

Structural optimization of carbonized polymer dots for modulation of electrochemical performance of aqueous electrolytes

HUANG Xinyao, WANG Jiahui, YANG Guoduo, JI Xin, SUN Haizhu*

*National & Local United Engineering Laboratory for Power Batteries, Faculty of Chemistry,
Northeast Normal University, Changchun 130024, China*

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Aqueous zinc ion batteries (AZIBs) are considered to be one of the most promising energy storage devices due to the advantages of high cost-effectiveness, safety, and environmental friendliness. However, they suffer from problems such as Zn dendrites growth and by-product generation. Carbonized polymer dots (CPDs) with polar groups as additive have been introduced to modulate the solvated structure of Zn^{2+} and reduce the water activity, promoting the uniform deposition of Zn and inhibiting the occurrence of side reactions. However, CPDs with different functional group contents from different precursor molar ratios variably affect the electrochemical performance of aqueous electrolytes. Therefore, in this work, we designed and synthesized CPDs with different molar ratios of the precursors (citric acid and urea) as electrolyte additives for AZIBs and explore the optimal molar ratios of the precursors. The Zn//Zn symmetrical cells using electrolytes with the optimal ratios CPDs achieve an extended cycle life over 615 h at $2 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$. This work offers great potential for future practical applications of CPDs.

Zn metal anode; Electrolyte additive; Carbonized polymer dots; Zn dendrites

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1. Introduction

Nowdays, aqueous Zinc (Zn) ion batteries (AZIBs) have attracted a lot of attention in the field of large-scale sustainable energy storage due to the high safety, low cost and eco-friendliness^[1]. However, there are still some problems with the practical application of AZIBs, such as the growth of Zn dendrites caused by uneven deposition of Zn^{2+} , hydrogen evolution reactions (HER) and chemical corrosions, which greatly reduce the Coulombic efficiency (CE) and cycle life of AZIBs^[2].

In order to solve the above problems, researchers have proposed various strategies^[3], such as surface protection of Zn anode^[4-6], electrolyte additives^[7-8] and development of polymer electrolytes^[9-10]. Among them, electrolyte additives are high cost-effectiveness and potential for scale-up application, they show unique advantages in interface modulation and performance optimization^[11], thus adding electrolyte additives is considered to be the most convenient, economical and efficient method. For example, Zhi et al.^[12]

proposed an effective inhibitor, phosphonoethanoic acid (PPGA), as an additive to ZnSO_4 electrolyte. They exposed Zn anode to PPGA solutions and conducted experiments for different time lengths, observing a progressive evolution of the by-products in terms of phase structure, crystallinity, and generation. The results suggest that PPGA tends to adhere to the Zn metal surface and interacts with the surrounding H_2O , thereby modulating Zn deposition and inhibiting side reactions. However, at present, most of the electrolyte additives have the disadvantages of single regulation function, high cost and poor environmental friendliness. Therefore, it is crucial to develop eco-friendly electrolyte additives that are multifunctional and cost-effective.

Eco-friendly, cost-efficient carbonized polymer dots (CPDs) are zero-dimensional carbon-based nanomaterials^[13]. CPDs have an organic-inorganic composite core-shell structure with an abundance of zincophilic surface functional groups, indicating that there are a large number of tunable active sites on the surface of CPDs, which can tightly bind to Zn^{2+} and thus control the growth and morphology of inorganic compounds^[14]. In addition, CPDs can be synthesized in large quantities from precursors and have

*Corresponding author. sunhz335@nenu.edu.cn (SUN Haizhu)
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excellent aqueous dispersibility and stability, making them a good choice as additives for aqueous electrolytes, with great potential for development in the field of electrochemical energy storage. For example, Liu et al.^[15] designed an eco-friendly natural biomass carbon quantum dots (BCDs) as the electrolyte additive for AZIBs. The formation of hydrogen bonds between BCDs and H₂O can reduce the activity of H₂O. Besides, the buffering and redistribution effects of BCDs can inhibit the growth of Zn dendrites and promote uniform Zn deposition. While significant progress has been achieved in applying CPDs as electrolyte additives, systematic investigations to optimize their molecular structures remain scarce, which is important for further exploring the role of CPDs on enhancing electrochemical performance of AZIBs.

