

A rechargeable Zn- poly(aniline-co-*m*-aminophenol) battery

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Abstract

Based on the cyclic voltammograms and impedance spectra of poly(aniline-co-*m*-aminophenol) in the electrolyte solution containing ZnCl_2 and NH_4Cl , a solution consisting of 2.0 M ZnCl_2 and 3.0 M NH_4Cl with pH 4.70 was employed as an electrolyte solution of a Zn-poly(aniline-co-*m*-aminophenol) battery. The battery was charged and discharged between 0.75 and 1.45 V at different current densities. The average charge voltage is about 1.15 V and the average discharge voltage is about 1.05 V, slightly depending on the charge–discharge current density. The capacity and energy densities of poly(aniline-co-*m*-aminophenol) are $137.5 \text{ A h kg}^{-1}$ and $152.5 \text{ W h kg}^{-1}$ for discharge process, respectively, which are much better than those of polyaniline at the same solution. After the test of the charge–discharge at different current densities, the battery was successively charged and discharged for 120 cycles. At one-hundred and twentieth cycle, the coulombic efficiency is 100% and the energy density of poly(aniline-co-*m*-aminophenol) only decreases by 9.2%, compared with the first cycle of the successive charge–discharge processes.

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1. Introduction

Conducting polymers possessing reversible redox characteristics provide a new kind of electrode materials for rechargeable batteries. Among conducting polymers, polyaniline is a promising candidate for the electrode material. This is because polyaniline has good redox reversibility, high stability in air and aqueous solutions and is easily prepared by using chemical or electrochemical method with low production costs. Especially, the reduced polyaniline can be protonated or oxidized spontaneously in the aqueous acidic solutions [1,2], which is very favorable for the capacity recovery of the battery. So, aqueous polyaniline batteries have been extensively studied and as a result the battery performance has been improved [3–15]. Most of the aqueous polyaniline batteries were constructed of a polyaniline cathode and a zinc anode with a solution containing of ZnCl_2 and NH_4Cl . In this case, the electrochemical activity of polyaniline is limited by the pH value of the electrolyte solution, since the zinc electrode undergoes easily corrosion in the electrolyte solution with lower pH values. This is a great disad-

vantage to the battery. To protect zinc from corrosion, the pH value of the electrolyte solution in the battery must be raised, which however, leads to a decay in the electrochemical activity of polyaniline. Generally, polyaniline has a little electrochemical activity at $\text{pH} > 4$ based on the result from cyclic voltammetry [16,17], but which is related to the buffer capacity of the electrolyte solution. For example, polyaniline in the solution consisting of ZnCl_2 and NH_4Cl still has a rather good electrochemical activity at $\text{pH} > 4$ because of a large buffer capacity [18].

An effective way for improving the pH dependence of polyaniline was carried out by using the sulfonation of polyaniline with fuming sulfuric acid [19,20] or concentrated sulfuric acid [21] and the copolymerization of aniline with *m*-aminobenzenesulfonic acid [22,23] to prepare self-doped polyanilines. They exhibit a good pH dependence of the conductivity in the aqueous solution and a good electrochemical activity over a wide pH range even in basic solutions. The self-doped polyaniline was used to fabricate the secondary Zn-polyaniline (self-doped) battery, which has a rather high specific energy [24].

As mentioned above, the copolymerization of aniline with a monomer having a functional group offers a good way for improving the polyaniline properties. Also, poly(aniline-co-*o*-

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aminophenol) has the good electrochemical properties at pH < 10.6 and rather high conductivity [25]. This is due to the fact that there is a functional group of the hydroxyl group on the copolymer chain, which can be oxidized and reduced reversibly. The reversible redox of the hydroxyl group on the copolymer chain not only produces some quantity of electricity but also provides proton exchange between the copolymer and the solution simultaneously. The latter plays an important role in adjusting pH value around the copolymer electrode, which leads to the improvement of the electrochemical activity of the copolymer.

Recently, we synthesized a new copolymer, poly(aniline-co-*m*-aminophenol). Its electrochemical properties such as pH dependence and usable potential range are even better than those of poly(aniline-co-*o*-aminophenol). Therefore, we tried to use poly(aniline-co-*m*-aminophenol) to fabricate a zinc-poly(aniline-co-*m*-aminophenol) battery. The electrolyte solution consisted of ZnCl₂ and NH₄Cl. In this paper, we report cyclic voltammetry and impedance measurements of poly(aniline-co-*m*-aminophenol) and the performance of the battery. The purpose for the study of cyclic voltammetry and impedance measurements is to determine the composition and pH value of the electrolyte solution used in the battery.

