Template method for dual network self-healing hydrogel with conductive property

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HIGHLIGHTS

• Chitosan-based self-healing hydrogels with dual-network crosslinks (hydrogen bonds and ionic linkage) were fabricated.
• The self-healing efficiency and compression deformation could attain 93.8% and 98.5%, respectively.
• Iron ions contributed to the self-healing property, compression resistance, and conductivity of the as-prepared hydrogels.

GRAPHICAL ABSTRACT

ABSTRACT

We herein present dual-network self-healing hydrogels via ionic cross-linking and hydrogel bonds based on chitosan molecular chain as a template. Chitosan and poly(acrylic acid) were reacted to form a dual-network structure via Fe3+ ion coordination and hydrogen bonds. The healing property mainly depends on the coordination between Fe3+ ions and —COOH or —NH2 groups of the polymer chains, as well as on the hydrogen bonds occurring among —OH, —NH2 and —COOH groups. Compared with the covalent cross-linking points, the ionic linkages and hydrogen bonds in the hydrogels were stochastic and dynamic. Hence, if the hydrogel becomes cracked or possesses a notch, the cross-linking points will be destroyed, while the new ones will be re-formed based on the dual-network of the hydrogel. The self-healing efficiency of the as-prepared hydrogel could reach 98.5% when the concentration range of 0.01–0.5 M Fe3+ aqueous solution was used. In addition, both the compression resistance and electrical conductivity of the hydrogel were enhanced with FeCl3 content increasing. Therefore, the as-prepared hydrogel exhibited excellent stretchability, compression resistance, self-healing performance without any external stimulus, and electrical conductivity.

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1. Introduction

Generally, the human skin, muscles, and ligaments exhibit self-repairing properties based on capsule, vascular, and intrinsic system effects. Inspired by biological systems in which damage triggers an automatic healing response, self-healing materials, which can repair damage when cracked, prolong their lifetime, and save costs, have gained increasing popularity [1–3]. Hence, self-healing materials have been attempted to be used in the fields of medical, heavy ions detection, catalyst [4,5], supercapacitor [6,7], drug delivery [8], and coating [9], just to mention a few.
Among self-healing materials, self-healing hydrogels exhibit a promising material as hydrogel is one of the most promising candidates in multiple bio-applications including drug delivery, tissue engineering [10,11], and cell proliferation temporary substrate [12]. Based on the cross-linking points, hydrogels can be divided into physical hydrogels which include cross-linking hydrogels with hydrogen bonding [13,14], electrostatic ionic force [15], π-π stacking [16], hydrophobic interaction, and host-guest inclusion [17], and chemical hydrogels which include dynamic covalent bonds, such as Schiff base linkages [18], acylhydrazone bonds [19,20], disulfide bonds [21], boron ester bonds (reaction of boronic acid with diol) [22], and non-dynamic covalent bonds that cannot be re-formed after breakage. However, of all the previously mentioned, the hydrogel with the dynamic cross-linking points, which contain either physical or dynamic covalent bonds, is more beneficial to healing after being destroyed or cut [23–25]. For example, Pan et al. [26] constructed polymer/graphene oxide (GO) nanocomposite hydrogels which achieved self-healing property based on ionic bonds between 2-(dimethylamino)ethylacrylamide and GO and the hydrogen bonds between acrylamide and GO. While Kang et al. [27] fabricated a supramolecular polymer film with water-insensitive self-healing ability via a mixture of strong and weak crosslinking hydrogel bonds. Furthermore, Nevejans et al. [28] elucidated self-healing property of poly(disulfide)s based on disulfide bonds.

