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Research Article

Investigation on Poly(acrylate-co-acrylamide)/polyaniline Conducting Hydrogel

Reetu Prabhakar and D. Kumar*

Department of Applied Chemistry and Polymer Technology
Delhi Technological University
Shahbad Daulatpur, Bawana Road, Delhi-110042, India

Abstract

In the present work, conducting poly(acrylate-co-acrylamide)/polyaniline (PAANa-co-AM/PANI) hydrogel was synthesized and its structural and electrical properties were studied in detail. The composite material was prepared by two step interpenetrating network solution polymerization of aniline inside the poly(acrylate-co-acrylamide) hydrogel matrix. The polymerization reactions were carried out in aqueous medium and in the presence of ammonium persulphate (APS) as initiator and N, N'-methylene bisacrylamide (NMBA) as crosslinker. Effects of crosslinker, initiator, aniline monomer, acrylamide amount and particle size of superabsorbent polymer on the synthesis and conductivity of (PAANa-co-AM/PANI) hydrogel were studied in detail. The structure and morphological analysis was done using Fourier Transform Infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) techniqes. The conductivity of P(AANa-co-AM)/PANI was found as high as 0.81 mS cm^{-1} with optimized synthesis conditions.

Keywords: copolymers; crosslinker; morphology; conductivity; swelling

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***Correspondence to**: D. Kumar, Department of Applied Chemistry and Polymer Technology, Delhi Technological University, Shahbad Daulatpur, Bawana Road, Delhi-110042, India; Email: dkumar@dce.ac.in

1. Introduction

Hydrogels are three dimensionally crosslinked water-swollen network systems formed by the polymerization of a hydrophilic monomer with the cross-linking agent [1]. Such polymer hydrogels show swelling and de-swelling behaviour under an external stimulus like pH, temperature and electric field, etc. [2-5]. Such characteristics of polymer hydrogels make them viable for applications in the field of functional materials such as biomedical materials, and as drug carriers [6-7]. Recently, superabsorbent hydrogels that have the ability to swell thousand times its dry weight have received much attention. These superabsorbent hydrogels retain their absorbed water even under pressure.

There had been only a few studies on conducting hydrogels based on superabsorbent polymers (SAP). Recently, researchers are focusing on superabsorbent polymers to extend their applications in the fields of supercapacitors, conducting materials and sensors, etc. [8]. Conducting polymer based hydrogels have been considerably focused upon and revealed to have special performances in numerous aspects [9-13]. Conducting hydrogels based on conducting polymers and superabsorbent polymers are generally synthesized *via* the swelling of polymer hydrogels in an acidic solution of monomers like aniline, followed by *in situ* polymerization in an aqueous solution of initiator and dopant [14].

There have been few reports on synthesis of conducting hydrogels based on superabsorbent polymers having conducting polymers or inorganic conducting materials as conducting fillers *via* two step interpenetrated method or free radical aqueous solution polymerization method. They proposed the interpenetrating networks of the synthesized hydrogel and further investigated the effect of synthesis parameters such as concentration of crosslinker, initiator, monomer, neutralization degree, reaction temperature and conducting material (aniline, pyrrole, graphite, copper etc.) on the conductivity of synthesized hydrogel [15-17]. Such multifunctional hydrogels can find applications as conducting materials, sensors, drug release materials and biomaterials [18].

In the present work, we have synthesized polyaniline embedded poly(acrylate-co-acrylamide) [P(AANa-co-AM)/PANI] conducting hydrogel *via* two step interpenetrating network polymerization method. The structural characterization was done using various characterization techniques. The effects of crosslinker, initiator, and aniline content on the electrical conductivity of the conducting hydrogel composites have been studied in detail. The swelling behaviour of hydrogel samples in different pH media and thermal stability of these hydrogels were also investigated.

2. Experimental

2.1 Materials

Acrylic acid (AA) and ammonium persulphate (APS) were procured from Merck, India. Aniline and crosslinker, i.e., N, N'-methylene bisacrylamide (NMBA) were purchased from SRL Pvt. Ltd., India while hydrochloric acid (HCl) was purchased from Fisher Scientific, India. All the solutions used in the experiments were prepared with double distilled water.

2.2 Synthesis of P(AANa-co-AM) superabsorbent polymer

Neutralization of acrylic acid (AA) was carried out by neutralizing the preset amount of an ice cooled AA with necessary concentration of NaOH in distilled water (12ml) under the stirring at room temperature. An optimum ratio of acrylamide and crosslinker NMBA (0.02-0.012 wt %) was added simultaneously to the neutralized AA solution. The above solution mixture was stirred for 10 min under nitrogen atmosphere and then the APS (0.05g) initiator was added to the above mixed solution under continuous stirring. The resulting hydrogel product was washed with distilled water several times and then cut into small pieces and dried under vacuum. Dried samples were crushed and passed through a sieve; a powdered P(AANa-co-AM) superabsorbent polymer was obtained.

