

# Swelling Properties of Superabsorbent Poly(acrylic acid-co-acrylamide) with Different Crosslinkers

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**ABSTRACT:** Poly(ethylene glycol) diacrylate (PEGDA) was synthesized and characterized with Fourier transform infrared. A superabsorbent based on poly(acrylic acid-co-acrylamide) (PAAM) was prepared by free-radical solution polymerization with various crosslinkers. The relationship between the absorbency of PAAM in distilled water and in 0.9% NaCl solutions and the swelling time was explored. The order of the equilibrium absorbency of PAAM with different crosslinkers in 0.9% NaCl solutions was glutaraldehyde (GDA) < PEGDA < *N,N'*-methylene bisacrylamide (NMBA) < divinylbenzene (DVB). Some swelling models were used to fit the swelling data. The second-order kinetic

model was best for describing the water absorption of PAAM with water absorption rate constants in the order of DVB < PEGDA < NMBA < GDA. The repeated absorbencies of PAAM in 0.9% NaCl solutions were also investigated. The repeated absorbency of PAAM with PEGDA as the crosslinker was better than that of PAAM with other crosslinkers. The repeated absorbency of PAAM was rather poor when DVB was used as the crosslinker, although the maximum absorbency was obtained. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 112: 602–608, 2009

**Key words:** crosslinking; hydrogels; swelling

## INTRODUCTION

Superabsorbent polymers (SAPs) are lightly cross-linked networks of hydrophilic polymer chains that can swell in water and hold a large amount of water while maintaining their physical and dimensional structures.<sup>1</sup> Because of their excellent water absorbency and good water retention capacity, SAPs have been applied in various fields such as medicine, personal hygiene, and agriculture.<sup>2</sup> Acrylic-based hydrogels and SAPs have been widely studied and applied in different biomedical fields because of their good swelling behaviors and chemical stability.<sup>3</sup>

In general, three-dimensional network of SAPs is formed through the use of crosslinkers in the prepara-

tion of SAPs, except for those that are self-cross-linking or are irradiated.<sup>2,4–6</sup> The crosslinkers can mainly be divided into the following types:<sup>2</sup> (1) monomers including two double bonds, which react with acrylate and acrylamide (AM) by free-radical polymerization, such as *N,N'*-methylene bisacrylamide, divinylbenzene (DVB), poly(ethylene glycol) diacrylate (PEGDA), and poly(ethylene glycol) dimethyl acrylate; (2) multifunctional compounds with the usual groups, which can react with  $-\text{COOH}$ ,  $-\text{OH}$ , or  $-\text{CONH}_2$ , such as diamine or polyamine, binary alcohol or polyalcohol, binary acid or polyacid, epoxide, glutaric dialdehyde, and ethylene glycol diglycidyl ether; and (3) multivalent metal ions such as  $\text{Fe}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Al}^{3+}$ , and  $\text{Cr}^{3+}$ , which mainly form electrovalent bonds with  $-\text{COO}^-$ .

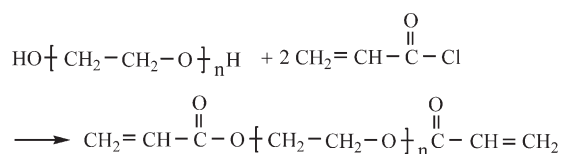
The absorbency of a superabsorbent is usually affected by the types and amounts of crosslinkers. According to reports on the preparation of SAPs, crosslinkers include *N,N'*-methylene bisacrylamide,<sup>7</sup> 1,4-butanediol diacrylate,<sup>7</sup> 1,4-butanediol dimethacrylate,<sup>8</sup> trimethylolpropane triacrylate,<sup>8</sup> DVB,<sup>9</sup> PEGDA,<sup>10</sup> glutaraldehyde (GDA),<sup>11</sup> ethylenediamine,<sup>12</sup> formaldehyde,<sup>13</sup> glycerine,<sup>13,14</sup> polyalcohols (including polyethylene alcohol, propanediol, and inositol),<sup>14</sup> poly(ethylene glycol) dimaleate,<sup>15</sup> mannitol,<sup>16</sup> xylitol,<sup>16</sup> ethylene glycol diglyceryl ether,<sup>17</sup> poly(ethylene

