Enhancement of energy density in organic redox capacitor by improvement of electric
conduction network

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Abstract

Organic compounds have attracted attention as inexpensive and environmentally friendly active materials for energy storage devices. Among them, quinonic compounds can achieve high power capabilities because of their fast redox reactions. To utilize the organic compounds, which have a poor electrical conductivity and which are soluble in the electrolyte, the construction of electric conductive network to hold the organic compounds is necessary. In this study, we enhanced the capacity in aqueous redox-capacitors with quinonic and hydroquinonic organic compounds supported in

nanoporous carbon by adding a small amount of another highly conductive carbon. The improvement of the electric conduction path enabled a high loading ratio of organic active material to nanoporous carbon (>70 wt%) in the electrode with a high utilization rate. It also achieved the high energy density of 30 Wh kg⁻¹ in the organic redox capacitor.

1. Introduction

Organic compounds have attracted attention as inexpensive and environmentally friendly active materials for electrochemical energy storage devices, such as electrochemical capacitors [1], Li-ion batteries [2], and other electrochemical storage devices [3]. These compounds can be elaborated from plants, and could solve uneven worldwide distribution of minor metal resources. Minor metals are essential elements in conventional active materials, such as RuO₂ and LiCoO₂. Therefore, the development of all-organic batteries [4-7], in which organic materials are employed as active materials in both electrodes, would contribute to the development of a more sustainable energy storage system.

Among organic compounds, quinonic compounds have fast redox reactions with two electrons in one molecule. This is a promising feature for active materials in high power

and high energy density energy storage devices. Quinonic compounds were used as the anode active materials in Li-ion batteries [8-15], active species in redox flow batteries [16-19], and active materials in electrochemical capacitors [20-29].

There are some intrinsic problems in applying quinonic compounds to energy storage devices with high utilization rates: the dissolution of the compound into the electrolyte and poor electrical conductivity. One solution to these problems is to support the organic compounds with porous carbon. The porous carbon can hold the compounds and prevent them from dissolving into the electrolytes. Moreover, it offers an electric conductive network to the organic compounds. In our previous study, we demonstrated aqueous redox capacitors using quinonic compounds, tetrachlorohydroquinone (TCHQ) and anthraquinone (AQ), in both electrodes [28]. Fundamentally different from conventional redox capacitors based on redox-active metal oxides, such as RuO_2 and MnO_2 [30], the organic redox capacitor has electrodes which consist of only light elements (hydrogen, carbon, oxygen, and chlorine). In this capacitor, nanoporous carbon, employed as the supporting material for the organic compounds, enabled a high quinonic compound utilization rate and maintained fast redox reactions. The developed redox capacitor has a high energy density of 20 Wh kg^{-1} , achieved by increasing the organic material content in the organic materials/carbon composite to 30 wt%.

Further enhancement of the capacitor energy density would require an increase in the organic compound content in the electrodes. However, the increment in the organic compound content can cause the insulation of the electrode and the reduction of the quinonic compound utilization rate. In our previous study, the carbon material to organic compound ratio was over 2:1 in order to effectively utilize organic compound reaction. However, further increase in the organic compound content resulted in a decrease in the utilization rate. Recently, Chen et al reported that, by using hierarchical porous carbon nanotube electrodes, the content of AQ in the AQ/carbon composite could be increased to 58 wt% in the redox capacitor [29]. However, even in their case, increasing the AQ content above 58 wt% resulted in the degradation of the specific capacitance.

In this study, we improved the electron conduction network in the electrode by adding a small amount of highly conductive carbons to the organic materials/activated carbon composites. By doing so, we achieved both a high utilization rate ($>70\%$) and high organic material loading ratio (70 wt% in the organic materials/activated carbon composites). A four point probe resistivity measurement was used to clarify the strong relationship between the organic compound utilization rate and the electrical conductivity in the electrode. The resultant redox capacitors exhibited high energy

densities of 30 Wh kg⁻¹.

