Adsorption of Cationic Dyes on Poly(acrylic acid-*co*-sodium acrylate-*co*-acrylamide) Superabsorbents

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ABSTRACT: Inverse suspension polymerization was carried out to synthesize poly(acrylic acid-*co*-sodium acrylate-*co*-acrylamide) superabsorbent polymers (SAPs) crosslinked with ethylene glycol dimethacrylate (EGDMA). The equilibrium swelling capacities of the SAPs, determined by swelling them in DI water, were found to vary with the acrylamide (AM) content. The SAPs were used to adsorb four cationic dyes (Acriflavine, Auramine-O, Azure-I and Pyronin-Y). The effect of AM content in the SAPs on the adsorption of the cationic dyes was investigated. Different initial concentrations of Azure-I were used with the same amount of the SAP to explore the effect of initial dye concentration on the adsorption. The effect of the adsorbent amount was investigated by taking different amounts of SAP with a fixed initial concentration of Acriflavine. The kinetics of the dye adsorption was modeled by a first order model and the equilibrium amount of the dye adsorbed, adsorption rate coefficients, removal efficiency and partition coefficients were determined. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 3892–3899, 2012

Key words: acrylic acid; superabsorbent; hydrogels; swelling; dyes; adsorption

INTRODUCTION

The crosslinked three-dimensional network structures of hydrophilic polymers, with the ability to absorb and retain liquids more than 100 times of their own weight, are known as the superabsorbent polymers (SAPs).[1] The SAPs contain ionic groups in the crosslinked network which cause large osmotic pressure difference between the polymers and the swelling medium.[2] Large inflow of water occurs to balance the osmotic pressure difference and the crosslinked networks exhibit very high equilibrium swelling capacities. Some successful commercial applications of the SAPs are in personal care products as diapers, sanitary napkins and adult incontinence products.[3] The ability to absorb large amount of water and other liquids and stimuli responsive behaviour depending on the swelling medium makes them useful in the biomedical field as drug delivery devises[4] and contact lenses.[5] They are also used for food packaging, water retention of soil[6] and water purification.[7–9]

In the field of water purification, SAPs have been widely used for the removal of metal ions[7,10,11] and dyes[8,9,12,13] from industrial wastes. The adsorption characteristics of SAPs depend on the nature of the charge on the adsorbent and the adsorbate, fraction of ionic repeat units in the SAPs, initial concentration of the adsorbate, pH and temperature of the medium, and the crosslink density of the polymer. Rodriguez and Katime[7] have studied the effect of pH and concentration on the uptake of metal ions by acrylic-acid itaconic acid hydrogels. The effect of treatment time, pH and temperature of the feed solution and effect of initial feed concentration on the dye uptake has been investigated for the radiation synthesized *N*-vinyl-2-pyrrolidone/methyl methacrylate and *N*-vinyl-2-pyrrolidone/acrylonitrile hydrogels.[8] In the study of adsorption of cationic dyes by acrylamide-maleic acid hydrogels, the effect of crosslinking agents trimethylolpropane triacrylate (TMPTA) and 1,4-butanediol dimethacrylate (BDMA) and the maleic acid content has been explored.[9] The effect of the anionic monomer content has also been reported for radiation synthesized acrylamide-maleic acid hydrogels,[1,14] *N*-vinyl 2-pyrrolidone-itaconic acid hydrogels,[1] acrylamide-acrylic acid hydrogels,[15] acrylamide-sodium acrylate,[16] and acrylamide-itaconic acid hydrogels.[12,17] Temperature and pH sensitive *N*-isopropylacrylamide-itaconic acid hydrogels have been used for the removal of cationic dyes.[18] γ-radiation synthesized poly(acrylamide/2-hydroxypropyl methacrylate/maleic acid) terpolymers have also been used for the adsorption of bovine serum albumin (BSA) from aqueous solutions.[19] Recently, hydrogel nanocomposites of

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acrylamide, itaconic acid and montmorillonite,[20] poly(2-(*N*,*N*-dimethylamino)ethyl methacrylate-*co*-2-acrylamido-2-methlypropane sulfonic acid-*co*-2-hydroxyethyl methacrylate) terpolymer/montmorillonite nanocomposite hydrogels[21] and starch-graft-acrylic acid/montmorillonite superabsorbent nanocomposite hydrogels[22] have been employed for the removal of basic dyes, acidic dyes, and metal ions, respectively.

This study involves the investigation of the adsorption of cationic dyes by poly(acrylic acid-*co*-sodium acrylate-*co*-acrylamide) superabsorbents. The ethylene glycol dimethacrylate crosslinked SAPs were synthesized by inverse suspension polymerization. The adsorption of various dyes was done in batch experiments. The effect of different dyes, initial dye concentration, amount of the SAP, and the AM content in the SAPs on the adsorption process were explored. The equilibrium swelling capacity, equilibrium amount of dye adsorbed, rate coefficients for the adsorption of dyes were determined from the first order model. The removal efficiency and partition coefficients were also calculated for various dye adsorption experiments.

