

In Situ Polymer Electrolyte Coating for Lithium–Sulfur Batteries

N. Zhumasheva¹, F. Sultanov^{1,2}, B. Tatykayev¹, Z. Shalabayev¹,
A. Belgibayeva¹, A. Dauletbay¹, E. Nurgaziyeva^{1*}

¹National Laboratory Astana, Nazarbayev University, Qabanbay batyr ave., 53, Astana, Kazakhstan

²Institute of batteries, Nazarbayev University, Qabanbay batyr ave., 53, Astana, Kazakhstan

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ABSTRACT

The transition from liquid electrolytes to solid-state electrolytes represents a key strategy for improving the safety and energy efficiency of lithium-sulfur (Li-S) batteries. However, poor interfacial contact and high resistance at the electrode-electrolyte interface remain critical barriers to commercialization. In this study, we report an *in situ* coating approach, where a crosslinkable polytetrahydrofuran-based solid polymer electrolyte (aPTHF-SPE) is directly formed on the surface of C@S cathodes via UV-curing. SEM and EDS analyses confirm the formation of a uniform polymer layer with a thickness of 35–46 μm , ensuring intimate interfacial contact and reduced void formation. Electrochemical tests demonstrate that the *in situ* coated cathodes deliver an initial discharge capacity of $\sim 250 \text{ mA}\cdot\text{h}\cdot\text{g}^{-1}$, with subsequent cycles exhibiting improved stability due to enhanced ionic transport and electrode activation. Despite gradual fading after extended cycling, the strategy significantly improves the electrode-electrolyte interface compared to uncoated cathodes. These findings highlight *in situ* polymer electrolyte coating as a promising and scalable method for addressing interfacial challenges in solid-state Li-S batteries, paving the way toward safer and higher-performance energy storage systems.

1. Introduction

The increasing demand for high-energy-density rechargeable batteries highlights the need for in-depth exploration of Li-S batteries, known for their exceptional energy storage capability. Among them, solid-state Li-S batteries stand out as a promising next-generation technology, offering both high energy density ($\sim 2600 \text{ Wh}\cdot\text{kg}^{-1}$) and enhanced safety [1, 2].

While solid-state Li-S batteries effectively mitigate polysulfide dissolution, volume expansion, and lithium dendrite formation, they still suffer from low ionic conductivity and poor electrode-electrolyte interface stability during charge/discharge, limiting their practical application. The transition from liquid electrolytes to all-solid-state electrolytes is considered one of the most promising strategies to enhance safety and reduce energy losses in lithium-ion and lithium-sulfur batteries. However, this transition poses several challenges,

including insufficient ionic conductivity and high interfacial resistance between the electrolyte and electrodes. The unique interfacial characteristics of the electrode/solid-state electrolyte (SSE) interface, compared with those of the electrode/liquid electrolyte interface, often lead to significant interfacial resistance. Ideally, the electrode/SSE interface should exhibit high chemical and mechanical stability, enable efficient ion transport, and maintain maximum contact area throughout cycling [3–5]. Nevertheless, this interface remains a major obstacle to the commercialization of all-solid-state lithium-sulfur batteries (ASSLSBs). High interfacial resistance at both the anode and cathode/SSE interfaces is a common issue. To improve the electrochemical performance of ASSLSBs, it is essential to ensure optimal interfacial compatibility, contact, and chemical stability [1, 6–8].

Polymer electrolytes can be classified into different types, including solid polymer electrolytes, gel electrolytes, and composite polymer electrolytes

*Corresponding author: E. Nurgaziyeva; E-mail address: elmira.nurgaziyeva@nu.edu.kz

incorporating ceramic fillers. In Li-S batteries, gel polymer electrolytes offer good ionic conductivity and flexibility but may suffer from limited mechanical strength and polysulfide dissolution. Polymer and composite polymer electrolytes are widely explored for Li-S batteries due to their potential to enhance ionic conductivity, mechanical stability, and interfacial compatibility.

However, poor ion transport at room temperature and weak interfacial contact with sulfur cathodes remain critical challenges that need to be addressed worldwide. Numerous efforts have been made to overcome these challenges, including polymer matrix modifications, the incorporation of functional additives, and the development of thin polymer electrolytes [9].

