

# Synthesis and Characterization of Na-Alginate/Acrylamide Hydrogel and Its Application in Dye Removal

Y. H. Gad, R. O. Aly, S. E. Abdel-Aal

National Center for Radiation Research and Technology, Polymer Chemistry Department, P.O. Box 29, Nasr City, Cairo, Egypt

Received 18 March 2010; accepted 12 August 2010

DOI 10.1002/app.33269

Published online 7 December 2010 in Wiley Online Library (wileyonlinelibrary.com).

**ABSTRACT:** Sodium-alginate/acrylamide (Na-alginate/AAM) cross-linked hydrogel was prepared by using gamma irradiation. The gel content and the water absorbency behavior of the obtained hydrogel were investigated. The thermal and morphological properties of the prepared hydrogel were studied using thermogravimetric analysis and scanning electron microscopy. The thermal stability of Na-alginate significantly changed when mixed with AAM. The adsorption of basic violet 7 dye on the prepared

hydrogel was studied under different experimental conditions. The adsorption process for the basic dye was proven pH dependent. The maximum amount adsorbed (mg/g) of the Na-alginate/AAM for the dye was found to be 78.1.0 mg/g at pH 9.0. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 120: 1899–1906, 2011

**Key words:** sodium alginate; acrylamide; irradiation; basic dye; adsorption

## INTRODUCTION

Sodium alginate [Na-Alg,  $(C_6H_7O_6Na)_n$ ] is a linear anionic polysaccharide occurring naturally in brown algae and belongs to carbohydrate group of polymers. It is mainly composed of  $\beta$ -D-1,4-mannuronic acid (M unit) and  $\alpha$ -L-1,4-guluronic acid (G unit).<sup>1–3</sup> The residues may vary widely in composition and sequence and are arranged in a pattern of blocks along the chain. The homopolymeric regions of M and G blocks are interspersed with regions of alternating structure (MG blocks).<sup>4–7</sup> The composition and extent of the sequences and the molecular weight determine the physical properties of the alginates. As a natural biopolymer, alginate has found increasing biotechnological and biomedical applications in view of its advantages, such as good biocompatibility, biodegradability, nontoxicity, nonimmunogenicity, chelating ability, and the possibility of chemical modification. One of the most important and useful properties of alginates is the ability to form gel in the presence of some multivalent metal ions such as calcium.<sup>8,9</sup> Grafting as a technique for modifying chemical and physical properties of natural polymers has attracted much interest.<sup>10,11</sup>

There are more than 100,000 kinds of commercially available dyes with over  $7 \times 10^5$  tons of dyes produced annually.<sup>12</sup> It is estimated that 2% of dyes produced annually are discharged in effluent from man-

ufacturing operations, whereas 10% is discharged from textile and associated industries.<sup>13</sup> Adsorption process/technique is widely used in the removal of contaminants from wastewaters. Liquid–solid adsorption operations are concerned with the ability of certain solids to preferentially concentrate specific substances from solution onto their surfaces.<sup>14</sup>

Because the cationic dyes used mostly in textile industry have large molecules, their adsorption onto activated carbon and other adsorbents may not be optimal. Polymeric adsorbents and hydrogel systems may offer distinct advantages in specifically defined cases. Hydrogels are cross-linked hydrophilic polymers that are swollen in water usually to equilibrium and they combine glassy behavior (in their dry state) with elasticity (when sufficient water is adsorbed). The behavior of highly swollen hydrogels is, of course, a function of the network characteristics (such as degree of swelling, diffusion coefficient, cross-link density, mesh size, etc.), which in turn is connected with chemical structures. Recently, a number of studies were reported with regard to the dye removal processes using various adsorbents.<sup>15,16</sup> Some groups have used various adsorbents for the removal of acidic and basic dyes from aqueous solutions.<sup>17</sup> In previous work, the swelling, thermal, and mechanical properties of poly(vinyl alcohol)/sodium alginate hydrogels synthesized by electron beam irradiation, metal sorption, and swelling characters of acrylic acid and sodium alginate based hydrogels synthesized by gamma irradiation were studied.<sup>18,19</sup> This work investigates the optimal conditions of adsorption of the basic violet 7 dye on the hydrogel sodium-alginate/acrylamide (AAM) prepared by gamma irradiation.

Correspondence to: Y. H. Gad (yasser2uk@yahoo.com).

## EXPERIMENTAL

### Materials

Acrylamide 99% produced by Aldrich, M.wt. = 71.08 was used without any purification. Na-alginate powder was supplied by Mulkerei, Germany. Pollutant: Basic dye: basic Violet 7 (Astrazon Red 6B), (C<sub>24</sub> H<sub>30</sub> N<sub>2</sub> Cl<sub>2</sub>) M. wt. = 416.

