

Effect of cross-linker and initiator concentration on the swelling behaviour and network parameters of superabsorbent hydrogels based on acrylamide and acrylic acid

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Abstract

Poly (acrylamide-co-acrylic acid) [poly (AAM-co-AAc)]-based hydrogels were prepared by varying the cross-linking agent concentration (NMBA) in the range of 0.0077% to 0.077%. The effect of the NMBA content on swelling parameters, such as water absorbency, swelling ratio on the hydrogel was studied by keeping two monomers content, that is, AAm and AAc constant. The effect of KPS as an initiator in the range of 0.1% to 0.5% on the water absorbency of the hydrogels was also studied. The swelling value could be modulated in a great range by varying the cross-linking agent (NMBA) or initiator (KPS) concentration at fixed monomer concentration. The synthesized hydrogels were characterized by FTIR, SEM and network parameters, such as average molecular weight between cross-links (M_c), cross-link density (q) and mesh size (ξ). The experimental results showed that the equilibrium water absorbency decreases from 707.23 to 389 with increasing the NMBA content from 0.0096% to 0.0385%. The network parameters, such as average molecular weight between cross-links (M_c) were decreased from 2.33×10^{12} to 2.30×10^{11} and cross-link density (q) decreased from 3.07×10^{-11} to 3.09×10^{-11} ; although mesh size (ξ) increased from 3.00×10^6 to 8.89×10^6 with increase in percentage of NMBA content from 0.009265% to 0.077%. The equilibrium water absorbency of various hydrogels were studied in distilled water and different types of saline solutions, which exhibited that hydrogels have higher equilibrium water absorbency in 0.009% saline (NaCl) solution than in 0.09% and 0.9% saline solutions. From the results it was clear that water absorbency increased from 302 to 573 g water/g sample with increasing the KPS content from 0.1 to 0.5%.

Keywords: Hydrogel • Cross-linker • Acrylamide • Acrylic acid • Super absorbent polymers • Network parameters • Initiator

Introduction

Desertification leading to lack of water for irrigation are serious problems for many regions of the world. If this is solved, even partially, will address the

food shortage problem in future. The use of super absorbent polymers (SAPs), which are synthetic and economical and have the ability to retain large amount of water even under high pressure or temperature can be an answer. Because of their excellent properties, these SAPs are already well established in various applications, such as disposable diapers, hygienic napkins, cement, drug delivery systems, sensors and agriculture [1]. In such applications high water absorbency or water retention is essential; therefore, [2, 3] attempts have been made to improve these absorbent polymers by enhancing their absorbency, gel strength and absorption rate by manipulating the synthesis conditions, such as solvent [4], cross-linking degree [5] and monomer composition. Their use for agriculture-based applications has shown promising results, however in order to reduce cost and efficacy, very high water absorbency and retention per unit weight of SAPs are essential. Use of expensive monomers/modifiers for increasing water absorbency will result in increased cost, rendering the resulting SAP economically unsuitable. In order to develop cheap SAP based on AAm and AAc, in the present study an effort was made to increase the water absorbency by optimizing the cross-linking parameters, such as initiator and cross-linker. Liu, ZS et al. [6] prepared high water absorbent co-polymers based on acrylic acid and acrylamide by solution polymerization. The acrylamide content was varied from 1.0% to 5% of the total monomer to synthesize the super absorbent polymers. Silva et al. [5] studied the effect of the cross-linking degree on the morphology of poly (NIPAAm-co-AAc) hydrogels using N,N' -methylene bisacrylamide as cross-linker, through SEM of dry hydrogels, changes from interconnected spherical pores, with the change in the cross-linking degrees from 3 to 5% resulting in significant change in swelling ratio. Chen et al. [7] prepared high water absorbent polyacrylates by concentrated aqueous solution polymerization within short time period, heating the monomer mixture in water bath. They investigated the influences of initiator content, bath temperature, cross-linker content (Cc), and degree of neutralization on water absorbency (Q). The effect of temperature variation during polymerization was thoroughly analyzed. In our earlier investigations [8, 9] the effect of acrylamide to acrylic acid ratio, and initiation mode (gamma irradiation and free radical initiator) was investigated by solution polymerization method.

In the present investigation, we report the synthesis of poly (acrylamide - co - acrylic acid) [poly (AAm-co-AAc)]-based superabsorbent hydrogels through solution polymerization, by varying the cross-linker concentration (N, N' -methylenebisacrylamide) and potassium per sulphate as an initiator. The acrylic acid was partially neutralized by sodium hydroxide (34.5%) in order to generate sodium acrylate partially from acrylic acid. The hydrogels with acrylamide content were prepared in order to lower the cost of resulting hydrogels. The chemical reaction is shown in Scheme 1. The acrylamide to acrylic acid ratio was selected (55:45) based on our previous results [9], as high swelling ratio was obtained for this ratio. The potassium persulfate was also used at a fixed

amount of 0.5%. The main objective of the study was to investigate the effect of cross-linker (N, N'-MBA) concentration and initiator (KPS) concentration in order to obtain high swelling hydrogels. The equilibrium swelling behaviour of the above hydrogels was studied in distilled water and different type of saline solutions. The network parameter, such as average molecular weight between cross-link (Mc) and cross-link density (q) was also determined to explain the swelling behaviour of hydrogels. The hydrogels were characterized by FTIR spectroscopy and nuclear magnetic resonance (NMR). The surface morphological studies of poly (AAm-co-AAc) were also conducted by scanning electron microscopy (SEM).

