Removal of Basic Dyes from Aqueous Solutions by Crosslinked-Acrylic Acid/Acrylamidopropane Sulfonic Acid Hydrogels

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ABSTRACT: In this study, Acrylic acid (AA)/2-acrylamido-2-methylpropane sulfonic acid (AMPS) hydrogels were prepared by free radical polymerization in aqueous solutions of AA, AMPS, and N,N-methylenebisacrylamide (NMBA) as crosslinker. Potassium persulfate (PPS)/potassium bisulfide (PBS) were used as initiator and accelerator pair. The water absorption capacities and dye adsorption properties of the hydrogels were investigated. Adsorption properties of the hydrogels were evaluated by depending on different adsorption conditions such as different initial dye concentration and contact time. The concentrations of the dyes were determined using UV/Vis Spectrophotometer at wavelength 530 nm for safranine T (ST) and 622 nm for brilliant cresyl blue (BCB). Adsorption kinetic studies showed that pseudo-first order kinetic model is suitable to explain the adsorption kinetic data of the hydrogels. Langmuir and Freundlich isotherm models were used to describe adsorption data. The result revealed that the adsorption of basic dyes onto hydrogels fit very well both Langmuir and Freundlich isotherms. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 1150–1159, 2009

Key words: hydrogel; acrylic acid; 2-acrylamido-2-methylpropane sulfonic acid; basic dye; adsorption

INTRODUCTION

Hydrogels represent tridimensional networks of hydrophilic polymers, which are able to swell in water and other biological fluids. A superabsorbant is defined as a material, which swells over 95% of the hydrogel composition.[1] The ability to swell in water depends firstly on the type of polymers and on the degree of crosslinking. Crosslinking can be either chemical (by covalent bonds) or physical (by ionic, hydrogen or Van der Waals bonds). Crosslinking with bifunctional agents to obtain hydrogels has been studied on a wide range of natural and synthetic polymers.[2–8] Hydrogels are becoming increasingly important because of their potential applications in industry. They are used for preparation of membranes in water purification and separation, catheters, contact lenses, agriculture, food industry, medicine, and biotechnology.[9,10]

Dyes are a kind of organic compounds, which can bring bright and firm color to other substances. Synthetic dyes usually have a complex aromatic molecular structure, which possibly comes from coal-tar–based hydrocarbons such as benzene, naphthalene, anthracene, toluene, xylene, etc. The complex aromatic molecular structures of dyes make them more stable and more difficult to biodegrade.[11,12] Today there are more than 10,000 dyes available commercially.[13] Synthetic dyes have been increasingly used in the textile, leather, paper, rubber, plastic, cosmetic, pharmaceutical, and food industries. The extensive use of dyes often poses pollution problems in the form of colored wastewater discharged into environmental water bodies. For some dyes, the dye concentration of less than 1 ppm in receiving water bodies is highly visible, so that even small quantities of dyes can color large water bodies. This not only affects aesthetic merit but also inhibits sunlight penetration and reduces photosynthetic action. In addition, some dyes or their metabolites are either toxic or mutagenic and carcinogenic.[14,15] The conventional methods for removal of dyes from wastewaters include coagulation and flocculation,[16] oxidation or ozonation,[17,18] membrane separation[19] and adsorption.[20] Amongst these numerous techniques of dye removal, adsorption is a procedure of prefer for the removal of organic compounds from wastewater.[21] Adsorption is an effective process for the removal of color from waste effluents. Recently, it was determined that crosslinked polymeric materials having functional groups such as carboxylic acid, amine, hydroxyl, and sulfonic acid groups could be used as complexing agents for removal of dyes from aqueous solutions.[22] When hydrogels come in contact with aqueous solutions, they adsorb and retain the dissolved substances. For this reason, they have
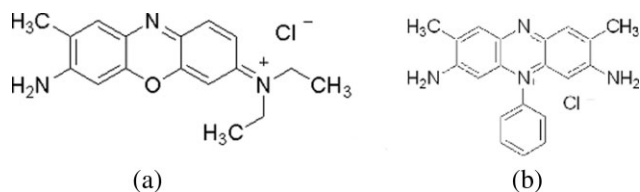
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Figure 1 Chemical structure of Safranine T and Brilliant Cresyl Blue (a) Safranine T; (b) Brilliant Cresyl Blue.

been in several studies proposed as water purification agents.[23–25] On the basis of the nature of the chemical groups attached on the polymer branches, hydrogels demonstrate selectivity towards dissolved substances. For example, the negatively charged —COO⁻ and —SO₃⁻ groups selectively attract and retain both organic and inorganic cations from aqueous solutions.[26]

In this study, acrylic acid (AA)/2-acrylamido-2-methlypropane sulfonic acid (AMPS) hydrogels were obtained by free radical polimerization in aqueous media. Their swelling properties were investigated in water. Also, they were used as a new adsorbent for the removal of Safranine T (ST), Brillant Cresyl Blue (BCB) from aqueous solutions.

EXPERIMENTAL

Materials

The monomers, acrylic acid (AA), 2-acrylamido-2-methlypropane sulfonic acid (AMPS); the crosslinking agent, N,N-methylenebisacrylamide (NMBA); the water soluble initiator and accelerator pair, potassium persulfate (PPS), and potassium bisulfide (PBS) were all purchased from Merck AG (Darmstadt, Germany). AA was purified by vacuum distillation (15 mmHg, 80°C). Basic dyes [Safranine T (ST) and Brillant Cresyl Blue (BCB)] were also obtained from Merck AG and their chemical structures are shown in Figure 1. Rest of the materials was chemically pure grade. All solutions and standards were prepared using distilled water.

