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DOL-ENHANCED ELECTROLYTES AS A ROUTE TO STABLE ANODES IN Li—V_2O_5 SYSTEMS

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Abstract. The development of stable anodes based on metallic lithium remains one of the key and most urgent challenges in creating lithium batteries with high specific energy and long cycle life. Increasing requirements for safety, efficiency, and durability make it important to study the interfacial processes that determine lithium-anode stability under various operating conditions and external influences. In this regard, the aim of the work is a comprehensive evaluation of the influence of electrolyte composition on the interfacial stability and electrochemical characteristics of the lithium anode in the Li—V_2O_5 system, as well as the identification of the most practical electrolyte formulation. The study analyzed four electrolytes based on a mixture of propylene carbonate and 1,2-dimethoxyethane (3:7): 1 M lithium difluoro(oxalato)borate (LiDFOB) and 1 M lithium bis(trifluoromethanesulfonyl)imide (LiTFSI), each tested with and without the addition of 15% 1,3-dioxolane (DOL). The applied methods included galvanostatic cycling at different current densities, recording of discharge curves, and impedance

spectroscopy for evaluating interfacial resistance and SEI-layer dynamics. Experimental results showed that the introduction of DOL significantly improves electrode processes by reducing interfacial resistance, increasing Coulombic efficiency, and ensuring more stable anode behavior. The best performance was demonstrated by the LiTFSI + 15% DOL electrolyte, which retained approximately 75% of its initial capacity near 300 cycles. The practical significance of the study lies in the fact that the identified patterns enable targeted optimization of electrolyte systems to create more reliable, energy-dense, long-lasting lithium batteries of the next generation.

Keywords: lithium anode; V_2O_5; electrolyte; LiDFOB; LiTFSI; 1,3-dioxolane; Coulombic efficiency; impedance spectroscopy

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Li–V_2O_5 ЖҮЙЕСІНДЕГІ ТҰРАҚТЫ АНОДТАРҒА ҚОЛ ЖЕТКІЗУГЕ АРНАЛҒАН DOL-МЕН МОДИФИКАЦИЯЛАНҒАН ЭЛЕКТРОЛИТТЕР

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Аннотация. Металл литий негізіндегі тұрақты анодтарды жасау жоғары меншікті энергиясы және ұзақ қызмет ету мерзімі бар литий-ионды аккумуляторларды әзірлеудегі аса өзекті және маңызды ғылыми міндеттердің бірі болып табылады. Қауіпсіздікке, тиімділікке және ұзақмерзімді жұмысқа қойылатын талаптардың артуылитийанодыныңәртүрліжұмысрежимдеріменсыртқыәсерлержағдайындағы тұрақтылығын анықтайтын межфазалық үдерістерді терең зерттеуді қажет етеді. Осыған байланысты бұл жұмыстың мақсаты – Li–V_2O_5 жүйесіндегі литий анодының межфазалық тұрақтылығына және электрохимиялық сипаттамаларына электролит құрамының әсерін кешенді түрде бағалау, сондай-ақ ең перспективалы және практикалық тұрғыдан маңызды электролит құрамын айқындау. Зерттеуде

пропиленкарбонат пен 1,2-диметоксиэтан қоспасы (3:7) негізінде дайындалған төрт электролит қарастырылды: 1 М литий дифтор(оксалато)борат (LiDFOB) және 1 М литий бис(трифторметансульфонил)имид (LiTFSI), олардың әрқайсысы 15% 1,3-диоксолан (DOL) қосылған және қосылмаған нұсқаларда зерттелді. Қолданылған әдістерге әртүрлі ток тығыздықтарында гальваностатикалық циклдеу, разрядтық қисықтарды тіркеу және межфазалық кедергіні, сондай-ақ SEI қабатының қалыптасу динамикасын талдау үшін импеданстық спектроскопия кірді. Эксперименттік нәтижелер функционалдық қоспа ДОЛ енгізу электродтық процестерді айтарлықтай жақсартатынын, фазааралық кедергіні төмендететінін, кулондық тиімділікті арттыратынын және анодтың тұрақты жұмысын қамтамасыз ететінін көрсетті. Ең жоғары нәтижелерді 15% ДОЛ қосылған LiTFSI электролиті көрсетіп, 300 циклдан кейін бастапқы сыйымдылықтың шамамен 75%-ын сақтады. Зерттеудің практикалық маңызы – алынған заңдылықтар литий-ион аккумуляторларының жаңа буыны үшін неғұрлым сенімді, энергиясы жоғары және ұзақмерзімді электролиттік жүйелерді мақсатты түрде оңтайландыруға мүмкіндік береді.