Over the past few decades, various research groups have developed polymer electrolytes with enhanced ionic conductivity and improved interfacial contact for Li-S batteries. PEO is a widely used polymer for polymer electrolyte fabrication due to its mechanical stability and excellent film-forming ability. However, its low ionic conductivity (10^{-7} – 10^{-5} S/cm) at room temperature, caused by its crystalline structure, limits its practical application in Li-S cells. Previous studies have shown that incorporating dual salts (LiTFSI/LiBOB), modifying the polymer matrix, and adding various fillers can significantly enhance ionic conductivity. Also different polymers have been studied as polymer electrolytes for Li-S batteries, including poly(ethylene oxide) (PEO) and poly(ethylene-methylene oxide) (PEMO) [10], poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-co-HFP) [11], and polyacrylonitrile (PAN)-based ‘polymer-in-salt’ electrolytes [12], among others. Nevertheless, the practical application of polymer-based electrolytes is still hindered by challenges such as low ionic conductivity at room temperature, poor lithium-ion transport, and instability at high voltages. Addressing these issues requires further optimization of polymer structure, composition, and electrolyte formulation to enhance electrochemical performance and long-term stability.

In this work, we aim to integrate the previously studied solid polymer electrolyte [13] with a Li-S battery, evaluating its ability to suppress polysulfide shuttling and enhance cycle stability. This research contributes to the field by addressing the critical challenge of polysulfide dissolution, which significantly hinders the long-term performance of Li-S batteries.

2. Experimental part

2.1. Synthesis of polymer electrolyte

The solid polymer electrolyte based on polytetrahydrofuran was synthesized following the method described in [13]. Previously, PTHF was first modified with acryloyl chloride, followed by the addition of PEGDA and ETPTA crosslinkers. Polymer electrolytes were fabricated by casting the mixture of a-PTHF, PEGDA and ETPTA with LiTFSI salt, and subsequently exposed to UV irradiation.

2.2. Synthesis of sulfur cathode

The synthesis procedure of the sulfur cathode was described in detail in our previous work [14]. Sulfur was immobilized onto the porous graphene composite (GPC) carbon matrix using a melt-diffusion method. First, sulfur was dissolved in a CS_2 /IPA (7:3) mixture under stirring, followed by the addition of GPC powder (4:6 ratio). The mixture was ultrasonicated for 20 min and stirred until solvent evaporation. The obtained material was then heated in an Ar atmosphere at 155 °C for 12 h (2.5 °C/min) and further annealed at 200 °C for 1 h to remove non-encapsulated sulfur. The obtained carbon-sulfur composite was subsequently mixed with acetylene black and PVDF (8:1:1) in NMP to form a slurry, which was then cast onto carbon-coated aluminum foil for C@S cathode fabrication.

2.3. In situ preparation of cathode-electrolyte system

In our work, an *in situ* coating method was employed to improve the contact between the cathode and the solid-state electrolyte. For the *in situ* coating, an C@S cathode and an aPTHF-based electrolyte were used. The preparation of the aPTHF-based solid polymer electrolyte (SPE) involved two main steps: acrylation of polytetrahydrofuran (PTHF) and subsequent processing to obtain the solid electrolyte. In the first step, PTHF was acrylated by converting its terminal groups into photosensitive acrylate groups.

In the second step, the aPTHF-based solid polymer electrolyte (SPE) was prepared by mixing a-PTHF with ethoxylated trimethylolpropane triacrylate (ETPTA) and polyethylene glycol diacrylate (PEGDA) in a mass ratio of 8:1:1. This mixture was stirred until homogeneous, after which lithium salt (LiTFSI) was added to the solution. Additionally, 2-hydroxy-

2-methylpropiophenone (HMPP) was added at a concentration of 3 wt% as a photoinitiator. The liquid polymer precursor aPTHF (200 μL) was applied onto the surface of the C@S cathode, then sandwiched between two glass plates and subjected to UV-curing (Spectroline, ENF-240C/FE, $\lambda_{\text{max}} = 365$ nm) for 10 min. After curing, an aPTHF polymer film was successfully formed *in situ* on the surface of the C@S cathode (Fig. 1). The resulting structure was then dried under vacuum for 48 h to remove residual solvents. The entire procedure was carried out under inert conditions in a glovebox to prevent contamination.