Instruments

Spectrophotometric measurements were carried out using Jenway 6105 UV/Vis spectrophotometer.

The infrared spectra of hydrogels were taken with Digilab Excalibur-FTS 3000MX model FTIR Spectrophotometer using KBr pellets.

Synthesis of hydrogels

The AA and AMPS-based hydrogels were prepared by free radical polymerization with NMBA as crosslinker in aqueous solution. Different amounts of

monomers (AA and AMPS) and the crosslinking agent (NMBA) were dissolved in 4 mL deionized water in 2 cm diameter glass tubes. After sealing the tubes with rubber caps, the solution was purged with nitrogen gas for 20 min to eliminate dissolved oxygen in the system, and the tubes were placed in a water bath at 70°C. Then, the required amounts of PPS and PBS were added as their solution in 1 mL water. The polymerization reactions were carried out for 2 h at 70°C. In all experiments, the total initial monomer concentration was fixed at 1M, the initiator concentration (PPS) was 1% (mole) with respect to total amount of monomers, and the amount of accelerator (PBS) was equal to the PPS in weight. At the end of the polymerization, the hydrogels were taken out from the the glass tubes by breaking the glass. The hydrogels were cut into discs in same length (~ 5 mm), put in distilled water, and were held in water at room temperature for 2 days. The water was replaced with fresh water twice in a day to remove any unreacted monomers and initiators. Afterwards, the hydrogels were dried under vacuum at 40°C for using in swelling and dye adsorption experiments. The feed compositions of the hydrogels are given in Table I.

Determination of water absorption capacities

The hydrogels were swollen in distilled water and water absorption capacities were determined by teabag method. To apply this method, a tea bag that was made of 250-mesh nylon screen, contains a known amount of dried hydrogel sample was immersed entirely in water and kept there to attain swelling equilibrium for a certain time (Q_t), then hung up for 1 min. to drain the excess solution, and weighed. The water uptake was calculated to the following equation.

$$Q_t = (W_{\text{wet}} - W_{\text{dry}})/W_{\text{dry}}$$

where Q_t is the swelling ratio for a given time, W_{wet} and W_{dry} are the weights of the swollen gel and the dry sample, respectively. In case of equilibrium swelling ratio, the Q_t symbol is given as Q_e.

TABLE I
The Feed Compositions of the Hydrogels

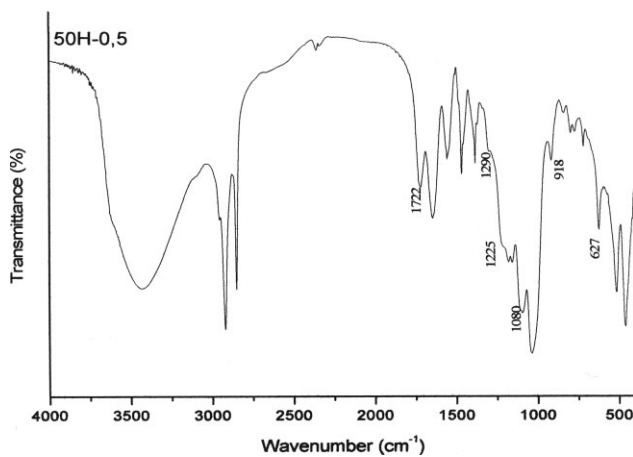| Hydrogel	AA (mole %)	AMPS (mole %)	NMBA (mole %)
10H-0.5	90	10	0.5
25H-0.5	75	25	0.5
50H-0.5	50	50	0.5
10H-1	90	10	1
25H-1	75	25	1
50H-1	50	50	1

Figure 2 FTIR spectra of copolymer.

Adsorption studies

All adsorption experiments were carried out at ambient temperature. Two different dye solutions were used to investigate the adsorption kinetics and adsorption isotherms. A fixed amount of hydrogel (0.05 g) was immersed in 250 mL ST or BCB solutions have a dye concentration of 500 mg/L. The amount of residual dye in the solutions was determined by spectrophotometry after desire treatment period. The adsorption capacities (mg dye/g hydrogel) of hydrogels were calculated using the following expression:

$$q_e = (C_i - C_e)V/m$$

where q_e is the adsorbed amount of dye per gram hydrogel, C_i and C_e the concentration of the dye in the initial solution and equilibrium, respectively (mg/L), V the volume of the dye solution added (ml) and m is the amount of the hydrogel used (g). In adsorption kinetic graphics the q_e symbol is given
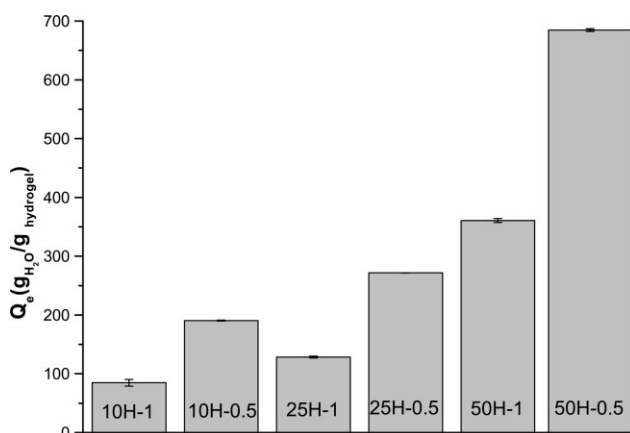
as q_t that illustrates the amount of dye per gram of hydrogel for a given time.