2. Experimental

Aniline was distilled before use. The other chemicals used were of reagent grade. Doubly distilled water was used to prepare solutions. The pH values of the solutions were determined by using a PXD-12 pH meter. The electrolytic cell consisted of two platinum foils and a saturated calomel reference electrode (SCE). The area of the working electrode was 4 × 4 mm, except for the electrode used in the battery.

A copolymer film, poly(aniline-co-*m*-aminophenol), was synthesized using repeated potential cycling between -0.10 and 0.95 V. The solution consisted of 0.34 M aniline, 0.012 M *m*-aminophenol and 2 M H₂SO₄. The preparation of poly(aniline-co-*m*-aminophenol) has been described elsewhere in more details [26]. A thick copolymer film of 22.6 mg deposited on the two sides of a platinum foil with the area of 1 cm² was used to fabricate a rechargeable Zn-poly(aniline-co-*m*-aminophenol) battery. A solution containing ZnCl₂ and NH₄Cl was used as the electrolyte solution of the battery. The charge and discharge of the battery were performed on an automatic battery charge-discharge unit (HJ-201B, Japan) and the data were recorded by using a YEW 3036 pen recorder. Cyclic voltammetry of the copolymer was carried out by using a CHI 407 electroanalysis instrument. The impedance measurements were conducted on an Autolab PGSTAT 30 instrument. Frequency sweeps extended from 10⁴ to 0.01 Hz using a signal of 10 mV, peak-to-peak. The copolymer film was pre-treated for six cycles using cyclic voltammetry in the potential range -0.20 to 0.80 V in the following corresponding solutions prior to recording cyclic voltammograms and impedance plots. The purpose is to establish pre-equilibrium of pH between the copolymer film and the tested solution. All experiments carried out at 20 ± 2 °C.

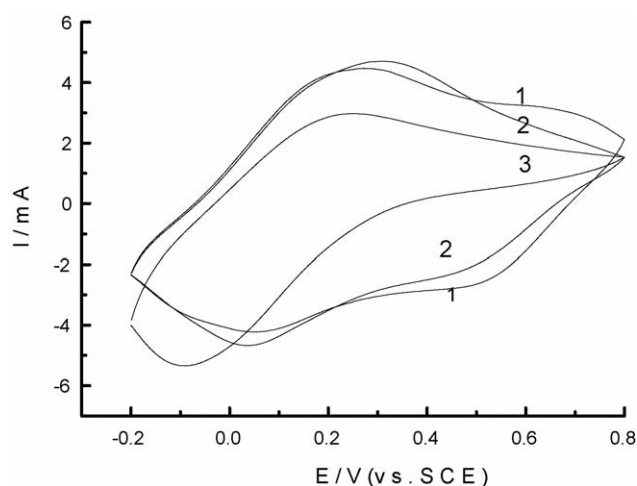


Fig. 1. The effect of pH on the cyclic voltammograms of poly(aniline-co-*m*-aminophenol) in a solution consisting of 2.0 M ZnCl₂ and 3.0 M NH₄Cl, (1) pH 4.20, (2) pH 4.70, (3) pH 5.20, at a scan rate of 60 mV s⁻¹.

3. Results and discussion

3.1. Cyclic voltammograms of poly(aniline-co-*m*-aminophenol)

To obtain information about the effects of the composition and pH value of the solution used in the battery on the electric activity of the copolymer, cyclic voltammetry of the copolymer was carried out in the different compositions of ZnCl₂ and NH₄Cl and different pH values. The scan rate was set at 60 mVs⁻¹. Curves 1–3 in Fig. 1 show the cyclic voltammograms of the copolymer in a solution consisting of 2.0 M ZnCl₂ and 3.0 M NH₄Cl with pH 4.20, 4.70 and 5.20, respectively. Two anodic peaks at 0.28 and 0.62 V and two cathodic peaks at 0.05 and 0.51 V occur on curve 1. However, a pair of redox peaks at higher potentials on curves 2 and 3 disappears, a cathodic peak at 0.05 V on curve 1 shifts to -0.08 V on curve 3 and their areas become smaller as the pH increases, indicating that the electrochemical activity of the copolymer decreases with increasing pH. But, the copolymer still holds the good electrochemical activity at pH 4.70 and in the potential range -0.20 to 0.80 V. The cyclic voltammograms of polyaniline and poly(aniline-co-*o*-aminophenol) demonstrate that a usable potential range is -0.20 to 0.50 V for polyaniline and -0.20 to 0.60 V for poly(aniline-co-*o*-aminophenol) [18], at the same experimental conditions as shown in Fig. 1. Hence, the usable upper potential of poly(aniline-co-*m*-aminophenol) is extended compared with polyaniline and poly(aniline-co-*o*-aminophenol). This is favorable for raising the charging voltage of the battery.

