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A study on water and dye sorption capacities of novel ternary acrylamide/sodium acrylate/PEG semi IPN hydrogels

Erdener Karadağ · Ömer Barış Üzüm

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Abstract Here, a novel ternary semi interpenetrating polymer networks (semi IPNs) have been synthesized. An effective role to develop mechanically strong polymeric materials has been through the preparation of interpenetrating polymer networks (IPNs). The highly swelling superabsorbent semi IPNs were prepared by introducing poly(ethylene glycol), (PEG) into an acrylamide/sodium acrylate, (AAm/SA) hydrogels. For swelling characterization, swelling experiments were performed in water at 25 °C, gravimetrically. Water uptake and dye sorption properties of AAm/SA hydrogels and AAm/SA/PEG semi IPNs were investigated as a function of chemical composition of the hydrogels. AAm/SA hydrogels and AAm/SA/PEG semi IPNs were used in experiments on sorption of water-soluble cationic dye such as "*Janus Green B*" (JGB). For sorption of JGB into AAm/SA hydrogels and AAm/SA/PEG semi IPNs were studied by batch sorption technique at 25 °C. For the analysis of sorption mechanism and for calculation of some binding parameters of JGB from aqueous solutions, some linearization methods such as Klotz, Scatchard, and Langmuir linearization methods have been used.

Keywords Acrylamide · Hydrogel · Swelling · Dye sorption · Interpenetrating polymer networks · Janus green B

Introduction

Hydrogels are macromolecular polymer networks immersed in a solvent, synthesized to exhibit large volumetric swelling in response to a variety of environmental stimuli. They are three-dimensional crosslinked polymeric structures that are able to swell in the aqueous environment. Materials with the ability to absorb water in high

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E. Karadağ · Ö. B. Üzüm (✉)
Department of Chemistry, Fen-Edebiyat Faculty, Adnan Menderes University, 09010 Aydın, Turkey
e-mail: ouzum@adu.edu.tr

amounts are again under investigation, because of their potential applications in bioengineering, biomedicine, food industry, communication technology, building industry, chromatography, water purification, separation processes, and agriculture. These polymers are often called polymer hydrogels and generally they are low crosslinked hydrophilic electrolytes [1–6]. An interpenetrating polymer networks (IPN) is defined as an intimate combination of two polymers, both in network form at least one of which is synthesized in the immediate presence of the other. IPNs are three-dimensional networks formed from homogeneous or heterogeneous polymers crosslinked in the presence of one another. Semi interpenetrating polymer network (semi IPNs) may be formed by the several methods. In these methods, one component is crosslinked, and the other is an uncrosslinked polymer. The mechanical properties of hydrogels may be improved by preparing semi IPNs, when the hydrogel network is prepared in the presence of a previously made polymer such as poly(ethylene glycol, polyacrylamide, poly(*N*-isopropyl acrylamide), poly(vinylpyrrolidone), poly(vinyl alcohol), or polyacrylic acid, etc. [7–9]. Water pollution due to organic compounds such as dye molecules and heavy metals remains a many serious environmental and health problem in living media. Dyes and aromatic molecules such as phenolic derivatives and polycyclic aromatic compounds are often found in the environment as a result of their wide industrial processing. Water pollutants in wastewater are known to be toxic and carcinogenic. Adsorption procedures are a way of one of the most widely used for pollutants such as dyes and organic compounds from industrial effluents. Adsorption is a well-known equilibrium separation process. Recently, new effective, efficient, and economic methods for water decontamination applications and for separation analytical purposes have been investigated [5]. The aim of this study is to investigate the swelling properties and sorption of dyes from aqueous solutions and to increase the water absorption capacity of a series of new interpenetrating polymeric networks that are crosslinked a “new” multifunctional crosslinker such as PEGDA and using a hydrophilic ionic/anionic comonomer such as SA with a polymer such as poly(ethylene glycol) (PEG) was selected. PEG has been used in a lot of biomedical applications for several purposes, for example, to provide better mechanical strength. PEG is water-soluble and is non-toxic for biological system. PEG-based hydrogels have good biocompatibility with biologically systems. PEG-based hydrogel systems have been used at many applications such as controlled release of some drugs as a biomaterial [10, 11] and as an adsorbent for removal of dyes [12, 13]. Then, for characterization, swelling properties and sorption properties of these hydrogel systems were studied.

