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Polyaniline/polyacrylamide conducting composite hydrogel with a porous structure

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ABSTRACT

A polyaniline/polyacrylamide composite hydrogel is synthesized, characterized and measured. Fourier transform infrared spectroscopy reveals that partial polyaniline chains have grafted on the nitrogen atoms of polyacrylamide. X-ray diffraction shows that typical polyaniline crystallization is formed in polyaniline/polyacrylamide composite, which is advantageous to increase the electrical conductivity of the composite hydrogel. UV–Vis spectra indicates the formation of high conductive emeraldine polyaniline salt in polyaniline/polyacrylamide composite. Scanning electron microscopy shows a typical porous structure in the composite hydrogel. The polyaniline/polyacrylamide hydrogel has a good conductivity of 0.6 S/cm and good release stability in acidic and neutral conditions.

1. Introduction

Polyaniline (PANI) is one of the most important conducting polymers, due to its easy synthesis, electrochemical behavior, thermal and relative environmental stability. The importance of stability, conductivity and diversity in determining electronic, magnetic, optical, structural and mechanical properties of PANI have been emphasized. Unlike other conjugated polymers, PANI has a simple and reversible acid/base doping/dedoping chemistry enabling control over properties such as free-volume, solubility, electrical conductivity and optical activity.

Superabsorbent polymers, such as polyacrylamide (PAM), have a typical three-dimensional (3D) network structure with a suitable degree of crosslinking (Buchholz & Graham, 1997). Not only it is able to absorb a large amount of water to form a stable hydrogel, but also the absorbed water is hardly removed under some pressure. About three decades ago (Fanta, Baker, Burr, Doane, & Russell, 1977; Fanta & Doane, 1986), superabsorbent polymers were introduced into the agriculture and diaper industries, and then their applications were extended to other industries where an excellent water holding property was of prime importance. Recently, many researchers focus their attentions on the superabsorbent polymer for developing new applications, such as conducting materials, biomaterials, sensors, release materials and wave-absorbing materials (Lan et al., 2006; Lin, Wu, Yang, & Pu, 2001; Omidian, Rocca, & Park, 2005; Tang, Lin, Wu, Zhang, & Hao, 2007). It is expected to prepare a multifunctional superabsorbent material to meet applications requirement by modifying, grafting, copolymerizing and other methods. However, the researches on conducting composite hydrogels based on the superabsorbent polymer are still few to see (Lin, Tang, & Wu, 2007; Lin, Tang, Wu, & Hao, 2007; Tang et al., 2007). A conducting hydrogel could be used in fuel cells, supercapacitor, dye sensitive solar cell and rechargeable lithium batteries (Iwakura, Murakami, Nohara, Furukawa, & Inoue, 2005; Kato, Okazaki, & Hayase, 2006; Kim, Kim, Kim, Park, & Ahn, 2008; Nazmutdinova et al., 2006; Wu, Lan, Wang, Hao, & Lin, 2006; Wu et al., 2006), due to its better conductivity property, colloid stability, low cost and simple preparation. The aim of this work is to synthesize a novel conducting composite hydrogel with a porous structure from PANI and PAM. The structure and properties are detailedly investigated using characterizations and measurements.

2. Experimental

2.1. Materials

Aniline monomer (ANI) was distilled under reduced pressure prior to use. Acrylamide monomer (AM) was used as received. Potassium peroxydisulfate (KPS) as a radical initiator for the synthesis reaction of polyacrylamide (PAM) and polyaniline (PANI), was purified by recrystallization from 66 wt% ethanol/water solution. *N*,*N*′-methylene bisacrylamide (NMBA) was a crosslinker for preparation of PAM. Hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4) and citric acid ($C_6H_8O_7$) were typical dopants for conducting PANI. Potassium hydroxide (KOH) was used for the adjustment of alkaline solution.