2.3 Synthesis of P(AANa-co-AM)/PANI hydrogel

The prerequisite amount of P(AANa-co-AM) powder was dipped in different weight ratio of aqueous aniline (0.5-2 wt%) solution until uniform dispersion of aniline takes place inside the matrix. Then, the aniline absorbed matrix was placed in APS/HCl solution at room temperature. After completion of reaction, the hydrogels were filtered and washed with enough distilled water. Thus, green P(AANa-co-AM)/PANI interpenetrating network hydrogel was prepared.

2.4 Measurement and characterization techniques

Structural analysis of PAANa-PANI and P(AANa-co-AM)/PANI gels were carried out on FTIR spectrophotometer (Thermo Nicolet, Model 380) using KBr pressed pellets in the wave number region of 4000 - 400 cm^{-1}. Morphological study of samples was carried out using the scanning electron microscope (Hitachi, S–3700N). Hydrogel samples were gold coated before the SEM analysis. The crystalline structure of the prepared hydrogels was recorded using X-ray diffractometer (D8 ADVANCE of Germany BRUKER Co.) using Cu Kα radiation. The diffracted angle ranged between 5 to 50°. Thermogravimetric analysis (TGA) of the dried hydrogel sample was carried out using thermal analyzer (TA instruments, Model Q50) in an inert medium up to 600 ℃ at a heating rate of 10 ℃/min. The electrical conductivity of P(AANa-co-AM)/PANI hydrogel sample was measured at room temperature by inserting a pocket conductivity meter (HANNA 8733) in a cylinder having swelled hydrogel sample. Swelling weight ratio (SR) of the hydrogel samples was calculated at different pH using the following equation:

$$\text{Swelling weight ratio} = (W_S - W_D)/\, W_S \qquad (1)$$

Where, W_S and W_D is the fully swelled and dry weight of hydrogel sample, respectively.

3. Results and Discussion

3.1 Structural analysis

Comparative FTIR spectra of PAANa/PANI and P(AANa-co-AM)/PANI are shown in Figure 1. PAANa/PANI exhibits broad and strong characteristic bands around 3434 cm^{-1} which arises due to N-H stretching of secondary amine, while an adjacent band at 3216 cm^{-1} is observed due to N-H stretching of primary amide groups. Other appearances of characteristic bands around 2933, 1704 and 1112 cm^{-1} can be attributed to the -CH_2 stretching, C=O bending and C-H bending of PAANa unit, respectively. The peaks at around 1621 and 1411 cm^{-1} are due to quinoid and benzenoid ring stretching of PANI, respectively [19]. These characteristic bands confirm the introduction of emeraldine PANI. Furthermore, P(AANa-co-AM)/PANI also represented absorption in the similar region but with comparatively smaller peaks, which could be due to lesser interaction between PANI and P(AANa-co-AM) composite.