Experimental

Materials

Acrylic acid (AAc), sodium chloride (NaCl) and acrylamide (AAm) were supplied by S.D. Fine Chem. Ltd, Mumbai, India. N, N'-methylenebisacrylamide (NMBA) was supplied by BDH, England. Potassium persulphate (KPS) was purchased from Romali, Mumbai, India. Sodium Hydroxide (NaOH) was supplied from Lab Chem. Industries, Delhi, India. Distilled water was used as solvent for all experiments.

Synthesis of poly (AAm-co-AAc)

A series of poly (AAm-co-AAc)-based superabsorbents were prepared by solution polymerization, in which cross-linking agent (NMBA) concentration was changed as 0.0077 %, 0.009625%, 0.01925%, 0.0385 %, and 0.077% (based on mole of total monomer) for the same monomer concentration (55: 45); which are designated by T_{1c1'}, T_{2c2'}, T_{3c3'}, T_{4c4'} and T_{5c5'}, respectively. KPS was taken 0.5% of total monomer as an initiator. Sodium hydroxide (34.5%) solution was used to neutralize AAc upto 4.5- pH [10].

Polymerization procedure

AAm, partially neutralized AAc, NMBA and KPS were dissolved in 60 ml distilled water. This reaction mixture was taken in a wide mouth, round bottom flask fitted with a mechanical stirrer. This flask was submerged in a thermostat oil bath at 80°C, stirring was continued until the reaction mixture became too viscous and gelled. The gel point was taken to be the time, at which bubbles no longer escaped, and the stirrer stopped. The flask was then taken out of the bath and allowed to cool at room temperature (32±3°C). The reaction product was removed and cut into small pieces (2-5 mm), and dried at 60°C to constant weight. The dried product was powdered with a hammer - type mini grinder and screened using a sieve. The particles were stored in stoppered bottles.

Water - absorbency measurement

A sample (25 mg) of the poly (AAM-co-AAc) hydrogel was immersed in distilled water (or saline solutions of different concentration) at room temperature until equilibrium was reached. Absorbency was determined by weighing the swollen gel (the gel was allowed to drain on a sieve for 15 min.), and the water absorbency was calculated using the following equation (1).

$$\text{Water absorbency 'Q'} = \frac{W - W_0}{W_0} \quad (1)$$

(g H₂O/ g sample)

Where, *W* and *W*₀ are the weights of the water - swollen gel and dry absorbents respectively. Absorbency is expressed in grams of water retained in the gel by one gram of dried gel.

Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra of poly (AAM-co-AAc) hydrogels were recorded with Perkin Elmer Spectrophotometer using solid pellet potassium bromide after complete drying of the sample under vacuum at 60°C upto constant weight.

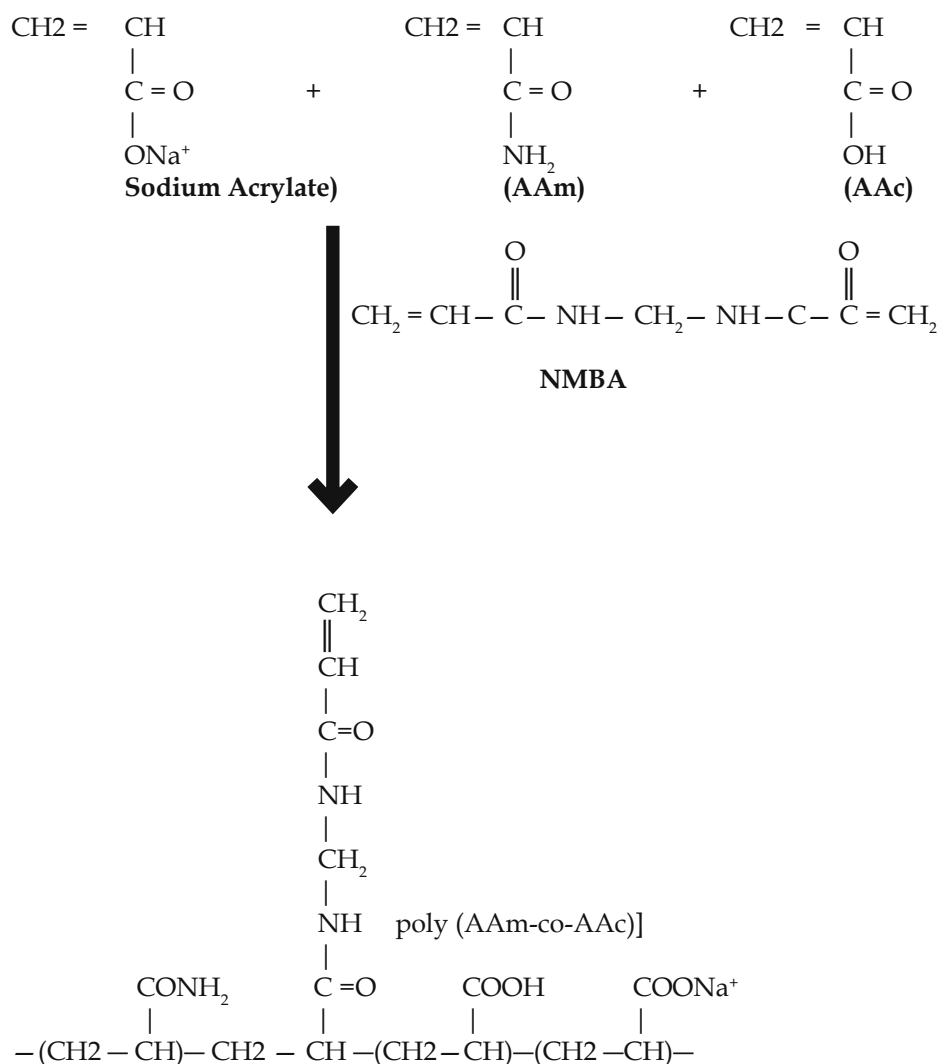
Differential scanning calorimeter (DSC)

