



Preparation and characterization of poly(vinyl alcohol)/sodium alginate hydrogel with high toughness and electric conductivity

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ABSTRACT

Development of bio-based hydrogels with good mechanical properties and high electrical conductivity is of great importance for their excellent biocompatibility and biodegradability. Novel electrically conductive and tough poly(vinyl alcohol)/sodium alginate (PVA/SA) composite hydrogel was obtained by a simple method in this paper. PVA and SA were firstly dissolved in distilled water to form the composite solution and the pure PVA/SA hydrogel was obtained through the freezing/thawing process. The pure PVA/SA hydrogels were subsequently immersed into the saturated NaCl aqueous solution to increase the gel strength and conductivity. The effect of the immersing time on the thermal and mechanical properties of PVA/SA hydrogel was studied. The swelling properties and the antiseptic properties of the obtained PVA/SA hydrogel were also studied. This paper provided a novel way for the preparation of tough hydrogel electrolyte.

1. Introduction

Hydrogels have attracted more and more attention for its diverse applications in various areas including tissue engineering, drug delivery, and soft machines (Li et al., 2016). Hydrogels are composed of three dimensional networks of crosslinked hydrophilic polymer chains possessing the ability to capture large amount of water. Hydrogels have the characteristic soft and rubbery consistency thus resembling living tissues (Anwar, Ahmad, Minhas, & Rehmani, 2017). Hydrogels can be fabricated from a wide range of natural polymers such as alginate sodium, chitosan, synthetic polymers such as poly(vinyl alcohol) (PVA), and the combination of natural and synthetic polymers. Synthetic polymers can offer good processability and mechanical properties while natural polymers have good biocompatibility, biodegradability and low toxicity. Blends of natural and synthetic polymers as bioartificial or biosynthetic polymeric material have attained remarkable interest for biomedical applications since last three decades because of their improved mechanical and thermal properties and biocompatibility compared to those of single component (Lin & Metters, 2006; Sionkowska, 2011).

Among these hydrogel-forming synthetic polymers PVA has attracted more and more attention because of its desirable physical properties i.e. excellent chemical resistance, biodegradability and biocompatibility (Qi et al., 2015). Moreover, PVA has been commercially produced on a large scale. PVA hydrogels have been widely used in surgical devices, synthetic cartilage, and drug delivery system.

Moreover, PVA hydrogel can be formed through both chemical and physical methods: the physical methods mainly just by repeated freezing and thawing processes producing physically crosslinked network without addition of any toxic crosslinking agents (Nugent and Higginbotham, 2007).

Sodium alginate (SA) is the sodium salt of alginic acid, a natural occurring polyanionic, non-toxic, biocompatible, non-immunogenic and biodegradable linear carbohydrate biopolymers derived from seaweed. SA is composed of two monomeric units, β -D-mannuronic acid (M-block) and α -L-guluronic-acid (G-block). SA has been widely used in biomedical gels owing to its biocompatibility, biodegradability, good gel-forming properties and low cost (Abd El-Ghaffar, Hashem, El-Awady, & Rabie, 2012; Gad, Aly, & Abdel-Aal, 2011; Huang et al., 2015; Park, Lee, An, & Lee, 2017; Safaei, & Taran, 2018; Thakur, Pandey, & Arotiba, 2016).

PVA/sodium alginate (PVA/SA) hydrogel could combine the advantages of PVA and SA. Many researchers have devoted efforts to prepare PVA/SA hydrogel (Anwar, Ahmad, Minhas, & Rehmani, 2017; Mahdavinia, Mousanezhad, Hosseinzadeh, Darvishi, & Sabzi, 2016; Martínez-Gómez, Guerrero, Matsuhira, & Pavez, 2017). Moreover, the PVA/SA hydrogels could be prepared by applying the freezing-thawing method (Hua et al., 2010). However, for many applications, the application of PVA/SA hydrogel is restricted due to the poor gel strength. There are many ways to improve the gel strength of PVA/SA hydrogel, i.e. incorporating nanoclays or nanoparticles (Yoon, Park, & Byun, 2012). Moreover, the conductive hydrogel also have attracted a lot of

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attention for their promising applications in the fabrication of bioelectrodes, implantable biosensors, electro-simulated drug release devices, and neural prostheses (Chen, Yang, Li, & Li, 2015; Shi, Ma, Peng, & Yu, 2015; Soeda, Yamagata, & Ishikawa, 2015). Currently many researchers have focused on the preparation of flexible and self-healable conductive hydrogel (Hou et al., 2017; Hur et al., 2014). However, the existing conductive hydrogel mostly suffer from the limitation of low mechanical robustness and low stretchability (Lin et al., 2016). Thus, the hydrogel with the combination of high mechanical properties and conductivity was seldomly reported. Adding the inorganic fillers is an effective way to improve the thermal, mechanical and electrical properties of PVA/SA materials. However, this method needs the expansive fillers and the hydrogel preparation process is complicated. Many researchers are working to find simple and effective ways to improve the mechanical and electrical properties of PVA/SA (Eghbalifama, Frounchi, & Dadbin, 2015; Ionita, Pandele, & Iovu, 2013; Jiao, Xiong, & Tao, 2016; Nie, Liu, & Wang, 2015).