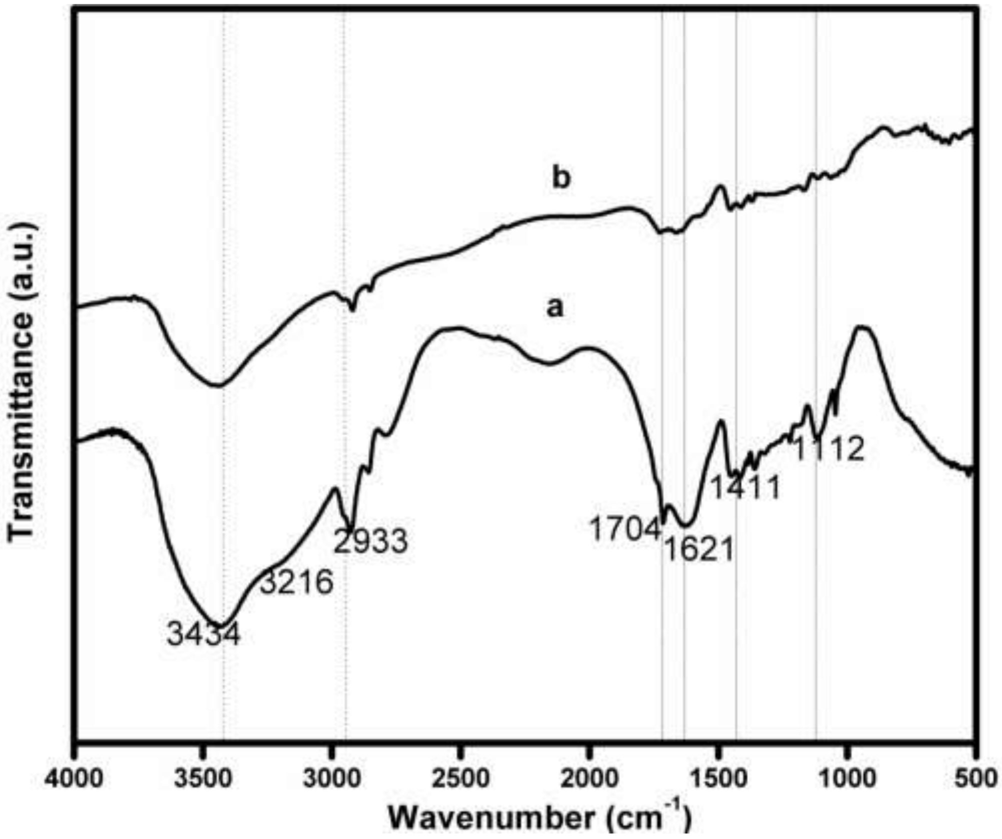

Figure 1 FTIR spectra of (a) PAANa/PANI and (b) P(AANa-co-AM)/PANI hydrogel

3.2 UV-Vis analysis

The UV-Visible study of PANI impregnated P(AANa-co-AM) hydrogel was carried out in the range of 200-800 nm. Figure 2. shows the absorption spectra of P(AANa-co-AM)/PANI with absorption peaks at around 275 and 322 nm. The absorption peak at 275 nm is attributed to the π-π* transition of benzenoid segment while, the absorption peaks (in Figure 2.) at 322 and 536 nm (broad peak) are attributed to the polaron and bipolaron transition in doped PANI. This indicates the incorporation and formation of PANI within the P(AANa-co-AM) networks [20-21].