2. Experimental

2.1. Preparation of organic electrodes

Nanoporous activated carbon (Maxsorb[®]MSC-30, Kansai Coke and Chemicals Co., Ltd.) was employed as the support for the organic compounds. From N₂ adsorption/desorption behaviors obtained using an automatic adsorption apparatus (BELSORP-18, BEL Japan, Inc.), it was determined that this carbon had a Brunauer Emmett Teller (BET) surface area of 3070 m² g⁻¹, a pore volume of 1.70 cm³ g⁻¹, and its major pore size in the nanometer scale (averaged pore size: ϕ 2.2 nm).

TCHQ and AQ (Tokyo Chemical Industry Co., Ltd.) were employed as redox active materials for positive and negative electrodes, respectively. The ideal capacities of TCHQ and AQ are 216 and 257 mAh g⁻¹ (organic compound details are shown in Supplementary Table S1). The organic compound and the nanoporous activated carbon were dispersed in acetone using sonication, and the organic compounds were adsorbed to the nanoporous activated carbon in the dispersion. This solution was then evaporated at 70 °C to obtain an organic active material/nanoporous activated carbon composite.

The electrode materials were prepared by mixing the composites, the conductive carbon (DENKA BLACK[®]FX-35, DENKI KAGAKU KOGYO Co., Ltd.) and polytetrafluoroethylene (PTFE), as a binder. The weight ratio of the composites to the

conductive carbon to the PTFE was 8:1:1. The conductive carbon consists of carbon nanoparticles—23 nm on average in size—that could be linked to form a chain. The specific surface area was $133 \text{ m}^2 \text{ g}^{-1}$. Scanning electron microscope (SEM) images of the AQ electrodes with a conductive carbon additive are shown in Supplementary Fig. S1.

2.2. Conductivity measurement using the four point probe method

A four point probe resistivity measurement was used to measure electrical conductivities of the electrode materials. Measurement samples were prepared in the form of a rectangular parallelepiped (height of 500–1000 μm , width of 5–10 mm, and the length between two inner probes used for the resistivity measurement was 4 mm) using electrode materials. AC impedance methods were conducted using a set of electroanalytical systems consisting of an electrochemical interface (SI1287, Solartron Analytical) and impedance/gain-phase analyzer (SI1260, Solartron Analytical).

2.3. Electrochemical measurement

2.3.1. Half-cell measurement

The electrochemical performances of AQ/nanoporous activated carbon electrodes with

various AQ loading ratios were measured using half-cell systems. The electrodes, prepared in the form of pressed pellets (ϕ 7 mm), were attached to Au mesh current collectors. The half-cell system consisted of the AQ composite electrode as a working electrode, a carbon electrode (the nanoporous carbon to the conductive carbon to PTFE weight ratio was 8:1:1) more than ten times heavier than the working electrode as a counter electrode, 70 ml aqueous H_2SO_4 solution (0.5 M) as an electrolyte, and a Ag/AgCl electrode as a reference electrode. N_2 gas was bubbled through aqueous H_2SO_4 solutions for 2 h to remove the dissolved oxygen before usage. After the system was assembled, the electrodes immersed in the electrolyte were placed in a vacuum to be degassed, and the electrolyte infiltrated the electrode.

We then initiated the measurements using a potentiostat/galvanostat (VMP3, Bio-Logic) in ambient conditions. The current was fixed at a 5 C charge/discharge rate based on the amount and ideal capacity of AQ. The cut-off potential for the working electrodes was -0.3 to 0.5 V vs. Ag/AgCl. N_2 gas was bubbled through the electrolyte during the measurement. A certain number of charge/discharge cycles was initially required to maximize the capacity of the composite electrode as shown in the Supplementary Information. We speculate that during the beginning cycles, the further infiltration of the electrolyte into the nanometer-sized pores of nanoporous carbon progresses and

activates the organic active materials. We employed the maximum capacity after a number of charge/discharge cycles to estimate the utilization rate.