Fig. 2 shows the cyclic voltammograms of the copolymer in the solutions consisting of 3 M NH₄Cl and different concentrations of ZnCl₂ with pH 4.70. As the concentration of ZnCl₂ increases from 2.0 to 3.0 M, the area of the cyclic voltammograms in Fig. 2 decreases a little. This means that the concentration of ZnCl₂ has a smaller effect on the electrochemical activity of the copolymer compared with pH value.

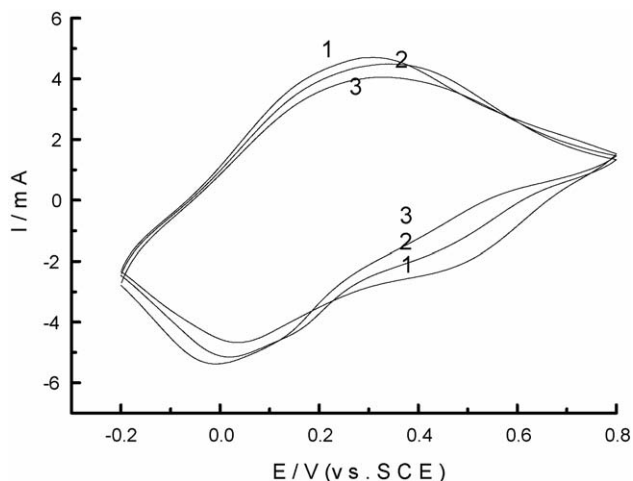


Fig. 2. Cyclic voltammograms of poly(aniline-co-*m*-aminophenol) in a solution containing 3.0 M NH_4Cl with different concentrations of $ZnCl_2$ with pH 4.70, (1) 2.0 M, (2) 2.5 M, (3) 3.0 M $ZnCl_2$, at a scan rate of 60 mVs^{-1} .

3.2. Impedance measurements of poly(aniline-co-*m*-aminophenol)

Here, we mainly discuss the effects of the potential, pH and composition of the solution on the impedance of the copolymer. The plots a–d in Fig. 3 are the impedance spectra of the copolymer at 0.20 (a), 0.30 (b), 0.40 (c) and 0.50 V (d), respectively. The solution consisted of 2.5 M $ZnCl_2$ and 3.0 M NH_4Cl with pH 4.20. All impedance plots in Fig. 3 are constructed of a semicircle with different charge transfer resistances (R_{ct}) and

a straight line. This indicates that the electrode reaction of the copolymer is under kinetic control at high frequencies and is under mass transfer control at low frequencies. The R_{ct} values of the copolymer at 0.20 and 0.30 V are very small. In this voltage region, the effect of the potential on the R_{ct} is very small. Even though the R_{ct} of the copolymer increases as the potential increases from 0.30 to 0.40 V, but the R_{ct} of the copolymer at 0.40 V is still not too large. However, the R_{ct} increases significantly as the potential increases from 0.40 to 0.50 V. This result is very similar to that of the cyclic voltammograms in Fig. 1, in which the area of the cyclic voltammogram starts to become smaller at the potential over 0.50 V. This is due to a great decrease of the conductivity, caused by overoxidation of the copolymer. The copolymer as well as polyaniline suffers overoxidation more easily at high pH values than that at low pH values. In order to compare overoxidation of the copolymer with that of polyaniline in a solution consisting of 2.0 M $ZnCl_2$ and 3.0 M NH_4Cl with pH 4.70, the cyclic voltammogram of polyaniline is shown in Fig. 4. It is obvious that the area of the cyclic voltammogram in Fig. 4 becomes very small in the potential of 0.50–0.80 V. This is quite different from that of the copolymer in the same experimental conditions (curve 2 in Fig. 1). This indicates that the ability of the copolymer against overoxidation is better than that of polyaniline. This is because the $-OH$ group on the copolymer chain can be oxidized and reduced reversibly, which plays an important role in adjusting pH value around the copolymer electrode. Such reversible redox diminishes overoxidation of the copolymer chain. Polyaniline used here was prepared in a solution consisting of 0.34 M aniline and 2 M H_2SO_4 , using repeated potential cycling between -0.10 and 0.95 V.

