



Double-side coated electrochemical actuator based on changes in volume of poly(acrylic acid) gel



Naoki Hisamatsu, Tomoya Iida, Takashi Yasui, Kazutake Takada*, Akio Yuchi

Graduate School of Engineering, Nagoya Institute of Technology, Gokiso, Showa, Nagoya 466-8555, Japan

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ABSTRACT

An actuator consisting of two poly(acrylic acid) gel films which sandwich a polyimide film coated with Au layers on both sides was developed. The actuator was driven by changes in the volume of the gel induced by redox reaction of *p*-hydroquinone (QH₂)/*p*-benzoquinone (BQ). The oxidation of QH₂ results in decrease in pH, which gives rise to collapse of the gel, whereas the reduction of BQ does the opposite phenomena. The Au film electrodes on both sides of the actuator were used as two electrodes setup. Thus, when the oxidation takes place at one electrode, the reduction does on the other electrode at the opposite side at the same time. In a solution of 5 mM QH₂ and 5 mM BQ containing 1×10^{-4} M NaClO₄ the actuator bent to left (anode) side at 1.3 V, and bent to right side after the reversal of the polarity. The actuator showed repeated stretching/bending at least 200 cycles. Further, it was able to actuate after 6 times of repeated drying/wetting of the gel.

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1. Introduction

There has been a great deal of interest in actuators based on soft materials because of potential applications such as nano- and micro-scale actuators, micromanipulators, and artificial muscles [1]. One of the most widely used materials is polymer gel because it can change its volume in response to variety of stimuli such as light intensity [2], temperature [3], pH [4], ionic strength [5], voltage/potential [6,7], and magnetic field [8]. Among them electric potential has been one of the most extensively used stimulus since it can be controlled easily and precisely and a setup is relatively small and simple. There has been reported a polymer gel actuator driven by redox reaction of species incorporated in the gel [9]. It can be operated at low potential of a few volts, which depends only on a redox species used.

We have recently developed a redox actuator consisting of a single poly(acrylic acid) gel film, which was immobilized on an film electrode (bimorph type), incorporating *p*-hydroquinone (QH₂) [10]. The actuator stretched upon oxidation of QH₂ at +1.0 V vs. Ag/AgCl in a 10 mM quinone solution containing 1×10^{-4} M NaClO₄, whereas it bent upon reduction of benzoquinone (BQ)

at −0.3 V. The mechanism of the actuation is explained as follows. Oxidation of QH₂ to BQ releases protons resulting in pH decrease, which, in turn, gives rise to shrinking of poly(acrylic acid) gel due to increase in osmotic pressure of the solution, decrease in hydrophilicity arising from protonation of carboxylates of the polymer chain, and/or decrease in electrostatic repulsion. On the other hand, reduction of BQ consumes protons resulting in pH increase, which gives rise to swelling of the gel due to the opposite phenomena. Since both reduced and oxidized forms of the quinone are soluble in aqueous solution, it has a low impact to the gel, which enhanced repeatability in comparison to the actuator consisting of poly(acrylic acid) gel and Cu²⁺ ion [9], which deposits on the electrode upon reduction. In addition the faster diffusion of proton improved the actuation rate.

However, in that study, the energy conversion efficiency was reported to be low (10^{-3} %). One of the reasons was ascribed to a loss of energy at a counter electrode of a three electrodes set up, which is not utilized in the actuation. In order to improve this, we developed an actuator consisting of two gel films which sandwich a polyimide film coated with Au layers on both sides. This configuration allows the actuator to be operated as a two electrode system. Thus, while oxidation QH₂ is taking place at one electrode, reduction of BQ can take place at the other electrode on the reverse side. Therefore, the actuator was expected to indicate higher performances such as energy conversion efficiency and actuation speed.

* Corresponding author.

E-mail address: takada.kazutake@nitech.ac.jp (K. Takada).

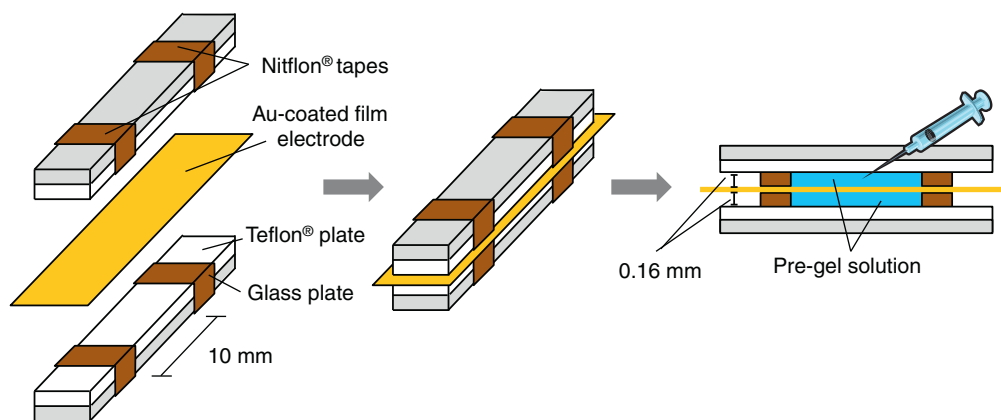


Fig. 1. Schematic illustration of preparation of poly(acrylic acid) gel films on a Au-coated film electrode.