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**Scheme 1** Synthesis of PEGDAs.

glycol) monoester,<sup>18</sup> kaolin,<sup>19</sup> and alumina.<sup>20</sup> *N,N'*-Methylene bisacrylamide (NMBA) has been used most widely because of its water solubility, short chains, and high activity in free-radical polymerization. However, a majority of NMBA is consumed in the initial periods of polymerization because of its high activity, which leads to rather difficult crosslinking in the latter stage of polymerization on account of the very small amount of NMBA left.

In this work, PEGDA and poly(acrylate-*co*-acrylamide) (PAAM) were synthesized, the absorbencies of PAAM with different crosslinkers in distilled water and 0.9% NaCl solutions were investigated, and the repeated absorbencies of PAAM in 0.9% NaCl solutions were also examined.

## EXPERIMENTAL

### Experimental materials

Acrylic acid (AA), AM, potassium persulfate (KPS), NMBA, and DVB were purchased from Shanghai Chemical Reagent Factory (Shanghai, China). Before use, AA was distilled *in vacuo*, and AM and KPS were recrystallized in acetone and in distilled water. The crosslinker GDA (Alfa Aesar and Chemistry Co., Tianjin, R. P. China) was used as received. Other reagents such as poly(ethylene glycol)s (PEGs) with molecular weight of 600, 1000, and 2000 (PEG600, PEG1000, and PEG2000, respectively), NaOH, and NaCl were also used as received.

### Synthesis of PEGDA

Acryloyl chloride (AACL) was synthesized according to the reported literature.<sup>21</sup> A tetrahydrofuran solution (120 mL) with 0.1 mol of dissolved PEG600 was put into a three-necked flask equipped with a reflux condenser and a drip funnel, and 0.15 mol of triethylamine was added dropwise under stirring; subsequently, 0.15 mol of AACL was added slowly. The resulting mixture was kept at room temperature for 24 h. The product was washed with saturated NaCl–NaHCO<sub>3</sub> solutions until the pH value of the effluent was 7, and it was dried with Na<sub>2</sub>SO<sub>4</sub> over 24 h. This was labeled PEGDA1. PEGDA2 and PEGDA3 were prepared in the same way as PEGDA1 from PEG1000 and PEG2000, respectively. These reactions are shown in Scheme 1. The PEGDA products (PEGDA1,

PEGDA2, and PEGDA3) were kept in a refrigerator at about –10°C.

### Preparation of PAAM

The mass ratio of AA to AM was 6 : 1. The total mass percentage of both monomers in the reaction mixtures was 25%. The mass percentages of KPS and the crosslinker, with respect to the total mass of both monomers, were 0.1 and 0.4%, respectively.

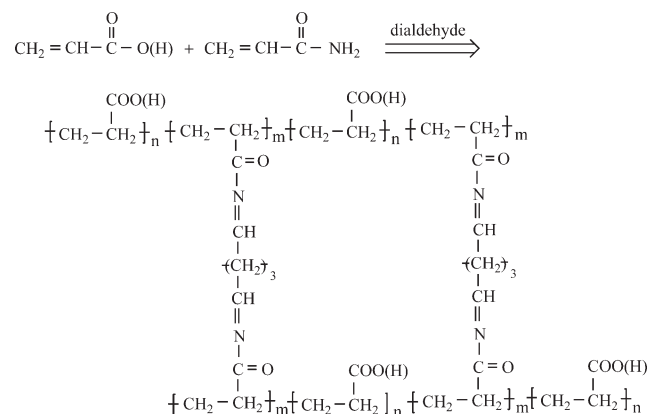
To obtain the desired neutralization, a defined amount of a 12 mol/L NaOH solution was added dropwise to a preweighed and stirred aqueous AA solution in a beaker cooled with an ice–water bath. Then, defined amounts of AM, GDA, and KPS were dissolved in distilled water, and they were successively added to the beaker. The reaction mixture was heated and polymerized at 60–80°C for 7 h. The time for increasing the temperature was 2 h for 60°C, 2 h for 70°C, and 3 h for 80°C. After the polymerization, the product was cut into small pieces and dried in a vacuum oven at 50°C until the mass of the product was constant. Finally, the dried product was pulverized and milled. All the samples were used with a particle size in the range of 60–100 mesh.