Herein, citric acid and urea were selected as precursors to synthesize CPDs with zincophilic functional groups by one-step solvothermal method. The obtained different CPDs were used as additives in electrolytes of AZIBs to investigate the modulation effect of CPDs synthesized with different molar ratios of precursors on the electrochemical performance of AZIBs. Due to the strong interaction of surface polar functional groups, such as —NH₂, —OH, —C=O with Zn²⁺ and H₂O, the solvation structure of Zn²⁺ is effectively regulated, while the activity of H₂O is simultaneously reduced^[16-17]. Furthermore, CPDs achieve durable regulation of Zn²⁺ and promote uniform deposition of Zn (See Figure 1). The Zn//Zn symmetric cells using electrolyte with optimal CPDs achieve an extended cycle life over 615 h at 2 mA·cm⁻² and 1 mAh·cm⁻².

2. Experimental section

2.1 Synthesis of CPDs

CPDs were synthesized from citric acid and urea in molar ratios of 1:10, 2:10, 3:10, 3:12 and 3:14, respectively. A certain amount of citric acid (mass 0.19, 0.38, 0.57, 0.57, 0.57 and 0.57 g, respectively) was weighed and dissolved in 10 mL of *N,N*-dimethylformamide (DMF) solution, then a certain amount of urea (mass 0.60, 0.60, 0.60, 0.72 and 0.84 g, respectively) was weighed and added to the above

solution and stirred at room temperature. After stirring for 30 min, the resulting solution was transferred to a sealed polytetrafluoroethylene (PTFE)-lined autoclave for solvothermal reaction at 180 °C for 12 h. After cooling to room temperature, excess ethyl acetate was added to the solution to induce precipitation. The product was washed several times with ethyl acetate, then dried in a vacuum oven to obtain the black powder.

2.2 Preparation of electrolytes

Ultrapure water (Macklin) was used to prepare aqueous electrolytes. The 2M ZnSO₄ electrolyte was formulated by dissolving 8.63 g ZnSO₄·7H₂O into 15 mL ultrapure water. Then 1 mg of CPDs with different molar ratios of citric acid and urea (1:10, 2:10, 3:10, 3:12 and 3:14) were added to the prepared 5 mL of 2M ZnSO₄ electrolyte, stirred for 12 h, and labeled, namely, 1C10U-CPDs, 2C10U-CPDs, 3C10U-CPDs, 3C12U-CPDs and 3C14U-CPDs.

2.3 Assembly of cells

The CR2032 coin-type Zn//Zn symmetrical cell was composed of two Zn foils, 2M ZnSO₄ electrolyte and 2M ZnSO₄ with different CPDs electrolyte and a glass fiber separator (Whatman GF/D). The CR2032 coin type Zn//Cu asymmetric cell consists of a Zn foil, a Cu foil, 2M ZnSO₄ electrolyte and 2M ZnSO₄ with different CPDs electrolyte, and a glass fiber separator (Whatman GF/D).

2.4 Materials characterization

The surface functional groups of CPDs and the characteristic functional groups in different electrolytes were characterized by Fourier transform infrared (FTIR) spectrum using Agilent Technologies Cray 630 FTIR. The Zn metal anod foils were characterized by scanning electron microscope (SEM, Hitachi).

2.5 Electrochemical measurements

Electrochemical impedance spectroscopy (EIS) was performed using a CHI660E electrochemical workstation with a measurement amplitude of 5 mV and frequencies from 0.01 Hz to 1.0 MHz. HER was tested using linear scanning voltammetry (LSV) with a scan rate of 1 mV·s⁻¹ and a scan range of 0 to -0.5 V and 0 to 3 V. Tafel plots were tested at 1 mV·s⁻¹ between -0.3 and 0.3 V (vs. Zn/Zn²⁺).