Experimental

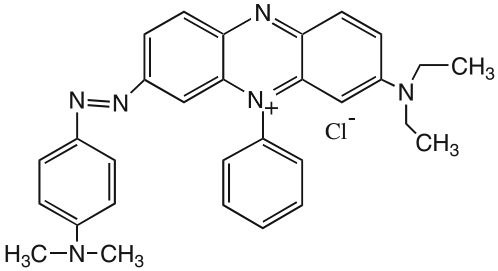
Acrylamide (AAm) as a monomer, sodium acrylate (SA) as a comonomer, poly(ethylene glycol) (PEG, $M_w = 4{,}000$), poly(ethylene glycol)diacrylate (PEG-DA, $M_n = 700$) as a crosslinker were supplied from Aldrich, Steinheim, Germany. The initiator, ammonium persulphate (APS) Merck, Darmstadt, Germany and the activator *N*,*N*,*N*′,*N*′-tetramethylethylenediamine (TEMED) was purchased from

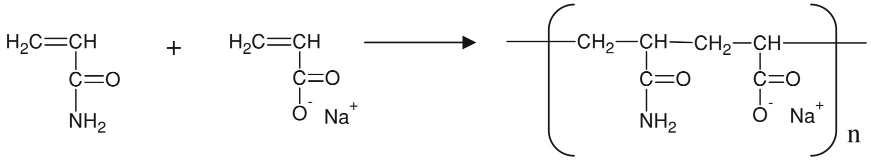
Merck Schuchardt, Germany. All chemicals were used as received. To prepare highly swollen acrylamide/sodium acrylate hydrogel systems, AAm weighing 1.0 g (14.07 mmol) was dissolved in 1.0 mL water. Then 0 mg, 10 mg/0.106 mmol, 20 mg/0.213 mmol, 30 mg/0.319 mmol, 40 mg/0.425 mmol, 50 mg/0.532 mmol, 60 mg/0.638 mmol, 70 mg/0.744 mmol, and 80 mg/0.851 mmol of SA were added to each AAm solutions, respectively. After these additions, for the synthesis, 0.040 mL/0.0640 mmol of PEGDA and 0.2 mL/0.0438 mmol aqueous solutions of APS (5.0 g APS/0.022 mol/100 mL water) and 0.25 mL/0.0167 mmol 1% concentration of TEMED were added to these aqueous solutions. The solutions were placed in PVC straws of 3-mm diameter. After gelation, fresh hydrogels obtained in long cylindrical shapes were cut into pieces of 3–4 mm in length. They were washed 4 days in distilled water to remove unreacted materials, blotted dry with filter paper, dried in air and vacuum, and stored for swelling studies. To prepare highly swollen AAm/SA/PEG semi IPNs, same method was used as mentioned above with addition of 0.25, 0.50, 0.75, and 1.00 g PEG to aqueous monomer solution per 1.0 g of AAm. For swelling studies, AAm/SA hydrogels and AAm/SA/PEG semi IPNs were accurately weighted and transferred into water. Water uptake with respect to time was obtained by periodically removing the samples from water; quickly blot drying and reweighing. The measurements were conducted at 25 ± 0.1 °C in a water bath. Batch sorption studies were proceeding in all sorption experiments. The sorption equilibrium included binding isotherm of the hydrogel/dye system, the determination of dye adsorbed, and percentage sorption. Cationic dye, Janus Green B (JGB) used in sorption studies, and some properties of JGB were given in Table 1. Solutions of JGB concentration range $1.25\text{–}2.00 \times 10^{-3}$ M in distilled water were prepared. AAm/SA hydrogels and AAm/SA/PEG semi IPNs containing 60 mg SA were used in a known volume of dye solution until equilibrium was reached. For SA effect on the dye sorption, aqueous solution of JGB of concentration of 2.00×10^{-3} M was used. After sorption, dye solution was separated by decantation from the hydrogels. Spectrophotometric method was applied to the dye solutions. Spectrophotometric measurements were carried out using a SHIMADZU UV 1601 model UV–VIS spectrophotometer at ambient temperature. The absorbances of these solutions were read at 660 nm for JGB [12, 14]. Distilled water was chosen as the reference. The equilibrium concentrations of the cationic dye solutions were determined by means of precalibrated scales.