Chitosan has received significant attention in biomedical sciences because of its mucoadhesivity, low cytotoxicity, biodegradability, antibacterial ability, and hemostatic ability [29–31]. Given that chitosan is derived from chitin by removing the acetyl groups, the hydrophilicity and appropriate amount of amine groups of chitosan depend on deacetylation degree [32]. Moreover, the amine and hydroxyl groups in the chitosan skeleton can be used for further functionalization [33], and formation of hydrogen bonds intramolecularly, intermolecularly and with groups such as —NH₂, —COOH, and —OH from other molecules. In addition, the amino groups of chitosan can also be protonated in acid aqueous solution and form composites with negatively charged molecules or materials [34], and can even form chelates with metal ions [35]. These properties make chitosan an ideal candidate for self-healing hydrogel material. For instance, Ren et al. [36] fabricated self-healing film using chitosan (CS) and dialdehyde functioned poly (ethylene glycol) (DF-PEG). Li et al. [37] fabricated injectable chitosan-based self-healing hydrogel for bioapplications. However, the chitosan-based hydrogels are soft and stretchable but with poor compressive capacity. So methods were explored to enhance the tough of the hydrogel based on chitosan. For example, Azevedo et al. [38] functionalized chitosan with catechol groups to form coordination bonds with Fe³⁺ ions and a chemical crosslinker was used to obtain the toughened self-healing hydrogel. Li et al. [39] also fabricated tough self-healing hydrogel as nano-fibrous aggregates of chitosan were formed in the hydrogel. Despite all these reported works, there is yet any literature report that uses physical cross-linking bonds to enhance the compression of chitosan-based self-healing hydrogel.

In this study, we report a method to fabricate self-healing hydrogels based on chitosan instead of synthesizing complex macromolecules with multi-steps under mild conditions. Generally, researching autonomous self-healing materials without external stimuli is still a challenging issue under ambient conditions. Herein, a template-based one-pot method for fabricating self-healing hydrogel is shown in Fig. 1. First, chitosan was dissolved in 1% (vol%) glacial acetic acid aqueous solution, and then acrylic acid (AA) and ferric ions (Fe(III)) were added. Herein, the chitosan molecule plays the role of a template, with AA molecules combining to the chitosan skeleton via hydroxyl bonds, while Fe(III) coordinates with —COOH of AA and —NH₂ of chitosan. After carrying out homogenous mixing, potassium sulphite (KPS) was used as an initiator to polymerize AA and form poly(AA) (PAA) through free radical polymerization. Thereafter, the double cross-linking hydrogel (labelled as CS-PAA-Fe(III)) was obtained where the first kinds of cross-linking involve intra-chain hydrogen bonding intra- or interchains of PAA and chitosan, as well as inter-chain hydrogen bonding between PAA and chitosan, while the second cross-linking is the coordination of Fe³⁺ with —COOH of PAA or —NH₂ of chitosan, or both. To observe the self-healing property of CS-PAA-Fe(III), the hydrogel was cut with a blade, and the freshly-cut section of the hydrogel re-contacted and healed without external stimulus.

2. Materials and methods

2.1. Materials

All reagents were of analytical grade and used without further purification. Chitosan (MW 400–2000 kDa, deacetylation degree > 95%) was purified by reprecipitation from the filtered 1% acetic acid solution with ammonium hydroxide. The precipitate was washed with water and dried under vacuum. The purified chitosan was thereafter dissolved in
1% acetic acid at room temperature. Potassium persulfate (K2S2O8) was recrystallized from distilled water, while acrylic acid (AA, chemically pure, Shanghai Wulian Chemical Factory, Shanghai, China) was distilled under reduced pressure before use. Except otherwise stated, all other reagents were used as received.

2.2. Synthesis of self-healing hydrogel based on chitosan

CS-PAA-Fe(III) hydrogels were synthesized by one-pot approach with chitosan as the macromolecular template; PAA, which was obtained by AA free-radical polymerization, as hydrogen bond cross-linker; FeCl3·6H2O as the ionic cross-linker; and K2S2O8 as the radical initiator. The detailed synthesis procedures are as follows: 8% (wt%) chitosan aqueous solution was prepared by mixing 1 mL of glacial acetic acid per 100 mL of distilled water. Then 3 mL AA and 0.5 mL FeCl3·6H2O aqueous solution that various concentrations (0, 0.01, 0.05, 0.1, 0.25, and 0.5 M) were added in the 5 mL of chitosan aqueous solution under vigorous stirring to obtain homogenous mixing, followed by the addition of 0.5 mL of KPS aqueous solution (0.2 M). After stirring for another 5 min, the solution was poured into a glass mold, and bubbles were removed under ultrasonication for 2 min. Subsequently, the mold was placed in an oven for approximately 3 h under 35 °C, after which the hydrogel CS-PAA-Fe(III) was obtained with the water content fixed at 61.2% (wt%). Before the uniaxial tensile measurement was conducted, all hydrogels were prepared in cylindrical shapes with 5 mm diameter and 60 mm length.