### Preparation of hydrogel

AAM containing 4, 8, and 12% Na-alginate (w/w) hydrogels were prepared by adding 15, 10, and 5 g dry AAM to 0.6 g dry Na-alginate, respectively; then, the mixture was dissolved in 30 mL distilled water and left for 3 hr in a water bath at 60°C until the formation of a homogenous paste. The paste was then transferred into a 20 cm<sup>3</sup> tightly closed glass tube and thereafter irradiated by gamma rays.

### Gamma radiation source

Irradiation of samples was carried out by using <sup>60</sup>Co gamma source installed at the National Center for Radiation Research and Technology (NCRRT), Egypt, with a dose rate of 1.19 Gy/sec with doses ranged from 5 to 30 kGy.

### Infrared measurement

Analysis by infrared spectroscopy was carried out using Mattson 1000, Pye-Unicam, England, in the range from 400 to 4000 cm<sup>-1</sup>.

### Thermogravimetric analysis

Shimadzu thermogravimetric analysis (TGA) system type TGA-50 H was used. The measurements were carried out in nitrogen atmosphere with a flow rate of 40 mL/min. The temperature range was from ambient to 700°C at a heating rate of 10°C/min.

### Scanning electron microscopy

The surface morphology was indicated by scanning electron microscope, and the micrographs were taken with a JSA-5400 Jeol (Japan).

### Measurements

#### Swelling measurement

The dried hydrogels of known weights were immersed at 25°C in various pH solutions until the swelling equilibrium took place (almost 24 hr). The hydrogels were removed, blotted quickly with absorbent paper, and then weighed. The following equation was used to determine water absorbency.

$$\text{Water absorbency} = (W_s - W_g)/W_g \quad (1)$$

where,  $W_s$  and  $W_g$  represent the weights (g) of wet and dry gel films (g), respectively.

#### Gel fraction of the prepared hydrogel

The gel fraction was calculated according to the following equation:

$$\text{Gel fraction (\%)} = (W_d/W_i) \times 100 \quad (2)$$

where  $W_d$  is the weight of insoluble part after extraction with water and  $W_i$  is the weight of dried gel before swelling.

#### Dye removal

*Adsorption studies.* The adsorption of basic violet 7 on the Na-alginate/AAM hydrogel was investigated in a batch system. Solutions of the dye, containing 25–700 mg/L, were prepared in purified water. The concentration range of the dye was prepared from a stock solution. Adsorption experiments were performed at 25°C ± 2°C for 8 hr for the sample of 50 mL and 1.0 g of Na-alginate/AAM. The unadsorbed dye in the supernatant was analyzed using a double-beam UV-vis spectrophotometer, Unicam UV-vis spectrometer (UV 2), United Kingdom, at the wavelength 540 nm. Amount of dye concentration adsorbed per unit mass of the hydrogel (mg/g) was calculated by using the following equation<sup>20</sup>:

$$q \text{ (mg/g)} = (C_0 - C) V/m \quad (3)$$

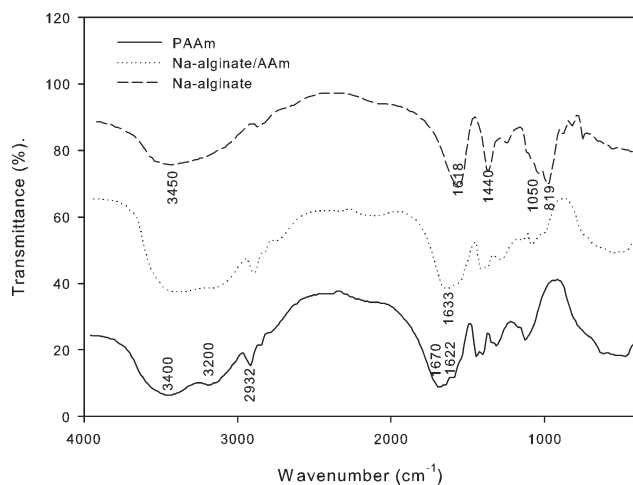
where  $q$  is the amount of dye adsorbed per adsorbent weight unit (mg/g),  $C_0$  and  $C$  are the concentration of the dye in the aqueous phase (mg/L) before and after the adsorption period, respectively,  $V$  is the volume of the aqueous phase (L), and  $m$  is the amount of the dry hydrogel used (g).

