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OPTIMIZING $ZNSO_4$-LI_2SO_4 ELECTROLYTES FOR STABLE AQUEOUS ZINC-ION BATTERIES

Abstract

Aqueous zinc-ion batteries (AZIBs) are considered as one of the most attractive candidates for safe, low-cost, and environmentally benign energy storage systems. However, the widespread implementation of these systems is still limited due to major problems with the zinc metal anode, including uncontrolled dendrite formation, hydrogen evolution, and the low reversibility of zinc plating and stripping. These issues lead to rapid capacity fading and shortened cycle life, highlighting the urgent need for electrolyte optimization as a simple and effective strategy to overcome anode instability. The purpose of this work was systematically investigated the electrochemical behavior of zinc anodes in a series of electrolytes with different $ZnSO_4$-Li_2SO_4 compositions, namely 2M $ZnSO_4$, 1.5M $ZnSO_4$ + 0.5M Li_2SO_4, 0.5M $ZnSO_4$ + 1.5M Li_2SO_4, 1M $ZnSO_4$ + 1M Li_2SO_4, and 2M Li_2SO_4. The electrochemical performance was evaluated using cyclic voltammetry (CV) and galvanostatic charge-discharge tests, and then post-cycle morphological characterization was performed using scanning electron microscopy (SEM). The results show that adding Li_2SO_4 to the $ZnSO_4$ electrolyte significantly changes the structure of the Zn^{2+} solution, thereby increasing the reversibility of zinc plating/stripping and suppressing dendrite formation. In particular, mixed electrolytes exhibit sharper voltage profiles and reduced polarization compared to single-salt systems. Among the tested formulations, the equimolar mixture of 1M $ZnSO_4$ + 1M Li_2SO_4 achieved the most balanced performance, delivering stable cycling and a uniform zinc dendrite morphology. This study highlights electrolyte engineering as a practical and scalable approach to stabilize zinc anodes, providing new insights into the design of high-performance aqueous zinc batteries for future large-scale energy storage applications.

Keywords: aqueous zinc-ion batteries, Zn anode, electrolyte, cyclic voltammetry, dendrite.

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Introduction

Electrochemical energy storage plays a pivotal role in enabling the large-scale integration of renewable energy and supporting the transition toward carbon neutrality. Among the current energy

storage technologies, lithium-ion batteries (LIBs) dominate the markets for portable electronics and electric vehicles, owing to their high energy density and dependable performance.

However, their broader deployment is hindered by critical challenges, including the high cost of lithium resources, uneven geographic distribution, and persistent safety concerns associated with flammable organic electrolytes. Moreover, the large-scale production of LIBs imposes significant environmental burdens, driving the pursuit of safer, more cost-effective, and sustainable alternatives [1–2].

In this context, aqueous zinc-ion batteries (AZIBs) have attracted considerable attention as next-generation energy storage devices. Zinc is abundant, inexpensive, and environmentally benign, with a global annual production exceeding 13 million tons. Its favorable electrochemical properties include a relatively low redox potential (- 0.76 V vs. SHE) and a high theoretical capacity of 820 mAh g^{-1}, making it promising for high-performance energy storage. In addition, aqueous electrolytes are intrinsically safe and nonflammable, in sharp contrast to the volatile organic solvents used in LIBs, which positions AZIBs as strong contenders for large-scale stationary and grid-level applications [3–7].

Despite these advantages, the practical application of AZIBs faces several serious challenges, the most important of which is the uncontrolled formation of zinc dendrites during repeated coating/stripping cycles. These dendritic structures can pierce the separators, causing internal short circuits, rapid power degradation, and low coulombic efficiency [8]. In parallel, side reactions such as hydrogen evolution, corrosion, and passivation of the zinc surface further impair long-term cycling stability [8, 10]. Overcoming these interfacial instabilities requires carefully engineered strategies to ensure uniform Zn^{2+} deposition and stable electrode-electrolyte interactions.