2.4. Characterization

The surface characteristics and the thickness of the prepared membranes of solid polymer electrolytes were analyzed using field emission electron microscopy (FESEM, SEM, JEOL JSM-7500F, ZEISS Crossbeam 540, Jena, Germany).

The coin cells were assembled with an C@S cathode (the mass loading of $2.5 \text{ mg}\cdot\text{cm}^{-2}$), with aPEP as the solid electrolyte, and Li metal as the anode to evaluate the electrochemical performance of the novel materials. Cyclic voltammetry (CV) was performed 1.3 V to 3.0 V at a scan rate of $0.1 \text{ mV}\cdot\text{s}^{-1}$ with the cell configuration Li/SPE-C@S at 80°C .

3. Results and Discussion

Scanning electron microscopy (SEM) was employed to evaluate the interfacial layers of the *in situ* coated C@S cathode and the solid polymer electrolyte (SPE). SEM images were obtained for the carbon-coated aluminum foil current collector, C@S cathodes before and after roll-pressing, as well as for the C@S cathode with the *in situ* coated SPE. Based on these SEM images, the thickness of the coatings was measured, revealing that the average thickness of the *in situ* coated polymer ranged from 35 to 46 μm . The SEM image of the unrolled C@S cathode on the current collector showed poor adhesion with visible gaps at the interface. In contrast, the roll-pressed C@S cathode demonstrated a well-bonded and tightly connected interface with the current collector.

The morphology and elemental distribution of the electrode-electrolyte interface were examined using cross-sectional SEM and EDS mapping (Fig. 2 a-c and Fig. 3 a-f).

The carbon-coated Al foil exhibits a smooth and dense morphology with an average thickness of $\sim 18 \mu\text{m}$ (Fig. 2a). Only minor surface scratches are visible from the rolling process, and the EDS analysis confirms a dominant Al signal with negligible impurities. This indicates the high purity of the foil