In this study, PVA/SA hydrogel was prepared by repeated freezing and thawing processes of PVA and SA complex solution and a simply way was developed to improve the gel strength of PVA/SA hydrogel. The research results showed that the PVA/SA hydrogel possessed the high gel strength and good shape recovery ability. The PVA/SA hydrogel prepared in this study were completely prepared at the absence of toxic chemical agent and this hydrogel would show a high potential application value in many areas such as bioelectrodes and artificial tissue engineering.

2. Materials and methods

2.1. Materials

PVA (1799, M_w : ~75,000 g/mol, > 99% hydrolysis) was purchased from Sichuan Vinylon Factory. SA (chemically pure, viscosity average molecular weight, $M = 6.4 \times 10^4$ g/mol, and M/G ratio equal to 0.46 for M groups and a fraction of 0.54 for G groups) and NaCl was provided by Sinopharm Chemical Reagent Co. Ltd. Distilled water was used throughout the experiment.

2.2. Hydrogel preparation

Firstly, 1.0 g PVA and 0.1 g SA were dissolved in 10 mL distilled water at 95 °C for 1 h to obtain a homogeneous and transparent solution. Then the solution was poured into a mold of desired dimension and cooled at -20 °C for 3 h, which was followed by thawing at 30 °C for 6 h. The freezing/thawing process was repeated twice. Then the virgin PVA/SA hydrogel was obtained (as designed as PVA/SA hydrogel). For the preparation of high toughness PVA/SA hydrogel, the virgin PVA/SA hydrogel was immersed into the saturated NaCl solution at 25 °C for different time and was designed as PVA/SA-x (x represented the immersing time in the unit of min).

2.3. Hydrogel characterization

2.3.1. Water content

The water content (WC) was calculated according to the following equation: $SR (\%) = (W_s - W_d) \times 100 / W_s$; where W_s was the weight of the original hydrogel sample and W_d was the weight of the dried hydrogel sample after drying at 100 °C for 24 h in the vacuum drying oven. Triplicate measurements were performed for each sample and the average result was used.

2.3.2. Swelling properties

The swelling behavior was evaluated by immersion in excess PBS solution (pH = 7.4). Dried scaffolds were weighed (W_d) and immersed in PBS solution at 37 °C. The swollen sample was taken out at a fixed time interval, the surface water was removed using filter paper and

samples were weighed (W_s). Experiments were conducted in triplicate. The swelling percentage of the hydrogels was calculated as follows (Verma, Verma, Kar, Ray, & Ray, 2007):

$$S(\%) = (W_s - W_d) / W_d \times 100$$

The Korsmeyer-Peppas kinetic model equation was as follows:

$$\frac{M_t}{M_\infty} = kt^n$$

where M_t was the mass of water absorbed at time t , M_∞ was the mass of water absorbed at equilibrium, k was the release rate constants, and n was the diffusion constant describing the mode of the penetrant transport mechanism.

2.3.3. Apparent density

The apparent density, ρ , was calculated according to the previously reported method (Ma, Du, & Wang, 2017). The volume of each hydrogel sample was calculated by measuring its height and the diameter. Each sample was weighted by an electric balance with 0.1 mg resolution. ρ was calculated from the equation:

$$\rho = \frac{W}{\pi \times (D/2)^2 \times H}$$

where W was the weight of the hydrogel, D was the diameter, and H was the thickness of the hydrogel. The final data were the mean of five measurements.

2.3.4. Scanning electron microscope (SEM)

The cross section of the PVA/SA hydrogel was observed on a SEM instrument (FEI Nova NanoSEM 230, America) with the acceleration voltage of 3.0 kV. The PVA/SA hydrogels were firstly freeze dried, brittle fractured in liquid nitrogen and then vacuum coated with gold.

2.3.5. X-ray diffraction (XRD) measurements

The XRD pattern of PVA/SA hydrogel was measured on an X'Pert PRO (Panalytical, Netherlands). The XRD radiation was generated using Cu $K\alpha$ radiation at 40 kV and 40 mA. The scanning speed was $0.209^\circ \text{ s}^{-1}$ with the step size of 0.0167° , and recorded in the angle range of 5–65° at the ambient temperature.

2.3.6. Differential scanning calorimetry (DSC)

About 5–10 mg of each polymer hydrogel was sealed in the aluminum pan. The measuring temperature was from -40 °C to 40 °C at a heating rate of 1 °C/min under nitrogen atmosphere by NETZSCH DSC 214.

2.3.7. Tensile testing

The stretching measurements were performed using a universal tensile tester (CMT 6503, MTS/SANS, China) at 25 °C and RH of 54%. The hydrogel sheets of 50 mm long, 5 mm wide, and 3 mm thick were stretched at a tension speed of 30 mm min^{-1} to calculate the tensile strength and elongation at break. For each sample, 5–7 specimens were tested and the average data were used.

2.3.8. Electric conductivity

The electric conductivity of samples were measured through an alternating current (AC) impedance method. The measuring system and the instrument was a CHI 660E electrochemical workstation (Chenhua, Shanghai). Current ranging was 200 mA, and the frequency range was from 1 Hz to 1 MHz. The samples were cut into 5 mm (width) \times 5 mm (length) \times 1 mm (thickness), and sandwiched by stainless steels (SS304). The testing condition was at room temperature 25 °C and the humidity was 30–35%. The values of conductivity were calculated from the impedance curves.

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