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2.2. Preparation of PANI/PAM conducting composite

In a typical synthesis, the distilled aniline monomers were dissolved in acrylamide solution with a synthesis conditions varying of acrylamide dosages 5–15 g, hydrochloric acid concentrations 0.5–2.5 mol L^{-1} and volumes 5–15 ml. The mixed solution was then vacuum-degassed for 30 min. NMBA varying from 0 to 0.16 wt% (mass ratio of NMBA to acrylamide) was added to the above mixture solution. Radical initiator and oxidant, KPS (molar ratio of KPS to aniline was 0.6–1.6) was added to the mixed solution consisting of aniline monomer, acrylamide monomer and crosslinker NMBA. Under a surrounding atmosphere, the reaction mixture was stirred and heated to 80 °C in a water bath for 2 h until all the KPS dissolved and green PANI formed. The resultant plasma was dried in 80 °C drier till the weight kept to a constant. Thus PANI/PAM conducting composites were obtained and preserved in a vacuum desiccator.

2.3. Measurement of electrical conductivity of PANI/PAM composite and hydrogel

For the measurement of electrical conductivity, the powdered conducting PANI/PAM porous composite of 5 g was prepared in pellet form (diameter: 13 mm, thickness: 1 mm) at pressure of 14 MPa using a Carver model C Press. Four probes method was used to measure conductivity of composite. The powdered conducting polymer of 3 g was immersed in distilled water of 500 ml at room temperature for at least 3 h to reach swelling equilibrium, which resulted in the absorption of water inside of the network of the PANI/PAM composite and the formation of a conducting hydrogel. The unadsorbed water was removed by filtrating over a 40-mesh stainless steel screen and hanging up for 25 min. The electrical conductivity of the hydrogel washed for three times was measured by inserting a Pocket Conductivity Meter (HANNA8733) in a cylinder containing a swollen sample of 30 g.

2.4. Release experiments

Swollen PANI/PAM hydrogel of 5 g was immersed in 50 ml hydrochloric acid (pH = 1.5), distilled water (pH = 6.8), and potassium hydroxide (pH = 11.4) solutions, respectively, and the release amount of PANI was obtained by measurement of absorbency of PANI at around 208 nm reflecting π–π^* electron transition within the benzenoid segments on a UV-3100 UV–VIS–IR spectrophotometer (Shimadzu Corporation, Japan). Each experiment was repeated three times with errors of approximately ±4.5% and an average value was obtained.

2.5. Characterizations

Powder X-ray diffraction (XRD) studies were performed using D8 ADVANCE X-ray diffractometer of Germany BRUKER Co., Cu Kα (λ = 0.15406 nm), running at 40 kV and 40 mA, scanning from 2° to 60° at a speed of 5 °min^{-1}. The PANI/PAM composite hydrogel sample was mounted on metal stub, coated with gold, its surface and cross-section was observed and photographed by a scanning electron microscopy (SEM) and identified by Fourier transform infrared spectroscopy (FTIR) on a Nicolet Impact 410 infrared spectrophotometer using KBr pellets. The sample was dispersed in distilled water and measured using a UV-3100 UV–Vis spectrophotometer (Shimadzu Corporation, Japan).

3. Results and discussion

3.1. FTIR spectra of the PANI/PAM composites

Infrared spectrography is a useful technique in characterizing structures of materials. It can be seen from Fig. 1a, the peaks at ~1565 and ~1475 cm^{-1} are attributed to C=N and C=C stretching mode for the quinoid and benzenoid rings, the ~1299 cm^{-1} band is assigned to the C—N stretching of a secondary aromatic amine. The band at ~1235 cm^{-1} can be interpreted as a C—N—C stretching vibration in the polaron structure and the band at ~1119 cm^{-1} is assigned to a vibration mode of the —NH^+= structure, which is formed during protonation (Kang, Neoh, & Tan, 1998). Out-of-plane deformations of C—H on 1,4-disubstituted rings are located at 818 cm^{-1}, and the absorption at 621 cm^{-1} is caused by the deformation of the benzene ring (Trchova, Sedenkova, Tobolkova, & Stejskal, 2004). For the PANI/PAM composite, the absorption peak at ~3200 cm^{-1} is belonged to the N—H bending, ~1669 cm^{-1} is for the C=O bending for PAM (AM band I), ~1608 cm^{-1} is attributed to N—H in-plane bending in —$CONH_2$ group (AM band II), peak at ~1412 cm^{-1} is corresponding to C—N stretching (AM band III). By comparison of the spectrum of the PANI/PAM hybrid, the absorption peak at ~1608 cm^{-1} (N—H in-plane bending) for the spectrum of PANI/PAM composite has weakened and shifted to ~1576 cm^{-1}, the peak reflecting C—N stretching at ~1412 cm^{-1} has also shifted to ~1453 cm^{-1}. This indicates that partial PANI grafts on the nitrogen atom of the PAM (Xiang & Xie, 1996). According to the FTIR analysis and comparability of PANI/PAM composite and emeraldine base PANI, it can be confirmed that the composite consists of PAM and emeraldine PANI.