To address these problems, researchers have explored multiple approaches. Anode surface modification through protective coatings or three-dimensional host structures reduces local current density and improves deposition homogeneity [9]. The design of functional separators with tailored porosity and ion selectivity has also been reported to regulate Zn^{2+} flux. On the cathode side, structural optimization aims to suppress dissolution and improve cycling durability. However, among all strategies, electrolyte engineering stands out as the most practical and scalable method. Previous studies have shown that tailoring electrolyte composition and solvation structures can regulate Zn^{2+} nucleation, suppress dendrite formation, and mitigate side reactions, thereby extending cycle life and advancing AZIBs toward practical deployment [10].

Electrolyte modification strategies have recently gained significant momentum. Conventional $ZnSO_4$-based electrolytes provide high ionic conductivity but are prone to dendrite formation and hydrogen evolution. To mitigate these drawbacks, foreign cations such as Li^+, Na^+, and Mg^{2+} have been introduced into $ZnSO_4$ solutions. These ions influence the structure of the electric double layer and modify Zn^{2+} nucleation kinetics, thereby promoting uniform deposition [11–12]. Among these additives, Li^+ has received particular attention. It can homogenize current distribution across the Zn surface, facilitate compact deposits, and stabilize interfacial chemistry. For instance, Dai et al. demonstrated that Li-containing electrolytes form a protective solid-electrolyte interphase (SEI) composed of $Li_2S_2O_7$ and Li_2CO_3, which mitigates dendrite propagation and suppresses parasitic hydrogen evolution [13]. Other studies have similarly reported that the incorporation of Li_2SO_4 improves Coulombic efficiency and extends cycling stability [14–15].

Beyond dendrite suppression, electrolyte engineering also modulates Zn^{2+} solvation chemistry and hydration structure. Recent reviews highlight that introducing Li^+ ions reshapes Zn^{2+} solvation shells, alters ion transport, and leads to favorable SEI compositions that stabilize electrode surfaces [16]. These findings underscore the importance of carefully designing the Zn^{2+}/Li^+ ratio to optimize both deposition morphology and interfacial chemistry.

Nevertheless, most previous reports have examined only narrow concentration ranges, leaving a lack of systematic comparisons across different $ZnSO_4$-Li_2SO_4 ratios. Furthermore, the precise mechanisms by which Li^+ alters Zn deposition-whether through competitive adsorption at the electrode interface, modification of Zn^{2+} solvation, or stabilization of SEI layers-remain under

debate [17]. Addressing these questions requires carefully designed studies that isolate the role of Zn^{2+}/Li^{+} ratio on electrochemical behavior and surface morphology.

Another critical but underexplored factor is the balance between dendrite suppression and available Zn^{2+} concentration. While dilution of Zn^{2+} by Li_2SO_4 may stabilize morphology and reduce side reactions, it can also lower the effective Zn^{2+} availability, limiting charge-storage capacity. Recent electrolyte design studies also emphasize the need to balance reversibility and capacity; in line with this, our results show that the equimolar Zn^{2+} /Li^{+} ratio offers the most effective compromise between dendrite suppression and charge storage [18].

The purpose of this work is to fill this gap by systematically investigating five electrolyte formulations: 2M $ZnSO_4$, 1.5M $ZnSO_4$ + 0.5M Li_2SO_4, 0.5M $ZnSO_4$ + 1.5M Li_2SO_4, 1M $ZnSO_4$ + 1M Li_2SO_4, and 2M Li_2SO_4. Using cyclic voltammetry, galvanostatic cycling, and post-cycling scanning electron microscopy, we examine how the Zn^{2+}/Li^{+} ratio influences reversibility, dendrite formation, and cycling stability.

Materials and methods

Electrolytes. Five types of aqueous electrolytes were prepared in order to evaluate the effect of Zn^{2+}/Li^{+} ratio on the electrochemical behavior of the cells. The electrolytes included: (i) 2M $ZnSO_4$, (ii) 1.5M $ZnSO_4$ + 0.5M Li_2SO_4, (iii) 0.5M $ZnSO_4$ + 1.5M Li_2SO_4, (iv) 1M $ZnSO_4$ + 1M Li_2SO_4 and 2M Li_2SO_4. All salts were of analytical grade and dissolved in deionized water under constant stirring until complete dissolution was achieved.