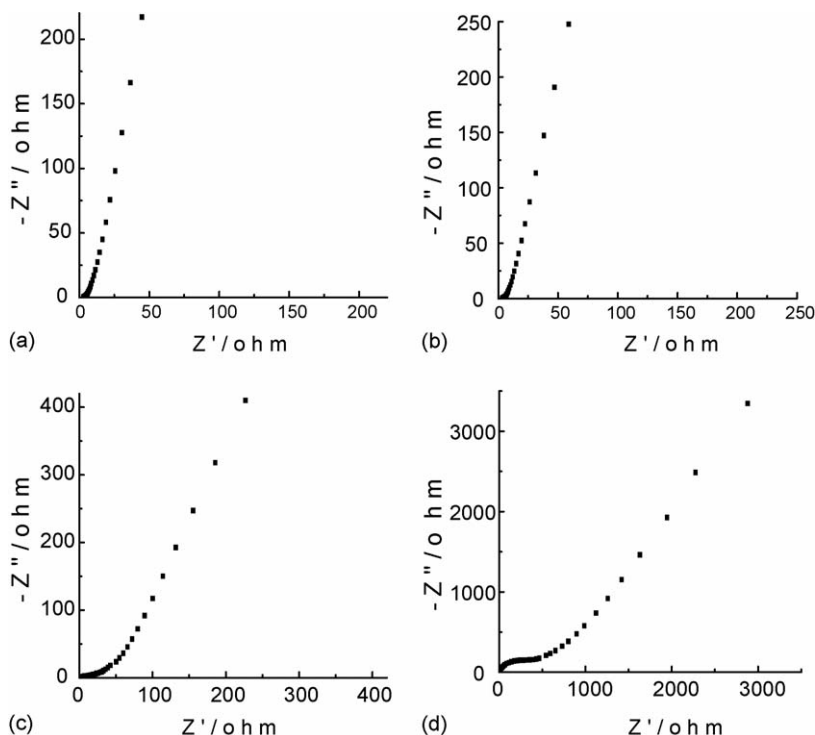


Fig. 3. The effect of the potential on the impedance of poly(aniline-co-*m*-aminophenol) in a solution consisting of 2.5 M $ZnCl_2$ and 3.0 M NH_4Cl with pH 4.20, (a) 0.20 V, (b) 0.30 V, (c) 0.40 V, (d) 0.50 V.

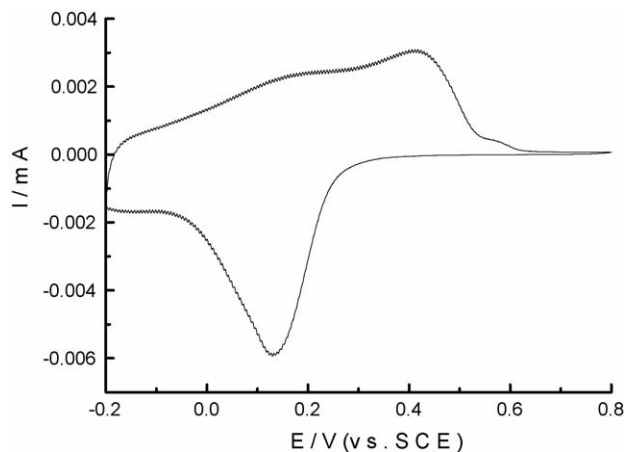


Fig. 4. Cyclic voltammogram of polyaniline in a solution containing 2 M ZnCl_2 and 3.0 M NH_4Cl with pH 4.70, at a scan rate of 60 mVs^{-1} .

Fig. 5 shows the effect of pH on the impedance of the copolymer in a solution consisting of 2.5 M ZnCl_2 and 3.0 M NH_4Cl at 0.40 V. It is obvious that the R_{ct} increases with increasing pH and the Warburg impedance almost disappears at pH 5.20, indicating that the electrochemical activity of the copolymer decreases with increasing pH. This result is in good agreement with that of cyclic voltammograms in Fig. 1, in which the area of the cyclic voltammogram of the copolymer is the lowest at pH 5.20.