DSC was carried out using a Perkin Elmer (Pyris Diamond) differential scanning calorimeter under the nitrogen atmosphere at a heating rate of 5°C/min up to 300°C.

Results and discussion

FTIR spectra

The FTIR spectrum of poly (AAM-co-AAc)-based superabsorbent polymer (T_{4c4}) was recorded and is presented in Fig. 1. In the FTIR spectrum, the absorbances appearing in the range of 3100–3500 cm⁻¹ are due to O-H and N-H bands. The absorbances at 1100–1225 cm⁻¹ can be attributed to C-N absorption. The weak peaks at about 2932 cm⁻¹ are C-H stretching vibrations, -CH₂- groups on the chain give absorption at 1460 cm⁻¹. The absorbances at 1663 and 1642 cm⁻¹ are of the -CONH₂ group absorption in gel and IR absorbances at 1570, 1400 and 1714 cm⁻¹ can be attributed to carbonyl absorption. The C=O bond of the amide group was observed at 1712.15 cm⁻¹ in gel. The absorbances at 1320, 1551 (-COONa) were characteristic peaks for -AAc- units and the absorbances at 3190, 3400 cm⁻¹ can be attributed to -NH₂ absorption in -AAM- units.



Scheme 1 General mechanism for polymerization of acrylamide and partially neutralized acrylic acid by solution polymerization

Nuclear magnetic resonance (NMR)

^{13}C -NMR spectroscopy is a very powerful tool for polymer structure characterization. The ^{13}C -NMR spectrum of poly (AAm-co-AAc) hydrogel (T_{4cd}) is shown in Fig. 2. It shows three resonance peaks at 40, 47, and 184 ppm respectively. Resonance peaks at 40 ppm and 47 ppm were assigned to

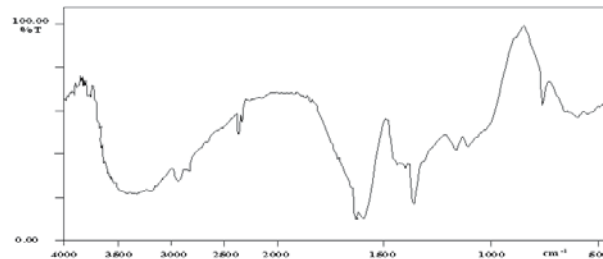


Fig. 1 FTIR spectrum of poly (AAm - co - AAc) hydrogel (T_{4c4})

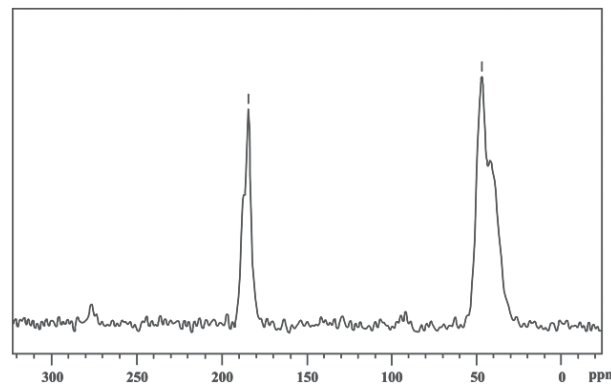


Fig. 2 NMR spectrum of poly (AAm - co - AAc) hydrogel (T_{4c4})

carbon from $-\text{CH}_2-$ and $-\text{CH}-$, respectively. Peaks at 184 ppm were assigned to carbon from $\text{C}=\text{O}$ group present in the poly (AAm-co-AAc) hydrogel. The results support the observations resulting from the FTIR spectrum of poly (AAm-co-AAc) hydrogel (T_{4C4}).