Electrodes. Zinc foil was used as the anode and cathode was lithium iron phosphate (LFP), which was prepared in the form of a slurry composed of active material (LFP), conductive carbon (AB), and polyvinylidene fluoride (PVDF) binder in a weight ratio of 8:1:1. The slurry then uniformly coated onto graphite foil using a doctor blade technique. The electrodes were dried under vacuum at 80 °C for 12 h.

Cell configuration. Electrochemical cells were assembled in a CR2032 coin-cell configuration. A glass fiber separator was used to physically isolate the Zn anode from the LFP cathode.

Electrochemical measurements. Cyclic voltammetry (CV) was conducted in the voltage range of 0.9-2.0 V at a scan rate of 0.2 mV s^{-1} to evaluate the redox processes of the LFP cathode and the reversibility of Zn plating/stripping. Galvanostatic charge-discharge tests were performed at a constant current density of 20 mA g^{-1} to assess specific capacity and cycling stability.

Characterization. To examine the influence of electrolyte composition on Zn deposition, the anode surface morphology was examined using scanning electron microscopy (SEM).

Results and discussion

The CV curves of Zn//LFP cells were recorded within a voltage window of 0.9-2.0 V at a scan rate of 0.2 mV s^{-1} (Figure 1).

In Figure 1a 2M $ZnSO_4$ electrolyte, the CV profile shows broad and poorly defined redox features. A cathodic peak appears at ≈1.07 V, while the anodic process is not fully captured.

Upon partial substitution with Li_2SO_4 (1.5M $ZnSO_4$ + 0.5M Li_2SO_4), the electrochemical response improves significantly. The reduction peak lies at ≈1.10-1.15 V, while the oxidation exhibits a shoulder at ≈1.42 V. With repeated cycling, the anodic peak stabilizes upon cycling, indicating reduced polarization and enhanced reversibility.

The equimolar mixture (1M $ZnSO_4$ + 1M Li_2SO_4) displays the sharpest and most symmetric peaks. The reduction peak is centered at ≈1.15-1.19 V, while the oxidation peak occurs at ≈1.34-1.48 V, yielding the smallest ΔE among all electrolytes. The strong current responses and nearly overlapping cycles demonstrate highly reversible Zn plating/stripping [19]. This composition thus provides the most favorable balance between Zn^{2+} concentration and Li^{+}.