Furthermore, adsorption isotherms were obtained by swelling the hydrogel samples in the solution of ST and BCB with various initial concentrations for 48 h. The range of the initial dye concentration was 100–500 mg/L, residual concentration of ST and BCB determined as described above.

RESULTS AND DISCUSSION

FTIR analysis

Since two monomers are acrylic base monomers, they have similar characteristic peaks, but because of their different functional groups (carboxyl group for AA, sulphonic acid group for AMPS) the FTIR spectra of the hydrogels could be able to give some information about the chemical structure of the hydrogels. As it seen from the Figure 2, the hydrogel sample has all characteristic peaks of carboxyl groups and SO_3H groups: 1722 cm^{-1}, $C=O$; 1290 cm^{-1}, $C-O$; 918 cm^{-1}, $-OH$ vibrations of carboxyl group; 1225 and 1080 cm^{-1}, SO_2 asymmetric and symmetric stretching vibrations; 627 cm^{-1}, $S-O$ stretching vibration. These so-called IR bands confirm the presence of AA and AMPS monomers in hydrogel structure.

Swelling behavior of the hydrogels

The swelling ratio of the hydrogels was measured in distilled water according to the conventional gravimetric procedure as described above. The equilibrium swelling ratios (Q_e) of the hydrogels were represented in Figure 3. The effect of the contact time on the water absorption capacities of the hydrogels was investigated, and the results were given in Figure 4. It was noticed that in about 4 h, all hydrogels reached their swelling equilibrium (Fig. 4).
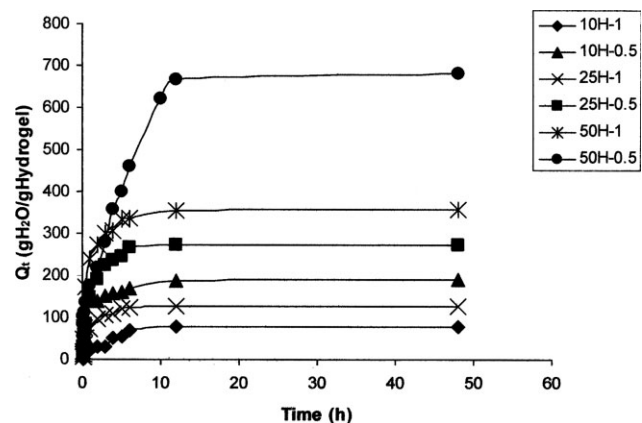

Figure 3 Equilibrium swelling values of the hydrogels.

Figure 4 The effect of contact time on swelling behavior of the hydrogels.
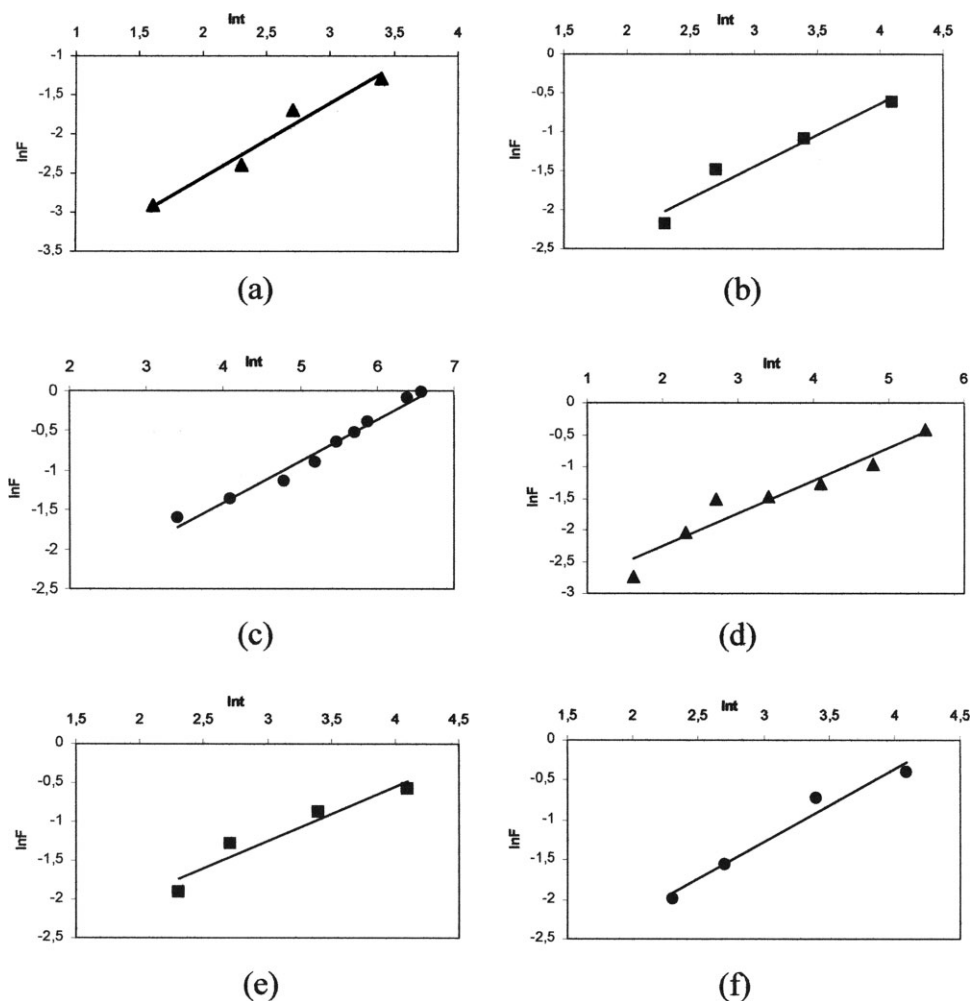
Figure 5 Swelling kinetic curves of the hydrogels. a) 10H-0.5; b) 25H-0.5; c) 50H-0.5; d) 10H-1; e) 25H-1; f) 50H-1.