Results and discussion

In this study, a novel semi interpenetrating network (semi IPN) hydrogel, composed of AAm with SA as comonomer, with PEG and a multifunctional crosslinker such as PEGDA was prepared. Highly swollen AAm/SA hydrogels and AAm/SA/PEG semi IPNs were synthesized by free radical solution polymerization (Fig. 1). Swelling experiments were performed in water at 25 °C, gravimetrically. A fundamental relationship exists between the swelling of a polymer in a solvent and the nature of the polymer and the solvent. The percentage swelling (*S*%) of the hydrogels in distilled water was calculated from the following relation,

Table 1 Some properties of Janus Green B

Name	Chemical formula	Molar mass (g mol^{-1})	λ_{max} (nm)	C.I. nr.
Janus Green B (JGB) (Union Green B)		511.07	660	11,050



Acrylamide (AAm) Sodium Acrylate (SA) Acrylamide/Sodium acrylate (AAm/SA)

Fig. 1 Representative chemical structures of monomers and AAm/SA copolymer

$$S\% = \frac{m_t - m_0}{m_0} \times 100 \tag{1}$$

where m_t is the mass of the swollen gel at time t and m_0 is the mass of the dry gel at time 0. The water intake of initially dry hydrogels was followed for AAm/SA hydrogels and AAm/SA/PEG semi IPNs crosslinked by PEGDA in water are shown in Figs. 2 and 3 as representative.

Figures 2 and 3 show that swelling increases with time up to certain level, and then levels off. This value of swelling may be called as the equilibrium percentage swelling ($S_{eq}\%$). $S_{eq}\%$ of AAm/SA hydrogels and AAm/SA/PEG semi IPNs is used for the calculation of swelling characterization parameters. $S_{eq}\%$ of AAm/SA hydrogels and AAm/SA/PEG semi IPNs are given in Tables 2 and 3. From Tables 2 and 3, it can be seen that swelling of AAm/SA hydrogels and AAm/SA/PEG semi IPNs are increased with increasing SA content in copolymeric structure. The most important effect is hydrophilicity of copolymeric gels. Hydrophilicity of AAm/SA and AAm/SA/PEG copolymers becomes greater than that of AAm, when addition of SA to the copolymeric structure. The ionic charge content in the polymeric structure is important. SA contains ionic units (–COO$^-$). The swelling degree of the hydrogels increases due to increase of the hydrophilic units on hydrogel structure (Fig. 1). It can be shown that a decreasing of the swelling value of AAm/SA/PEG

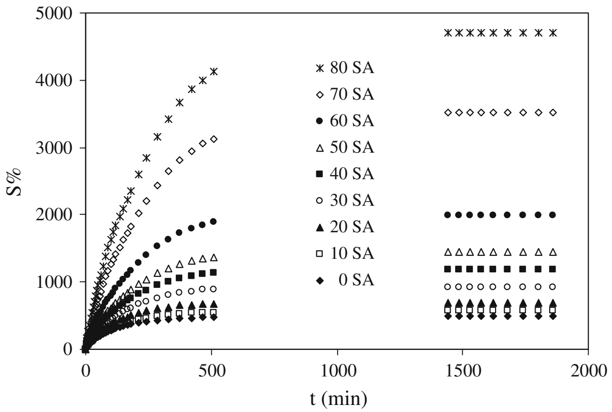


Fig. 2 Swelling isotherms of AAm/SA hydrogels

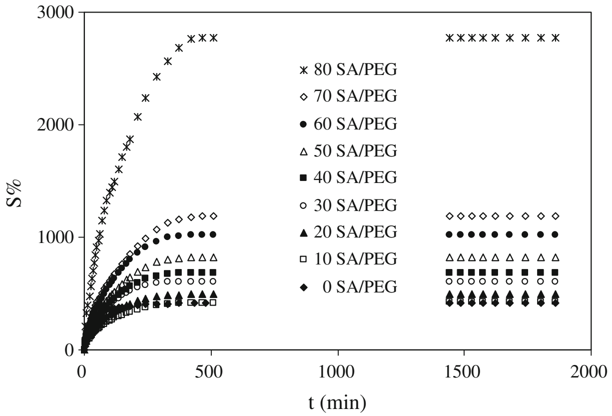


Fig. 3 Swelling isotherms of AAm/SA/PEG semi IPNs

Table 2 Values of the equilibrium percentage swelling (S_{eq}%) of AAm/SA hydrogels and AAm/SA/PEG (containing 0.5 g PEG) semi IPNs