Түйін сөздер: литий аноды; V_2O_5; электролит; LiDFOB; LiTFSI; 1,3-диоксолан; кулондық тиімділік; импеданстық спектроскопия

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DOL – МОДИФИЦИРОВАННЫЕ ЭЛЕКТРОЛИТЫ КАК ПУТЬ К СТАБИЛЬНЫМ АНОДАМ В СИСТЕМАХ Li–V_2O_5

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Аннотация. Разработка стабильных анодов на основе металлического лития остаётся одной из ключевых и актуальных задач при создании литиевых

аккумуляторов с высокой удельной энергией и длительным сроком службы. Повышенные требования к безопасности, эффективности и долговечности делают особенно важным исследование межфазных процессов, определяющих устойчивость литиевого анода при различных режимах работы и внешних воздействиях. *Целью* данной работы является комплексная оценка влияния состава электролита на межфазную стабильность и электрохимические характеристики литиевого анода в системе Li–V_2O_5, а также выявление наиболее перспективного и практически значимого состава электролита. В исследовании анализировались четыре электролита на основе смеси пропиленкарбоната и 1,2-диметоксиэтана (3:7): 1 М литий дифтор(оксалато)бората (LiDFOB) и 1 М литий бис(трифторметансульфонил)имида (LiTFSI), каждый — в варианте с добавлением 15% 1,3-диоксолана (DOL) и без него. *Методы* включали гальваностатическое циклование при разных плотностях тока, анализ разрядных кривых и импедансную спектроскопию для оценки межфазного сопротивления и динамики формирования SEI-слоя. *Экспериментальные результаты* показали, что введение функциональной добавки DOL значительно улучшает электродные процессы: уменьшает межфазное сопротивление, повышает кулоновскую эффективность и обеспечивает более стабильное поведение литиевого анода. Наилучшие характеристики продемонстрировал электролит LiTFSI + 15% DOL, сохранивший около 75% первоначальной ёмкости к 300-му циклу. *Практическая значимость* исследования заключается в том, что выявленные закономерности позволяют целенаправленно оптимизировать электролитные системы для разработки более надёжных, энергоёмких и долговечных литиевых аккумуляторов нового поколения.

Ключевые слова: литиевый анод; V_2O_5; электролит; LiDFOB; LiTFSI; 1,3-диоксолан; кулоновская эффективность; импедансная спектроскопия

Introduction. Lithium-ion batteries (LIBs) have transformed the landscape of energy storage, underpinning the rapid expansion of portable electronics, electric vehicles, and grid-scale storage systems due to their high energy density and extended cycle life (Tarascon & Armand, 2001, Goodenough & Park, 2013, Zhang et al., 2018). Yet, the growing demand for batteries with even higher energy densities and enhanced safety characteristics has revealed the limitations of conventional graphite anodes. In this context, lithium metal has emerged as a promising alternative anode material, owing to its exceptionally high theoretical specific capacity (3860 $mAh \cdot g^{-1}$) and the lowest redox potential among known electrode materials (–3.04 V vs. SHE) (Xu, 2014, Lin et al., 2017, Bruce et al., 2012).

Despite its advantages, the practical application of lithium metal anodes (LMAs) is hampered by several critical issues, including the formation of lithium dendrites, continuous parasitic reactions with electrolyte components, and the development of an unstable and resistive solid electrolyte interphase (SEI) (Zhang, 2013, Liu et al., 2019, Chen et al., 2021a). These phenomena contribute to rapid capacity fading, reduced Coulombic efficiency, and severe safety hazards. Therefore, stabilizing the lithium–

electrolyte interface is a key prerequisite for the successful deployment of lithium metal batteries in commercial applications (Wang et al., 2022).

Recent progress has shown that ether-based electrolytes, particularly those comprising dimethoxyethane (DME) and 1,3-dioxolane (DOL), exhibit improved compatibility with lithium metal due to their low viscosity and ability to form flexible and uniform SEI layers (Hong et al., 2025, Ren et al., 2020, Chen et al., 2021b). In parallel, the use of advanced lithium salts such as lithium difluoro (oxalato)borate (LiDFOB) has proven effective in constructing SEI layers rich in inorganic components like LiF, which reduce interfacial resistance and improve passivation stability (Wang et al., 2020, Li et al., 2019). Lithium bis(trifluoromethanesulfonyl)imide (LiTFSI), known for its superior ionic conductivity and electrochemical stability, is another widely explored salt, although it often requires additives or co-solvents to mitigate corrosion and side reactions (Li et al., 2024, Song et al., 2022).