The reaction scheme proposed for the synthesis of GDA-crosslinked PAAM is shown in Scheme 2. The reaction scheme for the other crosslinkers is similar to Scheme 2 for GDA.

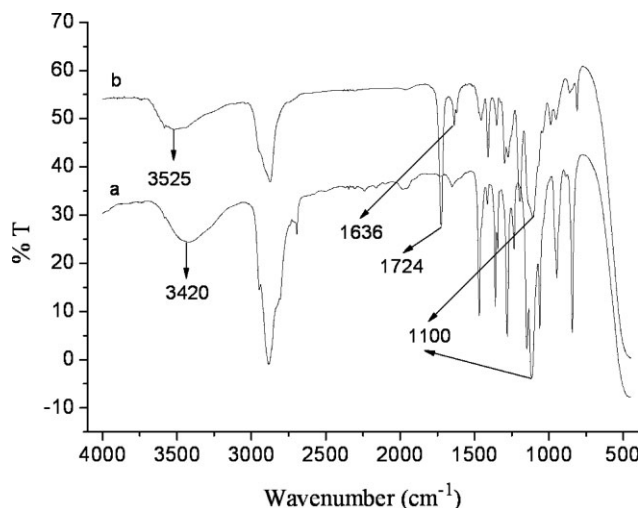
### Absorbency experiments

At room temperature, a weighed amount of PAAM (0.4 g) was immersed into about 800–1000 mL of distilled water or a 0.9% NaCl solution, and the swollen hydrogel was directly weighed after the desired time; then, the absorbency (*Q*) was calculated according to eq. (1):

$$Q = (m_2 - m_1) / m_1 \quad (1)$$



**Scheme 2** Synthesis of GDA-crosslinked PAAM.



**Figure 1** FTIR diagraphs of (a) PEG and (b) PEGDA.

where  $m_1$  is the mass of the dry PAAM sample (g) and  $m_2$  is the mass of the wet sample at different times of treatment (g). The maximum absorbency was considered to be the equilibrium absorbency (g/g of dry PAAM).

### Repeated absorbency experiments

At room temperature, about 1.5 g of PAAM was immersed into about 800–1000 mL of distilled water or a 0.9% NaCl solution over 6 h to achieve the equilibrium absorbency.  $Q$  was calculated according to eq. (1). The swollen hydrogel was directly washed with distilled water of different pH values and then was dried. The aforementioned swelling, washing, and drying procedures were repeated  $n$  times.

All the data on equilibrium absorbencies represent the mean values of three measurements.

## RESULTS AND DISCUSSION

### Fourier transform infrared (FTIR) of PEGDA

The strong and acute peak at  $1724\text{ cm}^{-1}$ , which is different from the peak at  $1760\text{ cm}^{-1}$  for AACl, proves that the  $\text{—OH}$  groups of PEG reacted with AACl, whereas the peak at about  $3500\text{ cm}^{-1}$  shows that there were still some  $\text{—OH}$  groups in the product; that is, the conversion of PEG was not 100%. There is not a strong peak at  $947\text{ cm}^{-1}$  for  $\text{C—Cl}$ , and this shows that AACl did not exist in the product. The peak at about  $1636\text{ cm}^{-1}$  for  $\text{C=C}$  in PEGDA further demonstrates that PEG reacted with AACl. The peak at about  $1100\text{ cm}^{-1}$  in both PEG and PEGDA suggests that  $\text{C—O}$  was not involved in the reaction; that is, the reaction occurred just at the end of the chain but not inside the chain (Fig. 1).