In general, the concentration of the co-monomer in the reaction solution affects the properties of the resulting copolymer. Figures 3 and 4 demonstrate the effect of AMPS contents on swelling behavior of the hydrogels. According to the figures it can be concluded that the higher the AMPS contents, the higher the Q_e values are (Q_e value of 50H-0.5 hydrogel is $\sim$ 1.5 times higher than that of 25H-0.5, and two times higher than that of 10H-0.5 hydrogel, and same for the hydrogels that have 1% NMBA). This can attributed the presence of a high amount of strong hydrophilic (SO$_3$H) groups improve the swelling behavior of the hydrogels.[27] Also in Figures 3 and 4, the effect of NMBA content on swelling behavior of the hydrogels can be seen. It is obvious that when the NMBA content is reduced from 1 to 0.5% Q_e values of the hydrogels increased. When crosslinkers are added to hydrogel systems, it is known that there would be a changing in the swelling value of hydrogels, because the molecules of crosslinkers are placed between the chains of monomer and comonomers. Then, the hydrophilic group

number is decreased, and consequently swelling value is decreasing.[28]

To determine the nature of diffusion of water into hydrogels the following equation was used:[29]

$$F = kt^n$$

In this equation, F denotes fractional uptake at time t, k is a constant related to the structure of the network, and n is the diffusional exponent, which is indicative of the transport mechanism. Fickian

TABLE II
Diffusion Characteristics of Hydrogels

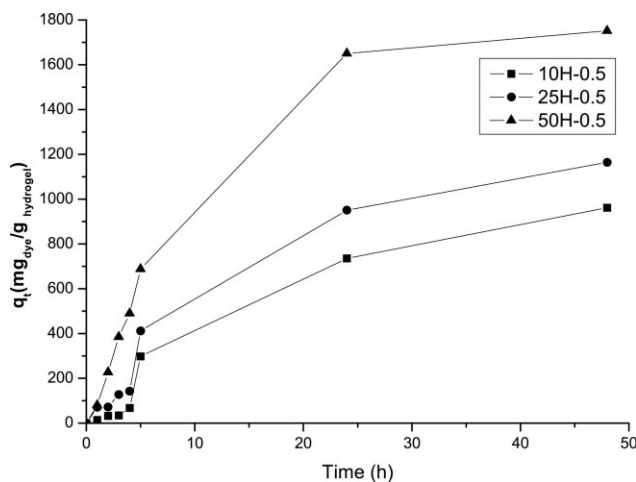| Sample	n	$k \times 10^{-2}$
10H-0.5	0.94	1.17
25H-0.5	0.81	2.02
50H-0.5	0.52	3.01
10H-1	0.52	3.76
25H-1	0.70	3.48
50H-1	0.91	1.79

Figure 6 The effect of contact time on ST adsorption of the hydrogels (0.5% NMBA).

Figure 8 The effect of contact time on BCB adsorption of the hydrogels (0.5% NMBA).

diffusion and Case II transport are defined by n values of 0.5 and 1.0, respectively. On the other hand, for non-Fickian diffusion n is between 0.5 and 1.0.[29]

The plots of ln F versus ln t are represented in Figure 5. The exponent n and k were calculated from the slopes and intercepts of the lines, respectively, and are listed in Table II. As seen in Table II, the values of the diffusion exponent range between 0.52 and 0.94 and are found to be over 0.5. Hence, the diffusion of water into the hydrogels is assumed to be a non-Fickian character.

Dye adsorption studies

Adsorption properties of the hydrogels were evaluated by depending on different adsorption conditions such as different initial dye concentration and contact time. The concentrations of the dyes were
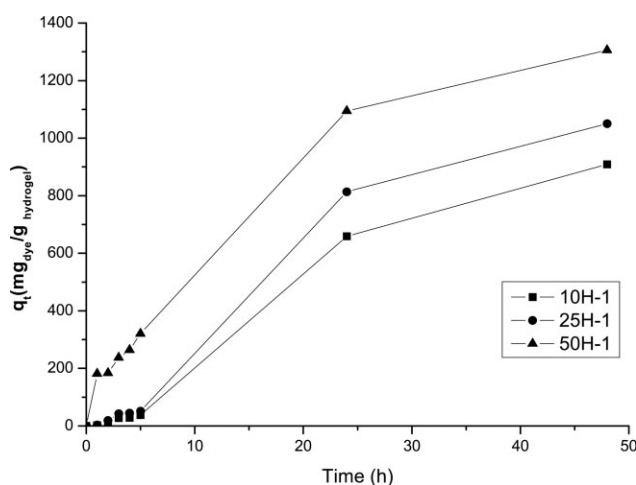
determined using UV/Vis Spectrophotometer at wavelength $\lambda = 530$ nm (ST) and $\lambda = 622$ nm (BCB).