EXPERIMENTAL

Materials

Acrylic acid (AA) and acrylamide (AM) were obtained from Merck Limited, India, and S.D. Fine-Chem Ltd., India, respectively. Ethylene glycol dimethacrylate (EGDMA), used as crosslinking agent, was purchased from Aldrich. Water soluble initiator, potassium persulfate (KPS), and sodium hydroxide, used for neutralization of acrylic acid to obtain sodium acrylate (SA), were procured from S.D. Fine-Chem Ltd., India. Methanol was purchased from Merck Limited, India. Water-in-oil surfactant, Span-80, was obtained from Rolex Chemical Industries (Mumbai). Dyes used for the adsorption studies, Azure-I, Acriflavine and Auramine-O were from S.D. Fine-Chem Ltd., India, and Pyronin-Y was from Rolex Chemical Industries (Mumbai). Milli-Q water was used for all the experiments.

Synthesis of the superabsorbent polymers

Inverse suspension polymerization was employed to synthesize the SAPs.[23] The detailed polymerization procedure and recipe is reported elsewhere[24] A four-necked round bottom flask having a reflux condenser, an inlet for N_2, and a temperature sensor was used for the polymerization. The reactants were added from the fourth inlet and mixed using a magnetic stirrer. The temperature of the flask was maintained using a temperature controller.

Sodium acrylate (SA) was obtained by 75% neutralization of AA by NaOH solution. The monomers AA, SA, and AM and the initiator KPS were dissolved in water to prepare the dispersed phase. Toluene was chosen as continuous phase and water-in-oil surfactant Span-80 and the tetra-functional crosslinking agent ethylene glycol dimethacrylate (EGDMA) were added to it. The dispersed phase was added dropwise to the continuous phase and the polymerization was carried out for 2 h at 80°C. The AM content was varied from 20 to 100 mol % and the synthesized polymers with 20, 40, 60, 80, and 100 mol % AM content were termed as AA/SA/AM-1, AA/SA/AM-2, AA/SA/AM-3, AA/SA/AM-4, and AM-1, respectively.

Fourier transform infrared spectroscopy (FTIR)

FTIR spectra for the synthesized SAPs were recorded on a Perkin Elmer Spectrum RX-I spectrometer. The spectra were recorded in transmission mode at a resolution of 4 cm^{-1} in the range of 4000–500 cm^{-1}.

Determination of swelling capacity of the superabsorbent polymers

To determine the swelling capacity of the SAPs, a known weight of the dry polymer was kept in a plastic basket and immersed in beakers containing 500 mL of DI water. The baskets were taken out of the swelling medium at different times and excess water was removed by wiping with tissue papers. The swollen polymers were weighed and then returned to the respective beakers for further swelling. The swelling capacity, S (g of water/g of SAP), is defined with respect to the weight of dry (W_d) and swollen (W_s) polymers as

$$S = \frac{W_s - W_d}{W_d} \quad (1)$$

Adsorption of dyes on SAPs

The adsorption of four cationic dyes, namely Acriflavine, Auramine-O, Azure-I and Pyronin-Y on the SAPs was investigated. The chemical formula, molecular weight, λ_{max}, CAS number, color index number, and molecular sizes for these dyes are listed in Table 1. The adsorption of the dyes was carried out in batch experiments in which 500 mL of the dye solution of required concentration was taken in a beaker and the pre-weighed amount of the dry SAP was added to it. During the experiments, the beakers were kept covered with aluminum foil and approximately 0.5 mL of the dye solution was taken out at various times. The samples were analyzed on a Shimadzu UV-1700 PharmaSpec UV-Vis spectrophotometer equipped

TABLE I
Some Characteristics of the Cationic Dyes Used for Adsorption Studies

Dye	Chemical formula	MW	λ_{max} (nm)	CAS number	CI number	Molecular size (Å^3)
Acriflavine	$C_{14}H_{14}ClN_3$	259.7	447	8048-52-0	46000	9.43 × 6.16 × 6.06
Auramine-O	$C_{17}H_{21}N_3HCl$	303.8	432	2465-27-2	41000	13.77 × 6.42 × 4.74
Azure-I	$C_{15}H_{16}ClN_3S$	305.8	646	531-55-5	52010	13.58 × 5.93 × 4.35
Pyronin-Y	$C_{17}H_{19}ClN_2O$	302.8	547	92-32-0	45005	13.37 × 6.02 × 4.37

with UVProbe 2.31 software. The samples were scanned in the wavelength range of 400–800 nm in the absorbance mode. The absorbance values at the λ_{max} (the wavelength corresponding to the maximum absorbance) were converted to the concentration using the predetermined calibration curves for each dye. The amount of dye adsorbed per unit mass of the SAP (q), % removal efficiency of the hydrogel (RE) and the partition coefficient (PC) are calculated using the following relations:

- $q = \frac{(C_0 - C)V}{W}$ = amount of dye adsorbed per unit mass of the SAP at time t (mg/g)
- $q_e = \frac{(C_0 - C_e)V}{W}$ = amount of dye adsorbed per unit mass of the SAP at equilibrium (mg/g)
- $\mathrm{RE}(\%) = \left(\frac{C_0 - C}{C_0}\right) \times 100$ = removal efficiency
- $\mathrm{PC} = \frac{C_0 - C}{C}$ = partition coefficient

where C_0 and C are the initial dye concentration and dye concentration at time t (mg/L). C_e is the equilibrium dye concentration (mg/L), V is the volume of dye solution (L) and W is the weight of the dry SAP (g). The amount of dye adsorbed per unit mass of the SAP (q), removal efficiency (RE) and partition coefficient (PC) indicate the efficacy of an adsorbent for the removal of dissolved materials from the solution.

RESULTS AND DISCUSSION

Fourier transform infrared spectroscopy

FTIR spectra of the SAPs are reported elsewhere.[24] The characteristic peaks of C=O of acrylamide and N—H stretching of acrylamide group were observed at 1667 and 3436.5 cm^{-1} and the peak at 1561 cm^{-1} was absent in AM-1 but observed in AA/SA/AM-1 and AA/SA-1 indicating the presence of COO^-Na^+ groups.

Equilibrium swelling capacity of the superabsorbents

The swelling of the SAPs followed the first order kinetics, given by following equation,

$$S = S_{eq}[1 - \exp(-k_s t)] \tag{2}$$

S and S_{eq} are the swelling capacities of the SAP at any time t and at equilibrium, and k_s is swelling rate constant. The equilibrium swelling capacities and the swelling rate constants of various SAPs are listed in Table 2. Among the SAPs, the equilibrium swelling capacity of the SAPs decreases with an increase in the mol % of AM. Increase in the non-ionic AM repeat units in the SAP results in the reduction in the number of anionic repeat units. As a result, the osmotic pressure difference between the polymer and the surrounding medium diminishes resulting in the lowering of the swelling capacity. The detailed description of the swelling and deswelling of the SAPs is given elsewhere.[24]

Adsorption of dyes on the SAPs

After the addition of the dry SAP to the dye solution, the swelling of the SAP and the adsorption of the dye take place simultaneously. The swollen polymer becomes colored by adsorbing the dye from the aqueous solution and the dye solution becomes clearer due to the removal of the dye. The SAPs adsorb dyes at a much faster rate in the beginning and the amount of dye adsorbed levels off after a certain period of time. By this time, the SAP has adsorbed the maximum possible amount of the dye and further de-coloration of the dye solution does not take place. The amount of dye adsorbed per unit mass of the SAP at equilibrium, q_e, depends on the content of AM in the SAP, initial concentration of dye, and the amount of SAP employed for the adsorption.

In this study, four cationic dyes, namely Acriflavine, Auramine-O, Azure-I, and Pyronin-Y were studied for adsorption. The adsorption of Azure-I on SAPs with 20, 40, 60, and 80 mol % AM was carried out. The superabsorbent polymers with the highest

TABLE II
Kinetic Parameters for the Swelling of Various SAPs

SAP	AA/SA (mol %)	AM (mol %)	S_{eq} (g of water/g of SAP)	k_s (h^{-1})
AA/SA/AM-1	80	20	621.7	0.408
AA/SA/AM-2	60	40	496.1	0.454
AA/SA/AM-3	40	60	285.0	0.892
AA/SA/AM-4	20	80	215.0	0.530
AM-1	0	100	30.1	0.902