2.3.2. Capacitor performance evaluation

The electrochemical performance of the organic redox capacitor was measured using a full-cell system. The system consisted of a AQ-composite electrode as a negative electrode, a TCHQ-composite electrode as a positive electrode, 70 ml aqueous H₂SO₄ solution (0.5 M) as an electrolyte, and a Ag/AgCl electrode as a reference electrode. On the basis of the ideal capacity of AQ (257 mAh g⁻¹) and TCHQ (216 mAh g⁻¹), we fixed the weight ratio of positive/negative electrodes to be 1.2 to balance the capacities of both electrodes.

Before using organic composite electrodes in the redox capacitor, we conducted a certain number of charge/discharge cycles for each electrode in the half-cell system to maximize their capacities. The details of such pretreatment for the electrodes are described in the Supplementary Information. After a number of charge/discharge cycles, each electrode was taken from the half-cell system and assembled in the redox capacitor. The measurement setup was the same as indicated in the previous section. The cut-off voltage was 0–1 V between the positive and negative electrodes. The C-rate was

calculated on the basis of the amount and the ideal capacity of AQ. The cycle performance evaluation was measured at 5 C. The rate performance evaluation was measured at C-rates of 0.5, 1, 2, 5, 10, and 20. The energy density calculations were based on the total weight of both electrodes.

3. Results and discussion

3.1. Improvement of the AQ utilization rate

Fig. 1 shows the specific discharge capacities for the AQ electrodes, with and without conductive carbon, as a function of the AQ content in the AQ/nanoporous carbon composites, using half-cell measurements. The AQ content shown in Fig. 1 was the organic active material ratio in the composites.

To estimate the specific discharge capacity derived only from the AQ reaction, we eliminated the electric double-layer (EDL) capacity originating from the nanoporous carbon from the total discharge capacity. The EDL capacity could be determined through the extrapolation of the discharge profile in the potential range in which AQ reactions do not occur. A detailed procedure to eliminate the EDL capacity is shown in our previous work [28]. The weight ratio of AQ to the nanoporous carbon is indicated on the second x-axis on top. The utilization rate of the AQ reaction, which was calculated based on the ideal capacity of AQ, 257 mAh g^{-1} , is indicated on the second y-axis on the right side.

In the case of the AQ electrodes without conductive carbon which were investigated in our previous study, the utilization rate significantly decreases when the AQ content goes above 40 wt%. On the other hand, in the case of the AQ electrodes with conductive

carbon, an over 60% utilization rate continued until the AQ content reached 80 wt%, corresponding to AQ to nanoporous carbon weight ratio of 4:1. This result indicated that the addition of the conductive carbons would maintain a high utilization rate at a high AQ content.

3.2. Relationship between utilization rate and electrical conductivity

To elucidate the contribution of the conductive carbon additive, the electric conductivities of the AQ electrodes were measured by taking four point probe resistivity measurement. In Fig. 2, we show the electrical conductivities of the AQ electrodes, with and without the conductive carbon, as a function of the AQ content in the AQ/nanoporous activated carbon composites. The electrical conductivity of the paste consisting of the conductive carbon and PTFE, 1.7 S cm^{-1} , was an order of magnitude larger than that of the paste consisting of the activated carbon and PTFE.

The electrical conductivity of the AQ electrodes without conductive carbon significantly decreased above an AQ content of 40 wt%. On the other hand, the AQ electrodes with conductive carbon show higher electrical conductivities than those without conductive carbon. This result indicated that the conductive carbon additive improved the electron conduction path in the electrode. Moreover, the electrical conductivity remained greater

than $10^{-1} \text{ S cm}^{-1}$ until the AQ content reached 70 wt%. This AQ content was over twice that of nanoporous carbon content by weight.

The electrical conductivities of the 80 wt% AQ electrode with conductive carbon ($4.4 \times 10^{-2} \text{ S cm}^{-1}$) and the 30 wt% AQ electrode without conductive carbon ($3.8 \times 10^{-2} \text{ S cm}^{-1}$) were comparable. These AQ electrodes were the highest loaded electrodes in each case with and without conductive carbon maintaining a high AQ reaction utilization rate. The relationship between the electrical conductivity and AQ content in the electrode was quite similar to the relationship between the utilization rate of the AQ reaction and the AQ content shown in Fig. 1. This similarity strongly suggested that the high electrical conductivity in the electrodes was necessary to improve the utilization rate of the organic active materials.

