{-1}), and k_id (mg g^{-1} $\mathrm{min}^{-1/2}$) are the rate constant for the pseudo-first-order, the pseudo-second-order and the intraparticle diffusion models, respectively. The values of k_1, k_2, and k_id at 25 °C were calculated from the slopes of the linear plot of $\ln(Q_\mathrm{e} - Q_t)$ versus t, $\frac{t}{Q_t}$ versus t and Q_t versus $t^{1/2}$, respectively, and the kinetic parameters obtained for adsorption of MB dye onto hydrogel were listed in Table 2. As it can be seen from Table 2, over all adsorption process fitted to the pseudo 2nd order kinetic model and at lower initial dye concentrations effect of intraparticle diffusion appeared to some extent. This behavior can be explained with the existence of chemisorptions. The structural nature of the adsorbent and the functional groups on it thought to be effective in such behavior. Actually this kind of behavior is in accordance with the literature that for such exchangers' adsorption kinetics is expected to follow second-order kinetics and/or intraparticle diffusion model [23]. At lower concentrations, adsorption kinetics also fitted to pseudo-first-order of lagergren (100 ppm) together with the second-order model. Generally, for such adsorbents, pseudo-first-order kinetic model is applicable to initial time course of the adsorption process but it does not represents the overall kinetic range [24]. The theoretical q_e values calculated from graphical interpretation of the first-order kinetic model were not found to fit experimental q_e values. For all initial concentrations, second-order kinetic model fitted better with high correlation coefficient and resulted in reasonable theoretical and experimental q_e values. This kind of behavior is also given in the literature for MB dye adsorption to the pyrophyllite adsorbent [25].

Table 2 Comparison of the pseudo-first- and second-order and intraparticle diffusion adsorption constants at different initial concentrations of MB

Kinetic model									
[MB] (ppm)	Pseudo-first-order				Pseudo-second-order			Intraparticle diffusion	
	Q_e (exp) (mg g^{-1})	Q_e (theor.) (mg g^{-1})	k_1 (min^{-1})	R^2	Q_e (theor.) (mg g^{-1})	$k_2 \times 10^3$ (g mg^{-1} min^{-1})	R^2	k_id (mg g^{-1} $\mathrm{min}^{-1/2}$)	R^2
100	24.1	16.07	0.028	0.965	25.64	3.719	0.998	1.810	0.978
200	57.3	41.68	0.035	0.958	62.50	1.600	0.999	4.756	0.881
300	80.0	47.70	0.059	0.939	83.33	3.600	0.999	4.302	0.778
400	109.4	66.02	0.035	0.938	125	1.030	0.999	6.800	0.882

Fig. 12 MB adsorption capacity of hydrogel after adsorption/desorption cycle [pH = 5.5; $T = 25$ °C; $t = 24$ h; $C_i = 25$ ppm; 10/90 IA/AMPS; 1% KPS; 10% MBAAm]



Reusability

The most an important properties of the adsorbent is its reusability after a particular process. For obtaining the reusability of the hydrogels, the adsorption–desorption cycle was repeated ten times with the same adsorbent and the results are shown in Fig. 12. It was observed that the adsorption capacity of hydrogels did not considerably change after ten adsorption–desorption cycles. This results shows that poly (AMPS-*co*-IA) hydrogels are good reusable adsorbent for the removal of MB from aqueous solution.

Conclusion

In this study, we prepared a hydrogel by copolymerization of AMPS and IA in aqueous medium, using MBAAm as crosslinker and KPS as initiator, and the adsorption properties of methylene blue (MB) from aqueous solution by the hydrogels was investigated by batch equilibration technique. As a result, the following conclusions were obtained.

1. Maximum removal percent of MB was obtained when polymerization was carried at the following conditions: IA/AMPS = 10/90 mol/mol; 1.0% KPS; 10% MBAAm; $t = 24$ h and $T = 50$ °C.
2. The adsorption of MB onto poly (AMPS/IA) hydrogel is favored at moderate-sized pH and lower temperature.
3. The kinetic studies indicate that the adsorption equilibrium of MB is $\sim$100 min and independent initial dye concentration (100–400 ppm).
4. Thermodynamic parameters suggest that the adsorption is a typical physical process, spontaneous, and exothermic in nature.

5. The regeneration of hydrogels without losing its original activity is found at least ten cycles.

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