### Absorbency of PAAM

#### Equilibrium absorbency of PAAM

Table I shows the relation of the absorbency of PAAM and the best masses of different crosslinkers. It reveals that the order of the mass of the crosslinker with respect to the mass of the monomers is  $\text{PEGDA1} < \text{NMBA} < \text{GDA} \ll \text{DVB}$  when the largest absorbency of PAAM in distilled water and 0.9% NaCl solutions is obtained. The order of the equilibrium absorbency of PAAM for different crosslinkers in distilled water and in 0.9% NaCl solutions is  $\text{GDA} < \text{PEGDA1} < \text{NMBA} < \text{DVB}$ . The amount of DVB used in preparing PAAM is the largest because the order of the solubility in water is  $\text{DVB} < \text{PEG1} < \text{GDA} < \text{NMBA}$ ; that is, the solubility of DVB in water is the lowest, whereas that of NMBA is the largest.

When GDA, having good solubility and reactivity, is used as the crosslinker, the  $\text{—CHO}$  groups of GDA react with the  $\text{—NH}_2$  groups of AM through the Schiff base during the polymerization to produce a nice three-dimensional network with large crosslinking density because there are a lot of crosslinking points. For a certain hydrogel network, the more crosslinking points there are, the smaller the network space will be. Furthermore, the number of hydrophilic groups in PAAM decreases when the  $\text{—CHO}$  group of GDA reacts with the  $\text{—NH}_2$  group of AM. Therefore, the absorbency of GDA-crosslinked PAAM is the lowest. The reactivity of PEGDA1 may be the lowest among the four types of the crosslinkers because of its long chains and most difficult molecular motion. In addition, the lower solubility of PEGDA1 with respect to GDA and NMBA could also cause small crosslinking points of PAAM. However, the long chain of PEGDA1 helps to produce a larger network space in the PAAM hydrogel and thus more embedded water in its network versus GDA- and NMBA-crosslinked PAAM. Theoretically, the absorbency of PAAM should be higher when PEGDA1 is used as the crosslinker rather than the others because the much longer

**TABLE I**  
Relationship of the Absorbency of PAAM and Best Amounts of the Crosslinkers

Crosslinker Species	NMBA	GDA	DVB	PEGDA600
[C]	$4.67 \times 10^{-5}$	$1.44 \times 10^{-4}$	$8.86 \times 10^{-3}$	$3.35 \times 10^{-5}$
$Q_{w,eq}$ (g/g)	2710	1072	3892	1970
$Q_{s,eq}$ (g/g)	133	104	172	121

[C] is the concentration of the crosslinkers with respect to the amount of the monomers (molar ratios);  $Q_{w,eq}$  and  $Q_{s,eq}$  are the equilibrium absorbencies of PAAM in distilled water and a 0.9% NaCl solution, respectively.