The ionic conductivity of electrolytes was tested by assembling a stainless steel sheet-electrolyte-stainless steel sheet blocking cells. The impedance values of the electrolytes were measured according to the EIS. The formula for calculating the ionic conductivity is as follows:

$$\sigma = \frac{L}{RS},$$

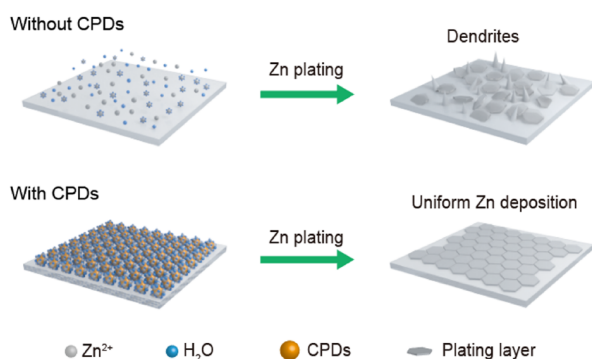


Figure 1 Schematic representation of Zn²⁺ deposition in different electrolytes

where σ represents the ionic conductivity ($\text{S}\cdot\text{cm}^{-1}$); L is the thickness of separator (cm); R stands for the impedance value (Ω); S is the contact area (cm^2).

3. Results and discussion

3.1 Structure characterization of zincophilic CPDs

The CPDs were prepared *via* simple solvothermal process, using citric acid as the source of —OH and —C=O groups, and urea providing —NH_2 groups. FTIR was used to test the CPDs and investigate the surface functional group of CPDs. As shown in Figure 2a, CPDs with different precursor molar ratios have the same type of surface functional groups. A broad absorption peak in the range of 3420 to 3320 cm^{-1} corresponds to the stretching vibrations of O—H groups. A strong absorption peak near 3200 cm^{-1} is attributed to the stretching vibration of N—H groups. The characteristic absorption peak at approximately 1650 cm^{-1} is associated with the stretching vibration of C=O . The characteristic absorption peaks at approximately 1600 to 1560 cm^{-1} and 1400 to 1340 cm^{-1} are associated with C=N and C—N stretching vibrations, respectively. A sharp absorption peak near 1100 cm^{-1} corresponds to the bending vibration of

C—O . These results indicate that the CPDs contain a variety of functional groups, which can serve as zincophilic sites to regulate Zn^{2+} deposition.

In order to evaluate the dispersibility and stability of CPDs in ZnSO_4 electrolyte, the electrolytes with $0.2\text{ g}\cdot\text{L}^{-1}$ of CPDs were prepared separately. As shown in Figure 2b, electrolytes are brownish yellow. From Figure 2c, the electrolytes containing CPDs with different precursor molar ratios all showed fluorescence phenomenon under ultraviolet (UV) illumination, which prove that CPDs are fully soluble and well dispersed in the ZnSO_4 electrolyte.

To illustrate the inhibitory effect of the introduction of CPDs on side reactions during Zn^{2+} deposition, FTIR was performed on ZnSO_4 electrolyte and electrolytes containing CPDs with different precursor molar ratios. In the ZnSO_4 electrolyte, the bending vibration of O—H is observed at 1635 cm^{-1} and the O—H stretching vibration is at 3200 cm^{-1} , and both the bending vibration and the stretching vibration of O—H are blueshifted after adding CPDs with different precursor molar ratios (See Figures 2d and 2e), indicating that the interaction between Zn^{2+} and H_2O is weakened by the introduction of CPDs, the amount of bound H_2O in the solvated structure of Zn^{2+} is reduced, and CPDs replace some of the H_2O to participate in the regulation of