SA/mg	0	10	20	30	40	50	60	70	80
Equilibrium percentage swelling (S_{eq}%)									
AAm/SA	492	579	693	918	1180	1448	1998	3524	4709
AAm/SA/PEG	416	422	491	605	681	818	1024	1190	2776

semi IPN systems when PEG has been added to the hydrogel systems (Table 3). Incorporation of PEG into the copolymer network leads to lower degrees of swelling. Here, it can be said that PEG chains was placed in the crosslinked polymeric systems, in stead of crosslinked AAm and SA molecules, it was seen that decreasing of the value of the equilibrium swelling percent, because of decreasing of hydrophilic character at crosslinked polymeric systems.

Table 3 Values of the equilibrium percentage swelling ($S_{\mathrm{eq}}\%$) of AAm/SA/PEG semi IPNs containing 60 mg SA

PEG/g	0	0.25	0.50	0.75	1.00
Equilibrium percentage swelling ($S_{\mathrm{eq}}\%$)	1998	1117	1024	997	860

Sorption studies

To observe the sorption of JGB, AAm/SA hydrogels and AAm/SA/PEG semi IPNs were placed in aqueous solutions of JGB and allowed to equilibrate for 4 days at 25 °C. At the end of this period, hydrogels and semi IPNs in the JGB solutions showed the dark coloration. However, acrylamide hydrogel did not show any dye sorption from solution.

In the sorption system at equilibrium, the total solute (dye) concentration; C_0 is following equation [15].

$$C_0 = C_{\mathrm{b}} + C \tag{2}$$

where C_{b} is the equilibrium concentration of the solute (dye) on the sorbent per liter solution (bound solute concentration) and C is the equilibrium concentration of the solute in the solution (free solute concentration). The value of the bound concentration may be obtained using Eq. 2. For a fixed free solute concentration, C_{b} is proportional to the polymer concentration on the binding system; the amount bound can therefore be conveniently expressed as the binding ratio r, defined by

$$r = C_{\mathrm{b}}/P. \tag{3}$$

Thus, with in and P is base mol (moles of monomer units) per liter solute represents the average number of molecules of solute bound each monomer unit at that free solute concentration [16, 17]. The binding data was interpreted on the basis of the uniform site-binding (u.s.b.) model, which in statistical-thermodynamic terms corresponds to a formation of an ideal localized one-dimensional monolayer of solute on the polymer chains [16, 17]. This leads to the hyperbolic (Langmuir) form of the binding isotherm, which applies to many polymer/solute (dye molecules) binding system.

$$r = \frac{nKC}{1 + KC} \tag{4}$$

where K is the binding constant, i.e., the equilibrium constant for the attachment of a molecule of dye onto a site by a specific combination of non-covalent forces. Here n is the site density (the limiting value of r for monolayer coverage) which is therefore of density of the sites along the polymer chain. To reciprocal of n is the site-size, u, which may be taken to represent either average number of monomer units occupied by the bound solute molecule, more generally the average spacing of solute molecules when the chain is saturated. The initial binding constant, K_{i} is the initial slope of the binding isotherm, and therefore the average binding strength of a solute molecule by a single monomer unit on an occupied chain. K_{i} is equal to the product nK.

To get the best values for the binding parameters from the experimental data, the linearization methods of Eq. 4 have been developed by some researches as Klotz, Scatchard, and Langmuir [15, 16].

Klotz method

If the isotherm (Eq. 4) is multiplied out and then divided throughout by C, K, n, r, this gives;

$$\frac{1}{r} = \frac{1}{n} + \frac{1}{nK}\frac{1}{C} \tag{5}$$

thus if this isotherm holds than a plot of $1/r$ versus $1/C$ will be straight line of slope $1/nK$, ordinate intercept $1/n$. This is very simple method of plotting, with the scatter in the r and C values reflected in the scatter in the ordinate and abscissa values, respectively. Its limitations are that the intercepts may be small and hence difficult to read off.