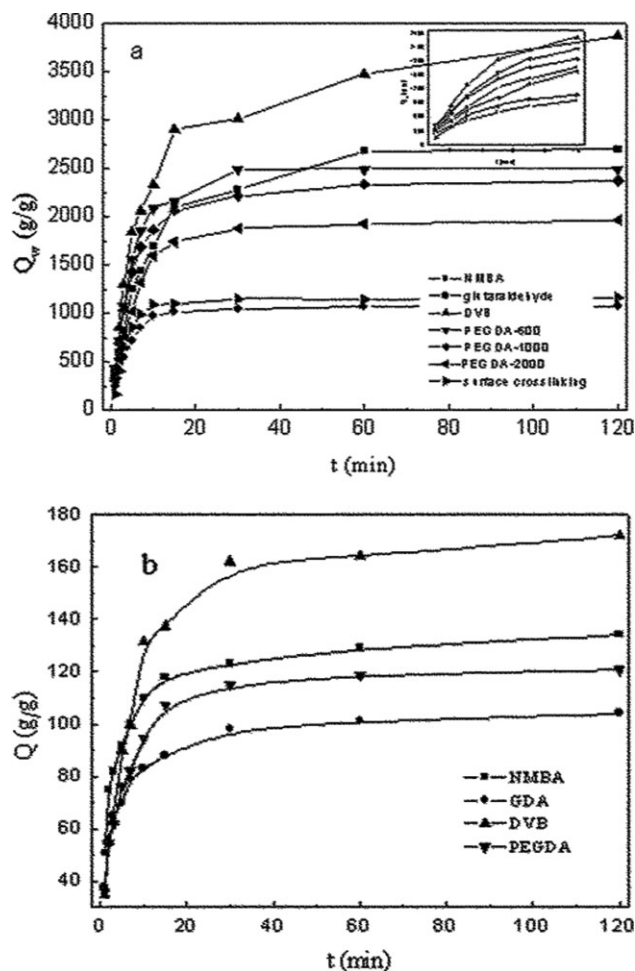
chain of PEGDA1 helps to construct a larger network. The experimental data, however, do not confirm these assumptions. This may be attributed to the fact that the network using PEGDA1 as the crosslinker collapses to some extent because of its too long chain. Therefore, the order of the absorbency is  $GDA < PEGDA1 < NMBA$ . DVB is slightly soluble in water; that is, only a small amount of the total DVB is dissolved in water. As the amount of DVB increases, the amount of DVB dissolved in water increases slowly. Therefore, the number of crosslinking points of the PAAM hydrogel is gradually increased, and the crosslinking density inside the PAAM hydrogel is uniform during the entire polymerization process. Thus, the largest absorbency is obtained with DVB as the crosslinker.

### Swelling of PAAM

The kinetics of the water sorption process have been investigated through monitoring of the changes in the physical dimensions of the swollen hydrogel or in the amount of water imbibed by the hydrogel at different times.

Figure 2 shows the relationship of the absorbency of PAAM in distilled water [Fig. 2(a)] and in a 0.9% NaCl solution [Fig. 2(b)] and the swelling time, and the standard deviations for these data are changed from about 5 to 12%.

The results show that the order of the absorbency of PAAM in distilled water in the first few minutes is  $DVB < PEGDA1 < PEGDA2 < NMBA < PEGDA3 < \text{surface crosslinking} < GDA$ , and the absorbency in 0.9% NaCl solutions is irregular in the first minutes, whereas the order of the equilibrium absorbency of different crosslinkers mentioned previously is  $GDA < \text{surface crosslinking} < PEGDA3 < PEGDA2 < PEGDA1 < NMBA < DVB$  in distilled water and  $GDA < NMBA < PEGDA3 < DVB$  in 0.9% NaCl solutions. The reason for the order of the absorbency in NaCl solutions in the first few minutes is not completely clear because the penetration pressure is affected by the existence of NaCl. However, in the initial swelling process, that is, for the first few minutes, it is controlled by the capillarity; in other words, the swelling of the dried hydrogel is dominated by the imbibing of water at the beginning of the swelling process. As we mentioned previously, DVB is hydrophobic, and this prevents water from entering the network at the beginning of the measurement. PEGDA3 is also hydrophobic, but there are a lot of hydrophilic atoms (oxygen), which more easily form hydrogen bonds in the chain of PEGDA3. Therefore, it is faster for water to absorb into the network with PEGDA as the crosslinker than with DVB. As for GDA and NMBA, the  $-\text{CONH}_2$  groups in the networks of both are hydrophilic, and they accelerate the entry of water



**Figure 2** Relationship of  $Q_w$  and  $t$  for different crosslinkers in (a) distilled water and (b) a 0.9% NaCl solution.  $Q_w$  is absorbency in distilled water.

into the hydrogel networks. The hydrogel is swollen with an increase in the swelling time, and the swelling process is mainly controlled by the expansion of the network; the large amount of absorbed water is the free water in the network space. Among these crosslinkers, the network space and hydrogel strength in DVB are the largest, so the absorbency of PAAM with DVB as the crosslinker increases more than that for other crosslinkers during the following swelling process.

