



Electrically responsive materials based on polycarbazole/sodium alginate hydrogel blend for soft and flexible actuator application



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ABSTRACT

The electromechanical properties, namely the storage modulus sensitivity and bending, of sodium alginate (SA) hydrogels and polycarbazole/sodium alginate (PCB/SA) hydrogel blends under applied electric field was investigated. The electromechanical properties of the pristine SA were studied under effects of crosslinking types and SA molecular weights, whereas the PCB/SA hydrogel blends were studied under the effect of PCB concentrations. The storage modulus sensitivity and bending of the pristine SA as crosslinked by the ionic crosslinking agent were found to be higher than those of the covalent crosslinking. The storage modulus sensitivity and deflection of the SA increased monotonically with increasing molecular weight. The highest electromechanical response of the PCB/SA hydrogel blends was obtained from the blend with 0.10% v/v PCB as it provided surprisingly the highest ever storage modulus sensitivity, $(G' - G'_0)/G'_0$ where G'_0 and G' are the storage modulus without and with applied electric field, respectively, at 18.5 under applied electric field strength of 800 V/mm.

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1. Introduction

Polymer actuator technology is being developed for large deformations by repetitive molecular motions. Polymer actuator fall into two groups (Mirfakhrai, Madden, & Baughman, 2007). The first group consists of electronic electroactive polymers (EAPs) in which the dimensional changes occur through electron shifting, an example is the piezoelectric polymer (Mirfakhrai et al., 2007). The second group consists of ionic electroactive polymers (IEAs) (Mirfakhrai et al., 2007) in which the movement of ions is required to make an actuation possible, examples are conductive polymers and polymer gels (Dias et al., 2015). A conductive polymer is one attractive choice to be used to enhance specific electrical and mechanical responses of polymer gels which function as the matrix phase as soft and flexible actuator applications.

Sodium alginates (SA) are anionic biopolymers extracted from brown seaweeds. They are unbranched polysaccharide (Kuen & Mooneya, 2012). Alginates have been extensively investigated and used for many biomedical applications, due to its biocompatibility, low toxicity, relatively low cost, and they can be prepared

through gelation method at room temperature (Kuen & Mooneya, 2012). Polymer hydrogels are three dimensionally crosslinked networks composed of hydrophilic polymers with high water content. Covalent or ionic crosslinking of hydrophilic polymers are typical approaches to form polymer hydrogels, and their properties are highly dependent on the crosslinking type and crosslinking density, in addition to the molecular weight and chemical composition of the polymers (Paradee, Sirivat, Niamlang, & Prissanaroon-Ouajai, 2012). In spite of many advantages, polymer hydrogels still possess many shortcomings, for instance, poor mechanical strength, low strain, low thermal stability, which have restricted their optimal and efficient realization in actuator applications. However, some of these shortcomings can be neutralized by blending with a conductive polymer (Tungkavet, Seetapan, Pattavarakorn, & Sirivat, 2012).

There are several a conductive polymer/biopolymer gel blends that have been developed as actuating materials. Examples are polycarbazole/silk fibroin hydrogel (Srisawasdi, Petcharoen, & Sirivat, 2015), polydiphenylamine/cellulosic gel (Kunchornsap & Sirivat, 2014), poly(3,4 ethylenedioxythiophene)/alginate (Paradee & Sirivat, 2014), and polypyrrole/gelatin hydrogels (Tungkavet et al., 2012).

Polycarbazole (PCB) is one of conductive polymers that contain two six-membered benzene ring fused on sides of a five-membered nitrogen containing ring. It can be synthesized through either

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electrochemical or chemical method (Gupta, Singh, & Prakash, 2010). Its applications are for light-emitting diodes, electrochromic displays, organic transistors, and rechargeable batteries (Harun, Saion, Kassim, Yahya, & Mahmud, 2007; Raj, Madheswari, & Ali, 2010; Gupta et al., 2010) due to its high electrical conductivity.

In this work, an electroactive material was fabricated from an SA hydrogel as the matrix phase and embedded with PCB particles. The effects of type and amount of crosslinking, PCB amount, and electric field strength were systematically investigated on the storage modulus response and bending.

2. Material and methods

2.1. Materials

Carbazole (CB) (Synthesis grade, Merck) was used as the monomer. Ammonium persulfate (APS) (AR grade, Sigma-Aldrich) was used as the oxidizing agent. Hydrochloric acid (HCl) 37% and dichloromethane (DCM) (AR grad, Labscan) were used as solvents. Cetyltrimethyl bromide (CTAB) (AR grade, Sigma-Aldrich) was used as surfactants. Ammonium hydroxide (NH₄OH) 30% (AR grade, Pan-reac). Sodium alginates of different molecular weights were used as matrices. Citric acid (AR grade, Merck) was used a covalent crosslinking agent and calcium chloride (AR grad, Fluka,) as an ionic crosslinking agent; both were obtained and used without further purification. Deionized water was used in all experiments.

2.2. Preparation of PCB

PCB was synthesized with CTAB added via interfacial polymerization method (Gupta et al., 2010) using CB monomer, APS, HCl, and DCM. The reaction was carried out at 25 °C. The APS (1.2 M) was dissolved in 50 ml of 0.5 M HCl solution as an aqueous phase. The CB monomer (60 mM) was dissolved in 50 ml of DCM with various monomer: CTAB mole ratios, to finally obtain a non-aqueous phase. Then, the aqueous and non-aqueous solutions were mixed for 24 h to induce CB monomers at the interface between the two immiscible phases. After filtering, the green precipitate was collected and dried in oven at 80 °C for 24 h. Dedoping PCB was performed by stirring the obtained PCB in NH₄OH solution of the PCB: NH₄OH mole ratio of 1:10 at 25 °C for 24 h. Then, PCB was doped by HClO₄ at the HClO₄: N_{PCB} mole ratios of 5:1, 10:1, 50:1, and 100:1.