2. Experimental

2.1. Materials

Acrylic acid (reagent grade, Wako Pure Chemical Industries) was distilled prior to use. All other reagents were of at least reagent grade quality and were used without further purification. Aqueous solutions were prepared with distilled-deionized water.

2.2. Synthesis of poly(acrylic acid) gel

Poly(acrylic acid) gel was synthesized following the previously reported procedure [10]. A pre-gel solution was prepared with acrylic acid (2.1 M) and *N,N'*-methylenebisacrylamide (21 mM) with ammonium peroxodisulfate (10.5 mM) and *N,N,N',N'*-tetramethylethylenediamine (16 μ M) as an initiator and an accelerator, respectively. For volume measurements cylindrical gels were prepared with a Teflon® tube of 3 mm inner diameter, to which the pre-gel solution was injected and heated at 70 °C under nitrogen for 3 h. The resulting gel was cut into cylindrical pieces (3 mm diameter \times 5 mm length), and immersed in water for at 4 weeks to remove unreacted reagents.

2.3. Preparation of poly(acrylic acid) gel modified film electrodes

Both sides of a polyimide film (5 mm \times 25 mm \times 12.5 μ m, Du Pont-TORAY) were roughened with sandpaper (#2000) to enhance Au adhesion, followed by etching with an ion sputter (SC-701, Sanyu Electron) at 7 mA for 5 min. A Au layer (ca. 100 nm) was sputtered on both sides of the polyimide film with an ion sputter at 7 mA for 5 min. The surfaces of Au layers on the film were modified with 2-aminoethanethiol by immersing the electrode in a 1×10^{-2} M aqueous solution. The Au-coated film was then immersed in a 2×10^{-2} M acrylic acid aqueous solution containing 1×10^{-2} M 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide to introduce vinyl group anchored on the film, which gives rise to immobilization of the gel on the Au electrode [9]. The film was then sandwiched by two Teflon plates (4 mm \times 20 mm \times 1 mm), which were supported with glass plates (4 mm \times 20 mm \times 1.4 mm). The both Teflon plates were pre-masked with two-ply Nitoflon® tapes (0.16 mm thick, Nitto Denko) to area of 4 mm \times 10 mm, so that there leaves a cavity (4 mm \times 10 mm \times 0.32 mm) in between the film and an unmasked part of the plate (Fig. 1). The above-mentioned pre-gel solution of 6.4 μ l was injected into the each spaces and gelation was carried out at 70 °C for 3 h under nitrogen. The resulting gel-modified film was dismounted from the Teflon plates and allowed to be dried in air. The film was cut into size of 1.5 mm \times 12.5 mm (gel coated area: 1.5 mm \times 3.0 mm). The cutting of the film also electrically isolate

the Au films on the both sides. The actuator was then immersed in pure water for at least 12 h to remove unreacted reagents and in a mixture of 5×10^{-3} M QH₂ and 5×10^{-3} M BQ containing 1×10^{-4} M NaClO₄ for over night to incorporate quinone.

2.4. General procedure

Volumes of the cylindrical gels (*V*) were determined by measuring their diameter and length after they reached at equilibrium in each solutions at various pH (at least 42 days). Volume ratio of the gel as synthesized (*V*/*V*₀) was calculated with respect to the volume of the gel as synthesized (*V*₀). Electrochemical experiments were carried out with a potentiostat (Hokuto HZ-5000, Hokuto Denko). For pH measurement upon quinone redox with two electrodes system, a Au-coated (both sides) polyimide film electrode (8 mm \times 9 mm \times 2 sides) similar to that described above was used as a working electrode. A same-sized Au coated polyimide film electrode was used as counter electrodes. The working electrode was separated from the counter electrode using a glass filter (G4). The pH of solution in the working electrode compartment (5 ml) was measured with a pH meter (TX-999, Tokou-Kagaku) with a glass electrode of 5.3 mm diameter (PCE105CW, Tokou-Kagaku). The actuator was operated as a two electrodes system, where Au layers on each side of the film were used as working and counter electrodes. The motion of the actuator was evaluated as a displacement of the top of the actuator along an arc assuming that it always describes an arc. All measurements were performed at room temperature (25 \pm 1 °C) and nitrogen gas was used to degas the solutions before use and flowed over the solutions during experiments.

3. Results and discussion

3.1. Dependence of gel volume on pH

Volumes of cylindrical poly(acrylic acid) gels were evaluated in solutions at various pH, which was adjusted with HCl or NaOH solution (Fig. 2). The gel showed abrupt increase in the volume over the pH range from 5.8 to 6.1, followed by gradual decrease. At lower pH carboxylates of the gel are protonated, giving rise to decrease in electrostatic repulsion and hydrophilicity, which, in turn, resulting in contraction of the gel. On the other hand, at higher pH since they are deprotonated, the converse phenomenon takes place, resulting in expansion. The decrease in the volume of the gel at higher pH can be attributed to an increase in a concentration of a cation from the base. The pH giving rise to the increase in the volume appeared to be higher and sharper than that of the gel with lower monomer (0.7 M) and cross-linker (7 mM) concentrations [11]. This

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