2.3. Characterization

Fourier transform infrared (FTIR) spectra was collected with the samples mixed in a KBr pellet using a Nicolet 5700 spectrometer in the region 4000–400 cm−1 (resolution: 2 cm−1, number of scans: 32), while X-ray photoelectron spectroscopy (XPS) data was collected using an AXIS HIS 165 spectrometer (Kratos Analytical, Manchester, UK) with a monochromatic Al Kα Xray source (1486.71 eV photons).

The conductivity measurements were performed on 5-mm-thick samples cut into 5 mm × 20 mm, using four-point probe measurements. The resistance values of the hydrogels and applied tensile strain were conducted using a DC resistance meter (UC2517).

The mechanical properties of the hydrogels were carried out using WDW-05 electromechanical tester (Time Group Inc, China) at room temperature. Hydrogels with a thickness of 2 mm cut into rectangle (50 mm in length (l), 5 mm in width (a), and 1 mm in thickness (b)) were used for the tensile stress test. The rate of extension was fixed at 60 mm·min−1. The nominal tensile stress (σ) and nominal tensile strain (ε) were determined by the rupture point of the stress-strain curve. The stress (σ) was calculated according to the equation: σ = F/ab where F is the recorded load and (ab) is the original cross-section of the specimen, respectively. The strain (ε) was calculated from the change of the fracture length (l) to the initial gauge length (λ0) of the measured sample, using the equation: ε = (l − λ0) × 100% while the fracture toughness was calculated by integrating the area underneath the stress-strain curve of each sample. The self-healing property of the hydrogel was tested after the original hydrogels were cut into two halves using blade at various healing time. One half of the hydrogel was dyed with Rhodamine B for visual discrimination purpose, after which the separated fresh hydrogel surfaces were re-contacted. The healing efficiency (HE) is defined as:

\[ HE = \frac{\varepsilon' \times 100}{\varepsilon} \] (1)

Where \( \varepsilon' \) is the tensile stress of self-healed hydrogel and \( \varepsilon \) is the tensile strength of original hydrogel (uncut hydrogel). The average values were calculated from at least three independent samples for each specimen.

The storage moduli and loss moduli of the hydrogel samples (cylinders, 20 mm in diameter and 2 mm in height) were measured on a Thermo HAAKE rotational rheometer (RS6000), silicone oil laid on the edge of the fixture to prevent water evaporation. The frequency sweep was performed over the angular frequency range of 0.1–10 Hz at a controlled regular strain of 1.0%. Furthermore, strain amplitude sweep was carried out to analyze elastic response of the hydrogels.

3. Results and discussion

3.1. Preparation of CS-PAA-Fe(III) hydrogel

Using chitosan as the molecular template, the polymer hydrogels were fabricated by simply adding AA and Fe3+ aqueous solution at various concentrations to chitosan aqueous solution. After KPS initiated the polymerization of AA, the hydrogel was obtained as shown in Fig. 1. The reaction temperature was 35 °C (Fig. S1). The color of hydrogels was orange (Fig. 2), which deepened with increasing Fe3+ concentration.

3.2. Chemical characterizations

Both FTIR and XPS were used to characterize the interaction of CS-PAA-Fe(III) hydrogel. FTIR was used to characterize the bonds and investigate the dynamic bonds formation in the hydrogels. The FTIR spectra of chitosan, PAA, CS-PAA-Fe(III)-0, and CS-PAA-Fe(III)-4 are shown in Fig. 3. The characteristic peaks of pure chitosan appeared at 3425, 1676, 1585, 1428, 1317, and 1159 cm−1 could be assigned to the asymmetric and symmetric vibrations of the hydroxyl group and 1657 and 1603 cm−1 for the amide I and II groups. The peaks shifted to 1624 and 1549 cm−1 confirmed the presence of -NH2 and -NH3+ both in CS-PAA-Fe(III)-0 hydrogel and CS-PAA-Fe(III)-4 [40,41]. The characteristic peaks at 3425, 1676, and 1433 cm−1 in the spectra of pure PAA can be clearly observed, which were attributed to the carboxyl groups in PAA. While the absorption peak at 1395 cm−1 could be assigned to the asymmetric and