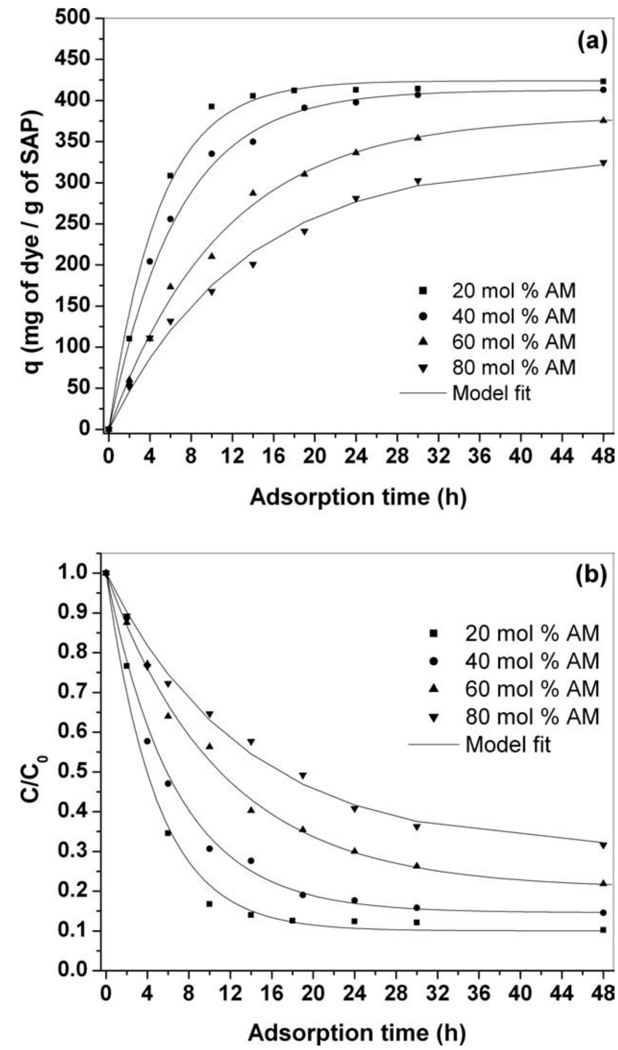

Figure 1 Effect of AM content in the SAPs on the adsorption of Azure-I. Variation in the (a) amount of dye adsorbed per unit mass of the SAP and (b) normalized residual concentration with adsorption time.

and the lowest swelling capacities (20 and 80 mol % AM content, respectively) were used to study the adsorption of cationic dyes Auramine-O, Azure-I, and Pyronin-Y. The effect of the initial concentration of the dye on the adsorption was investigated by taking various concentrations of Azure-I with the same amount of AA/SA/AM-2 (40 mol % AM). The effect of the amount of the adsorbent was observed by taking different weights of AA/SA/AM-2 (40 mol % AM) with the same initial concentration of Acriflavine.

First order model for the adsorption of dyes

Kinetic analysis of the adsorption of dyes on SAPs was done using a first order model.

$$\frac{dq}{dt} = k_a(q_e - q) \qquad (3)$$

where k_a is the rate constant for the adsorption (h^{-1}). At time $t = 0$, $C = C_0$, $q = 0$, thus

$$q = q_e[1 - \exp(-k_a t)] \qquad (4)$$

The experimental data for the adsorption of four cationic dyes, namely Azure-I, Acriflavine, Pyronin-Y, and Auramine, was fitted by using eq. (4).

Effect of AM content on the adsorption of dyes

To investigate the effect of AM content in the SAPs on the adsorption of cationic dyes, the adsorption of Azure-I was carried out with the SAPs having 20, 40, 60, 80, and 100 mol % AM. The adsorption of Auramine-O and Pyronin-Y was also conducted on the SAPs having the highest and the lowest swelling capacities, i.e., on the SAPs having 20 and 80 mol % AM, respectively.

Figure 1(a) shows the increase in amount of Azure-I adsorbed per unit mass of the SAPs (q), and Figure 1(b) shows the reduction in normalized residual concentration (C/C_0) of Azure-I with adsorption time. The q and C/C_0 for the adsorption of Auramine-O and Pyronin-Y are plotted in Figures 2(a,b) and 3(a,b), respectively. The adsorption of the dyes followed the first order kinetics and the rate parameters are listed in Table 3. It is evident from the Figure 1(a) and Table 3 that as the mol % of AM increases in the SAPs, the amount of dye adsorbed at the equilibrium per unit mass of the SAP (q_e) decreases. The AA/SA/AM superabsorbents have two kind of repeat units, anionic and non-anionic. AA/SA incorporates the anionic repeat units while AM is responsible for the nonionic repeat units in the SAPs. The cationic dyes are adsorbed by the SAPs due to the electrostatic attraction forces between the cationic dye molecules and the anionic groups in the SAPs. With the increase in the mol % of AM, the nonionic content of the polymer increases and the number of anionic repeat units reduces. Thus the amount of the dye adsorbed at equilibrium reduces due to the decreased number of interacting sites in the adsorbent. This behaviour is observed for all the three cationic dyes used in the present study (Table 3). The SAP with 100 mol% AM does not have any anionic repeat units and, therefore, it did not adsorb any of the cationic dyes in the present study. These results are in accordance with the studies of radiation synthesized copolymeric hydrogels of acrylamide, *N*-vinyl-2-pyrrolidone, itaconic acid, and maleic acid,[1] TMPTA, and BDMA crosslinked superabsorbent acrylamide/maleic acid hydrogels,[9] γ radiation synthesized acrylic/maleic acid hydrogels,[14] acrylamide-acrylic acid hydrogels synthesized by ^{60}C-γ radiation[15] and glutaraldehyde and divinylbenzene crosslinked