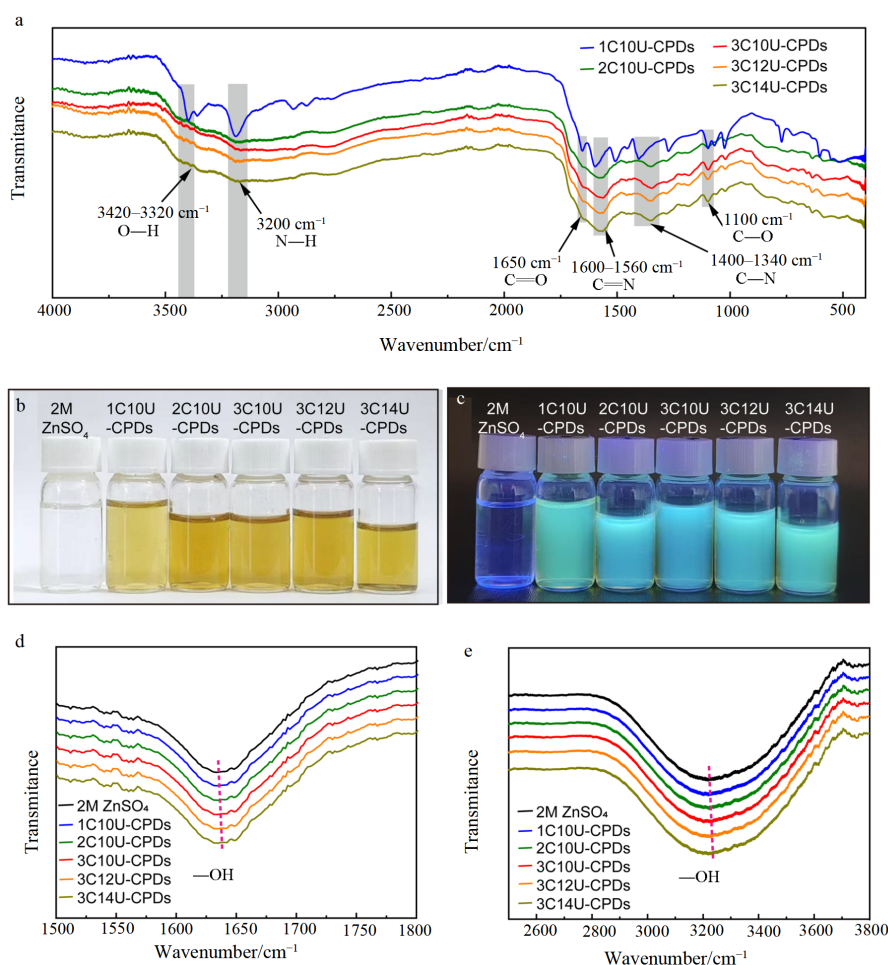


Figure 2 FTIR spectrum of CPDs (a), optical images at initial state (b) and under UV light (c), and FTIR spectrum of electrolyte containing CPDs with different precursor molar ratios (d and e)

Zn^{2+} solvated structure. Besides, CPDs disrupt the hydrogen bonding network of H_2O and reduce the activity of H_2O , thus effectively mitigating the occurrence of side reactions.

3.2 Optimization of CPDs structure

Zn//Zn symmetric cells were assembled for cycling performance tests to explore the optimal molar ratio of CPDs precursors. As shown in Figure 3a, at $0.5 \text{ mA}\cdot\text{cm}^{-2}$ and $0.5 \text{ mAh}\cdot\text{cm}^{-2}$, the electrolyte with 3C10U-CPDs exhibits the longest cycle life (506 h), significantly outperforming the ZnSO_4 electrolyte (28 h) and electrolytes with 1C10U-CPDs (69 h), 2C10U-CPDs (82 h), 3C12U-CPDs (150 h), and 3C14U-CPDs (134 h). Furthermore, as shown in Figure 3c, at $2 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$, the electrolyte with 3C10U-CPDs still has the best cycling performance, with 615 h of stable cycling. It is worth noting that the ZnSO_4 electrolyte and electrolytes with other CPDs can short-circuit after cycling for a certain period of time due to the instability of the Zn anode, which can lead to battery failure. Figures 3b and 3d show the voltage-time curve with specific cycles, the electrolyte with 3C10U-CPDs shows a lower voltage hysteresis, indicating that the introduction of 3C10U-CPDs has the most significant improvement in the cycling stability of Zn//Zn symmetric cells.

In order to further investigate the effect of CPDs with different precursor molar ratios on the electrochemical performance of AZIBs, Zn//Cu cells were assembled for CE tests. As shown in Figure 4a, at $2 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$, the cells in the electrolyte with 3C10U-CPDs remain stable for 137 cycles. Besides, as shown in Figure 4c, at $4 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$, the cells in the electrolyte with 3C10U-CPDs remain stable for 512 cycles. However, the cells in ZnSO_4 electrolyte just remain stable

for 71 cycles. In contrast, the cells in the electrolytes with 1C10U-CPDs, 2C10U-CPDs, 3C12U-CPDs, and 3C14U-CPDs stabilized the cells for 297, 53, 360, and 173 cycles, respectively. The electrolyte with 3C10U-CPDs show a higher average CE of 99.18% at $2 \text{ mA}\cdot\text{cm}^{-2}$ after 130 cycles and 99.22% at $4 \text{ mA}\cdot\text{cm}^{-2}$ after 510 cycles. The ZnSO_4 electrolyte and electrolytes with CPDs containing other precursor molar ratios show low CE, the results can be attributed to the gradual growth of Zn dendrites, causing the poor cyclic stability. This further suggests that the incorporation of 3C10U-CPDs improves Zn anode plating/stripping performance more effectively.