![Fig. 2. Photographs of the CS-PAA-Fe(III) hydrogels with various Fe3+ concentration range from 0.5 M to 0 M.](image)

![Fig. 3. FTIR spectra of chitosan, PAA, CS-PAA-Fe(III)-0, and CS-PAA-Fe(III)-4.](image)
symmetric stretching vibrations of the carboxyl group of PAA both in CS-PAA-Fe(III)-0 hydrogel and CS-PAA-Fe(III)-4 hydrogel. The shift observed for these peaks confirmed that CS and PAA can react together to form hydrogen bonds [40]. Additionally, a new small peak at 1740 cm$^{-1}$ was observed in CS-PAA-Fe(III)-4 hydrogel, possibly due to the interactions between the carboxyl groups of PAA and Fe$^{3+}$ [42]. Hence, the hydrogel has a dual-network structure, which contains hydrogen bond and ionic cross-linking. The N 1s XPS of chitosan and CS-PAA-Fe(III)-4 hydrogel are shown in Fig. 4. The binding energies for N significantly changed between chitosan and the as-fabricated CS-PAA-Fe(III)-4 hydrogel. The N 1s band of chitosan at 399 eV was assigned to free amino groups (NH$_2$) [43], which was also involved in hydrogen bonding (NH$_2$-O) [44], and might come from the inter- or intra- molecular hydrogen bonds. Moreover, the N 1s binding energy attained a band 401.1 eV, a value compatible with the ionic NH$_3^+$ chemical state [43]. This finding might be attributed to the interaction of chitosan with glacial acetic acid when dissolved. The CS-PAA-Fe(III)-4 sample expressed a new band at around 402 eV for N 1s, which was assigned to the chelation between the amino groups and iron ions (NH$_2$-Fe) [44]. The results of the FTIR and XPS analyses showed that the hydrogel both possessed hydrogen bonds and ionic cross-linkings.

3.3. Mechanical properties of the CS-PAA-Fe(III) hydrogels

To analyze the mechanical properties of CS-PAA-Fe(III) hydrogels, a series of CS-PAA-Fe(III) hydrogels was prepared (Table S1). The photographs and mechanical properties of the CS-PAA-Fe(III) hydrogels obtained, are presented in Figs. 5 and 6. Usually, gels based on physical cross-linking are mechanically weak and soft due to the lack of strong interaction between the polymer chains [45–47], thus, apart from the dependence on the hydrogen bonds in this work, Fe$^{3+}$ ions was also employed to obtain ionic linkages, which could improve the mechanical properties [48]. The compression property of the prepared hydrogel is shown in Fig. 5. Fig. 5(a) and (c) present the photographs of the compression process, whereas Fig. 5(b) shows the compression stress-strain diagram of CS-PAA-Fe(III) hydrogels at different concentrations of Fe$^{3+}$. It could be observed that the compression strain of the hydrogels increased with the increasing concentration of iron ions.
Without iron ions, the compression stress and strain were 13.5 MPa and 89%, respectively. However, after adding 0.01 M of Fe(III), the compression stress increased to 21 MPa and when 0.05 M Fe\textsuperscript{3+} was used, the stress decreased back to 4.5 MPa. This phenomenon may be due to the fact that when Fe\textsuperscript{3+} was initially added at lower concentration (0.01 M) into the hydrogel, Fe\textsuperscript{3+} ions mainly coordinated with –COOH, but further addition of the Fe\textsuperscript{3+} ions resulted into expanded coordination with –NH\textsubscript{2} on the molecular chain of chitosan whose bonds are weaker than that of coordination with –COOH, thereby leading to the decrease in compressive capacity [49]. Furthermore, an increase in the Fe\textsuperscript{3+} ions beyond 0.05 M, resulted in enhanced compressive capacity of the hydrogel [50]. This result shows that when the concentration range of 0.01 M to 0.5 M Fe\textsuperscript{3+} aqueous solution was used, the compressive deformation increased from 89% to 98.5%, which can be attributed to the more dynamic ion cross-links for stress dissipation [51], indicating that Fe\textsuperscript{3+} can support the shape and increase the compression resistance property of the hydrogel.