In the case of the PANI/PAM composites doped HNO_3, H_2SO_4 and citric acid (Fig. 1b), although absorption peaks have a slight shift compared with the composite doped HCl, it can be proved that these acids have doped into the PANI to form PANI/PAM conducting composites, respectively.

3.2. XRD pattern of the PANI/PAM composites

It is very important to study the crystal structure of conductive PANI. Because highly ordered chain structure of conductive polymer will endow highly electrical conductivity. Pouget, Jozefowica, Epstein, Tang, and MacDiarmid (1991) investigated the crystal structure of PANI prepared through conventional polymerization in detail, suggesting a pseudo-orthorhombic crystal form. In this study, we discuss the crystal structure of PANI component in PANI/PAM composite.

XRD patterns of PANI/PAM composite doped HCl (black line), PANI doped HCl (red line) and pure PAM (green line) are shown in Fig. 2. It can be seen that pure PAM shows a typical noncrystalline pattern, while in the case of the PANI/PAM composite, XRD pattern is nearly consistent with that of typical crystalline PANI powders synthesized using conventionally chemical methods. The diffraction peaks at 2θ = 6.02°, 17.03°, 20.29°, 24.03°, 25.39° and 28.56° are corresponding to the (0 0 1), (0 1 0), (1 0 0), (1 1 0), (1 1 1) and (0 2 0) reflections of the emeraldine PANI, respectively. The peaks centered at 2θ = 20.29° and 24.03° are ascribed to the periodicity parallel and perpendicular to the PANI chains. From the XRD pattern of PANI/PAM composite, the PANI has highly ordered crystal structure, which is expected to exhibit high electrical conductivity.

3.3. UV–Vis spectra of the PANI/PAM composites

UV–Vis spectra of resultant PANI/PAM composite (black line + inset a), emeraldine PANI (red line + inset b) and pure PANI (green line + inset c) in deionized water are shown in Fig. 3A. It can be seen that the PANI/PAM solution is bottle-green without any deposits, and the emeraldine PANI solution presents emerald green and even system, while for the pure PANI solution, large amount of deposits appear. There absorption peaks at 208, 300 and 796 nm, are observed in emeraldine PANI spectra. The