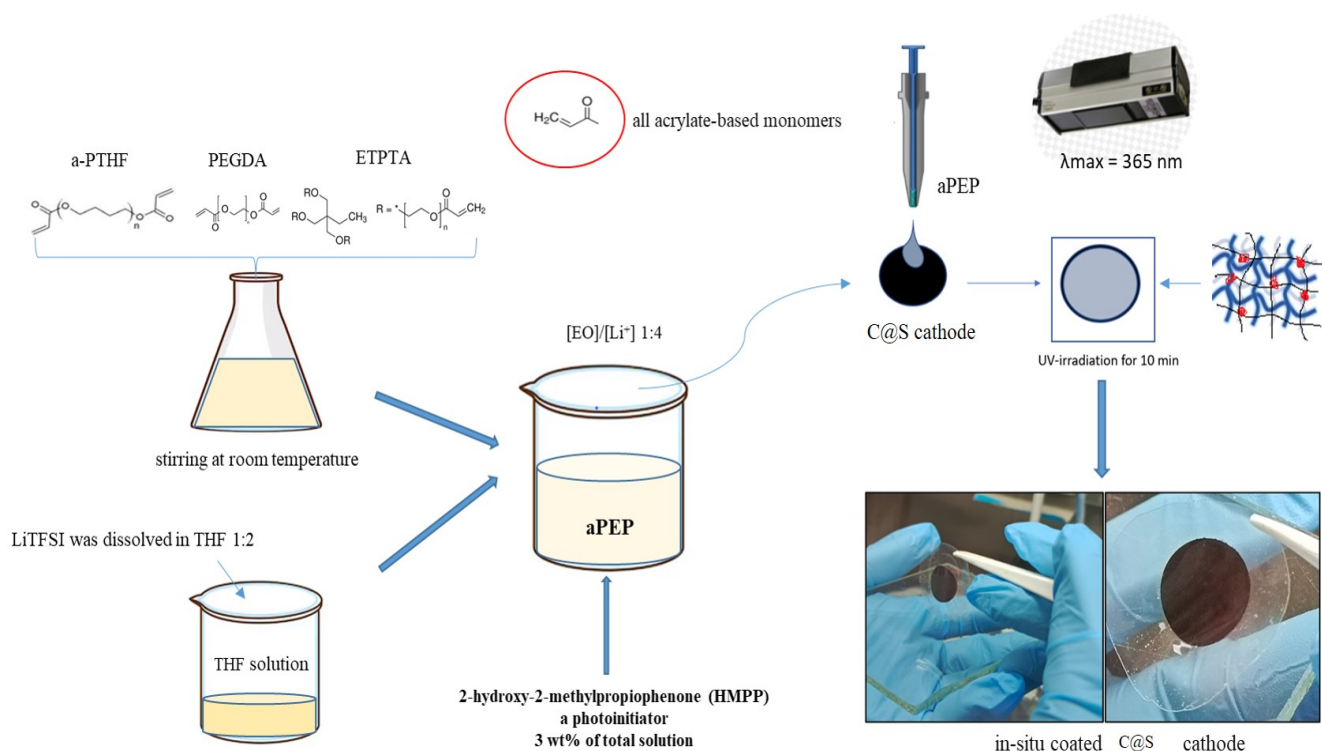


Fig. 1. Schematic illustration of the *in situ* preparation of the cathode-electrolyte system.

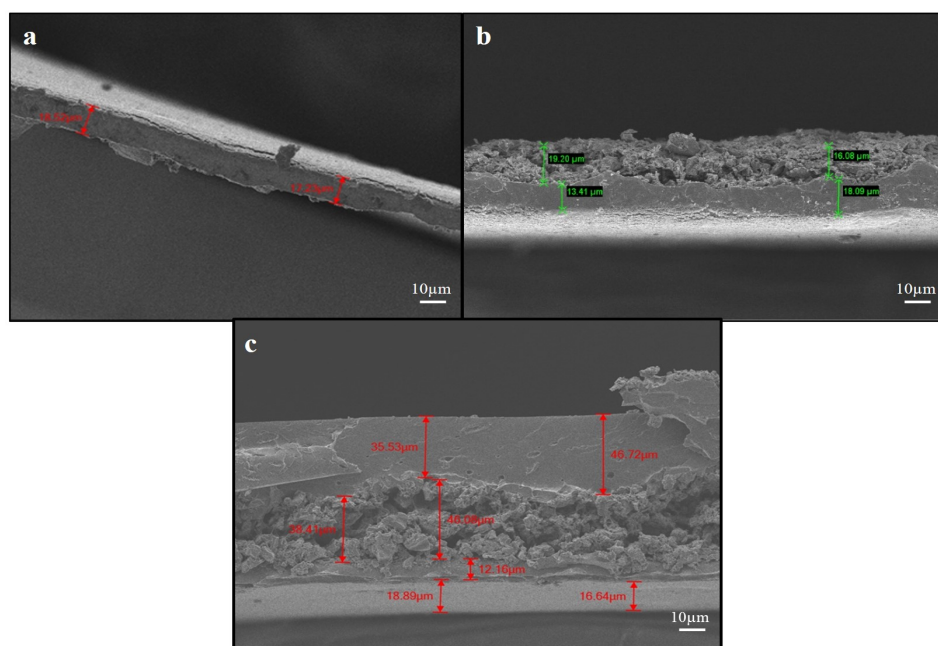


Fig. 2. (a) Al foil, (b) cathode C@S-roll pressed on the Al foil, (c) *in situ* coated aPTHF-based solid polymer electrolyte layer.

and its suitability as a stable current collector. After roll-pressing, the C@S composite cathode forms a porous, granular layer with thicknesses ranging from $\sim 16.08 \mu\text{m}$ to $19.20 \mu\text{m}$ (Fig. 2b). The interface with the Al foil appears well adhered, suggesting strong mechanical contact induced by roll pressing. The porous microstructure is advantageous for electrolyte penetration, which can improve ion transport during cycling. However, slight variations in thickness indicate the need for further optimization of coating and pressing conditions. The *in situ* coating process results in a continuous polymer electrolyte film covering the cathode, with a thickness varying between $\sim 35.6 \mu\text{m}$ and $\sim 46.7 \mu\text{m}$ depending on the cross-sectional position (Fig. 2c). The SPE layer exhibits a dense and crack-free morphology, indicating good film-forming capability and strong interfacial adhesion. The conformal coating not only encapsulates the cathode particles but also fills interfacial voids, thereby improving physical contact between the cathode and electrolyte.