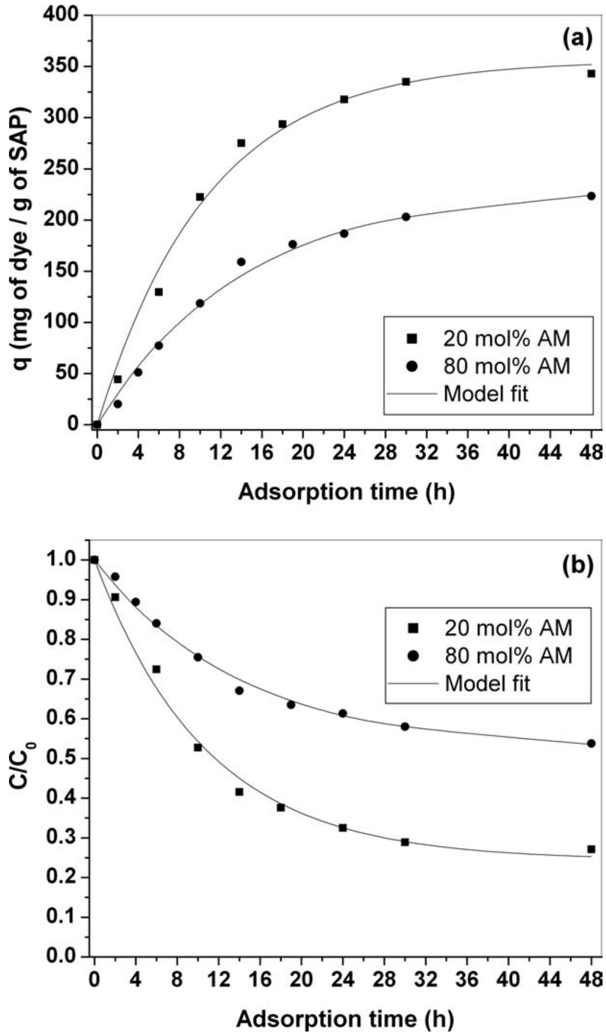

Figure 2 Effect of AM content in the SAPs on the adsorption of Auramine-O. Variation in the (a) amount of dye adsorbed per unit mass of the SAP and (b) normalized residual concentration with adsorption time.

acrylamide and sodium acrylate hydrogels,[16] where the amount of dye adsorbed per unit mass of dry hydrogel increased with increase in the anionic monomer content of the hydrogel at equilibrium. Experiments were conducted using anionic dyes like Orange G and Remazol Brilliant Blue R. Owing to the strong electrostatic repulsive forces between the anionic groups, the anionic dyes were not adsorbed by any of the SAPs. Similar results have been reported for the acrylamide/maleic acid hydrogels where anionic dyes Orange-II, Azocamine B, Alizarine yellow G and Erytrosin B were not adsorbed by the hydrogels.[9]

Effect of initial concentration of dye

The SAP AA/SA/AM-2 (40 mol% AM) has been used to investigate the effect of initial dye concentration on the adsorption of dyes. For this purpose, the same amount of AA/SA/AM-2 was added to the aqueous solutions of Azure-I (25, 100, and 250 mg/L). The variation in the amount of the dye adsorbed per unit mass of the SAP (q) and the normalized residual concentration of dye solution (C/C_0) with the adsorption time are plotted in Figure 4(a,b), respectively. Table 3 lists the rate parameters obtained by fitting the first order adsorption model to the experimental data. With the increase in the initial concentration of the dye, the amount of dye adsorbed per unit mass of the SAP at equilibrium (q_e) increased and the normalized residual concentration decreased. The removal efficiency (RE (%)) and the partition coefficient (PC) also decreased with increasing initial concentration of dye. Similar results have been obtained for the uptake of metal ions by acrylic acid-itaconic acid hydrogels.[7]