The nucleation overpotential of the Zn//Cu half-cells were also obtained. As shown in Figure 4b, at $2 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$, the Zn^{2+} in the Zn//Cu cells with electrolyte containing 3C10U-CPDs only exhibit a low nucleation overpotential of 22.5 mV, which is lower than that observed in the ZnSO_4 electrolyte (58.6 mV). From Figure 4d, at $4 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$, the nucleation overpotential of Zn^{2+} in the electrolyte with 3C10U-CPDs (50.8 mV) is lower than that in the ZnSO_4 electrolyte (52.6 mV). In addition, under both of the above conditions, the nucleation overpotential of Zn^{2+} in the electrolyte containing 3C10U-CPDs was lower than the nucleation overpotential in the electrolyte containing CPDs with other precursor molar ratios. These indicate that the introduction of 3C10U-CPDs significantly lowers the energy barrier for Zn^{2+} nucleation and promote the uniform deposition of Zn^{2+} .

3.3 Influence of 3C10U-CPDs on Zn deposition behavior

In order to investigate the effect of 3C10U-CPDs on Zn deposition, SEM tests were used to observe the morphology of Zn anode after 2 h and 4 h of deposition in ZnSO_4

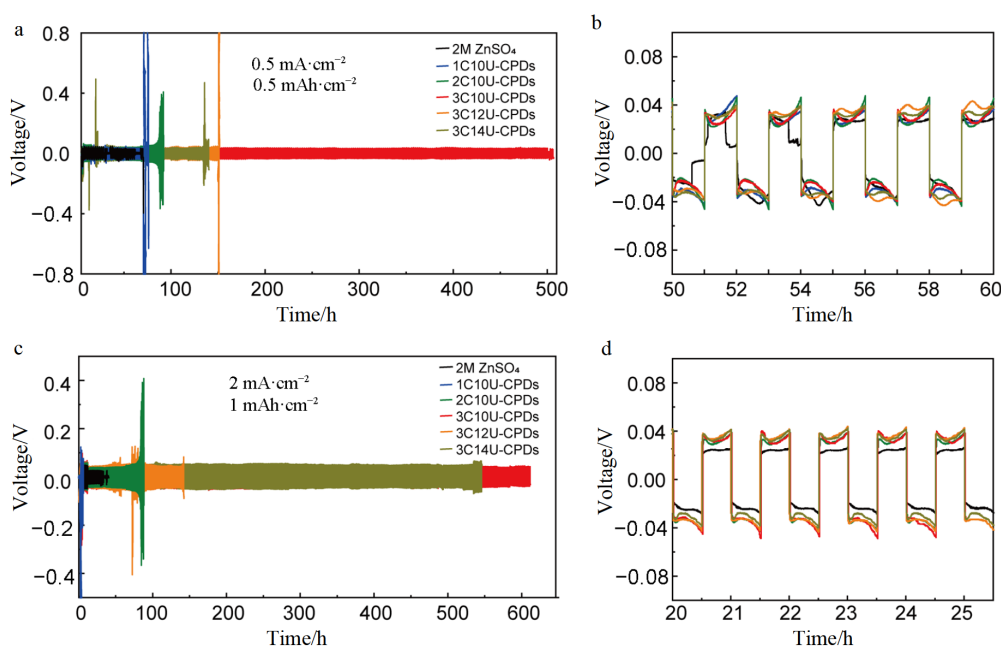


Figure 3 Cycling properties and magnified graphs at specific number of cycles of Zn//Zn symmetric cells in ZnSO_4 electrolyte and electrolytes with different CPDs at $0.5 \text{ mA}\cdot\text{cm}^{-2}$ and $0.5 \text{ mAh}\cdot\text{cm}^{-2}$ (a and b) and at $2 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$ (c and d)