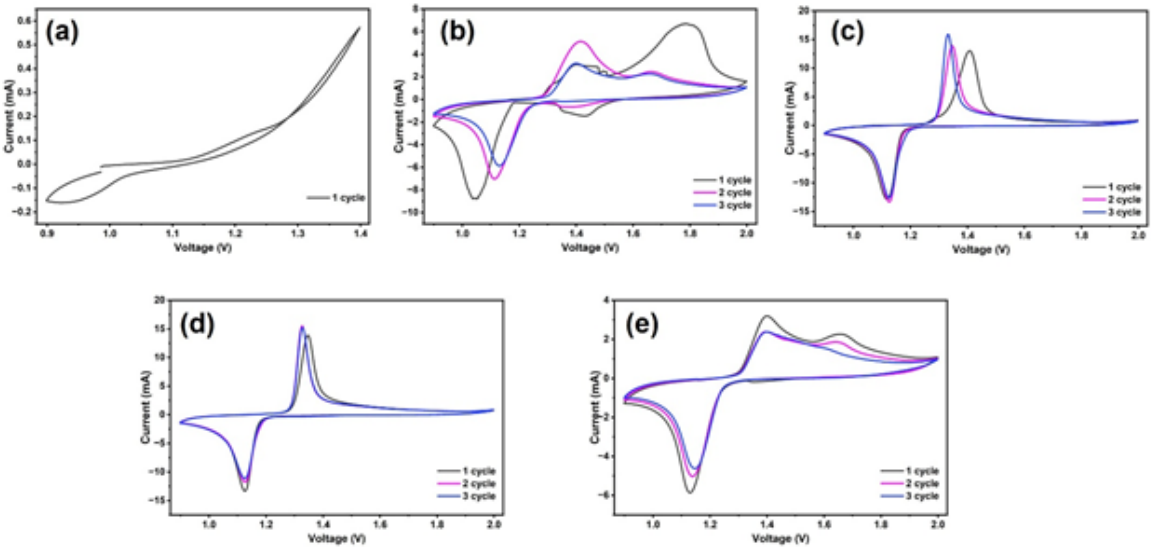

Figure 1 – Cyclic voltammograms of Zn//LFP cells: a) 2M $ZnSO_4$, b) 1.5M $ZnSO_4$ + 0.5M Li_2SO_4, c) 1M $ZnSO_4$ + 1M Li_2SO_4, d) 0.5M $ZnSO_4$ + 1.5M Li_2SO_4 and e) 2M Li_2SO_4

Further increasing the Li_2SO_4 fraction (0.5M $ZnSO_4$ + 1.5M Li_2SO_4) maintains sharp and reversible peaks at 1.12-1.16 V and ≈ 1.34-1.40 V. However, the overall current densities are lower than in the equimolar electrolyte, reflecting reduced Zn^{2+} availability. This suggests a trade-off between dendrite suppression and charge storage capability when Zn^{2+} concentration is diluted.

In the absence of $ZnSO_4$ (2M Li_2SO_4), the CV curves no longer display clear Zn^{2+}/Zn redox peaks. Instead, broad anodic responses are observed at ≈1.40-1.50 V with an additional weak shoulder. This confirms that Li_2SO_4 alone cannot sustain Zn plating/stripping and mainly drives parasitic side reactions.

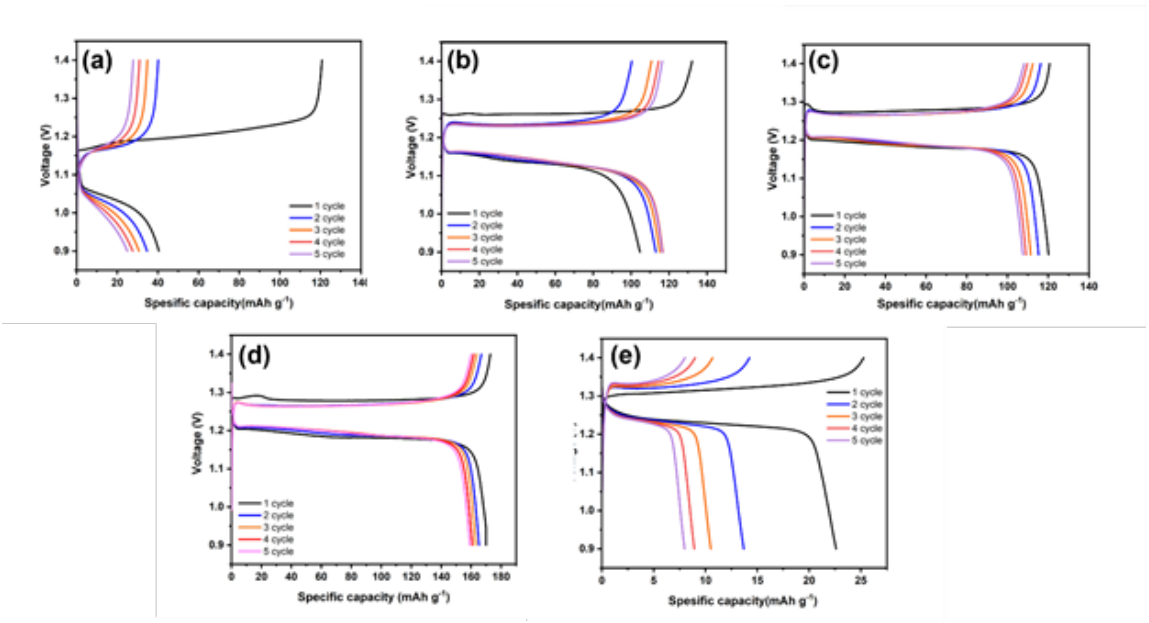

Figure 2 – Charge-discharge profiles of Zn//LFP cells in different electrolytes between 0.9-1.4 V