The effect of medium pH on the amount adsorbed (mg/g) by Na-alginate/AAM hydrogel was investigated within the pH range 2.0–9.0 at 25°C. The batch experiments were conducted using different amounts of Na-alginate/AAM hydrogel between 0.25 and 2.0 g/L at 25°C, pH 9.0. Sodium chloride (NaCl) was used as a background electrolyte up to 1.0M to detect the influence of ionic strength on the dye removal.

## RESULTS AND DISCUSSION

### Infrared analysis

Fourier transform infrared spectroscopy is of importance to study the molecular structure. The width and intensity of spectral bands, as well as the position of peaks are all sensitive to environmental



**Figure 1** Fourier transform infrared spectra of PAAm, Na-alginate, and Na-alginate/AAM (with 4% Na-alginate irradiated at 20 kGy).

changes and the conformations of the macromolecule on a molecular level. The Fourier transform infrared spectrum of the prepared hydrogel is different from that of the pure polymers. Figure 1 shows the infrared spectra for sodium alginate (Na-alginate), sodium alginate/AAM hydrogel, and polyacrylamide (PAAm) within the wavelength range of 4000–400  $\text{cm}^{-1}$ . Sodium alginate shows a broad peak at 3450  $\text{cm}^{-1}$  for the OH group, two peaks at 1618 and 1440  $\text{cm}^{-1}$  for the  $\text{COO}^-$  group, and one sharp peak at 1050  $\text{cm}^{-1}$  for the C-O group. Meanwhile, the characteristic peak of sodium alginate appeared at 819  $\text{cm}^{-1}$  for Na-O was absent in the hydrogel spectra. PAAm exhibits bands at 3400 and 3200  $\text{cm}^{-1}$  assigned to stretching vibration of N-H, 1670  $\text{cm}^{-1}$  for C=O stretching, and 1622  $\text{cm}^{-1}$  for N-H bending. The band at 2932  $\text{cm}^{-1}$  for C-H stretching and within 1500–1300  $\text{cm}^{-1}$  for C-H bending were also detected. The spectra of the (Na-alginate/AAM) hydrogel are characterized by the presence of the absorption bands typical for the pure components. However, the hydrogel formation is emphasized by the hydrogen bond formation between  $\text{COO}^-$  groups of sodium alginate and  $-\text{CONH}_2$  groups of AAm by the shift at 1633  $\text{cm}^{-1}$  assigned to the asymmetrical stretching vibration of  $\text{COO}^-$  groups coupled with the peaks at 1670 and 1622  $\text{cm}^{-1}$ . Furthermore, the bands of PAAm centered at about 3400 and 3200  $\text{cm}^{-1}$  accounted for the stretching vibration of  $\text{NH}_2$  groups that involve in both inter- and intramolecular hydrogen bonds, broadened, and coupled with the OH band of sodium alginate at 3450  $\text{cm}^{-1}$ .<sup>21,22</sup> Scheme 1 represents the mechanistic proposal of PAAm/Na-alginate hydrogel formation using gamma irradiation.<sup>23</sup>

### Thermal stability of PAAm/Na-alginate

The thermal stability of Na-alginate and the irradiated PAAm and PAAm/Na-alginate copolymer was

investigated using TGA (Fig. 2). It is obvious that there is a significant change in the thermal stability of Na-alginate when blended with AAm. The thermal stability of cross-linked PAAm is higher than that of both Na-alginate and PAAm/Na-alginate. The results show that the low thermal stability of alginate accelerated the thermal degradation of PAAm. To determine the thermal stability of PAAm, Na-alginate, and (PAAm/NaAlg) hydrogel, the temperature for the maximum weight loss ( $T_{\text{max}}$ ) and the temperature for half-life ( $T_{1/2}$ ) were found directly from its thermograms given in Figure 2.