electrolyte and in the electrolyte with 3C10U-CPDs at a current density of $0.5 \text{ mA}\cdot\text{cm}^{-2}$, respectively. As shown in Figure 5a, a large number of flake-like Zn dendrites with random directions appear on the Zn anode surface at a capacity of $1 \text{ mAh}\cdot\text{cm}^{-2}$. When the deposition capacity is increased to $2 \text{ mAh}\cdot\text{cm}^{-2}$, the irregular Zn dendrites continue to grow and become denser (See Figure 5c). This uneven Zn surface enhances the “tip effect”, promoting the accumulation of by-products and leading to short-circuiting of the cells. On the contrary, after plating $1 \text{ mAh}\cdot\text{cm}^{-2}$ in the electrolyte containing 3C10U-CPDs, as shown in Figure 5b, the surface of the Zn foil shows a smooth, uniform and

dense morphology. The deposition Zn is homogeneous without obvious dendrite. Figure 5d also shows smooth surface of the Zn foil after plating $2 \text{ mAh}\cdot\text{cm}^{-2}$, which proves that 3C10U-CPDs can effectively induce the uniform deposition of Zn, thus realizing a dendrite-free Zn anode.

3.4 Influence of 3C10U-CPDs on the electrochemical performance of AZIBs

First, the interfacial stability of different electrolytes was investigated using EIS measurements. As shown in Figure 6a, the charge transfer resistance of cells with electrolytes

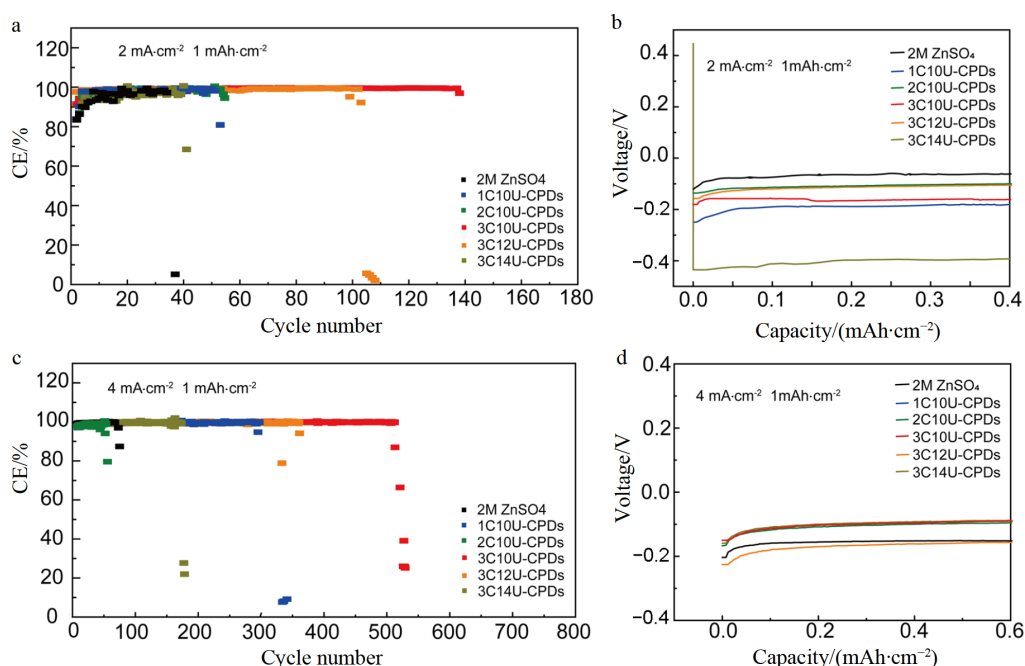


Figure 4 CE and nucleation overpotentials of Zn/Cu cells in ZnSO_4 electrolyte and in electrolytes with different CPDs at $2 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$ (a and b) and at $4 \text{ mA}\cdot\text{cm}^{-2}$ and $1 \text{ mAh}\cdot\text{cm}^{-2}$ (c and d)