Network studies

The average molar mass between cross-link 'Mc' is used for characterization of cross-linked polymers. It is directly related to cross-link density. Flory and Rehner laid out a theory for calculation of Mc [11, 12]. According to the theory,

$$Mc = -V_1 \times d \frac{V_s^{1/3} - V_s/2}{\ln(1 - V_s) + V_s + \chi V_s^2} \quad (2)$$

Where, V_1 is the molar volume of the solvent (mL mol^{-1}), d is the polymer density (g ml^{-1}), V_s is the volume fraction of polymer in the swollen gel, and

χ is the Flory-Huggins interaction parameter between solvent and polymer. The density of various dried poly (AAM-co-AAc) hydrogels was already calculated according to Chen, J. et al. [13]. The molar volume of water at 25°C is 18 cm³ mol⁻¹ and density of water at this experiment temperature is 0.9907 gm ml⁻¹. Flory-Huggins interaction parameter between solvent and polymer were calculated according to Wei Xue et al. [14]. The swelling ratio 's' [12] is equal to reverse of volume fraction of polymer in swollen gel 'V_s'. The cross-linking ratio 'q' is defined as the mole fraction of cross-linked units [15].

$$q = \frac{M_o}{M_c} \quad (3)$$

Where, M_o is the molar mass of the repeating unit and can be calculated by following equation (4).

$$M_o = \frac{n_{AAm} \times M_{AAm} + n_{AAc} \times M_{AAc} + n_{CL} \times M_{CL}}{n_{AAm} + n_{AAc} + n_{CL}} \quad (4)$$

Where, n_{AM}, n_{AA} and n_{CL} are mole number of AM, AA and cross-linker. M_{AM}, M_{AA} and M_{CL} are the molecular weight of the AM, AA and cross-linker respectively.

Assuming isotropic swelling of the gel and using the flory characteristic ratio, C_n, for calculation of the end-to-end distance, the mesh size of a swollen polymeric network can be calculated using equation (5) [16].

$$\xi (\text{Å}) = \left[\frac{2 C_n M_c}{M_o} \right]^{1/2} \lambda V_s^{-1/3} \quad (5)$$

Where λ is the carbon - carbon bond length (1.54 Å) and the value of flory characteristic ratio 'C_n' is 6.32.

The network parameters were determined for poly (AAM-co-AAc) hydrogels having varying NMBA concentration are shown in Table 1, which shows the value of, polymer volume fraction in swollen gel, swelling ratio, average molecular weight between cross-link 'M_c', cross-link density 'q' and mesh size 'ξ (Å)'. Cross-link density is one of the most significant structural parameter for a family of hydrogel, which influenced the water absorption capacity [17]. It was clear from Fig. 3 that when water absorption capacity increased from 389 to 730; then cross-link density of the poly (AAM-co-AAc) copolymer also decreased from 3.09 × 10⁻¹⁰ to 3.07 × 10⁻¹¹. Fig. 4 shows that average molecular weight between cross-links decreased from 2.33 × 10¹² to 2.30 × 10¹¹ with increase in percentage of NMBA content from 0.009265% to 0.077%. Mesh Size of the

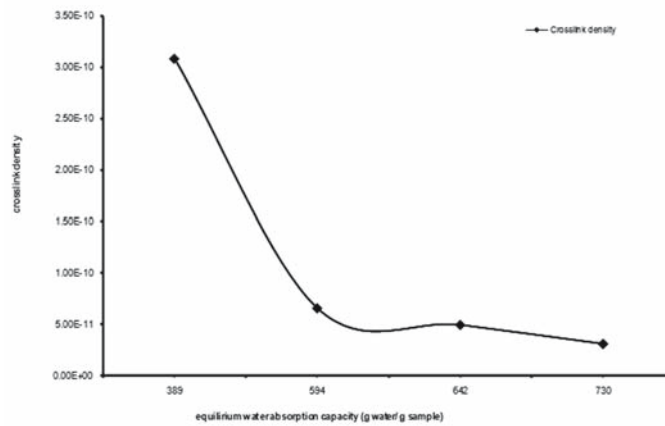


Fig. 3 Effect of equilibrium water absorption capacity of poly (AAM-co-AAc) hydrogels on cross-link density

hydrogels also affected the swelling ratio. It increased from $8.89 \times 10^6 \text{ \AA}$ to $3.00 \times 10^6 \text{ \AA}$ with increase in NMBA concentration from 0.009265% to 0.077% (Fig. 5).

Effect of cross-linker concentration

The effect of cross-linking agent on water absorbency is shown in Fig. 6. When cross-linking agent was 0.0077% , then resultant copolymeric hydrogel (T_{1c1}) was not cross-linked and after swelling, it converted into a viscous liquid with water.