The design of mixed-solvent systems combining high-permittivity components such as propylene carbonate (PC) with low-viscosity ethers offers a rational approach to balance ion transport, SEI formation, and chemical stability. Moreover, the choice of cathode material plays a crucial role in defining electrolyte requirements. Vanadium pentoxide (V_2O_5) stands out for its high theoretical capacity and layered structure enabling multivalent redox reactions, but it is also highly sensitive to electrolyte-induced degradation, especially in the presence of aggressive solvents or decomposition products (Hu et al., 2023, Kim et al., 2025).

To address these challenges, this study focuses on evaluating the electrochemical performance of lithium metal electrodes in Li–V_2O_5 systems using ether-based electrolytes with and without DOL co-solvent and two different lithium salts (LiDFOB and LiTFSI). The results provide new insights into the role of electrolyte formulation in stabilizing lithium metal interfaces and enabling long-term cycling in high-energy battery systems.

Materials and methods. All chemicals and materials were used as received without further purification. Lithium difluoro(oxalato)borate (LiDFOB, >99.5%, Solvionic) and lithium bis(trifluoromethanesulfonyl)imide (LiTFSI, >99.9%, Sigma-Aldrich) were used as lithium salts. The solvents propylene carbonate (PC, 99.7%, Sigma-Aldrich), 1,2-dimethoxyethane (DME, 99.5%, Sigma-Aldrich), and 1,3-dioxolane (DOL, 99.8%, TCI) were dried over 3 Å molecular sieves and handled under an inert atmosphere.

Vanadium pentoxide (V_2O_5, 99.2%, Alfa Aesar) was used as the active cathode material. Conductive additives included multi-walled carbon nanotubes (MWCNTs, >95%, CheapTubes Inc.) and Super C45 carbon black. The binder was polyvinylidene fluoride (PVDF, Solef 5130), dissolved in N-methyl-2-pyrrolidone (NMP, 99.5%, Merck). Aluminum foil (20 μm thick, MTI Corp.) served as the current collector, and lithium metal foil (150 μm thick, Ø15.6 mm, China Energy Lithium Co.) was used as the anode.

Cathode Preparation

The cathode slurry was composed of V_2O_5 (91 wt%), MWCNTs (1 wt%), Super C45 carbon black (3 wt%), and PVDF (5 wt%) dispersed in NMP. The mixture was magnetically stirred at room temperature for 12 hours to obtain a homogeneous paste.

The slurry was cast onto aluminum foil using a Doctor Blade Coater (MTI AFA-II-V), with the blade height set to 200 μm to ensure uniform film thickness. The coated electrodes were dried at 100 °C under vacuum for 12 hours. After drying, the electrodes were calendered using a hot rolled to a final thickness of 100–120 μm and punched into 14 mm diameter discs. Prior to cell assembly, the discs were additionally dried under vacuum at 110 °C for 6 hours to remove residual moisture.

Electrolyte Preparation

Four electrolyte formulations were prepared based on a PC: DME solvent mixture (3:7 by volume) with 1 $mol \cdot L^{-1}$ concentration of either LiDFOB or LiTFSI. Two of the formulations included 15 vol% of DOL as a co-solvent:

1. 1 M LiDFOB in PC: DME (3:7);
2. 1 M LiDFOB in PC: DME (3:7) + 15% DOL;
3. 1 M LiTFSI in PC: DME (3:7);
4. 1 M LiTFSI in PC: DME (3:7) + 15% DOL.

Each solution was stirred for 12 hours, filtered through a 0.22 μm PTFE syringe filter, and stored in sealed glass vials under argon.

Cell Assembly

CR2032-type coin cells were assembled in an argon-filled glovebox (SPECS GB02M, Russia) equipped with oxygen and moisture sensors. The glovebox-maintained oxygen levels below 1.0 ppm and moisture content below 0.1 ppm, which was essential for handling lithium metal and ether-based electrolytes. Celgard 2325 microporous polypropylene membranes were used as separators. Approximately 100 μL of electrolyte was added to each cell. Cells were sealed using a hydraulic crimper and allowed to rest for 12 hours prior to testing to ensure proper electrolyte wetting. For each electrolyte formulation, 2–3 cells were assembled to verify reproducibility.

Electrochemical Testing

Galvanostatic cycling was performed using a BioLogic VMP-300 multichannel potentiostat in the voltage range of 2.2–3.8 V. Rate capability tests were carried out at C/2, C, 2C, and 5C, followed by a return to C/2 to assess capacity recovery. Long-term cycling was conducted at C/2 for 300 cycles. Coulombic efficiency was calculated as the ratio of discharge to charge capacity in each cycle.