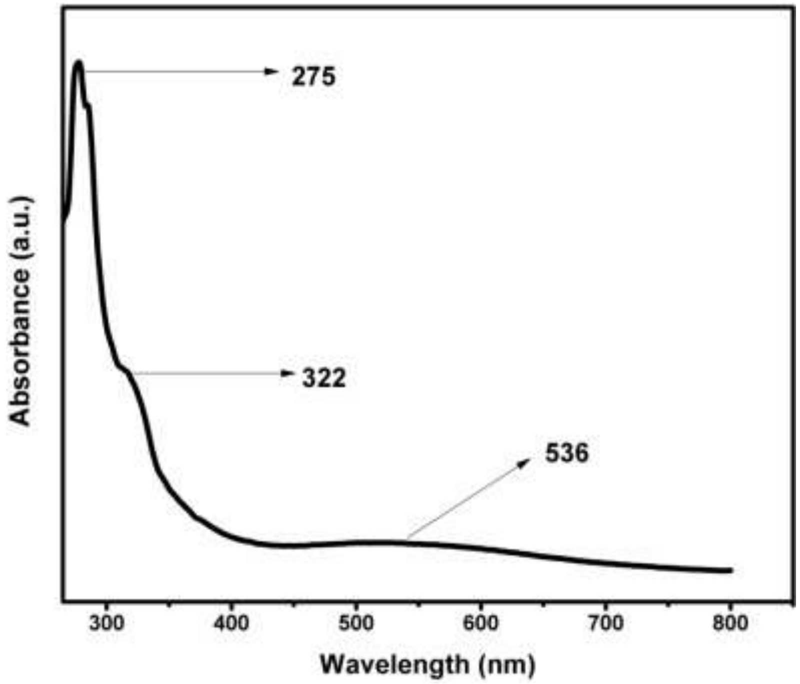

Figure 2 UV-Vis spectra of PANI impregnated P(AANa-co-AM) hydrogel

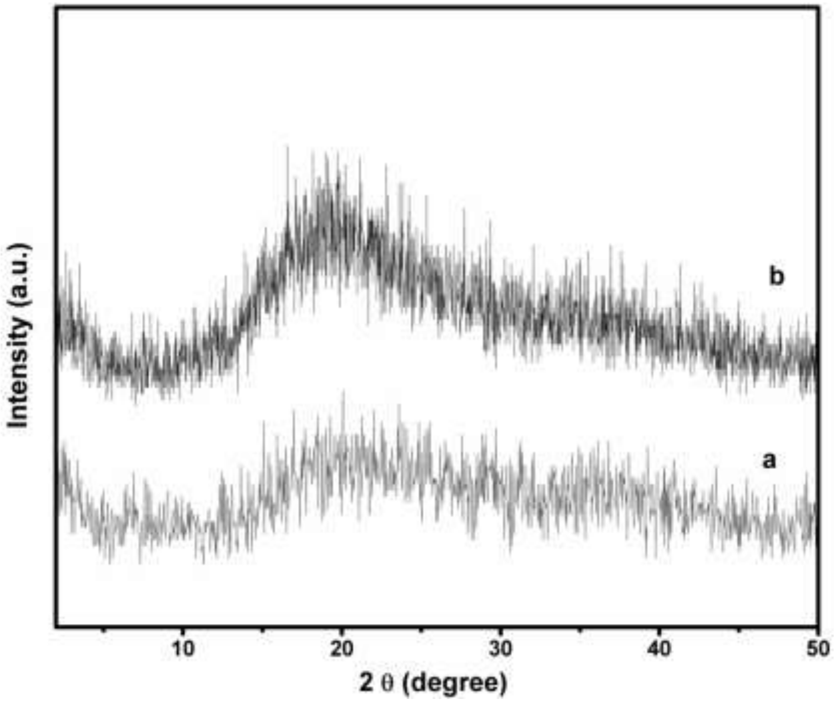

Figure 3 XRD pattern of dried (a) P(AANa-co-AM) and (b) PANI impregnated P(AANa-co-AM) hydrogel