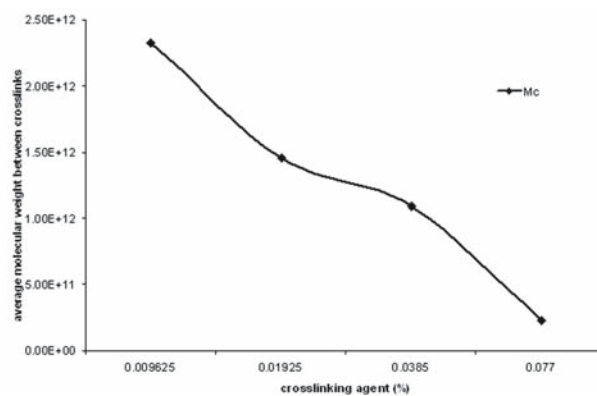


Fig. 4 Effect of cross-linking agent on average molecular weight between cross-links of poly (AAM-co-AAc) hydrogels

When the cross-linking agent was increased in the range of 0.0096% to 0.0385%, the copolymers showed equilibrium water absorbency (g water/ g sample) in the range 707.23 to 389. The T_{2C2} hydrogels with 0.0096 % exhibited highest swelling ratio 707.23; but it is disintegrated in small pieces during swelling; rendering

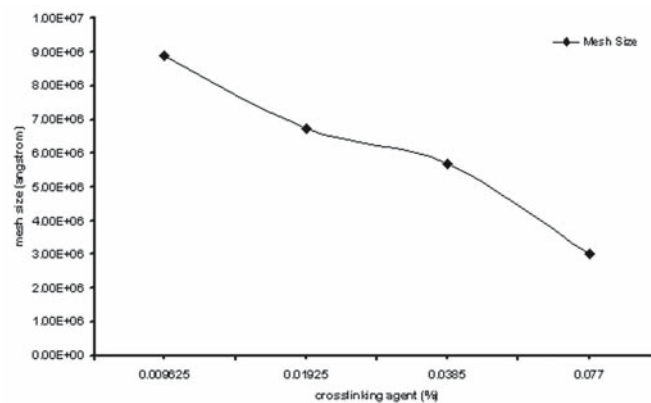


Fig. 5 Effect of cross-linking agent on mesh size of poly (AAm-co-AAc) hydrogels

it unsuitable for practical applications. Therefore, it can be stated that highest swelling ratio (cross-linker concentration) 0.0118% with reasonable strength was 594. The water absorbency decreases with increase in cross-linking agent. This is in conformity with Flory's water absorbency equation (6) [18]

$$q_m^{5/3} \approx [(i/2 v_u S^{*1/2})^2 + (1/2 - \chi_1) v_1] / v_e / V_o \quad (6)$$

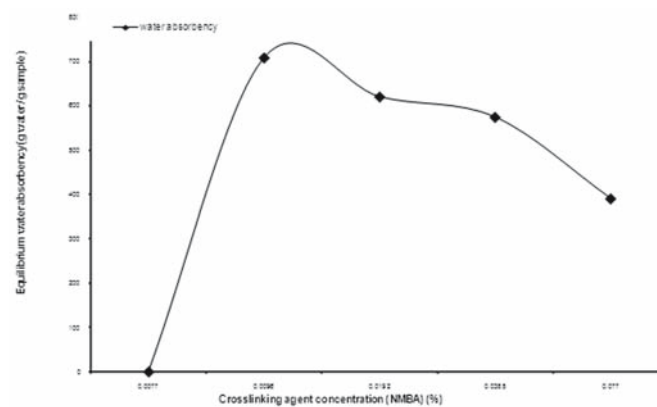


Fig. 6 Effect of cross-linking agent (NMBA) on equilibrium water absorbency of poly (AAm-co-AAc) hydrogel

Where, q_m is swelling ratio; i/v_u the concentration of the fixed charge of unswollen networks; S^* , ionic strength of the swollen solution; χ_1 the polymer – solvent thermodynamic interaction parameter; v_1 , the molar volume of the swelling agent; v_e/V_o , the cross-linking density which refers to the number of effectively cross-linked chains in unit volume.

The relation between q_m and Q is shown in equation (7) when water is the swelling agent:

$$q_m = Q\rho_p / \rho_s + 1 \quad (7)$$

where, ρ_p and ρ_s are the density of the polymer and swelling solvent, respectively. Usually, Q is very large and ρ_p is higher than ρ_s for an acrylic based superabsorbent. Then, the unit 1 in equation (7) can be omitted and we have

$$q_m \approx Q\rho_p / \rho_s \quad (8)$$

For a given chemical structure of a superabsorbent and a given solution, $[(i/2 v u S^{*1/2})^2 + (1/2 - \chi_1)/V_1]$ in equation (6) could be regarded as constant (assumption 1). From equation (6) and (7), equation (8) can be obtained:

$$Q \approx K_1 (v_e/V_o)^{-0.6} \quad (9)$$

Where, k_1 is a constant.