Elemental maps further confirm the successful integration of the multilayer structure (Fig. 3). Aluminum (Al, blue, Fig. 3g) is localized at the bottom current collector layer without diffusion into the overlying layers. Sulfur (S, magenta, Fig. 3b) is homogeneously distributed throughout the C@S cathode region, while carbon (C, red, Fig. 3c) and oxygen (O, green, Fig. 3d) signals are present in both the cathode and polymer electrolyte, reflecting contributions from the RHG matrix and the aPTHF-based polymer. The uniform elemental distribution demonstrates that the cathode maintains its

composite integrity and that the SPE forms a conformal coating over the entire cathode surface (Fig. 3 e, f). The combined SEM and EDS results reveal that the Al foil remains intact and uncontaminated, acting as a reliable current collector. The C@S cathode forms a well-adhered, moderately porous layer on the Al foil, providing pathways for ionic transport. The *in situ* coated aPTHF-based polymer electrolyte establishes a uniform and defect-free overlayer, ensuring strong interfacial contact and homogeneous element distribution.

These findings validate the effectiveness of the fabrication strategy and highlight the potential of the *in situ* coated aPTHF-based solid polymer electrolyte to stabilize the electrode-electrolyte interface, suppress polysulfide dissolution, and enhance long-term cycling stability in solid-state battery systems.

The CV profiles (Fig. 4a) of the RHG@/aPTHF-based SPE/Li cell display characteristic redox peaks associated with the reversible conversion of sulfur species during cycling at 80°C [15]. In the 2nd–6th cycles, the cathodic peaks corresponding to the reduction of sulfur to long-chain and subsequently short-chain polysulfides are well defined, while the anodic peaks related to the re-oxidation of polysulfides to elemental sulfur are also evident. The nearly overlapping CV curves after the 3rd cycle indicate good electrochemical reversibility and stable redox kinetics within the electrode–electrolyte system [9]. This suggests that the conformal polymer coating contributes to improved interfacial stability and efficient utilization of active sulfur species.