To validate the CV results, the cycling performance of Zn//LFP cells was further examined under repeated galvanostatic charge-discharge (Figure 2 and 3). The charge–discharge curves show clear differences across electrolytes. Pure $ZnSO_4$ exhibits large polarization and low capacity, while partial Li_2SO_4 substitution improves reversibility and sharpens voltage plateaus. The equimolar 1M $ZnSO_4$ + 1M Li_2SO_4 delivers the highest and most stable capacity with minimal hysteresis. In contrast, Li_2SO_4 alone provides steep sloping curves with very low storage, confirming the absence of reversible Zn^{2+} electrochemistry and indicating that the observed capacity mainly originates from side reactions rather than effective charge storage. In terms of cycling performance, the 2M $ZnSO_4$ electrolyte exhibits, the capacity rapidly declines from ~40 mAh g^{-1} to around 10 mAh g^{-1} within 15 cycles. A similar rapid fading is observed in the 2M Li_2SO_4 electrolyte, which delivers <10 mAh g^{-1} after only a few cycles. By contrast, the mixed $ZnSO_4$-Li2SO_4 electrolytes exhibit markedly better cycling stability. Both the 1.5M $ZnSO_4$+ 0.5M Li_2SO_4 and 0.5M $ZnSO_4$+ 1.5M Li_2SO_4 compositions stabilize around 85-95 mAh g^{-1} after initial activation, retaining most of their capacity over 30 cycles.

The best performance is achieved with the equimolar 1M $ZnSO_4$+ 1M Li_2SO_4 electrolyte, which maintains ~165-169 mAh g^{-1} [20] over 20 cycles with minimal fading, confirming highly reversible Zn plating/stripping and excellent interfacial stability. These results strongly corroborate the CV findings: while single-salt electrolytes suffer from poor reversibility and rapid capacity decay, mixed Zn-Li electrolytes, especially the equimolar formulation, ensure stable and reversible cycling behavior.

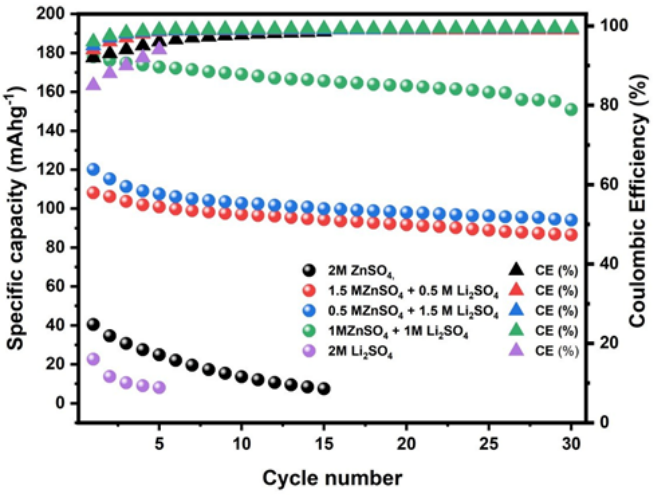

Figure 3 – Cycling performance of Zn//LFP cells in different electrolytes

In addition to capacity retention, the coulombic efficiency (CE) of the cells was evaluated to assess the reversibility of the electrochemical processes (Figure 3). The mixed $ZnSO_4$ – Li_2SO_4 electrolytes exhibit consistently high CE values close to ~98–100% throughout cycling, indicating highly reversible Zn plating/stripping behavior.

In particular, the equimolar 1M $ZnSO_4$ + 1M Li_2SO_4 electrolyte demonstrates the most stable CE (~99 – 100%), confirming minimal parasitic reactions and enhanced interfacial stability. In contrast, the 2M $ZnSO_4$ electrolyte shows slightly lower CE values, which can be attributed to side reactions such as hydrogen evolution and dendrite formation.

These results further confirm that electrolyte optimization with Li_2SO_4 effectively improves both reversibility and cycling stability of aqueous Zn-ion batteries.