Figure 6–9 illustrate the effect of adsorption time on the adsorption efficiency. The adsorption rates and capacities of the hydrogels for basic dyes increase with the increase of the adsorption time. As it seen from the figures, the dye adsorption capacity of 50H-0.5 hydrogel is ~ 1.5 times higher than that of 25H-0.5, and two times higher than that of 10H-0.5 hydrogel in both kinds of basic dye solution. This adsorption capacity reduction was also observed in case of hydrogels that contain 1% NMBA (the adsorption capacity of 50H-1 > 25H-1 > 10H-1 for both dye solutions). It can be easily said that increasing the AMPS content causes an increase in dye adsorption rates and capacities of the hydrogels. This can be attributed the presence of a high amount of strong hydrophilic (SO_3H) groups improve the

Figure 7 The effect of contact time on ST adsorption of the hydrogels (1% NMBA).

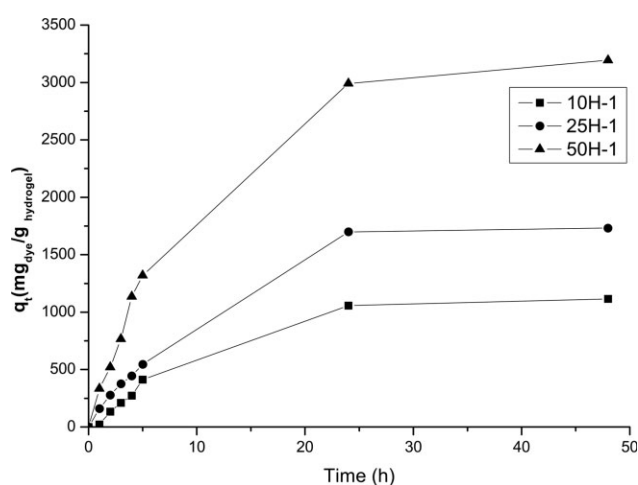**Figure 9** The effect of contact time on BCB adsorption of the hydrogels (1% NMBA).
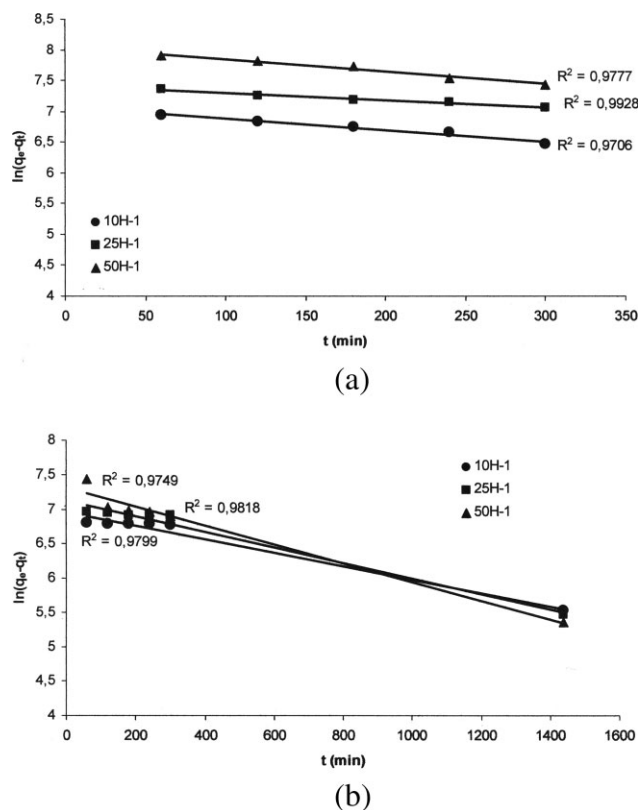
Figure 10 Pseudo-first order plots for BCB and ST onto hydrogels. a) BCB; b) ST.

dye adsorption rates and capacities of the hydrogels. Also it was observed that, the dye adsorption capacities of the hydrogels lowered $\sim$ 1.5 times when the NMBA mole content increased from 0.5 to 1% in hydrogels that have equal AMPS content.

On the other hand, adsorption capacities of hydrogels for ST and BCB decrease with following order: BCB > ST (adsorption of BCB is approximately two times higher than that of ST). It is concluded that the main adsorption of both dye is occurred by the electrostatic interactions of $-SO_3H$ groups in the hydrogel and N^+ group of the dyes. Since the N^+ group is more isolated in ST (by the aromatic groups), (Fig. 1),

lower adsorption capacity is an expectable event in treatment process of ST than that of BCB.

Kinetic studies

Pseudo-first order and pseudo-second order kinetic equations were used to test the experimental data for examine the controlling mechanism of adsorption processes. The pseudo-first order kinetic model was suggested by Lagergren for the adsorption of solid/liquid systems and its linear form can be formulated as:[30]

$$\log(q_e - q_t) = \log q_e - (k_1 t / 2.303)$$

where q_t is the adsorption capacity at time t (mg/g) and k_1 (min^{-1}) is the rate constant of the pseudo-second order-first adsorption was applied to this study of ST and BCB adsorption. The k_1 values and regression coefficients were calculated from the linear plots of $\log(q_e - q_t)$ versus t (Fig. 10). The linearity of these plots indicates the applicability of the pseudo-first order rate expression. The k_1 value calculated from the slope of the line in Figure 10. The k_1, the calculated q_e values and regression coefficients R^2 are given in Table III. The calculated q_e values agree with the experimental data in case of pseudo-first order kinetics. This case suggested that the adsorption data are represented by pseudo-first order kinetics.