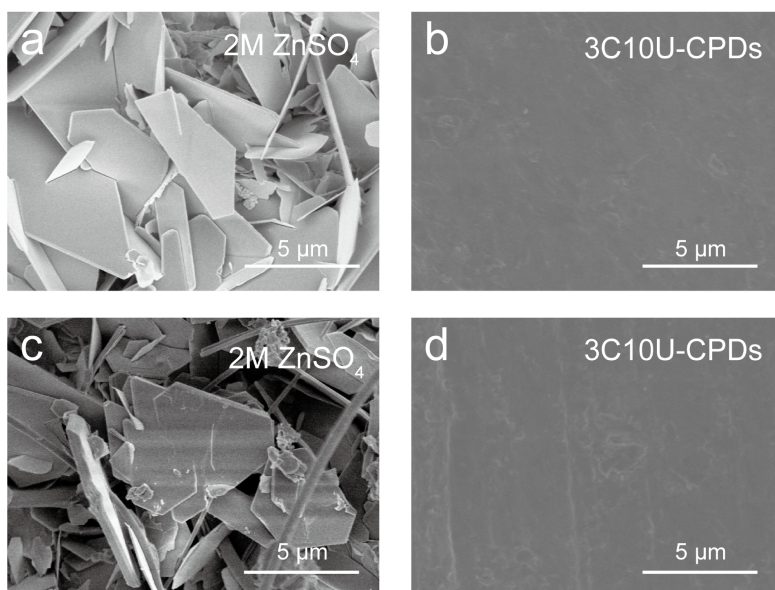


Figure 5 SEM images of Zn deposition at $0.5 \text{ mA}\cdot\text{cm}^{-2}$ in ZnSO_4 electrolyte at $1 \text{ mAh}\cdot\text{cm}^{-2}$ (a), in electrolyte with 3C10U-CPDs at $1 \text{ mAh}\cdot\text{cm}^{-2}$ (b), in ZnSO_4 electrolyte at $2 \text{ mAh}\cdot\text{cm}^{-2}$ (c), and in electrolyte with 3C10U-CPDs at $2 \text{ mAh}\cdot\text{cm}^{-2}$ (d)

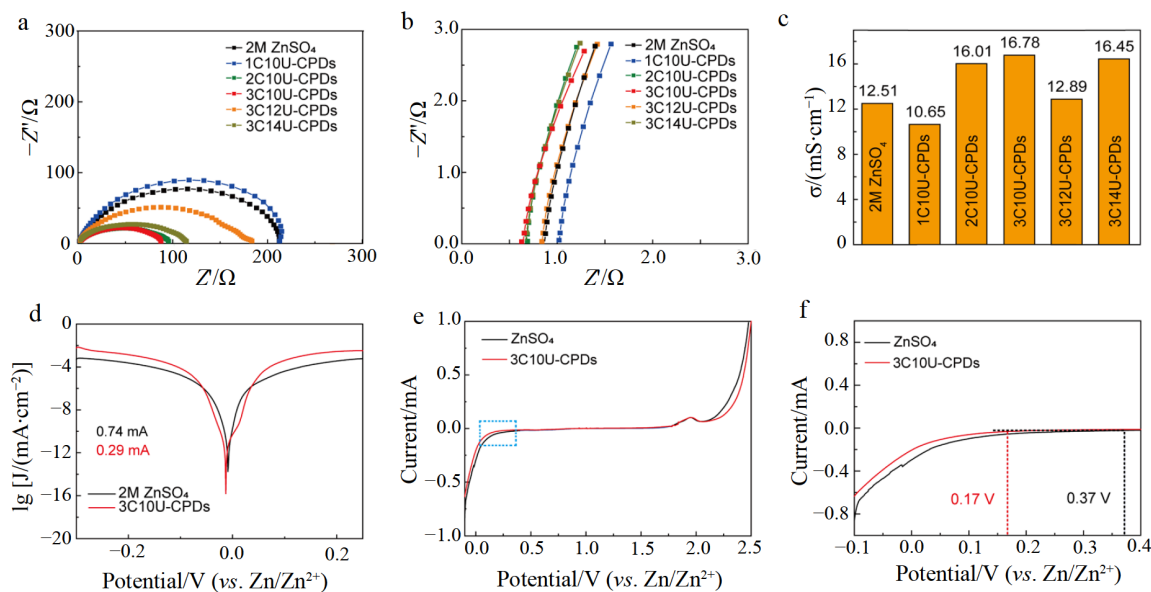


Figure 6 EIS spectra of the Zn//Zn cells at initial state in ZnSO₄ electrolyte and in electrolytes with different CPDs (a), ionic conductivity test (b and c), the Tafel plots (d), LSV curves (e), and magnified graphs of LSV curves (f) in ZnSO₄ electrolyte and in electrolytes with 3C10U-CPDs

containing 3C10U-CPDs is significantly lower than that of cells with ZnSO₄ electrolyte and electrolytes with CPDs in other precursor molar ratios, indicating that cells in the electrolyte with 3C10U-CPDs have a faster internal ion transport ability, which allows the reaction between electrons and Zn²⁺ to occur rapidly at the solid-liquid interface, thus improving the Zn deposition process.