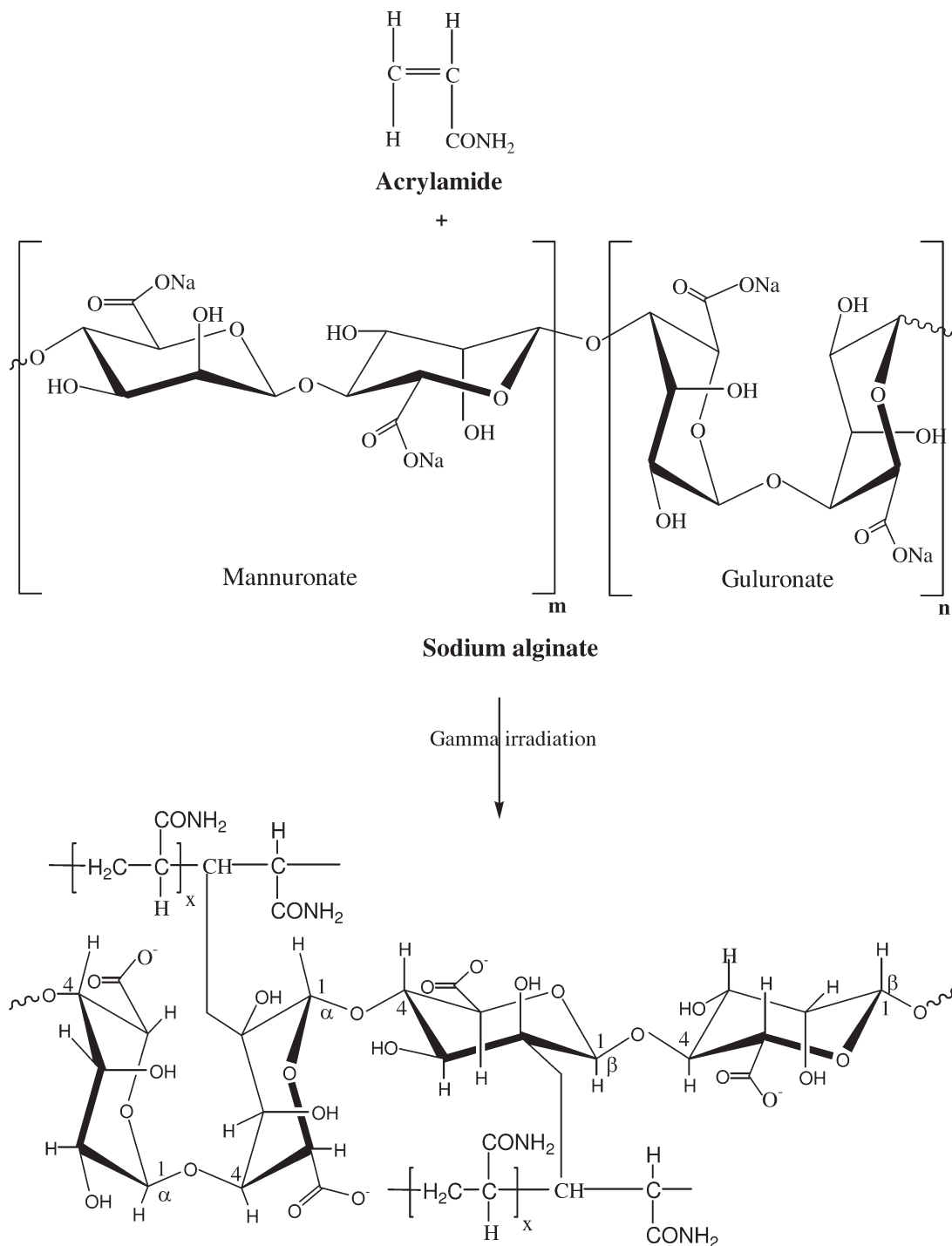
PAAm gave 25% residue at 600°C. The temperature for maximum weight loss of PAAm is  $T_{\text{max}} = 396^\circ\text{C}$  and  $T_{1/2} = 418^\circ\text{C}$ . There are three decomposition stages at 60–100°C, 259°C, and 396°C, the former being attributed to moisture loss. Weight loss at 259°C could be related to thermal processes involving the degradation of PAAm chains. The third stage at 396°C was indicative of the occurrence of more extensive thermal degradation processes.

For Na-Alginate, the temperature for maximum weight loss is  $T_{\text{max}} = 238^\circ\text{C}$ , and the temperature for half-life is  $T_{1/2} = 300^\circ\text{C}$ , and Na-Alginate gives 30% residue at 600°C. Na-Alginate shows a peak at 95°C attributed to the loss of water absorbed. The peak at 238°C indicates the occurrence of thermal degradation of intermolecular side chain. The thermogram of Na-Alginate shows two stages of weight loss. The first weight loss at  $\sim 60\text{--}120^\circ\text{C}$  was due to the loss of water, and the second weight loss started at about 238°C, implying that the occurrence of different extent of thermal degradation of Na-Alginate. The third weight loss at 578°C may be due to a more extensive thermal degradation. The greatest thermal degradation peak shifted to the higher temperature, resulting from the addition of PAAm to Na-Alginate.

TGA curve of (PAAm/Na-Alginate) hydrogel shows the greatest weight loss at 346°C. As shown in Figure 2, it is understood that the thermal stability and the temperature for maximum weight loss of (PAAm/Na-Alginate) hydrogel is between those of Na-Alginate and PAAm. When a less-stable species combined with a species of higher thermal stability, (PAAm/Na-Alginate) hydrogel gained new thermal characteristics.  $T_{\text{max}}$  for (PAAm/Na-Alginate) hydrogel is 346°C and  $T_{1/2}$  is 384°C.