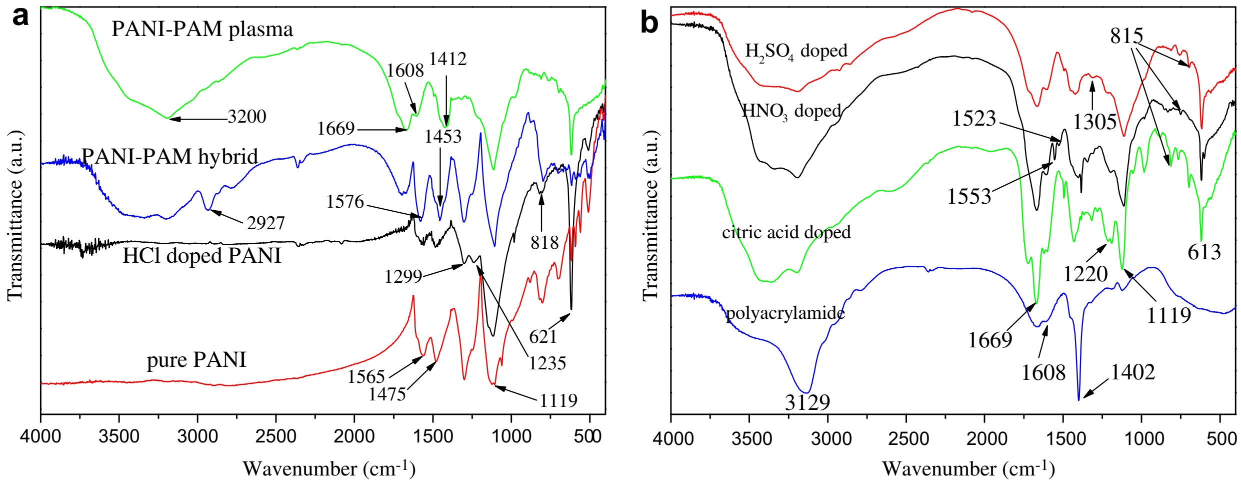

Fig. 1. (a) FTIR spectra of PANI doped HCl (black line), pure PANI (red line), PANI/PAM hybrid (blue line) and PANI/PAM composite (green line); (b) FTIR spectra of PANI/PAM composite doped H_2SO_4 (red line), HNO_3 (black line), citric acid (green line) and PAM (blue line), respectively. (For interpretation of color mentioned in this figure the reader is referred to the web version of the article.)

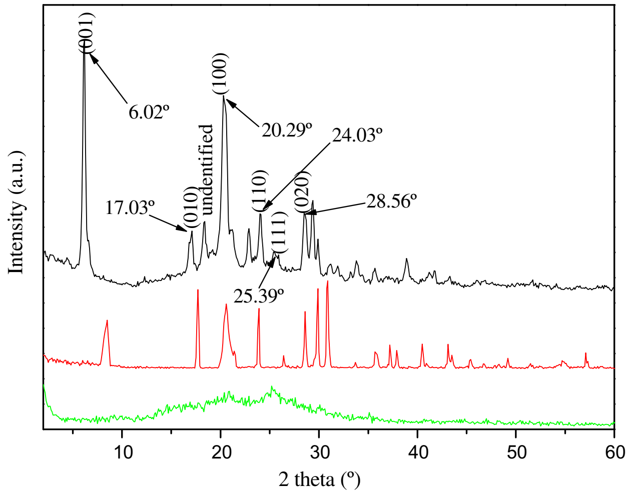

Fig. 2. XRD patterns of PANI/PAM composite doped HCl (black line), PANI doped HCl (red line) and pure PAM (green line), respectively. (For interpretation of color mentioned in this figure the reader is referred to the web version of the article.)

208 nm peak arises from π–π^* electron transition within the benzenoid segments, while the absorption peaks at 300 and 796 nm originate from the charged cationic species known as polarons (Buchholz & Graham, 1997). The almost consistence UV–Vis absorption spectra and color of the solution of PANI/PAM composite with that of emeraldine PANI indicates the formation of high conductive PANI in composite and PAM oligomer. The emeraldine oxidation state of PANI in PANI/PAM composite contain half imine and half amine nitrogens, and can be represented by the formula $[\text{—}C_6H_4\text{—}N{=}C_6H_4{=}N\text{—}C_6H_4\text{—}NH\text{—}C_6H_4\text{—}NH\text{—}]_n$ (Huang & Kaner, 2004).

Compared with the PANI/PAM composite doped HCl, the absorption spectra of PANI/PAM composites doped HNO_3, H_2SO_4 and citric acid (Fig. 3B) show red shift in both 208 and 300 nm absorption bands. For the sample doped citric acid, the absorption band at infrared region (>850 nm) disappears. This band around 850 nm, also known as the free carrier tail, is similar to that of highly conductive polymers, which confirms the expanded coil conformation of deposited PANI resulting in the polaron concentration increase and π-electrons delocalization.