The kinetic data were further analyzed using Ho's pseudo-kinetics model. It can be expressed as:[30]

$$t/q_t = 1/(k_2 q_e^2) + 1/q_e$$

The k_2 (the rate constant of the pseudo-second order adsorption), the calculated q_e values and regression coefficients R^2 are given in Table III. It can be seen from the Table that a clearly difference of q_e between the experiment and calculation was observed. Moreover, the regression coefficient R^2 values for the first order kinetics are higher than that of the second order kinetics. This model is not suitable to explain the adsorption data.

TABLE III
Kinetic Parameters for the Adsorption of BCB and ST onto hydrogels

Dye	Sample	q_e (exp)	Pseudo-first order model			Pseudo-second order model		
			k_1 (min^{-1})	q_e (calculated) (mg g^{-1})	R^2	k_2 (g mg^{-1}min^{-1})	q_e (calculated) (mg g^{-1})	R^2
BCB	10H-1	1057	0.0018	1170	0.9706	2.10×10^{-7}	2500	0.8644
	25H-1	1699	0.0012	1665	0.9928	2.05×10^{-7}	3300	0.9687
	50H-1	3000	0.0020	3135	0.9777	2.18×10^{-7}	5000	0.9734
ST	10H-1	910	0.0010	1050	0.9799	3.3×10^{-5}	81	0.5229
	25H-1	1050	0.0011	1242	0.9818	9.5×10^{-5}	74	0.9523
	50H-1	1306	0.0014	1493	0.9749	4.47×10^{-4}	67	0.8536

TABLE IV
Freundlich Constants of Adsorption Isotherms for ST and BCB onto Hydrogels

Hydrogel	Safranine T			Brilliant Cresyl Blue		
	K_f (mg/g)	n	R^2	K_f (mg/g)	n	R^2
10H-0.5	48.9980	1.9285	0.9800	41.1926	1.3936	0.9411
25H-0.5	127.8336	2.4923	0.9647	81.3588	1.5260	0.9360
50H-0.5	162.9429	2.1543	09696	138.6745	1.2949	0.9178
10H-1	49.8705	2.1104	0.9950	7.3165	1.0800	0.9648
25H-1	74.9798	2.1643	0.9860	20.3711	1.2218	0.9761
50H-1	194.6980	2.7182	0.9257	123.2678	1.6037	0.9754

Adsorption isotherms

An adsorption isotherm describes the relationship between the amount of substance adsorbed on the substrate and the concentration of dissolved adsorbate in the liquid at equilibrium. Equations often used to describe the experimental isotherm data are those developed by Freundlich, by Langmuir. The Freundlich and Langmuir isotherms are the most commonly used to describe the adsorption characteristics of adsorbent used in water and wastewater.

In experiments of equilibrium adsorption isotherm, a fixed amount of 0.05 g adsorbents were immersed into 250 mL of aqueous solutions ST and BCB have different concentrations (100–500 mg/L).

Freundlich isotherm

The Freundlich equations were used to fit the equilibrium isotherms. The Freundlich equation[31] is the earliest known relationship describing the adsorption equation. Freundlich isotherms were obtained by different initial dye concentrations (100–500 mg/L) and 0.05 g hydrogel dose for a constant time of 24 h. The adsorption isotherms data were correlated with the Freundlich equations and the Freundlich constants K_f (mg/g) and n (intensity of adsorption) were calculated from the following equations:

$$q_e = K_f \times C_e^{1/n}$$

$$\ln q_e = \ln K_f + 1/n \ln C_e$$

where q_e is the amount of dye adsorbed (mg/g) onto hydrogel. The parameters of Freundlich isotherm, K_f,

n, and the regression coefficients R^2 are given in Table IV. Linear plots of log q_e versus log C_e for the different initial dye concentrations illustrated that the adsorption follows the Freundlich isotherm (Fig. 11). Such conclusion can be drawn from data R^2 that the Freundlich adsorption law is applicable to be adsorption of ST and BCB onto hydrogel (Table IV). The n values between 1 and 10 indicate beneficial adsorption.[31] For the adsorption of ST and BCB onto hydrogel, the n values were above to beneficial adsorption ($n > 1$). The adsorption capacities of hydrogels for ST and BCB have the following order: BCB > ST.