Scatchard method

Multiplying Eq. 4 by K, n, r and rearranging gives;

$$\frac{r}{C} = Kn - Kr \tag{6}$$

so that in this case a plot of r/C versus r should be a straight line of slope $-K$, ordinate intercept nK.

Langmuir method

Multiplying the Klotz form of Eq. 4 by C gives;

$$\frac{C}{r} = \frac{1}{nK} + \frac{C}{n} \tag{7}$$

so that here a plot of C/r versus C should be a straight line of slope $1/n$, ordinate intercept $1/nK$.

The Klotz, Scatchard, and Langmuir plots of AAm/SA hydrogels and AAm/SA/PEG semi IPNs are shown in Figs. 4, 5, and 6, respectively, and the binding parameters are calculated from the intercept and slopes of the binding isotherm methods. The binding parameters K_i, K, n, and u are listed in Table 4 for hydrogels and semi IPNs. In Table 4, the final column contains the derived values of the Ô, the maximum fractional occupancy attained experimentally, calculated from the definition of fractional occupancy Ô:

$$\hat{O} = r/n \tag{8}$$

Using the value of r at the maximum experimental-free dye concentration and with the site density obtained for the (u.s.b.) model. The calculated parameters determined using Klotz, Scatchard, and Langmuir linearization methods are nearly

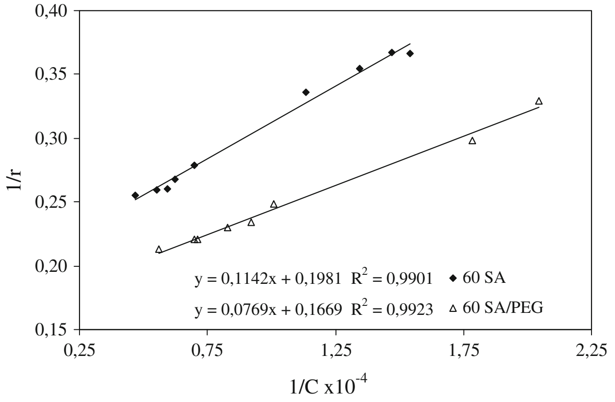


Fig. 4 Klotz plots of AAm/SA hydrogels and AAm/SA/PEG semi IPNs with JGB

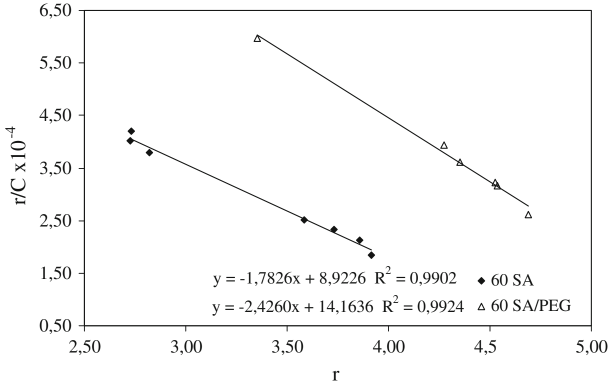


Fig. 5 Scatchard plots of AAm/SA hydrogels and AAm/SA/PEG semi IPNs with JGB

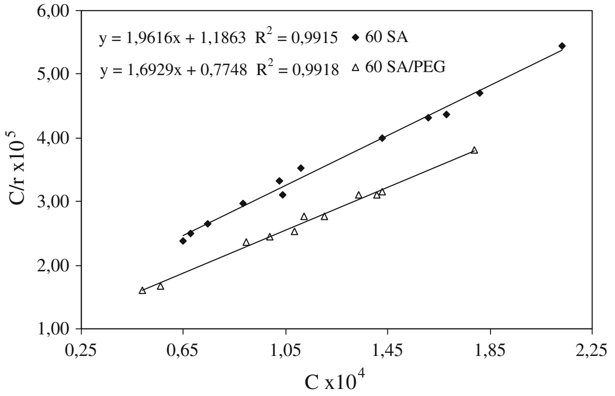


Fig. 6 Langmuir plots of AAm/SA hydrogels and AAm/SA/PEG semi IPNs with JGB

Table 4 Some binding parameters of AAm/SA hydrogels and AAm/SA/PEG semi IPNs with JGB