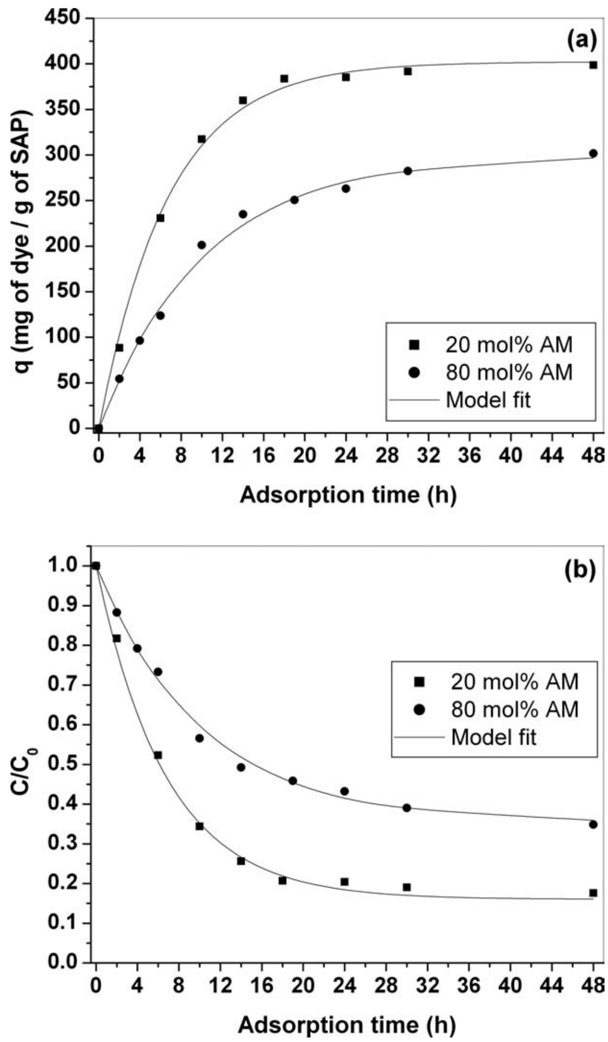

Figure 3 Effect of AM content in the SAPs on the adsorption of Pyronin-Y. Variation in the (a) amount of dye adsorbed per unit mass of the SAP and (b) normalized residual concentration with adsorption time.

TABLE III
Kinetic Parameters for the Adsorption of Cationic Dyes on the SAPs

Mol % of AM	q_e (mg/g)	k_a (h^{-1})	RE (%)	PC
Effect of AM content on the adsorption of Azure-I; Initial concentration dye = 100 mg/L; Amount of the SAP = 0.1 g				
20	423.95	0.206	88.35	7.58
40	412.61	0.150	85.41	5.85
60	381.21	0.089	76.24	3.21
80	330.64	0.076	66.13	1.95
Effect of AM content on the adsorption of Auramine; Initial concentration dye = 100 mg/L; Amount of the SAP = 0.1 g				
Mol % of AM	q_e (mg/g)	k_a (h^{-1})	RE (%)	PC
20	355.77	0.093	74.33	2.90
80	330.64	0.076	66.13	1.95
Effect of AM content on the adsorption of Pyronin-Y; Initial concentration dye = 100 mg/L; Amount of the SAP = 0.1 g				
Mol % of AM	q_e (mg/g)	k_a (h^{-1})	RE (%)	PC
20	402.24	0.148	85.76	6.02
80	299.70	0.100	59.82	1.49
Effect of initial dye concentration; Amount of the SAP (AA/SA/AM-2) = 0.1 g; Dye used = Azure-I				
C_0 (mg/L)	q_e (mg/g)	k_a (h^{-1})	RE (%)	PC
25	100.59	0.114	95.32	20.36
100	412.61	0.150	85.41	5.85
250	1169.12	0.095	84.99	5.66
Effect of the amount of SAP; SAP = AA/SA/AM-2 (40 mol% AM); Dye = Acriflavine (100 mg/L)				
W (g)	q_e (mg/g)	k_a (h^{-1})	RE (%)	PC
0.0250	506.09	0.105	25.71	0.35
0.1000	418.19	0.074	84.22	5.34
0.2500	191.53	0.101	95.96	23.74

Effect of the amount of the superabsorbent polymer

Different amounts of the SAP AA/SA/AM-2 (40 mol% AM) were used to study the effect of adsorbent dosage on the adsorption of Acriflavine. For this purpose, 0.025, 0.100, and 0.250 g of dry SAP was added to 500 mL of 100 mg/L of Acriflavine solutions. The variation in the amount of the dye adsorbed per unit mass of the SAP and the normalized residual dye concentration against time are plotted in Figure 5(a,b), respectively, and the rate parameters are listed in Table 3. With increasing loading of the adsorbent, the residual normalized concentration decreased, removal efficiency (RE) increased, and the partition coefficient increased but the amount of dye adsorbed per unit mass of the SAP decreased. In fact, the RE% and PC were the lowest for the 0.025 g of the dry SAP (25.71% and 0.35, respectively) and were the highest for 0.250 g of the dry SAP (95.96% and 23.74, respectively) among all the experiments. This indicates that the higher amount of the adsorbent resulted in higher number of anionic adsorption sites, which was responsible for the higher removal efficiency of the dye from the aqueous solutions.