Langmuir isotherm

The Langmuir model is probably the best known and most widely applied sorption isotherm. It may be represented as follows:

$$q_e = q_o b C_e/(1 + bC_e)$$

$$1/q_e = 1/q_o + 1/q_o bCe$$

The Langmuir constants q_o and b are related to the adsorption capacity and the energy of adsorption, respectively. The essential characteristics of Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter:

$$R_L = 1/(1 + bC_o)$$

Where b is the Langmuir constant and C_o is the highest initial dye concentration (mg/L).[31] The value

TABLE V
Langmuir Constants of Adsorption Isotherms for ST and BCB onto Hydrogels

| Hydrogel	Safranine T				Brilliant Cresyl Blue			
	q_o (mg/g)	b (L/mg)	R^2	R_L	q_o (mg/g)	b (L/mg)	R^2	R_L
10H-0.5	1083.34	0.0192	0.9699	0.10	2737.37	0.0074	0.9609	0.21
25H-0.5	1546.89	0.0125	0.9880	0.14	3911.37	0.0081	0.9819	0.20
50H-0.5	2373.98	0.0171	0.9766	0.11	7326.38	0.0146	0.9951	0.12
10H-1	943.40	0.0105	0.9815	0.16	1997.41	0.0035	0.9813	0.36
25H-1	1122.35	0.0211	0.9684	0.09	3357.87	0.0038	0.9974	0.35
50H-1	1538.98	0.0294	0.9573	0.06	3034.98	0.0200	0.9824	0.09
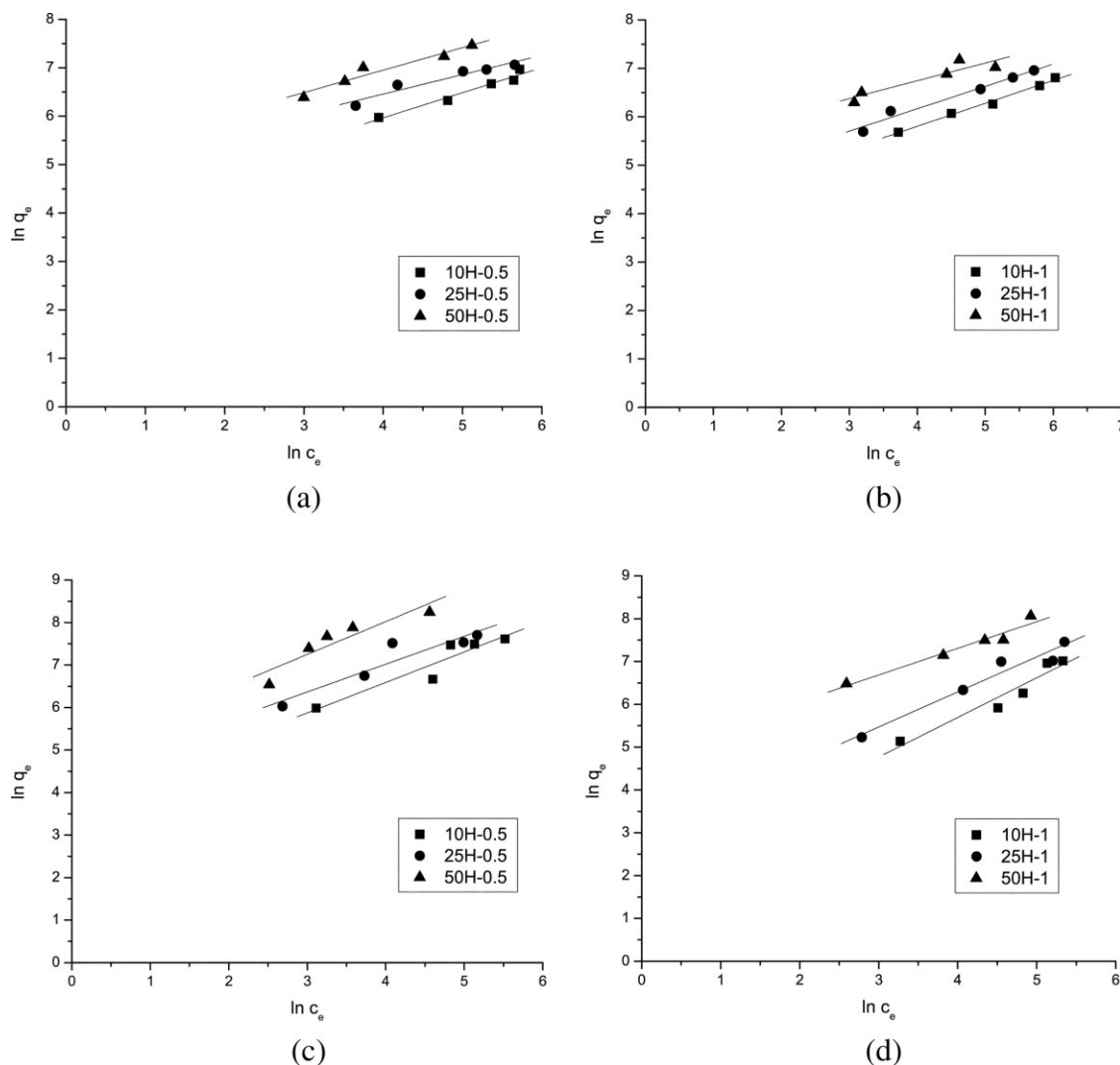
Figure 11 Freundlich isotherm for the adsorption of basic dyes onto hydrogels. a) Hydrogels (10H-0.5, 25H-0.5, and 50H-0.5), Basic dye (ST); b) Hydrogels (10H-1, 25H-1, and 50H-1), Basic dye (ST); c) Hydrogels (10H-0.5, 25H-0.5, and 50H-0.5), Basic dye (BCB); d) Hydrogels (10H-1, 25H-1, and 50H-1), Basic dye (BCB).

of R_L indicates the type of the isotherm either to be unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$).[32] The parameters of Langmuir isotherm, q_o, b, R_L, and the regression coefficients R^2 are given in Table V and Figure 12. The R_L values calculated from above expression, lies in the range of 0–1, indicates favorable adsorption of ST and BCB on hydrogels (Table V).