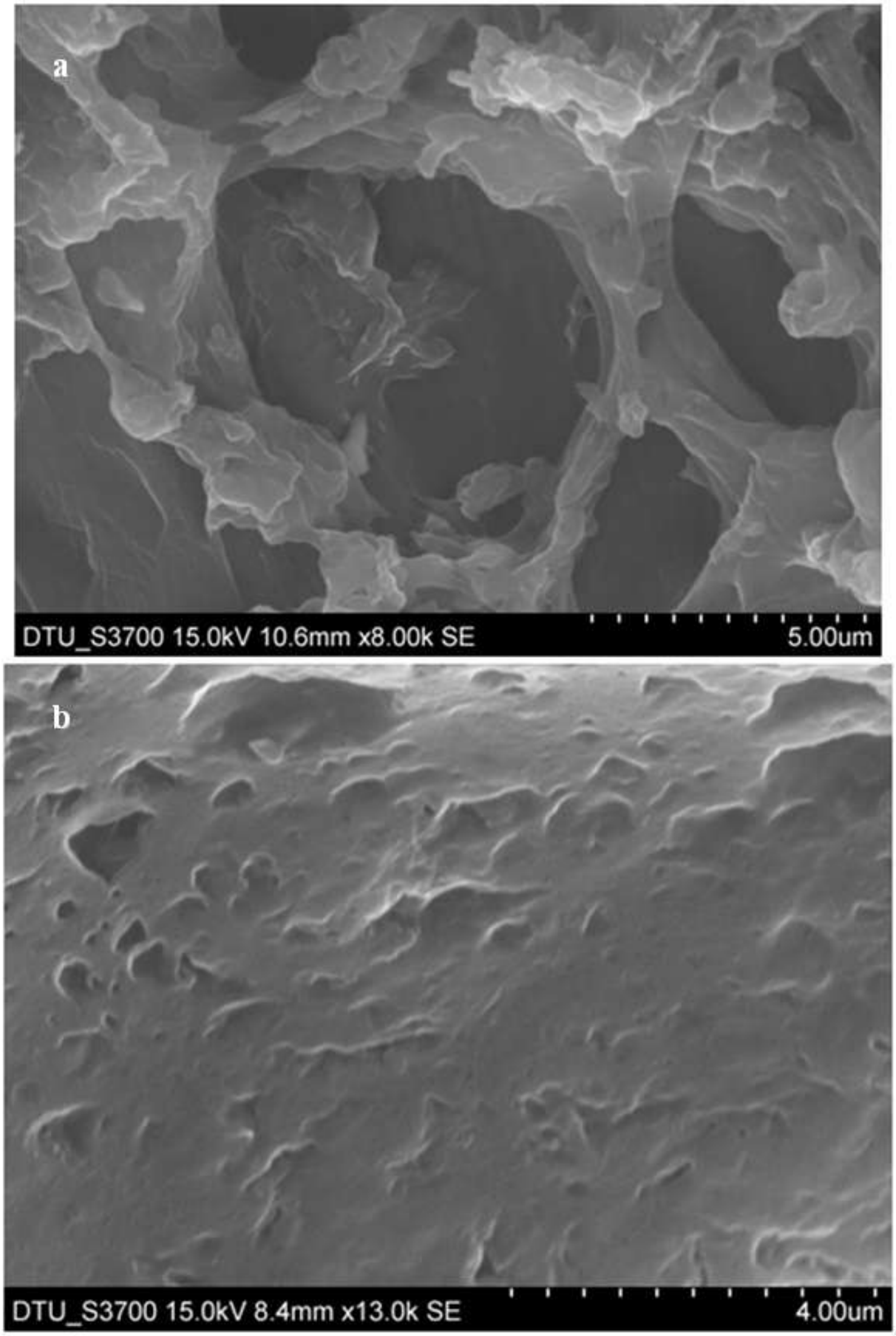

Figure 4 SEM micrographs (a) native P(AANa-co-AM) and (b) P(AANa-co-AM)/PANI hydrogel

3.3 XRD analysis

XRD patterns of P(AANa-co-AM) and PANI embedded P(AANa-co-AM) hydrogel matrix are shown in Figure 3. PANI embedded P(AANa-co-AM) gel shows a broad peak around 25^0 which is the characteristic peak of amorphous PANI (Fig. 3b) [22]. And, such a peak was not observed in the native gel (Figure 3a). This, indicates the successful interaction of PANI chains within P(AANa-co-AM) polymer matrix in P(AANa-co-AM)/PANI composite hydrogel [23].

3.4 SEM analysis

Morphological SEM micrographs of P(AANa-co-AM) and PANI embedded P(AANa-co-AM) hydrogel matrix are shown in Figure 4. It can be seen from figure that P(AANa-co-AM) shows a dense and compact structure, while composite hydrogel P(AANa-co-AM)/PANI shows a porous and crosslinked network structure, suggesting the connection and formation of PANI chain within the P(AANa-co-AM) polymer matrix.

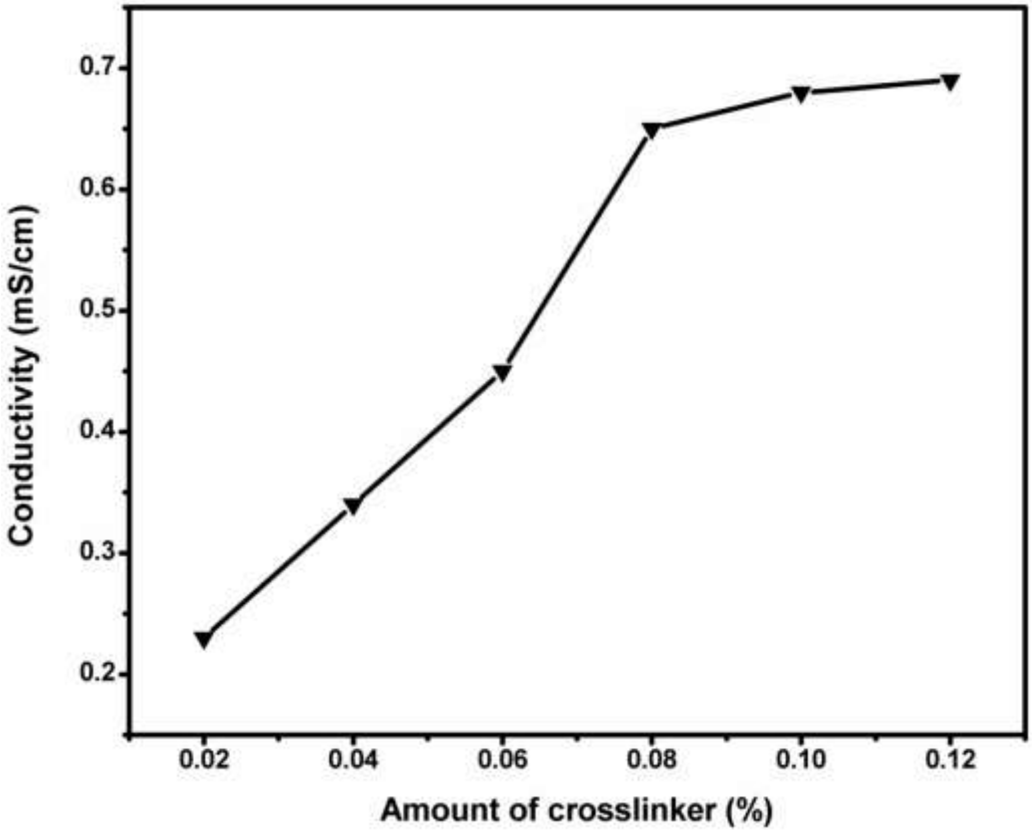

Figure 5 Effect of crosslinker on electrical conductivity of P(AANa-co-AM)/PANI hydrogel

3.5 Optimization of synthesis parameter

In order to develop a crosslinked hydrogel with good conductivity, an appropriate ratio of precursor SAP materials such as crosslinker, aniline monomer, initiator, and particle size is very important.