Here, we discuss how the conductive carbon additive contributes to the improvement of the utilization rate of the organic active materials. As mentioned above, the quinonic organic materials showed poor electrical conductivities, and an electron conduction path to the organic materials was required for its utilization in electrochemical devices.

In Fig. 3, we show an image of the improved electric conductive network with the conductive carbon additive. The pore volume of nanoporous activated carbon was $1.70 \text{ cm}^3 \text{ g}^{-1}$. If AQ could be allocated into the pore, the limit of the AQ adsorption was

approximately 70 wt%. However, the use of a loading method based on the adsorption phenomenon in liquid solutions resulted in the insufficient penetration of the AQ molecule into the pores of activated carbon that were too small. This limited AQ adsorption to just 35 wt% in AQ/nanoporous activated carbon composites (Supplementary Fig. S2). Therefore, it is supposed that the excess AQ in the composite was located on the surface of the activated carbon particles rather than the pores of the activated carbon. This excess AQ with low electrical conductivity on the surface prevents the electrical connection between the carbon particles, and reduces the utilization rate of organic active materials. On the other hand, in the electrode with the conductive additive, the carbon nanoparticles with high electrical conductivity bridge the activated carbon, forming electron conduction paths in the AQ/nanoporous activated carbon composite electrode. As a result, a high AQ reaction utilization rate could be achieved even at a high AQ loading ratio.

3.3. Evaluation of the electrochemical performance of redox capacitors

We have demonstrated a redox capacitor which consisted of a TCHQ-composite electrode (composite of 70 wt% TCHQ content with conductive carbon) as a positive electrode and an AQ-composites electrode (composite of 70 wt% AQ content with

conductive carbon) as a negative electrode. Fig. 4 shows the charge/discharge profile of the redox capacitor measured at 5 C per ideal AQ capacity (0.72 A g^{-1} -Negative electrode). The plateau potentials of the positive and negative electrodes corresponded to the redox potentials of TCHQ and AQ, respectively. The operating voltage was approximately 0.65 V. The coulombic efficiency was above 99%, indicating that redox reactions of AQ and TCHQ were reversible. In this case, the EDL contribution was negligible, and the utilization rates of TCHQ and AQ redox reactions, which were estimated based on the total capacity, were 75% (162 mAh g^{-1} -TCHQ) for the positive electrode, and 77% (198 mAh g^{-1} -AQ) for the negative electrode. These high utilization rates mean that the reversible two-electron reactions of quinonic compounds were conducted in both electrodes.

Fig. 5 shows the rechargeable energy densities of the redox capacitor as a function of current density. For a comparison, our previous redox capacitor results are also shown in Fig. 5 [28]. The redox capacitor in that study had electrodes without conductive carbon that had 30 wt% organic material content, and the pure EDL capacitor had electrodes consisting of nanoporous carbon and PTFE [28].

Even at high current densities, the energy densities of the redox capacitors developed in this study were higher than those of other capacitors. It means that a fast

quinonic/hydroquinonic compound redox reaction could be maintained, even in the electrode highly loaded with organic compounds. The energy density of the redox capacitor was 29 Wh kg^{-1} at a current density of $7.2 \times 10^{-2} \text{ A g}^{-1}$ -Negative electrode-. This value was over three times larger than the energy density of the pure EDL capacitor, and exceeded that of the RuO_2 -based aqueous electrochemical capacitor (26 Wh kg^{-1} based on the electrode material) [31]. Moreover, when we change the cut-off voltage to 0–1.4 V, the energy density reached 33 Wh kg^{-1} at $7.2 \times 10^{-2} \text{ A g}^{-1}$ -Negative electrode- (charge/discharge profile of the redox capacitor in the 0–1.4 V range is shown in Supplementary Fig. S5). By using the redox reaction of highly loaded organic compounds effectively, the energy density of the aqueous redox capacitor using organic compounds was enhanced. In this fundamental study, we employed electrolyte in excess, and the energy density calculations were based on the total weight of both electrodes. For future practical use of the redox capacitor, comparing the energy density of the packaged capacitor using a minimized amount of electrolyte with that of the commercial capacitors would be important.