### Scanning electron microscopy

Figure 3 shows the surface morphology of PAAm and PAAm/Na-alginate hydrogel prepared at the irradiation dose 20 kGy by using gamma irradiation source. From the present two micrographs, it can be seen that PAAm exhibited a smooth dense surface without any pores, whereas the prepared PAAm/



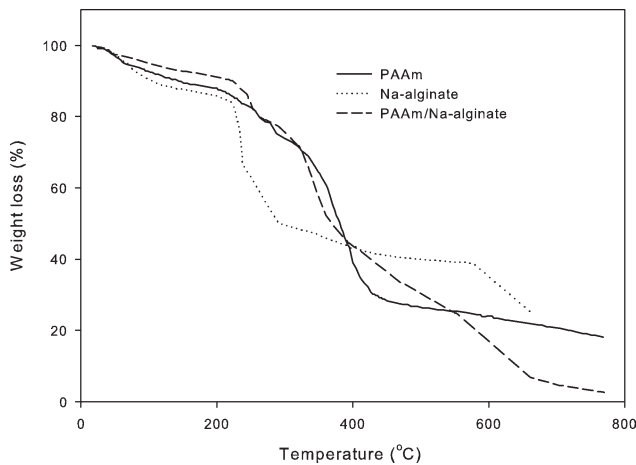
**Scheme 1** The mechanistic proposal of PAAm/Na-alginate hydrogel formation using gamma irradiation.

Na-alginate hydrogel possesses a macroporous surface structure.

#### Gel fraction of the prepared hydrogel

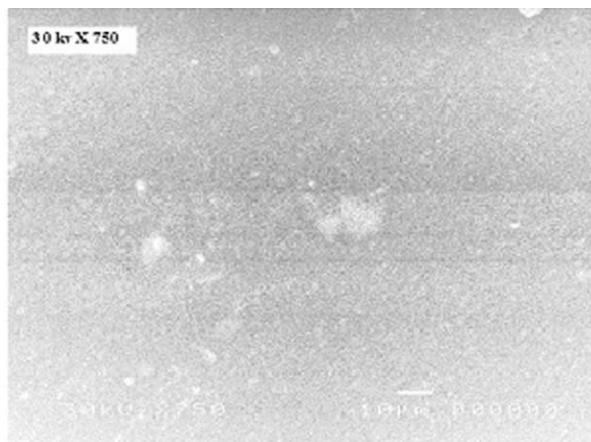
There are many factors affecting the polymer cross-linking, including polymer composition and irradiation dose. The impact of gel content of Na-alginate/AAM, prepared at different copolymer compositions

and irradiation dose, was investigated, as shown in Figure 4. It is clear that the gel content increases with increasing irradiation dose; this enhancing cross-linking to some extent. However, within the chosen range of Na-alginate content, 4–12%, as the Na-alginate content increases, the gel content was found to decrease at a similar radiation dose. It is well known that polysaccharide, such as Na-alginate, undergoes chain scission by radiation. Conversely,

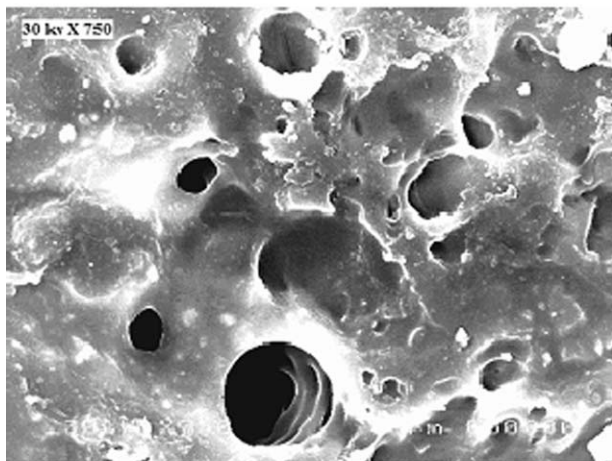


**Figure 2** The TGA thermograms of PAAm, Na-Alginate, and (AAm/Na-Alginate) hydrogel.

AAm undergoes cross-linking by irradiation. Therefore, the presence of sodium alginate may affect the overall polymer cross-linking process and, conse-