### Swelling model analysis

Water diffusion into polymer networks, that is, the relationship between the water absorbency and swelling time, can be described by the following equation:<sup>22</sup>

$$\frac{Q_t}{Q_{eq}} = kt^n \quad (2)$$

In this expression,  $k$  is the proportionality constant related to the structure of the network;  $n$  is the

**TABLE II**  
Simulation Parameters of Eq. (2) for the Swelling of PAAM Crosslinked by Different Crosslinkers in 0.9% NaCl Solutions ( $Q_t < 0.6Q_{eq}$ )

Crosslinker type	$k$	$n$	$R$
NMBA	0.52731	0.047	0.983
GDA	0.50136	0.050	0.931
DVB	0.27105	0.101	0.982
PEGDA	0.41633	0.066	0.970

diffusion exponent;  $t$  is the swelling time; and  $Q_t$  and  $Q_{eq}$  are the amounts of water absorbed at  $t$  and at equilibrium, respectively.

The logarithmic form of eq. (2) is used to determine the values of  $n$  and  $k$  from the slope and the intercept, respectively. If  $n$  is less than 0.5, the swelling process is controlled by the Fickian diffusion mechanism. If  $n$  varies between 0.5 and 1, diffusion and macromolecular relaxation equally control the swelling process. We have used the aforementioned model to simulate the swelling curves when  $Q_t$  is less than  $0.6Q_{eq}$ . The simulation parameters are shown in Table II. From Table II, we can find that  $n$  is less than 0.5, and this suggests that the swelling process is controlled by a Fickian diffusion mechanism. The correlation coefficient ( $R$ ) values are all less than 0.99, and this suggests that eq. (2) cannot fit the experimental data very well.

Mahdavinia et al.<sup>1</sup> described the swelling process of a hydrogel by the first-order kinetics:

$$Q_t = 1 - \exp(-Kt) \quad (3)$$

where  $K$  is the swelling constant. A plot of  $Q_e$  is equilibrium absorbency,  $Q_{eq}$ , which is the same as  $Q_{eq}$  in equation 2, versus  $t$  should be linear with a slope of  $-K$ . Table III shows that the swelling data of PAAM with NMBA and PEGDA3 as crosslinkers fit the first-order kinetics well. However, it is not suitable for GDA and DVB. The order of  $-K$  is  $GDA \leq DVB < NMBA < PEGDA3$  ( $Q_t < 0.8Q_{eq}$ ), and this demonstrates that the swelling of PAAM with PEGDA as the crosslinker is fastest, whereas the swelling of PAAM with GDA is slowest.

Recently, Sag and Aktay<sup>23</sup> and Saraydin et al.<sup>24</sup> suggested a theoretical model for the diffusion-con-

**TABLE III**  
Simulation Parameters of Eq. (3) for the Swelling of PAAM Crosslinked by Different Crosslinkers in 0.9% NaCl Solutions ( $Q_t < 0.8Q_{eq}$ )

Crosslinker type	$K$	$R$
NMBA	0.1013	0.997
GDA	0.0784	0.969
DVB	0.0973	0.969
PEGDA	0.1148	0.998

trolled swelling of polymeric films. At relatively high degrees of swelling, first-order kinetics do not give a good approximation of the real behavior. It has been demonstrated that for second-order swelling kinetics, the reciprocal of the average rate of swelling ( $t/Q$ ) is related to the time of treatment by the following linear equation:

$$t/Q = A + Bt \quad (4)$$

where  $A$  is the reciprocal of the initial swelling rate ( $A = 1/k_Q Q_{eq}^2$ ),  $B$  is the inverse of the degree of swelling at equilibrium, and  $k_Q$  is the rate constant of swelling.