The ionic conductivity tests were performed using EIS measurements (See Figures 6b and 6c). As shown in Figure 6c, the ionic conductivity of the electrolyte with 3C10U-CPDs was calculated to be up to 16.78 mS·cm⁻¹, while that of the ZnSO₄ electrolyte was only 12.51 mS·cm⁻¹. The increase in ionic conductivity indicates that the introduction of 3C10U-CPDs greatly improves the ion transport kinetics of the electrolyte.

Then the corrosion behavior of Zn anode in different electrolytes was revealed by Tafel tests. As shown in Figure 6d, after the addition of 3C10U-CPDs to the ZnSO₄ electrolyte, the corrosion current is reduced from 0.74 to 0.29 mA, which proved that the addition of 3C10U-CPDs could effectively inhibit the corrosion reactions. Furthermore, Figures 6e and 6f show that, at the same current density, the reduction potential decreases from 0.37 to 0.17 V after adding 3C10U-CPDs, indicating that the introduction of 3C10U-CPDs effectively inhibits the HER.

4 Conclusion

In this work, CPDs with different precursor molar ratios were successfully prepared using a one-step solvothermal method. Experimental results show the optimal molar ratio of citric acid and urea used for the synthesis of CPDs is 3:10 (named as 3C10U-CPDs). The electrolyte with 3C10U-CPDs exhibits the best electrochemical performance. At

2 mA·cm⁻² and 1 mAh·cm⁻², the Zn//Zn symmetrical cells using the electrolyte with 3C10U-CPDs achieve an extended cycle life over 615 h and the CE of Zn//Cu asymmetrical cells in the electrolyte with 3C10U-CPDs can be stabilized for 512 cycles. Besides, the electrolyte with 3C10U-CPDs can effectively inhibit Zn corrosion and the growth of Zn dendrites, thus improving the long-term cycling stability of AZIBs. This work explores the optimal molar ratio of citric acid and urea required for the synthesis of CPDs, which can be used to further modulate the electrochemical performance of AZIBs and offers great potential for future practical applications.

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碳化聚合物点的结构优化用于调控水系电解液的电化学性能

黄馨瑶, 王佳慧, 杨国夺, 冀鑫, 孙海珠*

动力电池国家地方联合工程实验室, 东北师范大学化学学院, 吉林 长春 130024

摘要 近年来, 水系锌离子电池因具有高理论比容量、高安全性、低成本和环境友好等优点, 在大规模储能领域展现出了广阔的应用前景。然而, 水系锌离子电池在实际应用中仍面临一些挑战, 如锌枝晶生长、析氢反应和锌腐蚀等副反应的发生。电解液添加剂被认为是解决上述问题的有效策略之一。碳化聚合物点(CPDs)表面具有丰富的亲锌官能团, 可有效调控 Zn^{2+} 的溶剂化结构, 降低水分子的活性, 从而抑制锌枝晶生长和副反应的发生。然而, 不同官能团含量的CPDs对水系电解液电化学性能的构效关系尚待阐明。本工作以柠檬酸和尿素为前驱体, 设计合成了CPDs作为水系电解液添加剂。同时, 通过调整柠檬酸和尿素的投料比, 进一步优化CPDs的结构, 从而探究出最佳的反应物物质的量比。最佳物质的量比合成的CPDs的电解液展现出了优异的电化学性能, 其 Zn/Zn 对称电池在电流密度为 $2\text{ mA}\cdot\text{cm}^{-2}$, 容量为 $1\text{ mAh}\cdot\text{cm}^{-2}$ 的条件下可稳定循环615 h。该工作拓宽了CPDs的实际应用范围。

关键词 锌金属负极; 电解液添加剂; 碳化聚合物点; 锌枝晶