Influence of crosslinker, initiator composition on the electrical conductivity of P(AANa-co-AM)/PANI

It has been observed from Figure 5 that absorption of oxidant/dopant and monomer solutions in the matrix hydrogel mainly depends on three dimensional crosslinked network structure of matrix. In this work, the influence of crosslinker NMBA has been investigated with its varying concentration such as 0.02 to 0.12wt %. It has been observed that when increasing the concentration of NMBA, conductivity value increases. This could be due to the increase in hydrophilicity of network structure, allowing more impregnation of aniline hydrochloride molecule inside the hydrogel matrix [24]. However, beyond an optimum concentration of crosslinker, conductivity tends to increase at a slow rate due to the compactness in the copolymer network structure and decreased pore size of network [25]. This restricts the diffusion and chain movement of aniline hydrochloride for the formation of conducting hydrogel, resulting in a smaller increase in the conductivity of the hydrogel sample. So, suitable concentration of crosslinker is required to generate conducting network structure for the smooth diffusion and formation of conducting PANI inside the hydrogel.

Again, since hydrogel is insulating in nature so, conductivity of conducting hydrogel depends on the existent of PANI chains inside the hydrogel matrix. The polymerization of aniline inside the hydrogel depends on the APS/aniline ratio. As shown in Table 1, concentration of APS with respect to aniline is an important aspect for the polymerization of aniline inside the hydrogel. Optimum concentration of APS was found to be as 0.52×10^{-2} mol L^{-1}; lower concentration of APS does not form the proper conducting channel, and also initiates the formation of aniline over the hydrogel's superficial surface which restricts diffusion of proper amount of aniline to the hydrogel resulting in the decrease of conductivity [26]. After attaining an optimum ratio with respect to APS, the conductivity value starts to decrease again. This is because; APS not only acts as initiator but also as an oxidant and a higher concentration of APS may result in an over oxidation of aniline which decreases the conductivity of synthesized hydrogel prepared thereby.

Table 1 Effect of APS concentration on conductivity of the P(AANa-co-AM)/PANI hydrogel

APS concentration (mol L^{-1})	Conductivity (mS/cm)
0.21×10^{-2}	0.343
0.52×10^{-2}	0.591
0.11×10^{-1}	0.482

Aniline concentration is another important factor which affects the conductivity of the hydrogel sample. The effect of aniline content on the conductivity (Table 2) of the matrix has been examined by varying the concentration range from 0.5 to 2wt % and studying its effect on conductivity value. The results summarized in Table 2 suggest that conductivity continues to increase with increasing the concentration of aniline. It is found that with increasing the aniline concentration, more aniline diffuses inside the matrix and form PANI salts which results in enhancement in the electrical conductivity of the hydrogel [27].

Table 2 Variation of conductivity at different concentration of aniline with optimum amount of crosslinker and initiator

Aniline Content (wt %)	Conductivity (mS/cm)	Filtrate conductivity (mS/cm)
0.5	0.50	0.13
1.0	0.71	0.17
1.25	0.73	0.21
1.5	0.77	0.23
2.0	0.81	0.28

Compared to their corresponding filtrate, it was found that hydrogel samples have more conductivity than filtrate of hydrogel sample (Table 2). This may be because of the fact that conductivity is generated inside the hydrogel matrix due to the polymerization of aniline to PANI chains inside the hydrogel samples.

Effect of acrylamide content

The copolymer acrylamide concentration also influences the preparation condition of conducting hydrogel sample. Excessive acrylamide content could result in high degree of polymerization to occur with additional crosslinks within the polymer networks. Formation of this additional crosslinks restricting an appropriate diffusion of aniline solution to form conducting channels, which results in decrease in the electrical conductivity of the hydrogel sample. In the present study, with optimum ratio of other ingredients the maximum conductivity was observed at 0.5 mol L^{-1} of acrylamide.