The hydrogels were also able to withstand other forms of deformation besides compression (Fig. 5(c)), and these include large stretching (Fig. 6(a)), and knotted stretching (Fig. 6(b)). Fig. 6(c) shows that after the CS-PAA-Fe(III)-4 hydrogel was cut into two pieces and one piece was dyed with rhodamine B, after which the cut surfaces were kept in contact at room temperature without other stimulation for 12 h; the healed gel can also withstand stretching almost similar to the original one (Fig. 6(a)). The result indicated the mobility of the polymer chains and the reconstruction of the metal ion coordination bonds in the two sections of the hydrogel. Furthermore, Fig. 6(d) illustrates the tensile stress-strain curves of the hydrogels with different Fe\textsuperscript{3+} contents, where it can be seen that the hydrogels possess a mechanical performance with 20–264 kPa tensile strength and 650%–2390% elongations at break. With the increasing Fe\textsuperscript{3+} content, the elongation decreases to 650%, and conversely, the fracture stress increases to 264 kPa. The dynamic Fe\textsuperscript{3+} ion coordination bonds can break and re-form during stretching with the unfolding and sliding of the polymer chains which endows the hydrogel with high stretchability [2].

The self-healing property was evaluated by employing the tensile test protocol. Fig. 7(a) shows the stress-strain curves of the original and healed CS-PAA-Fe(III) hydrogels with various Fe\textsuperscript{3+} contents after 12 h. The HE of the hydrogels (Fig. 7(b)) was obtained corresponding to Fig. 7(a), which was calculated using Eq. (1). Fig. 7(a) shows that in the absence of FeCl\textsubscript{3}, the HE of CS-PAA-Fe(III)-0 hydrogel was only 70.8%, whereas that of other hydrogels increased significantly on the addition of FeCl\textsubscript{3}, indicating that the diffusion of Fe\textsuperscript{3+} ions contributed to the self-healing efficiency of the hydrogel, thereby leading to the cross-linking of polymer networks at the interface, which assisted to the healing of the hydrogel [52]. The HE of the hydrogels increased with further increase of FeCl\textsubscript{3} concentration while sacrificing the strain of the hydrogels (Fig. 7(a)). This phenomenon indicated that the elastic properties of the hydrogels decreased because the high physical cross-linking density of polymers might block the stretching of hydrogels [52]. It was also discovered that CS-PAA-Fe(III)-4 hydrogel with Fe\textsuperscript{3+} concentration of 0.25 M exhibited optimal elastic properties and healing efficiency, hence CS-PAA-Fe(III)-4 was used as a typical example for a time dependent self-healing test. Fig. 7(c) displays the typical stress-strain curves for the CS-PAA-Fe(III)-4 gel after being cut into two pieces and re-contacted at different self-healing times. This result illustrates that the tensile strength increases with increasing healing time, with 62.2% of the original strength recovered after 4 h and –93.8% after 12 h. The excellent self-healing property of CS-PAA-Fe(III)-4 hydrogel could be explained as follows. As mentioned above, the hydrogel networks consist of two types of interactions, the hydrogen bonds and ionic interactions. On one hand, the abundant carboxylic groups in the PAA backbone can produce large numbers of intra- and intermolecular hydrogen bonds [53], and produce hydrogen bonds with chitosan molecules that possess -NH\textsubscript{2} in the chain in acid aqueous solution [34,40], and the hydrogen bonds intra- or intermolecularly among chitosan chains. These hydrogen bonds produced a network of polymer chains. On the other hand, the ionic cross-linking between Fe\textsuperscript{3+} ions and the carboxyl groups of PAA, and between Fe\textsuperscript{3+} ions and amino groups of chitosan produced an ionic network of polymer chains [49,53]. The natural dynamism of hydrogen bonds and ionic interactions plays a critical role in self-healing. Therefore, the interactions between PAA and Fe\textsuperscript{3+} ions, chitosan and Fe\textsuperscript{3+}, and PAA and chitosan across the interface repaired the damage and rejoined the two pieces of hydrogels.