Further, the surface morphologies of Zn anodes after 30 cycles in different electrolytes were examined by SEM (Figure 4). In the 2M $ZnSO_4$ electrolyte (Figure 4a), the surface is covered with large, irregular dendritic structures with lengths exceeding 1 μm (1.3–1.6 μm), indicating severe Zn dendrite growth and uncontrolled deposition. Such morphology explains the rapid capacity decay observed during cycling [16].

In the 1.5M $ZnSO_4$ + 0.5M Li_2SO_4 electrolyte (Figure 4b), the dendritic features are partially suppressed, but the surface still exhibits scattered plate-like deposits and roughness, reflecting incomplete stabilization. A more uniform morphology is obtained in the 1M $ZnSO_4$ + 1M Li_2SO_4 electrolyte (Figure 4c), where the Zn surface is smoother and dendritic protrusions are largely absent. This confirms that the equimolar mixture promotes homogeneous Zn nucleation and growth.For the 0.5M $ZnSO_4$ + 1.5M Li_2SO_4 electrolyte (Figure 4d), the Zn deposits are finer and less aggregated compared to pure $ZnSO_4$, though scattered needle-like features are still visible. The reduced Zn^{2+} concentration leads to thinner deposits, which may limit capacity despite improved stability. Finally, in the 2M Li_2SO_4 electrolyte (Figure 3e), the surface is dominated by irregular flakes and byproducts, lacking well-defined Zn deposits. This correlates with the poor electrochemical performance observed.

Taken together, the SEM results confirm that Li_2SO_4-modified electrolytes suppress dendrite formation and promote smoother Zn deposition, with the 1:1 $ZnSO_4$-Li_2SO_4 mixture producing the most uniform surface morphology, in line with electrochemical data.

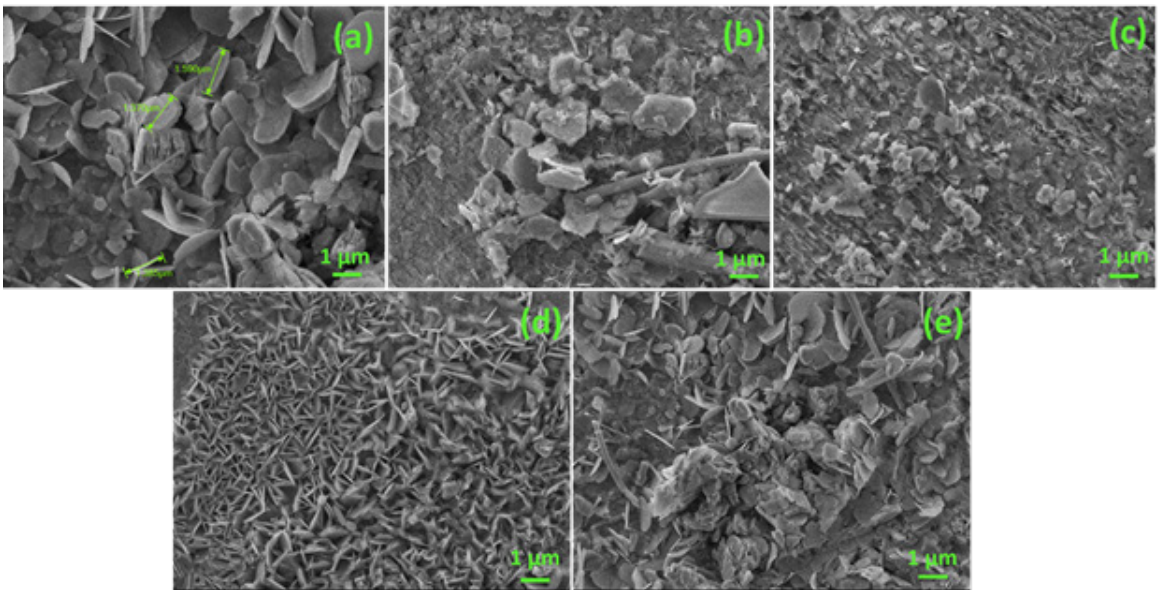

Figure 4 – SEM images of Zn anodes after cycling: a) 2M $ZnSO_4$, b) 1.5M $ZnSO_4$ + 0.5M Li_2SO_4, c) 1M $ZnSO_4$ + 1M Li_2SO_4, d) 0.5M $ZnSO_4$ + 1.5M Li_2SO_4 and e) 2M Li_2SO_4