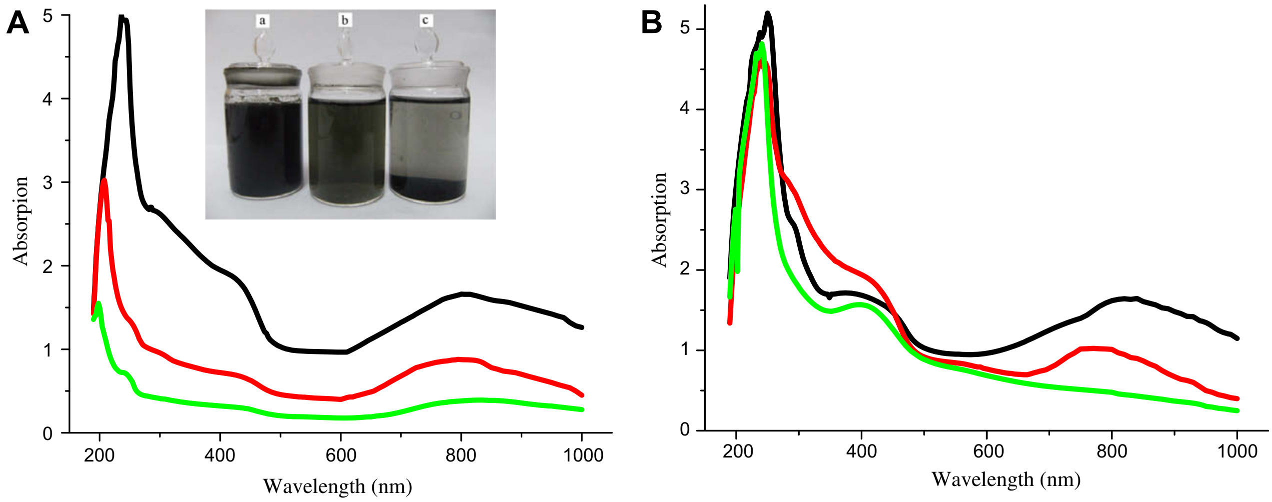

Fig. 3. (A) UV–Vis spectra of PANI/PAM composite (black line + inset a), emeraldine PANI powder (red line + inset b), pure PANI (green line + inset c); (B) UV–Vis spectra of PANI/PAM composites doped with HNO_3 (black line), H_2SO_4 (red line) and citric acid (green line), all the samples are dispersed with deionized water. (For interpretation of color mentioned in this figure the reader is referred to the web version of the article.)

3.4. Morphologies of PANI/PAM hydrogels

The PANI/PAM composite is immersed in deionized water for 24 h to reach swelling equilibrium to form a hydrogel. The swollen hydrogel is subsequently frozen in liquid nitrogen and then freeze–dried before SEM morphological investigation. Fig. 4 shows the typical SEM image of PANI/PAM hydrogel. It can be seen that a porous structure with is formed in PANI/PAM hydrogel and the pores are connective each other. Based on the porous structures of PANI/PAM hydrogels, the hydrogel is expected to have infiltration, swelling and drug release properties. A hydrogel with porous microstructure is expected to give rise to faster volume change, since the effective diffusion distance can be controlled by the average distance between neighboring pores (Arndt, Schmidt, & Reichelt, 2001). Suzuki and Hirasa (1993) synthesized bulky poly(methyl vinyl ether) (PMVE) hydrogels with a fine porous structure to obtain quick response gels. The swelling/deswelling of sponge-like gels is orders of magnitude faster than any other responsive gels of comparable dimension.

3.5. Conductivity and release of the PANI/PAM hydrogel

The electrical conductivity of the hydrogel depends on PANI chains, and the formation of the PANI chains is initiated by KPS, so the amount of initiator KPS affects the conductivity of the hydrogel. As are shown in Fig. 5, the electrical conductivity of the hydrogel (black line) increases with the increase of the mass ratio of KPS to aniline monomer in the range of 0.2–1.2, beyond a mass ratio of 1.2, the conductivity decreases. Clearly, a lower amount of KPS does not produce enough crosslink points to construct PANI chains and conducting channels, which results in the decline of the electrical conductivity of the hydrogel. On the other hand, KPS is not only an initiator, but also an oxidizer, excessive KPS causes a side reaction for oxidizing PANI, which leads to the devastation of the PANI chain in some extent, a conducting channel cannot run through the hydrogel effectively, therefore, the conductivity of the hydrogel decreases. According to Fig. 5, the mass ratio for KPS to aniline monomer 1.2 is the better. In comparison with PANI/PAM composite under the same preparation conditions, the hydrogel has the smaller electrical conductivity. It can be ascribed to the swelling of the PANI/PAM composite in the presence of distilled water. Once the PANI/PAM hydrophilic composite is immersed in distilled water, its volume will swell for several times, the PANI chains are dispersed and the conducting channels are weakened which results in smaller conductivity for the hydrogel than that for the composite. In optimized conditions, the PANI/PAM hydrogel shows electrical conductivity of 0.62 S/cm. It is accessible to obtain the hydrogels with high electrical conductivity by decreasing the swelling ratios of the composites.