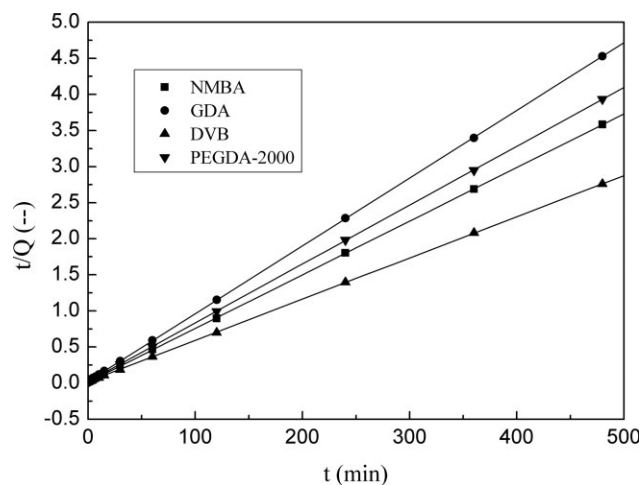
The swelling kinetics of PAAM with different crosslinkers have been fitted with the second-order diffusion model excellently; the simulation curves are shown in Figure 3, and the parameters are shown in Table IV.

Table IV shows that the order of  $k_Q$  does not agree with the inverse order of the theoretical absorbency. This may be because the network collapses a little when PEGDA3 is used as the crosslinker, whereas this phenomenon does not exist with the other crosslinkers.

#### Repeated absorbency

Nowadays, many areas, such as agriculture and architecture, need SAPs with good repeated absorbency after they are used a few times. In this work, we investigate the repeated absorbency of PAAM with different crosslinkers in 0.9% NaCl solutions. The experimental results are given in Figure 4, and the standard deviations for these data are changed from about 4 to 9%.

The repeated absorbency of PAAM decreases obviously with the repeated swelling times increasing, especially the second repeated absorbency. It



**Figure 3** Relationship between  $t/Q$  and  $t$  for different crosslinkers in 0.9% NaCl solutions.

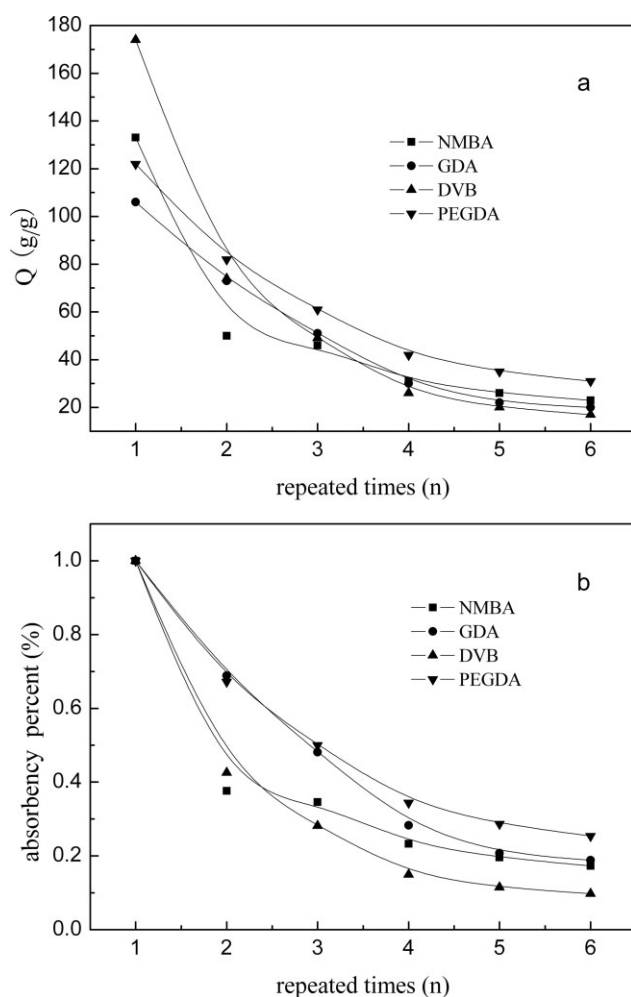
**TABLE IV**  
Simulation Parameters of Eq. (4) for the Swelling of PAAM Crosslinked by Different Crosslinkers in 0.9% NaCl Solutions