2.3. Preparation of SA hydrogels and PCB/SA blends

SA solutions were prepared from three SA molecular weights namely low molecular weight SA (LSA) of 2.83×10^5 g/mol, medium molecular weight SA (MSA) of 3.34×10^5 g/mol, and high molecular weight SA (HSA) of 4.57×10^5 g/mol in distilled water (1.0% v/v) at 25 °C under continuous stirring for 40 min (Kulkarni, Soppimath, Aminabhavi, Dave, & Mehta, 2000). The crosslinking of SA was carried out by the ionic and covalent crosslinking methods (Kuen & Mooneya, 2012). In case of the ionic crosslinking method, it was prepared by adding the appropriate volume of CaCl₂ solution to the SA solution at 25 °C under continuous stirring for 30 min with CaCl₂ concentrations varying from 0.0050, 0.0100, 0.0150, and 0.0200% v/v. In case of the covalent method, it was prepared by adding the appropriate volume of citric acid solution to the SA solution at 80 °C under continuous stirring for 30 min with citric acid concentration varying from 0.25, 0.50, 0.75, and 1.0% v/v. The SA mixture solutions were poured into plastic petri dishes (10 cm of diameter). The SA hydrogels were obtained after allowing water evaporation at 25 °C for a period of 2 days and kept at 4 °C for controlling water content in the SA hydrogel samples (~1.5 min in thickness).

The PCB/SA hydrogel blends were prepared by mixing PCB with a SA solution. PCB powder was dispersed in 70 ml SA solution

filled with 0.05 M of sodium dodecyl sulfate (SDS) as the surfactant (Srisawasdi et al., 2015) and with 0.015% v/v CaCl₂ as the ionic crosslinking agent. The PCB concentration was varied from 0.01, 0.05, 0.1, 0.5, 1.0, 3.0, and 5.0% v/v. The solution was then poured into a plastic petri dish for casting at room temperature for 2 days to obtain the hydrogel blend samples (~1.5 min in thickness).

2.4. Characterizations and testing

2.4.1. The electrical conductivity

The electrical conductivity of the synthesized polycarbazole was determined by pressing the PCB powder into disk pellets under the pressure of 7 kg (diameter of 10 mm and 0.2 mm thickness). Electrical conductivity was measured using a custom-built two-point probe meter connected with a voltage supplier, in which voltage was varied and the resultant current measured in the regime where the resultant current is linearly proportional to the applied voltage, or the linear Ohmic regime. The voltage and the current in the Ohmic regime were converted to the electrical conductivity via Eq. (1):

$$\sigma = 1/\rho = 1/(R_s \times t) = I/(K \times V \times t) \quad (1)$$

where σ is the specific conductivity (S cm⁻¹), ρ is the specific resistivity (Ω cm), R_s is the sheet resistance (Ω sq⁻¹), t is the thickness of the sample pellet (cm), V is the applied voltage (V), I is the measured current (A), and K is the geometric correction factor of the two-point probe meter, K as 4.29×10^{-4} . All sample thicknesses were measured by a thickness gauge (Peacock, PDN-20).

2.4.2. Fourier transform infrared spectrometer

A fourier transform infrared spectrometer, FT-IR (Nicolet, Nexus 670) was used to characterize functional groups of PCB powder, SA and PCB/SA films. For the PCB powder used the in KBr-pellet technique, optical grade KBr (Carlo ErBr Reagent) was used as the background material. The PCB powders were intimately mixed with dried KBr at the PCB:KBr weight ratio of 1:20, then they were compressed into pellets under the force of 7000 kg. The technique used the absorption mode with 32 scans and a resolution of ± 4 cm⁻¹, covering a wavelength range of 4000–400 cm⁻¹.

2.4.3. X-ray diffractometer

An X-ray diffractometer, XRD (Rigaku/Smartlab) was used to investigate the degree of crystallinity of the hydrogel samples (sample size: 2 cm² square disk with ~1.5 mm in thickness). The diffractometer was operated in the Bragg–Brentano geometry and fitted with a graphite monochromator and the diffracted beam using a 5°/min scan rate.

2.4.4. Thermal gravimetric analyzer

A thermal gravimetric analyzer (DuPont, TGA 2950) was used to determine the amount of water content and the decomposition temperatures of PCB powers, SA hydrogels and PCB/SA hydrogel blends. The thermal behavior was investigated by weighting each sample of 5–10 mg and loaded into a platinum pan and then heating it under nitrogen flow with a heating rate of 10 °C/min from 30 to 800 °C.

2.4.5. Scanning Electron Microscope

A Scanning Electron Microscope, SEM (Hitachi, S4800) was used to examine the morphological structure of PCB and the dispersion of PCB in SA hydrogels. Each sample was placed on the holder with an adhesive tape and with a thin layer of platinum using a JEOL JFC-1100E ion sputtering device for 1 min to observation under SEM. The scanning electron images were obtained by using the acceleration voltage of 10 kV with the magnification of 3.0 k.

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