3.4. Rheology measurements

The mechanical properties of the CS-PAA-Fe(III) hydrogels were also characterized using dynamic rheology measurements (Fig. 8). CS-PAA-Fe(III)-4 was used as the typical hydrogel. Fig. 8(a) shows the typical plots of storage (G\textsuperscript{′}) and loss (G\textsuperscript{″}) moduli versus oscillatory strain amplitude, ɛ\textsubscript{o}. As illustrated in Fig. 8(a), at ɛ\textsubscript{o} < 100%, the hydrogel showed elastic features, which was indicated by the constant value of both G\textsuperscript{′} and G\textsuperscript{″}, with G\textsuperscript{′} > G\textsuperscript{″}. Moreover, both moduli showed weak dependency on the angular frequency ω at ɛ\textsubscript{o} < 100%. When ɛ\textsubscript{o} > 100%, the G\textsuperscript{′} value decreased rapidly and G\textsuperscript{″} approached the value of G\textsuperscript{′}, indicating the collapse of the hydrogel network [52,54] thereby resulting in a loose network (tan \( \delta = G' \)/G″ ≈ 0.5). Moreover, a frequency sweep measurement was employed to further confirm the gel-like behavior of this
hydrogel. As shown in Fig. 8(b), \( G' \) is larger than the \( G'' \) in all the studied frequency range of 0.1–10 Hz by \( G' \) of \( \approx 20,000 \) Pa and \( G'' \) of 2000–3000 Pa. Moreover, \( \tan \delta = G''/G' \) below 0.2 shows weak dependency on the frequency change.

3.5. Conductivity of hydrogels

CS-PAA-Fe(III) hydrogels exhibited favorable conductivity. As shown in Fig. 9, a high LED luminance demonstrated the conductivity of CS-PAA-Fe(III)-4 hydrogel. However, the light was turned off after the hydrogel was cut into two pieces and the LED recovered its luminance immediately when the two cut surfaces were reconnected together. This phenomenon demonstrated the excellent repeatable restoration of the conductive performance of the CS-PAA-Fe(III) hydrogel. The electrical resistivity of the original CS-PAA-Fe(III)-4 hydrogel reached \( 1.22 \times 10^{-4} \) S/cm (Table S2). A 12 h contact resulted in a nearly full restoration of the electrical properties, with electrical conductivity reaching \( 1.0 \times 10^{-4} \) S/cm. Other samples also exhibited electrochemical...
properties, with the electrical conductivities ranging from \(0.42 \times 10^{-4}\) S/cm to \(1.67 \times 10^{-4}\) S/cm. Meanwhile, the conductivity was enhanced with the increasing FeCl\(_3\) content in the hydrogels. This result indicates that introduction of Fe\(^{3+}\) and Cl\(^{-}\) can enhance the conductivity of the hydrogel [55]. In the as-prepared hydrogel with FeCl\(_3\), the Fe\(^{3+}\) ions could chelate with chitosan [35] and also interact with carboxyl groups of PAA, while there are other free Fe\(^{3+}\) ions within the hydrogel network to sustain the dynamic nature of the ionic interaction [52,55], hence the conductivity of the hydrogels could be attributed to the diffusion of ions (Fe\(^{3+}\) and Cl\(^{-}\)) in the water-containing hydrogel network.

4. Conclusion

Overall, self-healing hydrogels, which contained chitosan, PAA, and Fe\(^{3+}\) ions were fabricated. The FTIR and XPS data revealed that the hydrogels possess dual network containing hydrogen bonds and ionic linkages. Based on the dual network of dynamic hydrogen bonds and ionic linkages, the hydrogels showed remarkable self-healing efficiency that can reach up to 93.8% after 12 h. Moreover, the compressive deformation of the hydrogels increased from 89% to 98.5% when the concentration of Fe\(^{3+}\) aqueous solution range from 0.01 M to 0.5 M was used. Furthermore, the addition of FeCl\(_3\) remarkably enhances the electrical conductivity of the hydrogels, which was increased with increasing FeCl\(_3\) content. Based on the remarkable properties exhibited by the as-prepared hydrogels, they have potential applications in the field of artificial skin and wearable devices.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matdes.2018.03.047.

Fig. 9. Photographs demonstrating the electrical conductivity of the CS-PAA-Fe(III) hydrogel.

References


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