	$K_i \times 10^{-4}$ (L mol^{-1})	$K \times 10^{-4}$ (L mol^{-1})	n	u	Ô
Klotz					
AAm/SA	8.76	1.73	5.05	0.1981	0.7758
AAm/SA/PEG	13.00	2.17	5.99	0.1669	0.7830
Scatchard					
AAm/SA	8.92	1.78	5.01	0.1998	0.7820
AAm/SA/PEG	14.16	2.43	5.84	0.1713	0.8031
Langmuir					
AAm/SA	8.43	1.65	5.10	0.1962	0.7682
AAm/SA/PEG	12.91	2.18	5.91	0.1693	0.7936

equal to each other. The linearization methods (Klotz, Scatchard, and Langmuir) can be used in the hyperbolic binding system for dye–polymers binding process. There can be many reasons for non-covalent interactions in the binding of JGB by hydrogels and semi IPNs. The main interactions between the hydrogel and the monovalent cationic dyes may be hydrophobic and hydrogen bonding. Specially, hydrogen bonding will be expected to occur between amine groups and nitrogen atoms on the dye molecules and the amine and carbonyl groups on the monomer unit of crosslinked polymer. Hydrophobic effects are especially aqueous solutions interactions which in the present case will involve aromatic ring on the dye molecules and the methine and methyl groups on the gel. There can be some other interactions such as dipole–dipole and dipole-induced–dipole interactions between the dye molecules and the hydrogel chains. In addition of these interactions, electrostatic interactions between cationic dye molecules and anionic crosslinked polymer chains are very important. There are many ionizable sites along the polymeric chain. So, it could have been expected some electrostatic interactions.

In later experiments, for equilibrium sorption studies, the dye removal capacity of the adsorbent, (q), can be investigated. The dye removal capacity, (q), of AAm/SA hydrogels and AAm/SA/PEG semi IPNs were evaluated using the following equation:

$$q = \frac{(C_b)v}{m} \tag{9}$$

where q is the dye removal capacity of AAm/SA hydrogels and AAm/SA/PEG semi IPNs (mol g^{-1}), v is the volume of the aqueous phase (L), m is the amount of dry AAm/SA hydrogels and AAm/SA/PEG semi IPNs (g), and C_b was defined in Eq. 2.

Uptake of dye was measured the effects of contents of SA. The amount of dyes sorbed onto unit dry mass of the gel was calculated for uptake of dye within the hydrogel in 2.00×10^{-3} mol dye in L of aqueous solutions, and presented in Table 5. Table 5 presents that the amount of dyes sorbed onto unit dry mass of the hydrogels and semi IPNs ($1.18–6.31 \times 10^{-4}$ mol g^{-1}), q, are increased. The amount of dyes sorbed onto unit dry mass of the hydrogels and semi IPNs gradually

Table 5 Some adsorption parameters of AAm/SA hydrogels and AAm/SA/PEG (containing 0.5 g PEG) semi IPNs in aqueous solutions of JGB

SA/mg	10	20	30	40	50	60	70	80
Dye removal capacity, $q \times 10^4$ (mol g^{-1})								
AAm/SA	1.66	2.36	3.41	4.40	5.10	5.30	5.70	6.31
AAm/SA/PEG	1.18	2.06	3.18	4.47	5.23	5.26	5.99	5.78
Adsorption percentage, Ads%								
AAm/SA	26	39	53	72	90	92	93	93
AAm/SA/PEG	18	34	49	65	82	90	91	92
Partition coefficient, K_d								
AAm/SA	0.35	0.63	1.13	2.55	9.49	11.12	12.98	13.47
AAm/SA/PEG	0.21	0.50	0.95	1.84	4.68	9.45	9.69	11.48

Table 6 PEG effects on some adsorption parameters of AAm/SA/PEG semi IPNs systems containing 60 mg SA in aqueous solutions of JGB