The adsorption isotherm data of both basic dyes well fit both Langmuir and Freundlich isotherms (Tables IV–V). Thus, it is concluded that the adsorption occurs by monolayer formation of basic dye molecules onto the hydrogel surface. The q_o values (Langmuir) increased by increasing the AMPS content. It is concluded that the positively charged dye molecules are directly adsorbed onto the surface through direct bonding with negatively charged SO_3^- groups. The increase the active sites on the surface, the increase the adsorption capacity. This conclusion

is also agreed with that discussed before in the effect of contact time in adsorption studies section. As it mentioned, increasing AMPS (in other words negatively charged active site) content on the surface increases the rate of adsorption of dyes. This can be attributed the presence of a high amount of strong hydrophilic (SO_3H) groups improve the dye adsorption rates and capacities of the hydrogels.

CONCLUSION

In this study, AA-*co*-AMPS hydrogels were obtained by free radical polymerization in aqueous media. They were characterized, and their swelling properties were investigated. The main purpose of this work is investigation of removal of the basic dyes from aqueous solutions with hydrogels. They were used as new adsorbents for the removal of Safranine T and Brillant Cresyl Blue from aqueous solution.
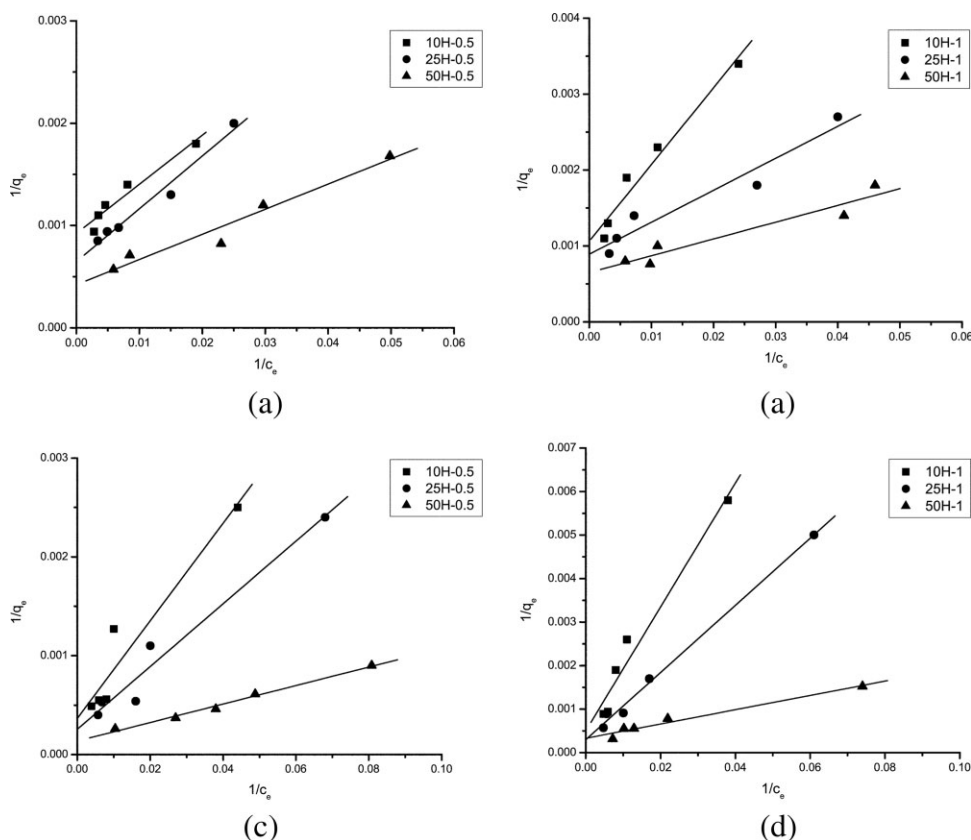
Figure 12 Langmuir isotherm for the adsorption of basic dyes onto hydrogels. a) Hydrogels (10H-0.5, 25H-0.5, and 50H-0.5), Basic dye (ST); b) Hydrogels (10H-1, 25H-1, and 50H-1), Basic dye (ST); c) Hydrogels (10H-0.5, 25H-0.5, and 50H-0.5), Basic dye (BCB); d) Hydrogels (10H-1, 25H-1, and 50H-1), Basic dye (BCB).

The isotherm data were fitted with Freundlich and Langmuir isotherms. The dye adsorption capacities of this sorbent for these basic dyes were better than other sorbents such as natural clays[31,33] and some other polymeric hydrogels[34] that have been given in the literature. The following conclusions can be drawn:

- Increasing AMPS content in hydrogel feed composition increases the dye adsorption capacity. This can attributed the presence of a high amount of strong hydrophilic (SO_3H) groups improve the dye adsorption capacities of the hydrogels.
- Adsorption capacities of hydrogels for ST and BCB decrease with following order: BCB > ST. This case was concerned with the isolation status of N^+ group in the basic dye molecular structure. The steric hindrances of the groups act an active role in adsorption process and causes important adsorption capacity reduction.
- Langmuir and Freundlich isotherm models were used to describe adsorption data. The result revealed that the adsorption of basic dyes onto hydrogels fit both Langmuir and Freundlich isotherms.

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