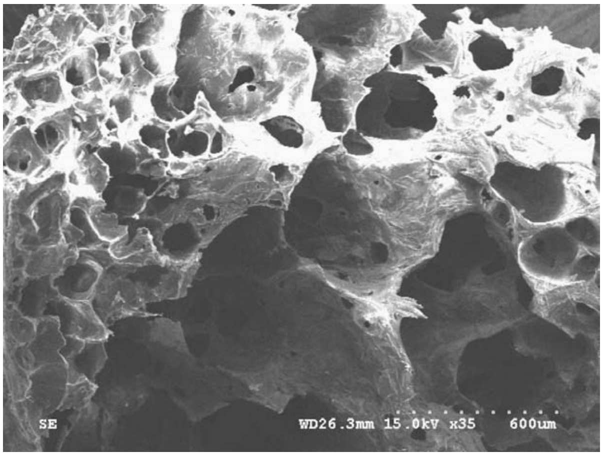

Fig. 4. SEM images PANI/PAM composite.

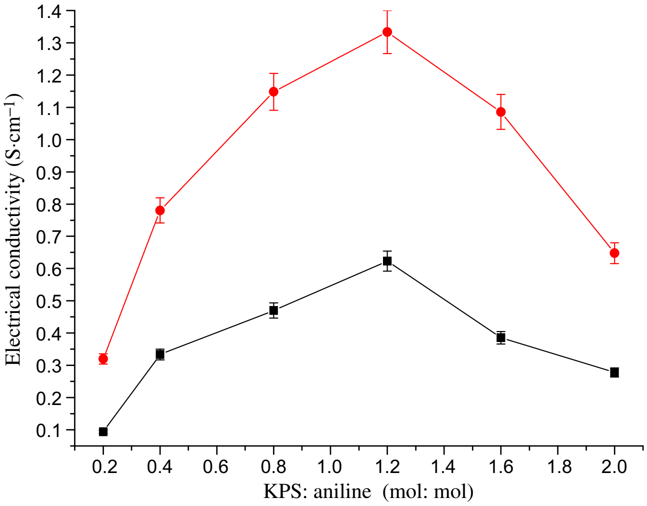

Fig. 5. Electrical conductivity of the PANI/PAM composite (red line) and hydrogel (black line) as a function of KPS dosages. (For interpretation of color mentioned in this figure the reader is referred to the web version of the article.)

The PANI release in surrounding solutions with various pHs are measured and plotted in Fig. 6. It can be seen that seldom PANI releases when the PANI/PAM composite is immerged strongly acidic (black line, pH = 1.5) or neutral aqueous solution (red line, pH = 6.8), but the release rate of PANI dramatically increase in alkaline medium (green line, pH = 14). As discussion above, in PANI/PAM composite or hydrogel, the acidic PANI chain is inlaid in the PAM network by physical or chemical crosslinking. In alkaline medium, the acidic PANI chain is neutralized. The interaction between PAM network and PANI chain is weakened, which results in the desquamation and releasement of PANI chain from the 3D