Comparison of PAANa-PANI and P(AANa-co-AM)/PANI composite hydrogel conductivity

A comparison of electrical conductivity between P(AANa-co-AM)/PANI and PAANa-PANI hydrogel was studied at optimal synthesis conditions. P(AANa)/PANI hydrogel was found to have greater value 3.54 mS/cm while PAANa-co-AM/PANI showed 0.81 mS/cm. The electrical conductivity of the P(AANa)/PANI hydrogel may be more due to the formation of more crosslinks of polyacrylamide in P(AANa-co-AM)/PANI hydrogel which restrict the diffusion of aniline hydrochloride solution and movement of PANI chains inside the hydrogel matrix, resulting in lowering the value of conductivity of P(AANa-co-AM)/PANI hydrogel.

Effect of particle size

Particle size of SAP is also another factor affecting the impregnation of aniline monomer within PAANa polymer matrix. It has been found that the particle size has little effect on the conductivity value, the composite hydrogel P(AANa-co-AM)/PANI prepared in 40 mesh particle size shows conductivity value in the range of 0.76 mS/cm while prepared in 60 mesh particle size shows maximum conductivity value in the range of 0.81 mS/cm at optimum synthesis conditions. So, it can be concluded that when the hydrogels are in a state of absorbing equilibrium, minimal effect of particle size can be seen on the conductivity value of hydrogel samples.

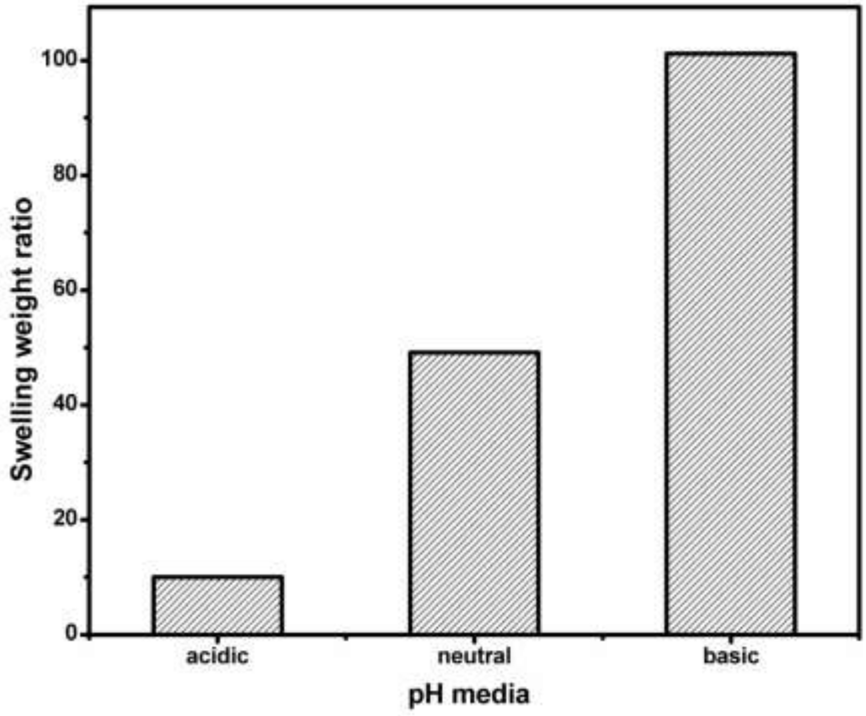

Figure 6 Swelling behaviour of PANI embedded P(AANa-co-AM) hydrogel at different pH

3.6 pH dependent swelling behaviour

The swelling equilibrium behaviour of P(AANa-co-AM)/PANI hydrogel was studied in different pH media. As shown in Figure 6, maximum swelling behaviour was observed in basic medium whereas, swelling was found to be minimum in acidic medium. In acidic medium, the hydrogel does not swell due to protonation of carboxylate groups, which disrupts hydrogel's network structure leading to the least absorption of hydrogel sample in this medium. While, in neutral medium functional groups or carboxylate/amine groups remain unionized, resulting in the formation of hydrogen bonding which decreases the swelling of hydrogel sample.

In basic medium, swelling of hydrogel increased due to the electrostatic repulsion among the corresponding carboxylate ions and amines. The two ionic species such as COO^- and $=NH^+$ are responsible for the stronger repulsion which causes the higher swelling in basic medium [28-30].

3.7 Thermal analysis

Figure 7 shows the TGA analysis of P(AANa-co-AM) and P(AANa-co-AM)/PANI hydrogels. TGA analysis shows the three distinct weight loss transitions as a function of temperature. As seen in this Figure, both P(AANa-co-AM) and P(AANa-co-AM)/PANI show the small weight loss around 100-120 ^{0}C due to the loss of moisture or water molecules [31]. P(AANa-co-AM) shows the second stage weight loss at around 220 ^{0}C which could be due to the elimination of low molecular weight compounds. The third transition is seen at about 350 ^{0}C due to the decomposition of polymer matrix.