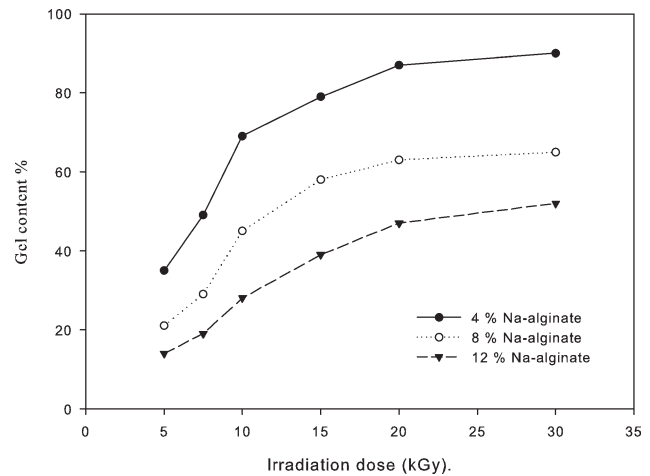


PAAm



PAAm/Na-alginate

**Figure 3** Scanning electron microscopy of both PAAm and PAAm/Na-alginate (irradiated at 20 kGy).

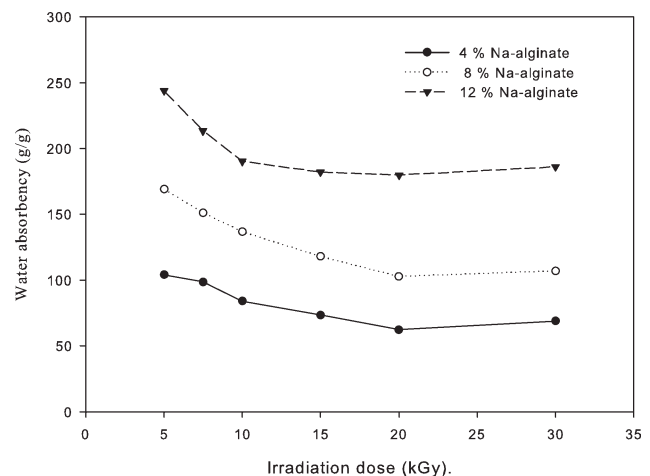


**Figure 4** Gel content (%) of Na-alginate/AAm hydrogel of different compositions prepared at various irradiation doses (kGy).

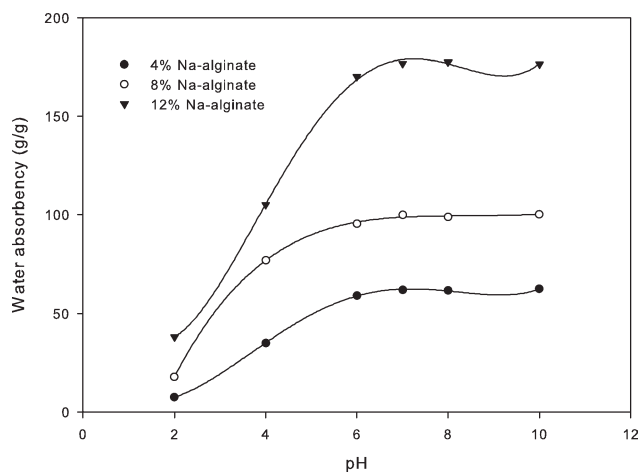
quently, may influence the properties of the obtained hydrogel. It should be noted that molecular movement of all hydrogel chains or their parts takes place in the presence of water, allowing the closing of distances between the macroradicals to recombine and enhance intermolecular and intramolecular recombination reactions.<sup>23</sup>

**Water absorbency**

Water absorption by the hydrogel prepared in different compositions was plotted as a function of irradiation dose (Fig. 5). The water absorbency was found to decrease as the irradiation dose increases. Additionally, water absorbency was also found to increase by increasing the Na-alginate content. The improvement in water absorbency with the increase in the hydrogel content can be mainly attributed to



**Figure 5** Water absorbency of Na-alginate/AAm hydrogel of different compositions prepared at various irradiation doses (kGy).



**Figure 6** Water absorbency of Na-alginate/AAm hydrogel of different pH prepared at irradiation dose (20 kGy).

the ionic character of Na-alginate. It is well known that progressive irradiation results in decrease in cross-linking, which leads to a general increase in swelling. Therefore, the hydrogel with 4% Na-alginate irradiated with 20 kGy was found to demonstrate nearly twice as much swelling as the records of 12% content irradiated at 10 kGy. Accordingly, Na-alginate/AAm hydrogel of 4% Na-alginate content irradiated with 20 kGy was admitted optimal.