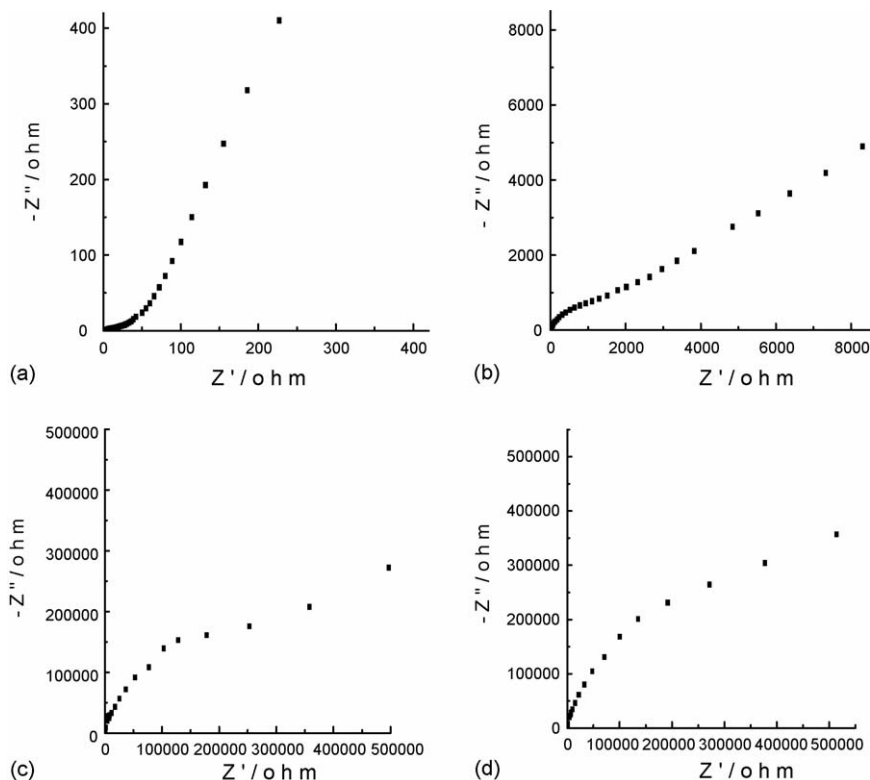


Fig. 5. The effect of pH on the impedance of poly(aniline-co-*m*-aminophenol) in a solution consisting of 2.5 M ZnCl_2 and 3.0 M NH_4Cl , (a) 4.20, (b) 4.70, (c) 5.00, (d) 5.20, at 0.40 V.

Fig. 6 shows the effect of the concentration of ZnCl_2 on the R_{ct} of the copolymer. The solutions consisted of 3 M NH_4Cl and 2.0 (a), 2.5 (b), 2.75 (c) and 3.0 (d) M ZnCl_2 with pH 4.70. The potential was set at 0.30 V. The R_{ct} increases with increasing the concentration of ZnCl_2 . This result is also indicative of the decrease in the electrochemical activity of the copolymer.

3.3. Effect of charge–discharge current density on the battery characteristics

Based on the result of the impedance measurements, a solution consisting of 2.0 M ZnCl_2 and 3.0 M NH_4Cl was used as the electrolyte solution of the battery, and upper charging voltage of the Zn-poly(aniline-co-*m*-aminophenol) battery was set at 1.45 V that is about 0.45 V (versus SCE) of the copolymer potential. Considering the corrosion of zinc and results from the effect of pH on the cyclic voltammograms and the impedance of the copolymer, the pH of the electrolyte solution was set at 4.70. In this case, the open-circuit voltage of the Zn-poly(aniline-co-*m*-aminophenol) battery is 1.34 V.

The structure and the electrode reaction of poly(aniline-co-*m*-aminophenol) are very complicated as well as polyaniline. Based on the fact that the electrode reaction of the copolymer is related to pH value and the IR spectrum of poly(aniline-co-*m*-aminophenol) shows the presence of the *m*-aminophenol unit [26] in the copolymer, so the redox reactions of the copolymer are assumed as follows:

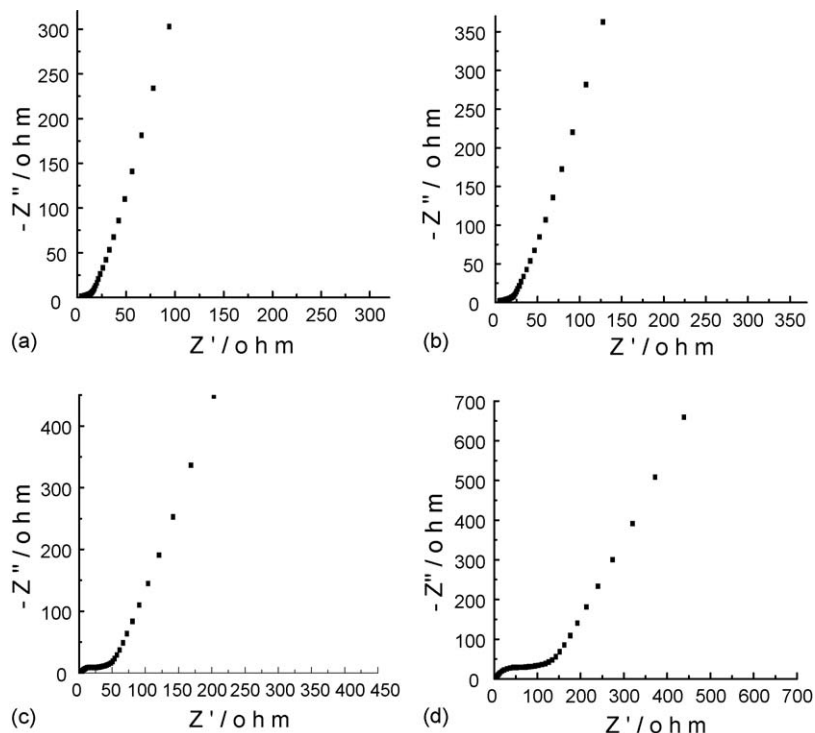
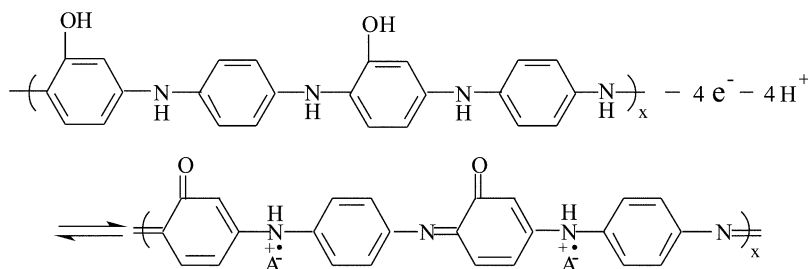


Fig. 6. The effect of the composition of the solution on the impedance of poly(aniline-co-*m*-aminophenol) in a solution consisting of 0.3 M NH₄Cl and different concentrations of ZnCl₂ with pH 4.70, (a) 2.0 M, (b) 2.5 M, (c) 2.75 M, (d) 3.0 M, at 0.30 V.



Where A⁻ is an anions that were doped into the copolymer.

The battery was charged and discharged between 1.45 and 0.75 V, and in order of 10, 5 and 1 mA. Curves 1 and 2 in Fig. 7 show the charge–discharge curves at 10 mA (5 mA cm⁻²) and 5 mA (2.5 mA cm⁻²), respectively. Fig. 8 shows the charge and discharge curves of the battery at 1 mA (0.5 mA cm⁻²). It is obvious that the decreasing rate of the battery voltage during the

discharging process is slower in the voltage of 1.25–1.10 V on curve 2 in Fig. 7 and 1.30–1.00 V in Fig. 8 than other voltage regions, depending on the charge–discharge current density, which is in good agreement with the result of the impedance measurements. This is because, in this voltage region, the charge–transfer resistance of the copolymer is the lowest in the whole discharging voltage region based on the result from the

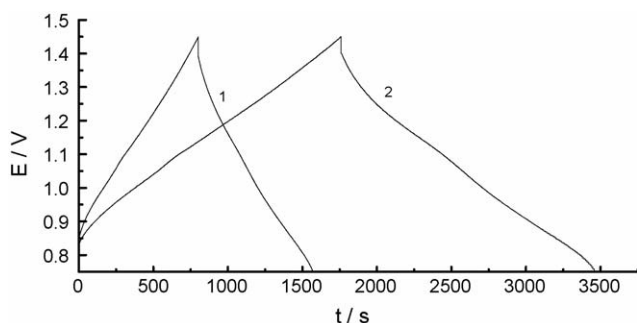


Fig. 7. Charge and discharge curves of the copolymer battery at a constant current, (1) 10 mA; (2) 5 mA.

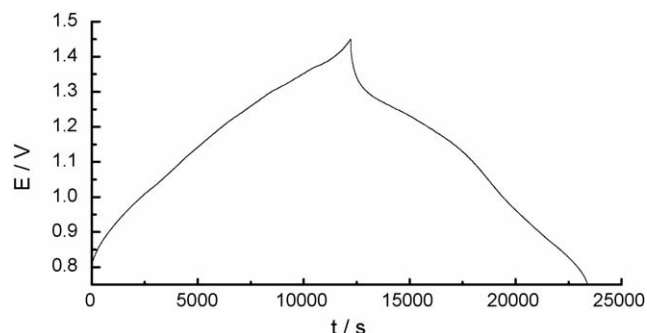


Fig. 8. Charge and discharge curves of the copolymer battery at a constant current of 1 mA.