To further understand the origin of the improved electrochemical performance, the possible mechanisms are discussed below. The improved electrochemical performance in Li_2SO_4 -containing electrolytes can be attributed to two possible mechanisms. First, Li^+ ions modify the Zn^{2+} solvation structure, reducing the number of free water molecules in the Zn^{2+} solvation shell. This suppresses parasitic hydrogen evolution and promotes more uniform Zn deposition. Second, Li^+ may contribute to the formation of a more stable solid-electrolyte interphase (SEI), composed of inorganic species such as Li_2CO_3 and Li_2SO_4 – derived compounds, which protects the Zn surface and inhibits dendrite growth. Although direct evidence of SEI formation was not obtained in this study, the improved cycling stability and smoother morphology observed by SEM indirectly support this hypothesis.

The obtained results are consistent with recent advances in aqueous zinc-ion batteries, which highlight the critical role of electrolyte engineering in improving Zn anode stability and electrochemical performance. Previous studies have shown that tailoring the Zn^{2+} solvation structure, cycling stability [21] and introducing Li^+ – based additives can effectively suppress parasitic reactions, enhance Coulombic efficiency, and mitigate dendrite growth [22]. In most reported systems, modified electrolytes typically achieve capacities in the range of 90–150 mAh g^{-1} depending on electrolyte composition [23]. In contrast, the equimolar $ZnSO_4$–Li_2SO_4 electrolyte (1M + 1M) investigated in

this work delivers a higher specific capacity of ~165–169 mAh g^{-1}, along with improved cycling stability and more uniform Zn deposition morphology.

These findings indicate that precise control of the Zn^{2+}/Li^{+} ratio plays a decisive role in optimizing ion solvation structure and interfacial processes, leading to superior electrochemical performance compared to previously reported electrolyte systems.

Conclusion

In this research, the electrochemical behavior of Zn//LFP cells was investigated in different aqueous electrolytes. The results show that mixed $ZnSO_4$–Li_2SO_4 electrolytes significantly enhance Zn^{2+} plating/stripping reversibility and cycling stability compared to single-salt systems. Among the tested formulations, the 1M $ZnSO_4$ + 1M Li_2SO_4 electrolyte exhibited the best overall performance, delivering the highest capacity, excellent retention, and uniform Zn morphology. Overall, our results demonstrate that electrolyte engineering with Zn-Li co-salts is a viable route to achieving stable aqueous Zn-ion batteries, as evidenced by the 1M $ZnSO_4$ + 1M Li_2SO_4 system delivering ~165-169 mAh g^{-1} with excellent retention.

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Conflict of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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ТҰРАҚТЫ СУЛЫ МЫРЫШ-ИОНДЫҚ АККУМУЛЯТОРЛАР ҮШІН $ZNSO_4 – LI_2SO_4$ ЭЛЕКТРОЛИТТЕРІН ОҢТАЙЛАНДЫРУ

Аңдатпа

Сулы мырыш-ионды батареялар (AZIBs) қауіпсіз, арзан және экологиялық таза энергия сақтау жүйелерінің болашағы зор түрлерінің бірі болып саналады. Алайда, мырыш анодының тұрақсыздығы, соның ішінде бақылаусыз дендриттік өсу, сутегінің бөлінуі және мырыштың қайтымсыз қапталу/шығарылу процестері олардың ауқымды қолданылуына кедергі келтіреді. Бұл мәселелер сыйымдылықтың тез төмендеуіне және қызмет ету мерзімінің қысқаруына әкеледі. Осыған байланысты электролитті оңтайландыру анодты тұрақтандырудың қарапайым әрі тиімді тәсілі ретінде ерекше мәнге ие. Жұмыстың мақсаты – әртүрлі құрамдағы $ZnSO_4$–Li_2SO_4 электролиттерінің (2 M $ZnSO_4$, 1.5 M $ZnSO_4$ + 0.5 M Li_2SO_4, 0.5 M $ZnSO_4$ + 1.5 M Li_2SO_4, 1 M $ZnSO_4$ + 1 M Li_2SO_4 және 2 M Li_2SO_4) мырыш анодына әсерін жүйелі түрде зерттеу. Электрохимиялық сипаттамалар циклдік вольтамметрия (CV), гальваностатикалық заряд-разряд сынақтары арқылы зерттелді, ал циклдеуден кейін анод бетінің морфологиясы сканерлеуші электрондық микроскопия (СЭМ) әдісімен