Effect of different dyes

Adsorption of cationic dyes Auramine-O, Azure-I, and Pyronin-Y on the SAPs having 20 and 80 mol % AM was investigated. The SAP having 20 mol % AM adsorbed the dyes in order of Azure-I > Pyronin-Y > Auramine-O and the values of q_e are 423.9, 402.2, and 356.0 mg of dye/g of SAP, respectively [Fig. 6(a), Table 3). The same order was followed by the dyes

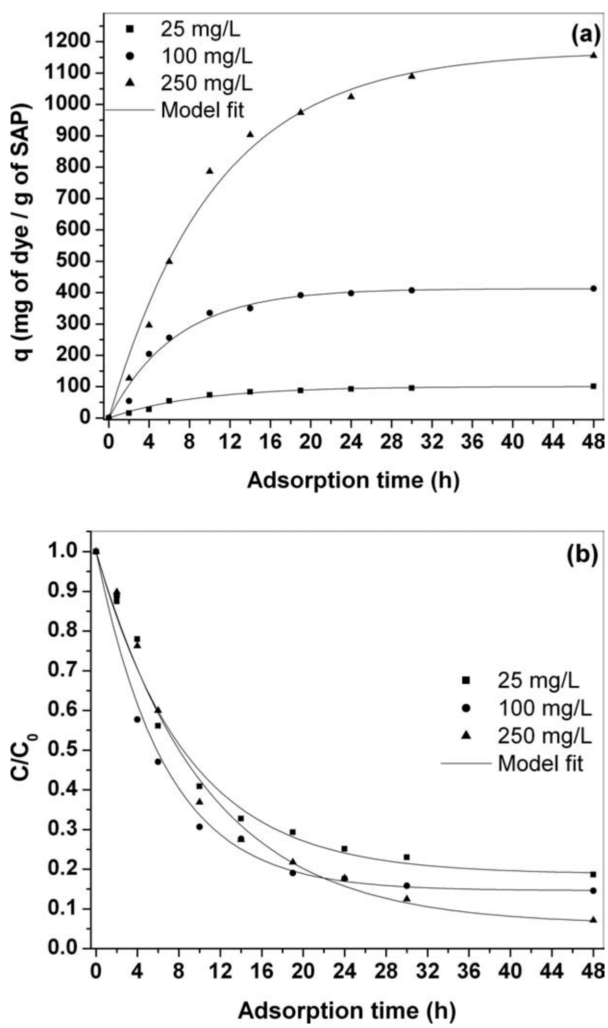

Figure 4 Effect of initial concentration of Azure-I on the adsorption of AA/SS/AM-2 (40 mol % AM). Variation in the (a) amount of dye adsorbed per unit mass of the SAP and (b) normalized residual concentration with adsorption time.

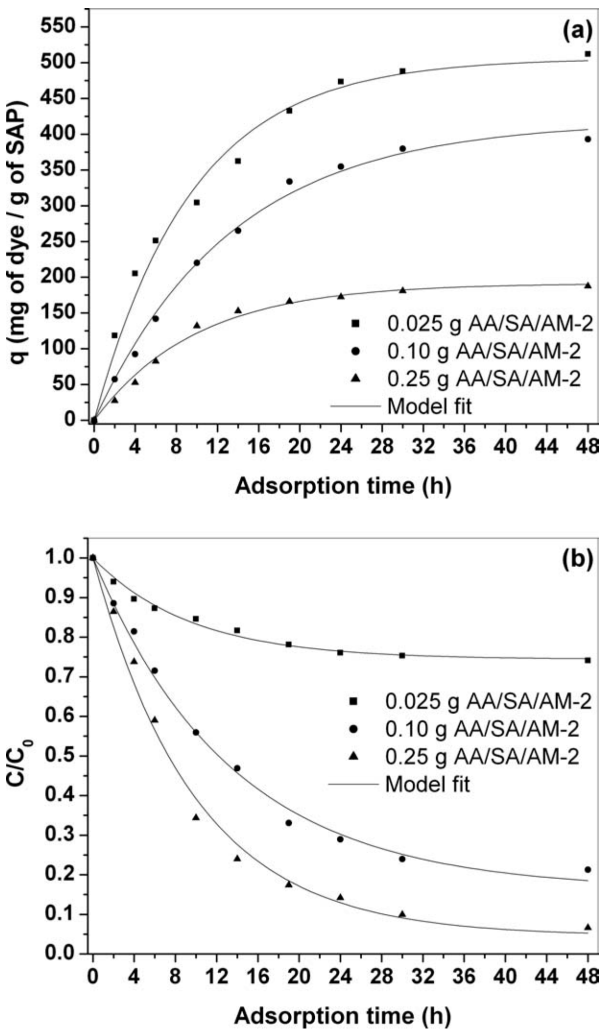

Figure 5 Effect of amount of AA/SA/AM-2 (40 mol % AM) on the adsorption of 100 mg/L Acriflavine. Variation in the (a) amount of dye adsorbed per unit mass of the SAP (b) normalized residual concentration with adsorption time.