PEG/g	0	0.25	0.50	0.75	1.00
Dye removal capacity, q (mg g^{-1})	5.30	5.63	5.26	5.84	6.11
Adsorption percentage, Ads%	92	87	90	88	73
Partition coefficient, K_d	11.12	6.54	9.45	7.25	2.70

increased with the increase of content of SA in hydrogels and semi IPNs. Again, Tables 5 and 6 present that the values of adsorption percentage, Ads% of the hydrogels and semi IPNs. These values were between 18.0 and 93.0%. Here, the same character was observed. It was seen that the sorption capacity of the hydrogels increase due to increase of the hydrophilic units on hydrogel structure. Partitioning of dissolved constituents between an aqueous phase and adsorbents in waters and sediments has commonly been described by an empirical partition coefficient that simply relates the total concentration of a dissolved species to the total concentration of the adsorbed species [18, 19]

$$K_d = \frac{C_b}{C} \tag{10}$$

where K_d is empirical partition coefficient at equilibrium. C_b and C were defined earlier. Partition coefficients of JGB between dye solution and hydrogels were calculated, and are shown in Table 5. In Table 5, K_d values of AAm/SA hydrogels are 0.35–13.47, but K_d values of AAm/SA/PEG semi IPNs are 0.21–11.48 with the incorporation of PEG groups into the hydrogels. In Table 6, it was seen that a decreasing of the values of partition ratio, K_d of AAm/SA/PEG semi IPNs with the adding of more PEG amount. In Table 6, K_d values of AAm/SA/PEG semi IPNs are 11.12–2.70. Here, in general, some K_d values of AAm/SA hydrogels and AAm/SA/PEG semi IPNs are higher than 1.0. So, it can be said that having K_d values bigger than 1.0 of AAm/SA hydrogels and AAm/SA/PEG semi IPNs could be used as potential water adsorbent in wastewater treatment or water purification processes [18, 19].

The ionic charge content in the polymeric structure is important. SA contains ionic units (–COO$^-$). The swelling degree of the hydrogels increases due to increase of the hydrophilic units on hydrogel structure (Fig. 1). Therefore, AAm/SA hydrogels and AAm/SA/PEG semi IPNs have many ionic groups that can increase interaction between the cationic dye molecules and anionic groups of hydrogels. The results of swelling studies are parallel character to the results of sorption studies. in both, it can be seen that swelling or sorption capability of AAm/SA hydrogels and AAm/SA/PEG semi IPNs are increased with increasing SA content in copolymeric structure. The most important effect is hydrophilicity of copolymeric gels. Hydrophilicity of AAm/SA and AAm/SA/PEG copolymers becomes greater than that of AAm, when addition of SA to the copolymeric structure. There can be shown that an important decreasing of values of Ads% and K_d of AAm/SA/PEG semi IPN systems when PEG has been added to the hydrogel systems (Table 6). Here, it could be said that PEG chains were placed in the crosslinked polymeric systems, instead of crosslinked AAm and SA molecules. When the PEG chains were located in the free space of crosslinked polymer networks, this was also caused of decreasing of the dye-binding sites. Incorporation of PEG into the copolymer network leads to lower values of sorption parameters.

Conclusion

Incorporation of hydrophilic group containing chemicals such as SA and a polymer such as PEG in AAm hydrogels can be obtained successively by free radical solution polymerization method. Multifunctional crosslinker such as PEGDA used at the polymerization process. AAm/SA hydrogels and AAm/SA/PEG semi IPNs showed high water absorbency. It was seen that swelling of AAm/SA hydrogels and AAm/SA/PEG semi IPNs increased with the increasing of content of SA. However, it is seen that a decreasing of values of S_{eq}% when the adding of PEG. This study has given the quantitative information on the sorption characteristic of JGB with AAm/SA hydrogels and AAm/SA/PEG semi IPNs. In this study, it has shown that AAm/SA hydrogels and AAm/SA/PEG semi IPNs have sorbed the monovalent cationic dyes such as JGB, while AAm do not. The amount of JGB sorbed onto unit dry mass of the gel is increased with the content of SA. Here, most of the K_d values of AAm/SA hydrogels and AAm/SA/PEG semi IPNs are higher than 1.0. So, it can be said that synthesized crosslinked AAm/SA hydrogels and AAm/SA/PEG semi IPNs could be used as potential adsorbent for cationic dye. Consequently, AAm/SA hydrogels and AAm/SA/PEG semi IPNs developed in this study may serve as a potential device for super water retainer, and adsorbent for cationic species. The utilization of these types of materials, in pharmaceuticals, agriculture, biotechnology, environment, separation, purification, and immobilization makes hydrogels more popular.

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