Table 1
Charge-discharge data of the Zn-poly(aniline-co-*m*-aminophenol) battery

Current (mA)	Average charge voltage (V)	Average discharge voltage (V)	Capacity density (A h kg ⁻¹)		Energy density (Wh kg ⁻¹)		Coulombic efficiency (%)
			Charge	Discharge	Charge	Discharge	
10	1.14	1.02	98.6	94.5	112.4	96.4	95.9
5	1.15	1.04	108.0	105.2	124.1	109.2	97.4
1	1.17	1.11	150.1	137.5	175.6	152.5	91.6

impedance measurements of the copolymer. The voltage region 1.00–1.30 V of the battery corresponds to about 0.00 and 0.30 V (versus SCE) of the copolymer potential.

Based on the charge–discharge results from Figs. 7 and 8 and the weight of the copolymer, the coulombic efficiency, average charge and discharge voltages, capacity and energy densities of the copolymer were listed in Table 1.

The average charge and discharge voltages of the battery in Table 1 were calculated based on the areas under the charge and discharge curves, current of the charge–discharge, charge time and discharge time. Table 1 shows that among the different charge and discharge current densities, the coulombic efficiency is the lowest at a constant charge–discharge current density of 0.5 mA cm⁻². This is caused by the self-discharge of the battery, because of long time for each charge-discharge cycle. The data of Table 1 indicate that the capacity and energy densities of the copolymer increase with decreasing current density. This is expected because of the electrode polarization. At a constant current density of 0.5 mA cm⁻², the capacity and energy densities of the copolymer in the discharge process are 137.5 A h kg⁻¹ and 152.5 Wh kg⁻¹, respectively. However, At a constant current density of 5 mA cm⁻², the capacity and energy densities of the copolymer in the discharge process are 94.5 A h kg⁻¹ and 96.4 Wh kg⁻¹ that are a little higher than the result of the Zn-polyaniline battery at a constant current discharge of 0.25 mA cm⁻² for polyaniline, in which the weight of polyaniline was 22.1 mg and also polyaniline was polymerized on a platinum foil of 1 cm² [27]. This indicates that the charge–discharge performance of the Zn-poly(aniline-co-*m*-aminophenol) battery at higher current density was pronouncedly improved compared with the Zn-polyaniline battery.

Since the capacity and energy densities of the Zn-polyaniline batteries are affected by the composition of the electrolyte solution in the battery, pH value, charge–discharge current density and charge–discharge voltage range, for simplicity we make a comparison between the Zn-poly(aniline-co-*m*-aminophenol) battery and Zn-polyaniline battery with the same electrolyte solution, but the pH value for the latter was 4.40. In this case, the capacity and energy densities of polyaniline were 78.5 A h kg⁻¹ and 86.1 Wh kg⁻¹, respectively, under a discharge current density of 0.25 mA cm⁻² [27]. For a Zinc-poly(aniline-co-*o*-aminophenol) battery with an electrolyte solution consisting of 2.5 M ZnCl₂ and 3.0 M NH₄Cl with pH 4.70 were 103.0 A h kg⁻¹ and 120.4 Wh kg⁻¹ at a constant discharge current density of 0.5 mA cm⁻² [18]. It is clear that the capacity and energy densities of poly(aniline-co-*m*-aminophenol) are 55.3% and 56.7% higher than those of polyaniline. Even though the

pH value of the electrolyte solution in the Zn-poly(aniline-co-*m*-aminophenol) battery is a little higher than that in the Zn-polyaniline battery, this small increment is favorable to protecting zinc electrode from corrosion. Table 1 shows that the capacity and energy densities of poly(aniline-co-*m*-aminophenol) are also higher than those of poly(aniline-co-*o*-aminophenol) at a constant current density of 0.5 mA cm⁻². This is because the pH dependence of the electrochemical activity of poly(aniline-co-*m*-aminophenol) is better than that of poly(aniline-co-*o*-aminophenol) and especially much better than that of the parent polyaniline. Also, the capacity density of poly(aniline-co-*m*-aminophenol) is higher than that of polyaniline in the Zn-polyaniline battery reported by Mirmohseni and Solhjo [12], in which the charge current density was 0.2 mA cm⁻², and it is close to that of the self-doped polyaniline (SDPA) that was used to fabricate Zn-polyaniline(self-doped) battery [24]. The later battery with a thin SDPA film has a maximum capacity of 146.4 Ah kg⁻¹ with a coulombic efficiency of 97–100% over at least 200 cycles between 0.8 and 1.6 V.