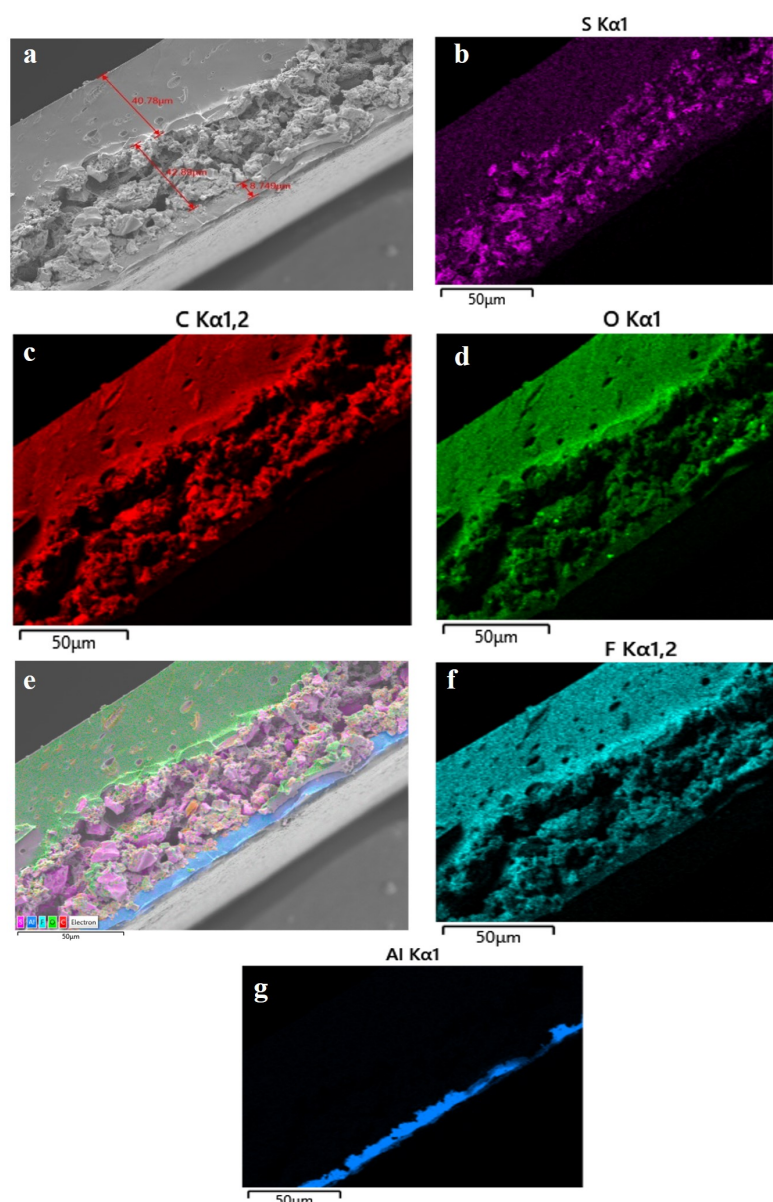


Fig. 3. SEM images: (a) ,cross-sectional image of *in situ* electrode-electrolyte interface (b) S, (c) C, and (d) O, (e) Energy-dispersive X-ray spectroscopy (EDS) analysis was performed on the C@S cathode with *in situ* coated SPE on carbon-coated aluminum foil, (f) F, (g) Al.

The charge-discharge curves at 80 °C (Fig. 4b) show two distinct discharge plateaus, consistent with the multistep reduction process of sulfur, while the charge process exhibits a corresponding plateau associated with sulfur reformation. The initial cycles demonstrate a higher specific capacity of about $250 \text{ mA}\cdot\text{h}\cdot\text{g}^{-1}$, which gradually decreases to $75 \text{ mA}\cdot\text{h}\cdot\text{g}^{-1}$. However, the subsequent cycles become more stable, indicating electrode activation and improved ionic transport within the solid polymer electrolyte. After 47 cycles, the specific capacity drops sharply. This capacity fading can be attributed to the deterioration of the electrode-electrolyte interface [16]. The capacity retention over multiple cycles remains relatively stable, highlighting the ability of the aPTHF-based electrolyte to suppress polysulfide

shuttling and maintain efficient electrochemical reactions. The nearly constant coulombic efficiency (>95% in most cycles) further supports the strong interfacial compatibility and high reversibility of the system. The performance of the *in situ* coated aPTHF-based solid polymer electrolyte at room temperature was limited due to its low ionic conductivity and insufficient interfacial contact with the electrode surfaces. The restricted segmental motion of the polymer chains at ambient conditions further hindered efficient ion transport, resulting in higher interfacial resistance and poor electrochemical performance [17]. Below are the preliminary cyclic voltammetry (CV) results for C@S cells with *in situ* coated aPTHF-based electrolyte | Li, along with the corresponding charge/discharge curves (Fig. 4).

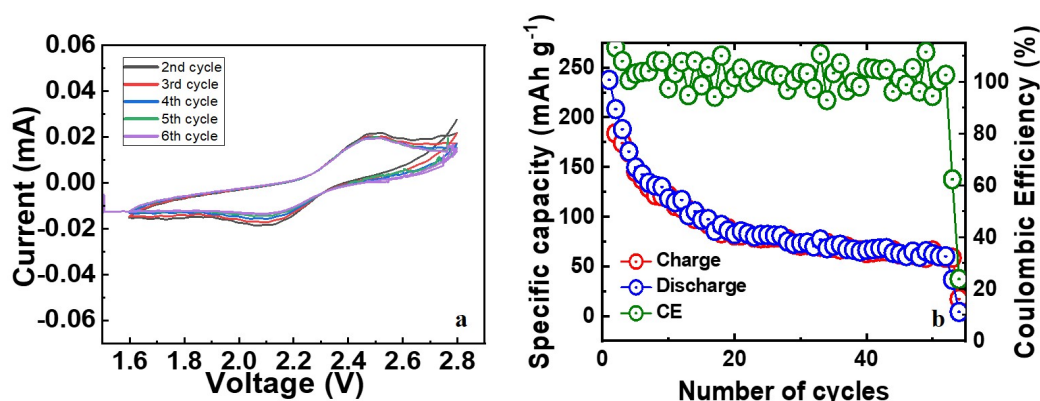


Fig. 4. Electrochemical performance of C@S/SPE/Li at 80 °C: (a) CV profile, (b) Ch/dCh curves.