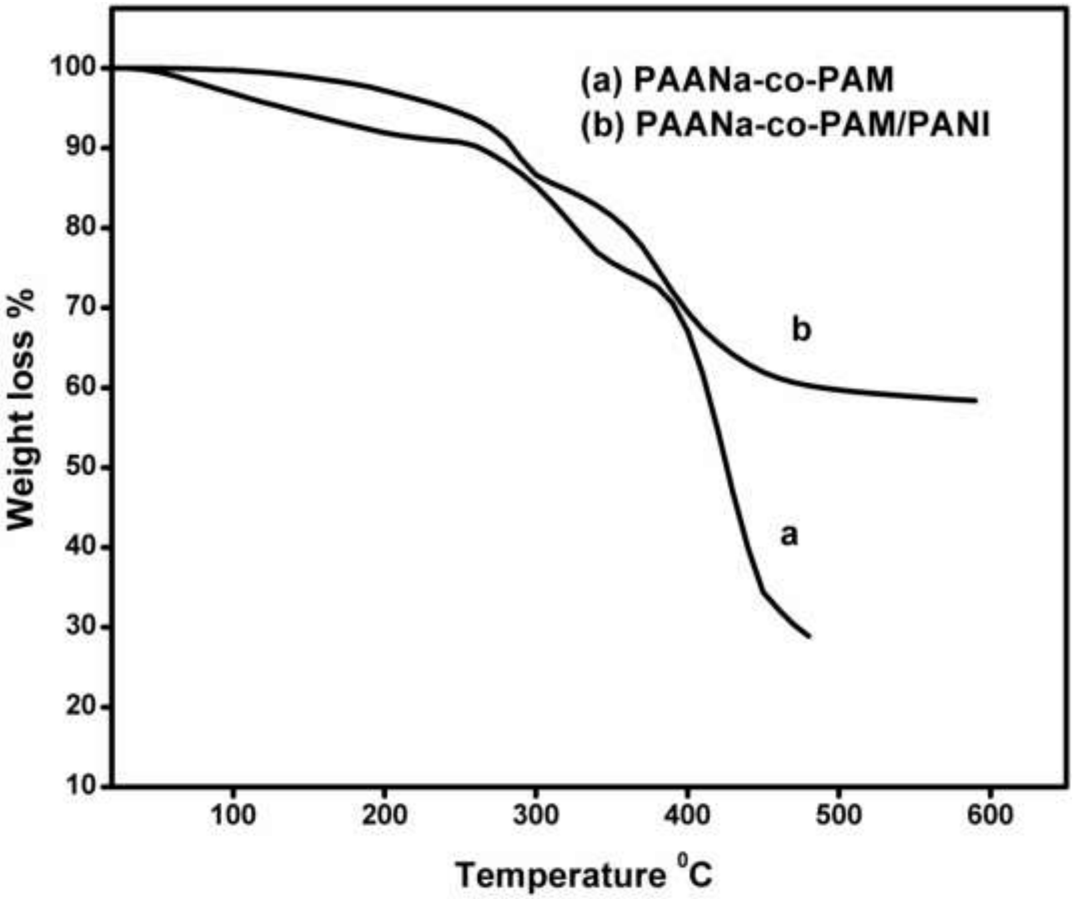

Figure 7 TGA thermograms (a) P(AANa-co-AM) and (b) P(AANa-co-AM)/PANI hydrogel

P(AANa-co-AM)/PANI composite hydrogel shows the second breakdown curve at about 240 ^{0}C associated with the loss of dopant molecule and elimination of low molecular weight substances [32]. The third weight loss at around 390 ^{0}C is due to the decomposition of the polymer. This indicates that the composite P(AANa-co-AM)/PANI hydrogel has better thermal stability as compared to P(AANa-co-AM) and it can be inferred that incorporation of PANI to the P(AANa-co-AM) has also improved the thermal stability along with an improvement in the electrical conductivity of the hydrogel sample.

4. Conclusion

A conducting polyaniline impregnated P(AANa-co-AM) hydrogel was successfully obtained *via* IPN aqueous solution polymerization by optimizing the synthesis conditions. The successful incorporation of PANI in P(AANa-co-AM) matrix was confirmed by using FTIR, UV and XRD analysis. Conductivity was found to be more in case of single PAANa/PANI based polymer than in case of PAANa-co-AM/PANI that was attributed to the presence of more crosslink in copolymer, blocking the efficient diffusion of aniline hydrochloride monomer to the matrix hydrogel.

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