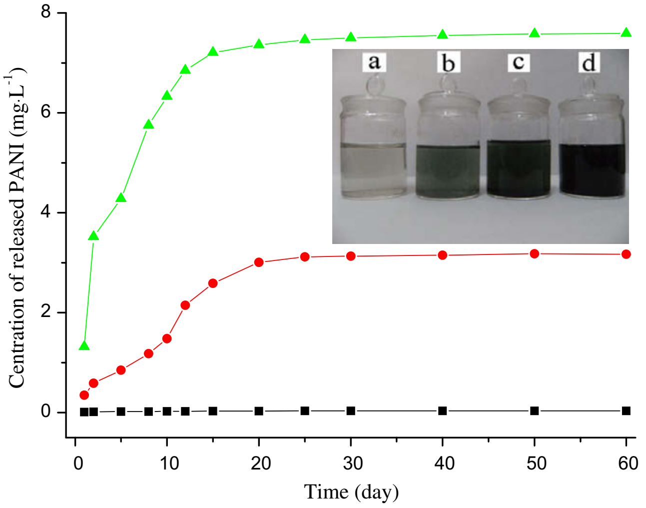

Fig. 6. PANI released from PANI/PAM hydrogel at pH of 1.5 (black line), 6.8 (red line), 11.4 (green line) and filtrate solution color at pH of 6.8 for a release time of 1 day (inset a), 5 days (inset b), 15 days (inset c), 60 days (inset d). (For interpretation of color mentioned in this figure the reader is referred to the web version of the article.)

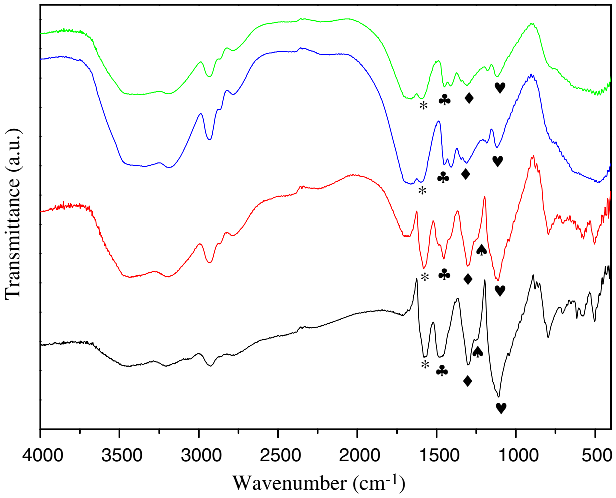

Fig. 7. FTIR of filtrate solution from PANI/PAM hydrogel for a release time of 1 (black line), 5 (red line), 15 (blue line) and 60 (green line) days, respectively. (For interpretation of color mentioned in this figure the reader is referred to the web version of the article.)

porous structure for PAM. In acidic medium, the acidic PANI chain and the interaction between PAM and PANI are keep, seldom PANI releasement appears. It is noticeable that the electrical conductivity of the PANI/PAM hydrogel decreases with the release of PANI, especially in alkaline medium, sice the conduction of the PANI/PAM hydrogel depends on the PANI chain.

The color alterations of filtrate solution under neutral condition for a release time of 1, 5, 15 and 60 days are shown in inset (Fig. 6). After one day release, the filtrate solution is nearly colorless (inset a), with prolongation of immersing time the color becomes emeraldine gradually (inset b). But once 15 days is reached, the color of the filtrate solution will be unchanged (insets c and d), which is consistent with results of PANI release measurement. From the FTIR spectra of the filtrate solutions for PANI/PAM hydrogel with different soakage times (Fig. 7), it can be seen that the peaks reflecting PANI characteristic absorption peaks at 1565, 1475, 1299, 1235 and 1119 cm^{-1} decrease with increasing of immersion time. The electrical conductivity of the PANI/PAM composite hydrogel also decreases with the diffusion of conductive PANI. So the PANI/PAM composite hydrogel is expected to be used in acidic or neutral surroundings to keep a high stability.

4. Conclusion

In this work, a novel PANI/PAM composite hydrogel was synthesized. A series of characterizations, such as FTIR, XRD, UV–Vis and SEM are carried out to identify the structure and morphology of the composite hydrogel. The results indicate that the PANI/PAM hydrogel has typical PANI crystallization and PAM porous framework structure. Owing to 3D porous structure and interaction between PAM and PANI for PANI/PAM hydrogel, the hydrogel shows a good electrical conductivity of 0.62 S/cm and hydrogel stability in acidic and neutral conditions.

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