adsorbed by the SAP with 80 mol % AM with the q_e values of 330.6, 299.1, and 231.7 mg of dye/g of SAP [Fig. 6(b), Table 3]. The dye removal efficiency, partition coefficient, and the amount of the dyes adsorbed per unit mass of SAP were in the order of Azure-I > Pyronin-Y > Auramine-O. Molecular sizes of the dyes were determined using ChemOffice (version 4.5) software. An inbuilt MOPAC program was used to carry out the energy minimization of dye structures[25] and the molecular sizes of the dyes, represented as length × height × depth, were determined (see Table 1). Although the molecular sizes and the molecular weights of the three dyes are almost the same, the amount of the dye adsorbed at equilibrium differs. These differences may arise due to the combined effect of the structure of the dye, the way the dye molecule enters the pores, and the canonical resonances in the dye molecules.

CONCLUSIONS

Ethylene glycol dimethacrylate (EGDMA) cross-linked poly(acrylic acid-*co*-sodium acrylate-*co*-acrylamide) superabsorbents were synthesized by inverse suspension polymerization. The equilibrium swelling capacities of the SAPs varied with the mol % of acrylamide in the superabsorbents. The adsorption of four cationic dyes, namely Acriflavine, Auramine-O, Azure-I, and Pyronin-Y, and anionic dyes on the SAPs was investigated. None of the SAPs adsorbed the anionic dyes and SAP with 100 mol% AM did not adsorb any dye. Due to the strong electrostatic attraction between the cationic dyes and anionic repeat units of the SAPs, all the cationic dyes were adsorbed. The kinetics of the adsorption of cationic dyes was modeled by the first order models and the rate parameters were determined. The amount of dye adsorbed per unit mass of the SAP at equilibrium and removal efficiency decreased with an

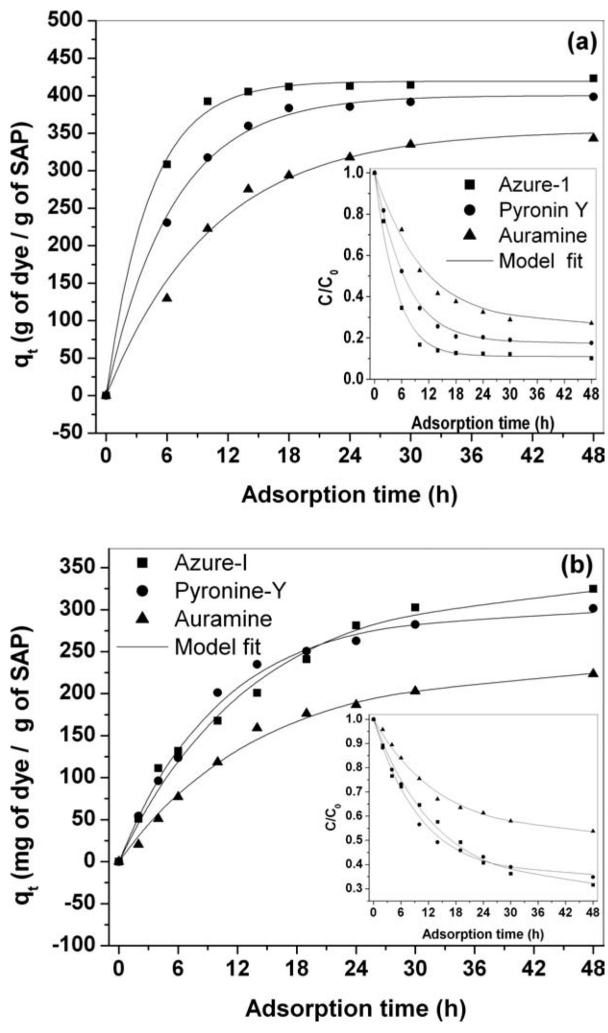

Figure 6 Comparison of adsorption of different cationic dyes by (a) AA/SA/AM-1 (20 mol % AM) (b) AA/SA/AM-4 (80 mol % AM). The inset shows the variation of the normalized concentration with time.

increase in non-ionic monomer content in the SAPs. The amount of dye adsorbed per unit mass of SAP increased with an increase in the initial concentration of the dye but the removal efficiency and partition coefficients decreased. An increase in the amount of adsorbent, for a fixed initial dye concentration, resulted in the lower amount of dye adsorbed per unit mass of the SAP but higher removal efficiency and partition coefficients.

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