However, we suppose that samples at different cross-linking agent concentration (C_c) have the same cross-linking efficiency (assumption 2), so that v_e/V_o is proportional to cross-linking agent concentration, and then we have

$$v_e/V_o = k_2 C_c \quad (10)$$

Where, k_2 is a constant relevant to cross-linking efficiency. From equations (9) & (10), we can obtain

$$Q \approx k_3 C_c^{-0.6} \quad (11)$$

Where, $k_3 = k_1 k_2^{-0.6}$. Equation (11) gives the relation between water absorbency Q and cross-linker content C_c .

Grapher 1.50 (Golden Software Inc.) was used to obtain the best-fit relation between Q and C_c . The following relation $Q = 2.45 C_c^{-0.6}$ was generated. The relation is drawn by a line in Fig. 6. The empirical relation fits the experimental results well. The exponent -0.600 in the empirical relation is equal to that in equation (11) and the two assumptions in the derivation of equation (11) are also proved to be reasonable. Flory's swelling equation is usually used as a qualitative interpretation of the relation between the swelling ratio and network parameter; however relationship between Q and C_c in the study testifies to the validity of Flory' swelling equation indirectly.

SEM observations

The surface morphology of synthesized superabsorbent hydrogels was examined under scanning electron microscope (SEM). The hydrogels coated with a thin layer of pure gold are 5150-sputter coater, and imaged in a SEM (LEO Electron microscopy, England).

SEM gives an idea of the microstructure of surface hydrogels. Fig. 7a, 7b & 7c are the SEM of T_{2c2} , T_{4c4} and T_{5c5} hydrogels. The hydrogel (T_{5c5}) containing 0.077% NMBA (cross-linking agent) showing some shrinkage appears in the SEM of the hydrogel (Fig. 7a). This hydrogel shows lowest water absorbency (389, g water/ g sample). While the SEM of hydrogel (T_{4c4}) containing 0.0385% NMBA, has 573 (g water/ g sample), water absorbency shows roughness, some pores (size, 1.44 – 11.16 μm) and some beads like formations (Fig. 7b). Hydrogel (T_{2c2}) containing 0.009625% NMBA, the hydrogel is microporous and shows large number of pores of large size (size, 1.77-15.99 μm) (Fig. 7c). These observations are in good agreement with our water absorbency observations and network parameters.

Effect of initiator

The chemically cross-linked poly (AAM-co-AAc) hydrogels (55:45) prepared by using 0.1%, 0.2%, 0.3%, 0.4% and 0.5% (mole of the total monomer) as an initiator (Potassium per sulphate). Fig. 8 shows the effect of the initiator content (C_i) on

Table 1. Network parameters of poly (AAM-co-AAc) hydrogels having 55% AAm to AAac feed ratio, prepared by varying Cross-linker content (NMBA)

Sample code	Swelling ratio ' Φ '	Polymer volume fraction in swollen gel ' V_s '	Interaction parameter Between polymer and solvent ' χ '	Average molecular weight between cross-links ' M_c '	Cross-link density ' q '	Mesh size ξ (\AA)
T_{1c1}	After swelling it convert into a highly viscous liquid with water	-	-	-	-	-
T_{2c2}	730	1.37×10^{-3}	0.500	2.33×10^{12}	3.07×10^{-11}	8.89×106
T_{3c3}	642	1.56×10^{-3}	0.501	1.45×10^{12}	4.93×10^{-11}	6.72×106
T_{4c4}	594	1.68×10^{-3}	0.501	1.09×10^{12}	6.56×10^{-11}	5.68×106
T_{5c5}	389	$0.002571 \times$	0.501	2.30×10^{11}	3.09×10^{-10}	3.00×106

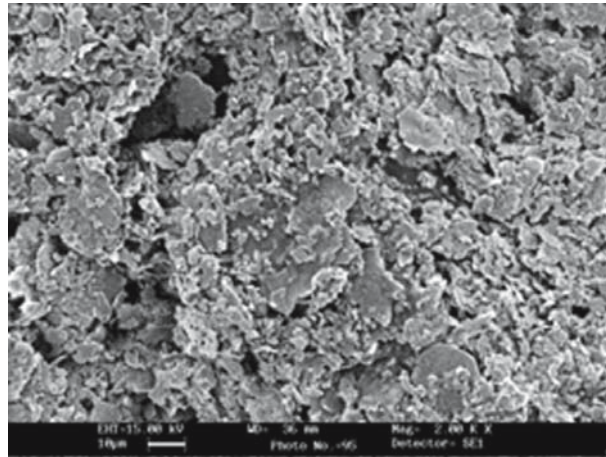


Fig. 7a Scanning electron micrograph of poly (AAm - co - AAc) hydrogel (T2c2)

the water absorbency. The water absorbency increases linearly with increase in the initiator content, when initiator content C_1 , used in the preparation of poly (AAm-co-AAc) hydrogels, increased in the range of 0.1 to 0.5% the water absorbency increased in the range of 302 to 573 g water/ g sample.