The combined CV and charge-discharge results confirm that the *in-situ* coated aPTHF-based solid polymer electrolyte effectively stabilizes the sulfur redox processes, reduces interfacial resistance, and promotes reversible electrochemical cycling. These findings demonstrate the potential of the designed electrode-electrolyte configuration for achieving improved performance in solid-state lithium-sulfur batteries.

4. Conclusion

The one-step *in situ* coating method of coating C@S cathode with solid polymer electrolyte was improved. The combined structural and electrochemical analyses confirm the successful fabrication of the C@S cathode integrated with the *in-situ* coated aPTHF-based solid polymer electrolyte. SEM and EDS analyses confirmed the formation of a dense, conformal polymer layer with a thickness of ~35–46 μm , providing strong adhesion to both the current collector and the cathode while eliminating interfacial voids. This structural integrity facilitated efficient ion transport, as reflected in the well-defined redox peaks and overlapping CV profiles, which indicate stable and reversible sulfur conversion reactions at 80 °C. The characteristic charge-discharge plateaus and high coulombic efficiency (>95%) further highlight the strong interfacial compatibility and suppression of polysulfide shuttling. The initial cycles demonstrate a higher specific capacity of about 250 $\text{mA}\cdot\text{h}\cdot\text{g}^{-1}$, which gradually decreases to 75 $\text{mA}\cdot\text{h}\cdot\text{g}^{-1}$. Although gradual capacity fading was observed during extended cycling, the overall performance of the cell highlights the effectiveness of the *in situ* coating strategy in enhancing interfacial stability, improving sulfur utilization, and advancing the development of high-performance solid-state Li-S batteries.

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About the Authors

N. Zhumasheva – researcher, National Laboratory Astana, Nazarbayev University, Astana, Kazakhstan
E-mail: nazerke.zhumasheva@nu.edu.kz
ORCID: 0000-0001-5266-3429

F. Sultanov – PhD, Associate Professor, Leading researcher, National Laboratory Astana, Institute of batteries, Nazarbayev University, Astana, Kazakhstan
E-mail: fail.sultanov@nu.edu.kz
ORCID: 0000-0002-7789-9398

B. Tatykayev – PhD, Leading researcher, National Laboratory Astana, Nazarbayev University, Astana, Kazakhstan
E-mail: batukhan.tatykayev@nu.edu.kz
ORCID: 0000-0001-5071-1968

Z. Shalabayev – PhD, Leading researcher, National Laboratory Astana, Nazarbayev University, Astana, Kazakhstan
E-mail: zhandos.shalabayev@nu.edu.kz
ORCID: 0000-0003-3465-8241

A. Belgibayeva – PhD, Leading researcher, National Laboratory Astana, Nazarbayev University, Astana, Kazakhstan
E-mail: ayaulym.belgibayeva@nu.edu.kz
ORCID: 0000-0002-3052-5241

A. Dauletbay – PhD, Senior Researcher, National Laboratory Astana, Nazarbayev University, Astana, Kazakhstan
E-mail: akbar.dauletbay@nu.edu.kz
ORCID: 0000-0003-0157-1449

E. Nurgaziyeva – PhD, Senior researcher, National Laboratory Astana, Nazarbayev University, Astana, Kazakhstan
E-mail: elmira.nurgaziyeva@nu.edu.kz
ORCID: 0000-0001-8673-4977

In situ* покрытие катода твердым полимерным электролитом для литий-серных аккумуляторов*Н. Жумашева¹, Ф. Султанов^{1,2}, Б. Татыкаев¹, Ж. Шалабаев¹, А. Белгибаева¹, А. Даулетбай¹, Э. Нургазиева^{1*}**¹National Laboratory Astana, Назарбаев Университет, ул. Кабанбай батыра, 53, Астана, Казахстан²Институт аккумуляторов, Назарбаев Университет, ул. Кабанбай батыра, 53, Астана, Казахстан**АННОТАЦИЯ**