Figure 6 shows the effect of pH on the water absorbency of hydrogel at different composition ratios. The data reveal that water absorbency is pH dependent. At pH 2, water absorbency of the hydrogel produced low levels. By increasing the pH value, water absorbency sharply increased until it reached a plateau (pH > 7). The results can be explained according to the following: at low pH, most of the carboxylate groups on the Na-alginate are converted to carboxylic acid groups, i.e., the ratio of the nonionized  $-\text{COOH}$  groups to the ionized ones ( $-\text{COO}^-$ ) increases. As a matter of fact, this process proceeds on the likelihood of the formation of inter- and intramolecular hydrogen bonds; as a result, the hydrophilicity of the copolymer decreases. On the other hand, at higher pH, the repulsion between the carboxylate groups, which contributes for swelling of the polymer, becomes predominant.<sup>24</sup>

### Effect of adsorption system parameters

#### Effect of initial concentration and pH on dye adsorption

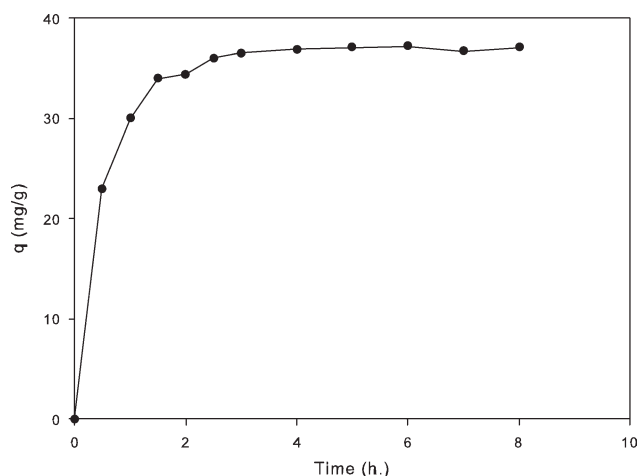
The initial dye concentration provides an important driving force to overcome all mass-transfer resistances of the dye molecules between the aqueous and solid phases. Hence, a higher initial concentration of dye is supposed to enhance the adsorption process. It should be noted that the initial dye concentration on an effluent was important because a given mass

of the adsorbent can adsorb only a fixed amount of dye. The experimental equilibrium adsorption isotherms of the basic violet dye onto the Na-alginate/AAm hydrogel are presented in Figure 7. The maximum amount of the dye adsorbed (m/g) by Na-alginate/AAm hydrogel was found to be 78.1 mg/g. Also, the data showed that, as the initial concentration of the dye increased from 25 to 700 mg/L, the amount adsorbed ( $q$ ) (mg/g) increased from 21.6 to 78.1 mg/g. This result showed that dye removal is basically concentration dependent.<sup>25</sup> It should be noted that the differences in amount adsorbed (g/g) of hydrogel for the basic dye should be caused by the chemical properties.

The effect of pH on the amount adsorbed (g/g) of the dye by Na-alginate/AAm hydrogel was tested in the pH range 2.0–9.0. As seen in Figure 7, at pH 2.0–4.0, the amount adsorbed (mg/g) was remarkably low probably due to weak electrostatic interactions between the molecules under condition of competition of the dye cations and  $\text{H}^+$  ions for the hydrogel active sites ( $-\text{COO}^-$ ,  $\text{Na}^+$ , and  $\text{NH}_2$ ). Furthermore, the amount of the dye adsorbed (mg/g) increased with increments in the pH value (pH 5.0–9.0); expectedly, the cationic functional groups of the tested basic dye interact with the anionic ( $-\text{COO}^- \text{Na}^+$ ) in sodium alginate as well as with and the free electrons of  $-\text{NH}_2$  in the AAm of the formed hydrogel.

#### Effect of amount of Na-alginate/AAm hydrogel and contact time

The dependence of dye adsorption on the applied amount of Na-alginate/AAm hydrogel was studied by varying the amount of adsorbent in the medium from 0.25 to 2.0 g while keeping other parameters constant, such as initial concentration of dye 500



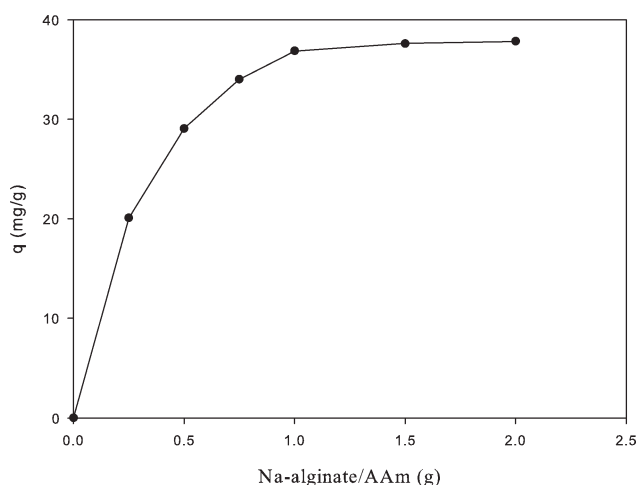
**Figure 7** Effect of initial concentration and pH on the amount of the basic violet dye adsorbed (mg/g) onto Na-alginate/AAm hydrogel. Temperature: 25°C; solution volume: 50 mL; amount of Na-alginate/AAm hydrogel: 1.0 g; and adsorption time: 3 hr.