The relation between the average kinetic chain length (ν) and concentration of the initiator in free radical polymerization is given by the following equation (12):

$$\nu = \frac{1}{2} k_p (f k_i k_t)^{-1/2} [I]^{-1/2} [M] \quad (12)$$

Where, k_p , k_t , and k_i are the rate constants for propagation, initiator and termination respectively; f , the efficiency of initiation by the initiator; and $[I]$ and $[M]$ the initial concentration of the initiator and monomer respectively. The molecular weight decrease with increase of the initiator concentration according equation (12).

The relation between the swelling ratio and network structure parameter for the swelling of ionic networks was given by Flory [18] usually used as the two equivalent equations (6) and (13):

$$q_m^{5/3} \approx \left[\left(\frac{1}{2} \nu u S^{*1/2} \right)^2 + \left(\frac{1}{2} - \chi_1 \right) \nu_1 \right] \times (Mc/\rho_p) (1-2Mc/Mn)^{-1} \quad (13)$$

Where, Mc is the average molecular weight of the network chains, ρ_p the density of the polymer and Mn , is the average molecular weight of the network chains. $(1-2Mc/Mn)$ in equation (13) expresses the correction for network imperfection resulting from the chain ends [11]. The network imperfection is higher when Mn is smaller, and this also means there are more chain ends in the networks.

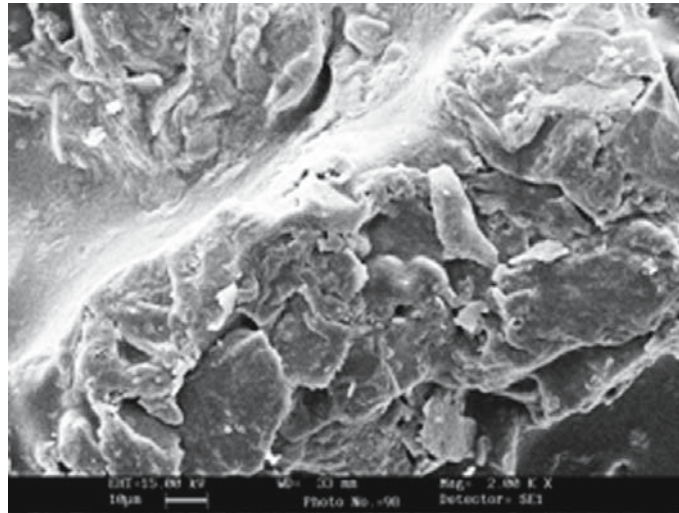


Fig. 7b Scanning electron micrograph of poly (AAm - co - AAc) hydrogel

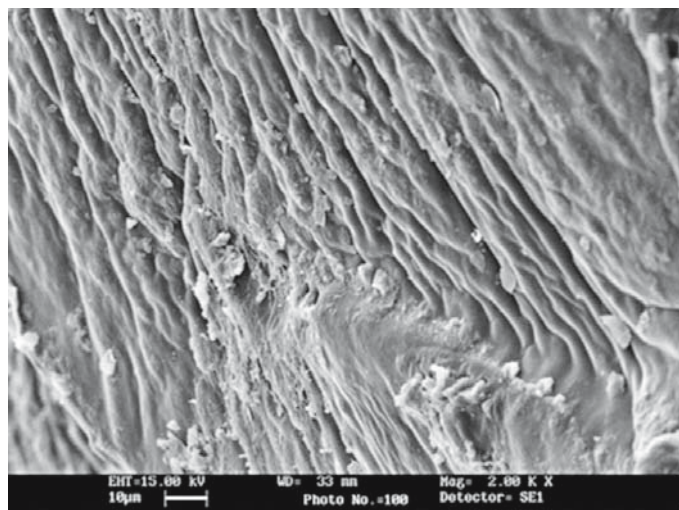


Fig. 7c Scanning electron micrograph of poly (AAm - co - AAc) hydrogel (T5c5)

From equation (13), it is predicted that networks with more chain ends have higher swelling ratios, q_m , when M_c is the same.

The reduction of molecular weight in free-radical polymerization with increased initiator content will result in increase of the relative amount of the polymer chain ends in the networks. Therefore, the experimental results in Fig. 8 are in conformity with equation (13) and directly support the Flory network theory.