Переход от жидких к твердофазным электролитам рассматривается как ключевая стратегия для повышения безопасности и энергоэффективности литий-серных (Li-S) аккумуляторов. Однако плохой межфазный контакт и высокое сопротивление на границе электрод-электролит остаются критическими препятствиями для коммерциализации. В данной работе представлен метод *in situ* покрытия, при котором сшиваемый на свету твердый полимерный электролит на основе политетрагидрофурана (aPTHF-SPE) формируется непосредственно на поверхности катодов C@S методом УФ-отверждения. Анализы SEM и EDS подтвердили образование равномерного полимерного слоя толщиной 35–46 мкм, обеспечивающего плотный межфазный контакт и уменьшение образования пустот. Электрохимические испытания показали, что катоды с *in situ* покрытием демонстрируют начальную разрядную емкость около 250 мА·ч·г⁻¹, при этом последующие циклы характеризуются улучшенной стабильностью благодаря усиленному ионному транспорту и активации электрода. Несмотря на постепенное снижение емкости при длительном циклировании, предложенная стратегия значительно улучшает интерфейс электрод-электролит по сравнению с непокрытыми катодами. Полученные результаты подчеркивают, что *in situ* покрытие полимерным электролитом является перспективным и масштабируемым методом для решения межфазных проблем в твердотельных Li-S аккумуляторах, открывая путь к более безопасным и высокоэффективным системам накопления энергии.

Ключевые слова: твердые полимерные электролиты, PTHF, *in situ* покрытие, поперечный анализ, литий-серные аккумуляторы.

Литий-күкірт аккумуляторлары үшін *in situ* қатты полимерлі электролитпен катодты қаптау**Н. Жумашева¹, Ф. Сұлтанов^{1,2}, Б. Татықаев¹, Ж. Шалабаев¹, А. Белгібаева¹, А. Даулетбай¹, Э. Нұрғазиева^{1*}**¹National Laboratory Astana, Назарбаев университеті, Қабанбай батыр д., 53, Астана, Қазақстан²Аккумуляторлар институты, Назарбаев университеті, Қабанбай батыр д., 53, Астана, Қазақстан**АҢДАТПА**

Сұйық электролиттерден қатты электролиттерге көшу литий-күкірт (Li-S) аккумуляторларының қауіпсіздігін және энергия тиімділігін арттырудың негізгі стратегияларының бірі болып саналады. Алайда электрод-электролит шекарасындағы нашар жанасу және жоғары кедергі оларды коммерцияландырудағы басты кедергілердің бірі болып қала береді. Бұл жұмыста *in situ* қаптау әдісі ұсынылады: фотосәулемен торланған политетрагидрофуран негізіндегі қатты полимерлі электролит (aPTHF-SPE) C@S катодының бетінде тікелей УК-сәулелендіру әдісі арқылы отырғызылады. SEM және EDS талдаулары қалыңдығы 35–46 мкм біртекті полимер қабатының түзілуін растады, ол тығыз шекаралық жанасуды қамтамасыз етеді және қуыстардың пайда болуын азайтады. Электрохимиялық сынақтар *in situ* қапталған катодтардың бастапқы разряд сыйымдылығы шамамен 250 мА·сағ·г⁻¹ екенін көрсетті, ал келесі циклдер иондардың тасымалдануының күшеюі мен электродтың активтенуінің арқасында тұрақтырақ болды. Ұзақ циклдеуде сыйымдылықтың біртіндеп төмендеуіне қарамастан, ұсынылған стратегия қапталмаған катодтармен салыстырғанда электрод-электролит шекарасының сапасын айтарлықтай жақсартады. Алынған нәтижелер *in situ* полимерлі электролит қаптау әдісінің қатты күйлі Li-S аккумуляторларындағы электролит-электрод қабаттарының жанасу мәселелерін шешудегі болашағы зор әрі ауқымды әдіс екенін көрсетеді, бұл қауіпсіз әрі тиімді энергия сақтау жүйелеріне жол ашады.

Түйін сөздер: қатты полимерлі электролиттер, PTHF, *in situ* қаптау, қима талдауы, литий-күкірт аккумуляторлары.