3.4. Effect of charge–discharge cycles on the performance of the battery

For a rechargeable battery, the charge–discharge cycles are very important. Therefore, after the above charge-discharge experiments at different current densities, the battery was employed for the successive charge–discharge test. The current density was controlled at 2.5 mA cm⁻² for both the charge and discharge processes. Curves 1 and 2 in Fig. 9 are the first and one-hundred and twentieth charge–discharge cycles. The exper-

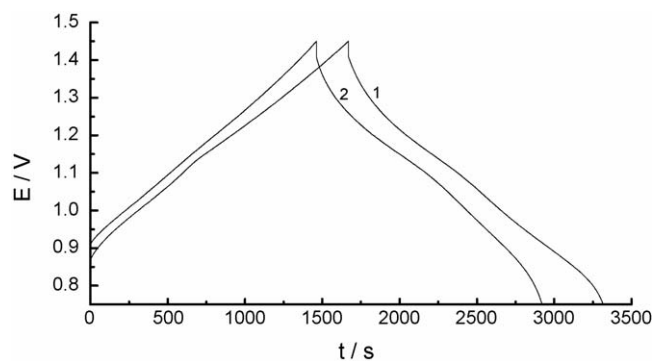


Fig. 9. Effect of the charge and discharge cycles on the performance of the copolymer battery, (1) the first cycle; (2) 120th cycle, at a constant current of 5 mA.

Table 2
Successive constant current charge–discharge data of the battery

Cycle	Average charge voltage (V)	Average discharge voltage (V)	Capacity density (A h kg ⁻¹)		Energy density (W h kg ⁻¹)		Coulombic efficiency (%)
			Charge	Discharge	Charge	Discharge	
First	1.16	1.05	102.5	101.3	119.5	106.8	98.8
120th	1.17	1.08	89.8	89.8	105.3	97.0	100

imental data of the successive charge–discharge cycles are listed in Table 2.

Comparison of the data in Table 2 and Table 1 shows that the capacity and energy densities for the first cycle in Table 2 are a little lower than those in Table 1 at a constant current density of 2.5 mA cm⁻². This is caused by a decay of the electrochemical activity of the copolymer after several cycles. Its reason has been discussed earlier. However, the coulombic efficiency increases from 97.4% to 98.8%. This may be caused by the decrease in the amount of aniline and *m*-aminophenol adsorbed on the thick copolymer surface as the charge–discharge proceeds, which leads to a diminution in a loss of quantity of electricity during the charging process. This is because the copolymer film used here was very thick, aniline and *m*-aminophenol adsorbed on the copolymer surface were more difficult to be removed completely from the film. To example this suggestion, after synthesis, a thick copolymer film was first washed by using 0.05 M H₂SO₄ solution and then immersed in 2 M H₂SO₄ solution over night. Finally, a bare platinum as a working electrode was immersed in the later solution for cyclic voltammetry at a scan rate of 60 mVs⁻¹. The result shows that an anodic peak appears at 1.01 V on the cyclic voltammogram (omitted here). This peak is caused by the oxidation polymerization of aniline and *m*-aminophenol, because of no appearance of an anodic peak at 1.01 V on the cyclic voltammogram of the bare platinum electrode in 2 M H₂SO₄ solution. At one hundred and twentieth charge–discharge cycle, the coulombic efficiency is 100%, and its energy density only decreases by 9.2% compared with the first cycle in Table 2. This means that the battery has a good recharge performance.

4. Conclusion

The electrolyte solution consisting of ZnCl₂ and NH₄Cl has a higher buffer capacity that is advantageous to the reversible redox of poly(aniline-co-*m*-aminophenol) at higher pH values. However, the concentration of ZnCl₂ could be controlled at somewhat lower concentrations. This is because once the ZnCl₂ solid appears on the copolymer electrode surface, which will leads to a pronounce increase in the charge transfer resistance of the copolymer. The pH value of the electrolyte solution used in the battery not only affects the electric activity of the copolymer, but also affects the corrosion rate of the zinc electrode. Therefore, the pH value of the electrolyte solution used in the battery would be considered from both the electric activity of the copolymer and the zinc corrosion. The capacity and energy

densities of poly(aniline-co-*m*-aminophenol) is higher than that of the parent polyaniline. This is due to a –OH group on the copolymer chain, which can be oxidized and reduced reversibly. Such reversible redox on the copolymer chain plays an important role in adjusting pH value around the copolymer electrode and contributes some of quantity of electricity to the battery. Even though the structure of poly(aniline-co-*m*-aminophenol) is presented above, its structure is not as simple as we assumed here, a more complex situation must be considered. Therefore, the further work on the copolymer structure is necessary.

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