талданды. Нәтижелер $ZnSO_4$ электролитіне Li_2SO_4 тұзын енгізу Zn^{2+} иондарының сольватациялық ортасын өзгертетінін көрсетті, соның арқасында мырыштың қайтымдылығы артып, дендриттердің түзілуі бәсеңдеді. Әсіресе, аралас электролиттер бір компонентті жүйелермен салыстырғанда төмен поляризацияны және айқынырақ кернеу қисықтарын көрсетті. Олардың ішінде 1 M $ZnSO_4$ + 1 M Li_2SO_4 теңмолярлы құрамы ең оңтайлы нәтижелерге қол жеткізді: тұрақты циклдеу, төмен интерфейстік кедергі және біртекті, дендритсіз морфология. Осылайша, электролиттік инженерия мырыш анодтарын тұрақтандырудың тиімді әдісі болып табылады және болашақта сулы мырыш-ионды батареяларды ірі көлемді энергия сақтау мақсатында дамытуға жаңа мүмкіндіктер ашады.

Түйін сөздер: сулы мырыш-ионды аккумуляторы, Zn анод, электролит, циклдік вольтамметрия, дендрит.

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ОПТИМИЗАЦИЯ ЭЛЕКТРОЛИТОВ $ZNSO_4 – LI_2SO_4$ ДЛЯ СТАБИЛЬНЫХ ВОДНЫХ ЦИНК-ИОННЫХ БАТАРЕЙ

Аннотация

Водные цинковые аккумуляторы (AZIBs) рассматриваются как одни из наиболее перспективных кандидатов для безопасного, недорогого и экологически чистого накопления энергии. Тем не менее их практическое применение ограничено нестабильностью цинкового анода, вызванной неконтролируемым ростом дендритов, выделением водорода и низкой обратимостью процессов осаждения/растворения цинка. Эти факторы приводят к быстрой деградации емкости и сокращению срока службы батареи. В связи с этим оптимизация электролита является эффективным и доступным подходом к стабилизации анода. Целью работы является систематическая оценка поведения цинкового анода в электролитах различного состава 2M $ZnSO_4$, 1.5M $ZnSO_4$ + 0.5M Li_2SO_4, 0.5M $ZnSO_4$ + 1.5M Li_2SO_4, 1M $ZnSO_4$ + 1M Li_2SO_4, и 2M Li_2SO_4. Электрохимические характеристики исследованы методами циклической вольтамперометрии (CV), гальваностатического циклирования и после испытаний морфология поверхности анализировалась с помощью сканирующей электронной микроскопии (СЭМ). Данные показали, что добавление Li_2SO_4 в раствор $ZnSO_4$ существенно изменяет сольватационную оболочку Zn^{2+}, улучшая обратимость процессов осаждения/растворения и подавляя рост дендритов. Смешанные электролиты продемонстрировали более четкие вольт-амперные профили и меньшую поляризацию по сравнению с одноионными системами. Наиболее сбалансированные характеристики достигнуты в электролите 1M $ZnSO_4$ + 1M Li_2SO_4 : обеспечены стабильное циклирование, низкое межфазное сопротивление и однородная бездендритная морфология. Таким образом, инженерия электролита является практическим и масштабируемым решением для стабилизации цинковых анодов и открывает путь к созданию высокоэффективных водных аккумуляторов для крупномасштабных систем хранения энергии.

Ключевые слова: водные цинк-ионные аккумуляторы, Zn-анод, электролит, циклическая вольтамперометрия, дендрит.