Crosslinker type	$k_q$ [g of gel/(g of water min) $\times 10^3$ ]	$Q_{\text{theoretical}}$ (g of water/g of gel)	$Q_s(\text{exp})$ (g of water/g of gel)	R
NMBA	3.33	135	134	0.9999
GDA	4.16	106	104	0.9999
DVB	1.33	175	172	0.9999
PEGDA	3.03	123	121	0.9999

$Q_{\text{theoretical}}$  = theoretical absorbency.  $Q_s(\text{exp})$  is the experimental absorbency in 0.9% NaCl solution.

has also been shown that the repeated absorbency of PEGDA3-crosslinked PAAM is the best, and that of DVB-crosslinked PAAM is the worst. If the superabsorbent is swollen in salt solutions, water molecules turn into combined water from free water by diffusing into the interior of the network to form hydrogen bonds with the hydrophilic groups of PAAM. The combined water cannot be taken off completely during the drying process of PAAM, and this causes

the reduction of the osmotic pressure between the inside and outside of the network when the superabsorbent is swollen again. The decreasing swelling driving force induces a decrease in the absorbency. With the number of repeats, the PAAM absorbency decreases. Furthermore, when water penetrates the network, the network collapses to a certain extent because of the disappearance of mutual interactions such as hydrogen bonds of the interchains or intrachains of PAAM during the drying of PAAM. The more times PAAM is dried, the more the network collapses. For the hydrophobic DVB bearing a polar phenyl group, physical crosslinking is formed with hydrophobic functional groups during the drying process. With the number of repeated swellings of the superabsorbent, the number of the physical crosslinking points increases, and the remaining network space becomes smaller.



**Figure 4** (a) Repeated absorbency and (b) absorbency percentage of PAAM crosslinked by different crosslinkers in 0.9% NaCl solutions.

## CONCLUSIONS

PEGDA was synthesized and characterized with FTIR. A PAAM superabsorbent was prepared by free-radical solution polymerization with KPS as the initiator and various crosslinkers. The relationship of the absorbency of PAAM in distilled water and in 0.9% NaCl solutions with the swelling time has been discussed. The order of the mass of the crosslinker with respect to the total mass of both monomers is  $\text{NMBA} < \text{GDA} < \text{PEGDA1} < < \text{DVB}$  when the largest absorbency of PAAM in distilled water and 0.9% NaCl solutions is obtained. The order of the equilibrium absorbency of PAAM for different crosslinkers in distilled water and 0.9% NaCl solutions is  $\text{GDA} < \text{PEGDA1} < \text{NMBA} < \text{DVB}$ . The absorbency of PAAM in distilled water in the first minutes increases in the order of  $\text{DVB} < \text{PEGDA1} < \text{PEGDA2} < \text{NMBA} < \text{PEGDA3} < \text{surface crosslinking} < \text{GDA}$ . However, the absorbency in 0.9% NaCl solutions is irregular in the first minutes. Some swelling models have been used to fit the swelling data. It has been shown that the second-order kinetic model is the best for describing the water absorption of PAAM, with the water absorption rate constants in the order of  $\text{DVB} < \text{PEGDA3} < \text{NMBA} < \text{GDA}$ .

The repeated absorbency of PAAM in 0.9% NaCl solutions has been investigated. It has been revealed that the repeated absorbency of PAAM with PEGDA3 as the crosslinker is better than that with the other crosslinkers. The repeated absorbency is rather poor when DVB is used as the crosslinker, although the maximum absorbency is obtained.

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