Water absorbency in a saline solution

Poly (AAm-co-AAc)-based superabsorbents are ionized hydrogels whose swelling behaviour depends on both the characteristics of the chemical structure and the swelling medium. The swelling of the absorbents in saline solutions was appreciably decreased comparing to the values measured in distilled water. This well-known phenomenon, commonly observed in the swelling of ionic strength [11], is often attributed to charge screening effect of the additional cations causing a non-perfect anion-anion electrostatic repulsion, led to a decreased osmotic pressure difference between the hydrogel network and external solution. Equilibrium water absorbency of the various poly (AAm-co-AAc) hydrogels in the distilled water, 0.009%, 0.09%, and 0.9% saline (NaCl) solutions are shown in Fig. 9. Equilibrium water absorbency of the hydrogels in 0.9 % saline solution was smaller because its ionic strength is greater than that in the distilled water. The ions of the NaCl act as 'contaminator' ions and become positioned along the polymer chain at the carboxylate sites. The NaCl ions partially neutralize these sites and limit the potential sites for hydrogen bond formation between the polymer and water molecules. In addition, there will be less repulsion between the cross-linked chains of the polymer, thus enabling less water to be absorbed. The same behaviours were also reported by Mahdavinia et al. [19].

Conclusion

A series of poly (AAm-co-AAc)-based superabsorbent hydrogels were synthesized by solution polymerization with same AAm:AAc ratio and initiator; but cross-linker concentration varied over ten-fold.

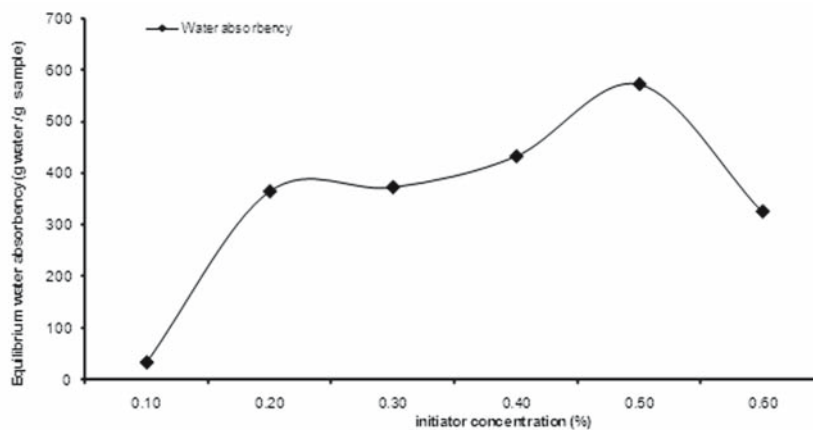


Fig. 8 Effect of initiator concentration on the equilibrium water absorbency of the poly (AAm-co-AAc)

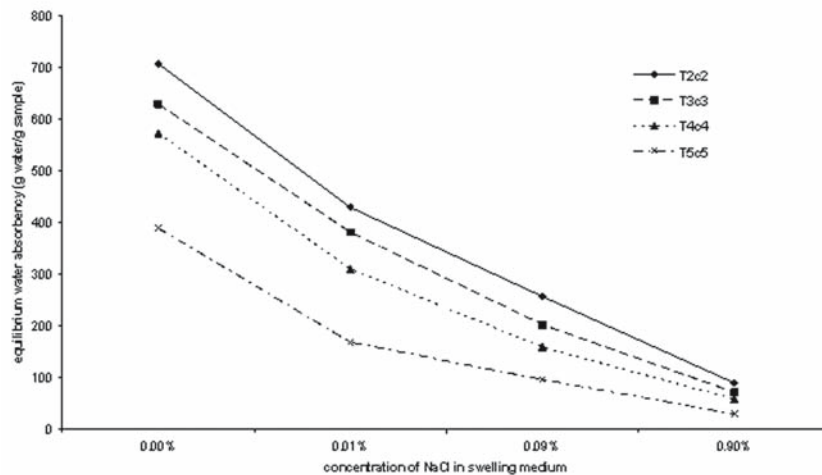


Fig. 9 Effect of NaCl solution of different concentration on equilibrium water absorbency of poly (AAM-co-AAc) hydrogels

- The experimental results showed that the equilibrium water absorbency decreases from 707.23 to 389 with increasing the NMBA content from 0.0096 % to 0.0385%.
- T_{2c2} hydrogel exhibited highest water absorbency (719 g H_2O /g sample) but it disintegrated in small pieces during swelling due to having low mechanical strength and T_{4c4} hydrogel showed water absorbency 573 g water/g sample but it doesn't disintegrated in small pieces during swelling due to high mechanical strength.
- The network parameters, such as average molecular weight between cross-links (M_c) and cross-link density (q) were decreased; although mesh size (ξ) increased with increase in percentage of NMBA content as cross-linker.
- The water absorbency is increased almost linearly with increase in the initiator content i.e. (KPS); The poly (AAM-co-AAc) hydrogels have higher equilibrium water absorbency in 0.009% saline (NaCl) solution than in 0.09% and 0.9 % saline solutions due to ionic strength.

Acknowledgements

The authors gratefully acknowledged to Mr. Anil Kumar Saini and Smt. Rekha, IIC; IIT, Roorkee for the thermal analysis and SEM analysis of the samples; and The Head, RSIC, CDRI, Lucknow for recording the FTIR spectra.

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