mg/L, pH 9.0, and contact time 3 hr. As seen in Figure 8, the removal efficiency corroborates the fact that the higher the dose of adsorbent in the adsorption medium, the greater is the availability of exchangeable sites. The amount adsorbed (mg/g) of basic violet dye on Na-alginate/AAM hydrogel increased from 42.5 to 79.6 mg/g. As the adsorbent dose increases, surface area and available sites for the dye molecules also increase, and, consequently, better adsorption takes place. Thus, the amount of the dye adsorbed (mg/g) increased with the increase in the amount of Na-alginate/AAM hydrogel and reached an equilibrium value around 1.0 g of amount of the hydrogel.

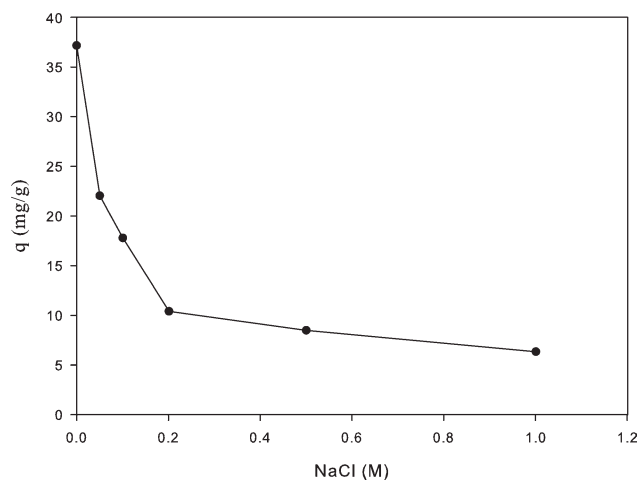
The equilibrium adsorption time of the basic violet dye on Na-alginate/AAM hydrogel was primarily investigated for 8 hr. Figure 8 demonstrates high initial slope for the adsorption curves indicating the rapid initial uptake. This may be because at the beginning of the sorption process all the reaction sites are vacant and, hence, the extent of removal is high. After a rapid initial uptake, there was a transitional phase in which the rate of uptake was rather slow till uptake reaches almost a constant value. Consequently, the adsorption of the dye reached an equilibrium value in 3 hr.

#### Effect of ionic strength

The adsorption of basic violet dye from aqueous solution onto Na-alginate/AAM hydrogel is primarily influenced by the surface charge of the adsorbent and the degree of ionization of the adsorptive sites.<sup>26</sup> Sodium chloride is currently used in textile dyeing processes because it promotes adsorption of dyes by the textile fibers. Figure 9 shows that NaCl produced an important decrease in the performance of the hydrogel, emphasizing that the sorption capacity of



**Figure 8** Effect of amount of Na-alginate/AAM hydrogel (g) and contact time (hr) on the amount of basic violet dye adsorbed at initial dye concentration: 500 mg/L; medium pH: 9.0; solution volume: 500 mL; amount of Na-alginate/AAM: 0.25–2.0 g; and temperature: 25°C.



**Figure 9** Effect of NaCl concentration (M) on the amount of the basic violet dye adsorbed (mg/g) onto Na-alginate/AAM hydrogel. Dye concentration: 500 mg/mL; temperature: 25°C; medium pH: 9.0; solution volume: 50 mL; amount of Na-alginate/AAM hydrogel: 1.0 g; and adsorption time: 3 hr.

the adsorbent depends on the ionic strength of the solution. By increasing ionic strength the adsorption of the dye was decreased, the electrical double layer surrounding the adsorbent surface is thought to be compressed and results in dramatic decrease in the basic violet dye adsorption.

## CONCLUSIONS

In this study, we prepared Na-alginate/AAM hydrogel with different composition ratios by using gamma irradiation. The hydrogel of 4% Na-alginate, irradiated at 20 kGy, was used for removal of basic violet 7 dye from its aqueous solution. Adsorption process was shown to be highly efficient for color removal from wastewaters because of its sludge-free clean operation, simplicity and flexibility of design, and complete removal of dyes even from dilute solutions. The percentage uptake of the basic violet dye on the prepared hydrogel was found to be concentration and pH dependent. The maximum amount adsorbed was 78.1 mg/g at pH 9.

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