Although this study focused on the investigation into the contribution of conductive carbon additives in improving the energy density of redox capacitors, the improvement of cycle lifetime is still a challenge. The retention rate of the redox capacitor was only

39% after 1000 cycles at a C-rate of 5 (Supplementary Information). This retention rate is worse than that of the established electrochemical capacitors, such as EDL capacitors, RuO₂-systems, and MnO₂-systems, all of which withstand more than 1000 charge/discharge cycles. One of the promising solutions to the problem, which might be caused by the dissolution of organic compounds into electrolyte, is the optimization of the structure of organic compounds. For example, the attachment of hydrophobic functional groups, such as a chloro group, can suppress the solubility of an organic compound in the aqueous solution. In our previous study [28], when we employed 1,5-dichloroanthraquinone (DCAQ) instead of AQ, the degradation of the rechargeable energy density became ignorable even after 1000 charge/discharge cycles (at a C-rate of 5 for 20 days). It is supposed that a suitable organic molecule design will enable the further enhancement of the cycle lifetime.

As the share of renewable energy sources (e.g. solar and wind power), whose outputs inherently vary greatly, increases in total energy production, large scale energy storage devices are necessary for smart grid systems to stabilize between electric demand and supply. Our redox capacitor is useful in large scale energy storage devices for smart grids because of three features. The first is its superior electrochemical performance. Our redox capacitor has both high energy and high power capabilities and can thus

respond to the rapid variability of the renewable sources to smooth them [32]. The second is low manufacturing cost. Different from the application to transport vehicles, cost per energy is more important for grid scale energy storage devices than gravimetric/volumetric energy density. Our metal-free redox capacitor can be manufactured with a low material cost. The third is high safety. Our redox capacitor is driven with non-flammable aqueous electrolyte.

4. Conclusions

To further enhance the energy density of metal-free redox capacitors based on organic active materials, we investigated the relationship between the electric conductive network and the utilization rate of organic materials. The improvement of the electric conductive network through the addition of highly conductive carbon achieved both a high organic material loading ratio (>70 wt%) and high organic material reaction utilization rate (>70%). The developed redox capacitors exhibited a high energy density of 30 Wh kg⁻¹, maintaining the fast redox reaction of quinonic/hydroquinonic compounds. This capacitor will contribute to the development of large scale energy storage systems for smart grids because of its high electrochemical performance, low manufacturing cost, and high safety.

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Figure captions

Fig. 1 The maximum capacities for anthraquinone (AQ) electrodes with conductive carbon and without conductive carbon [28] as a function of the AQ content in the AQ/nanoporous activated carbon composites. The current was fixed at a 5 C charge/discharge rate based on the amount and the ideal capacity of AQ.

Fig. 2 Electrical conductivities of AQ electrodes with conductive carbon and without conductive carbon as a function of the AQ content in the AQ/activated carbon composites.

Fig. 3 Image of the electric conductive network improved by the addition of conductive carbon.

Fig. 4 Charge/discharge profile of the developed redox capacitor during the second charge/discharge cycle. The current was fixed at a 5 C charge/discharge rate based on the amount and the ideal capacity of AQ.

Fig. 5 The rechargeable energy densities of the redox capacitor in which organic materials made up 70 wt% of the contents of each electrode with a conductive carbon (C.C.) additive (operation voltage: 0–1 V and 0–1.4 V) at the second charge/discharge cycle as a function of current density. Our previous results for the redox capacitor in which organic materials made up 30 wt% of contents of each electrode without a

conductive carbon additive (0–1V), and the pure EDL capacitor (0–1V) are also shown [28].

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