Electrochemical Impedance Spectroscopy (EIS)

EIS measurements were performed before and after the first three formation cycles. The frequency range was from 100 kHz to 10 mHz with an amplitude of 10 mV. All impedance measurements were conducted at room temperature (25 ± 1 °C). Data were analyzed using equivalent circuit models in EC-Lab software.

Results and discussion. The primary objective of this study was to investigate the impact of electrolyte composition on the stability of the lithium metal electrode in Li–V_2O_5 systems under varying discharge rates and extended cycling conditions. Four electrolyte systems were evaluated:

1. 1 M LiDFOB in PC: DME (3:7);
2. 1 M LiDFOB in PC: DME (3:7) + 15% DOL;
3. 1 M LiTFSI in PC: DME (3:7);
4. 1 M LiTFSI in PC: DME (3:7) + 15% DOL.

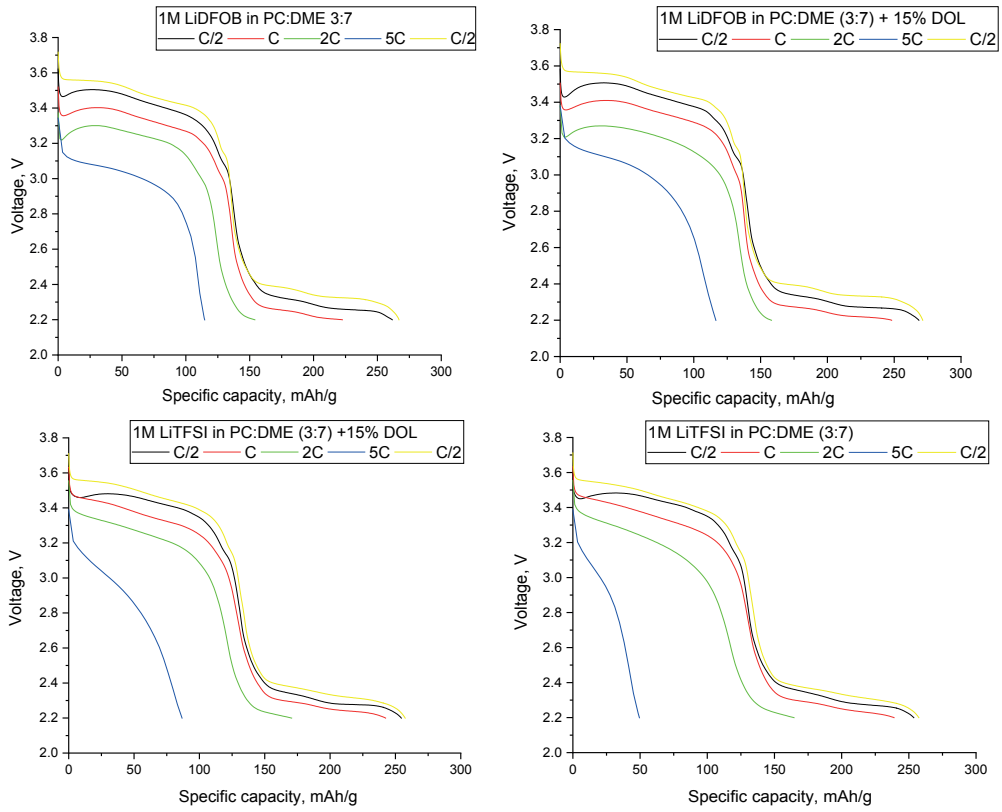

Figure 1 - Discharge voltage profiles of Li—V_2O_5 cells with different electrolytes at varying discharge rates: (a) 1 M LiDFOB in PC:DME (3:7); (b) 1 M LiDFOB in PC:DME (3:7) + 15% DOL; (c) 1 M LiTFSI in PC:DME (3:7) + 15% DOL; (d) 1 M LiTFSI in PC:DME (3:7)

Figure 1 presents the discharge voltage profiles at current densities of C/2, 1C, 2C, and 5C for the different electrolytes. While all systems demonstrated similar specific capacities at low discharge rates (C/2), significant differences emerged under higher current loads. The LiTFSI-based electrolyte without DOL exhibited the most rapid performance degradation with increasing current, showing pronounced polarization and capacity drop, especially at 2C and 5C. In contrast, electrolytes containing DOL-particularly the LiDFOB + DOL formulation-retained higher capacities and flatter voltage plateaus, indicating improved lithium-ion conductivity and lower interfacial resistance at elevated rates.

These results suggest that DOL plays a critical role in facilitating lithium-ion transport and suppressing interfacial polarization at high rates, likely due to its low viscosity and ability to form an elastic, ion-conductive SEI layer. The superior performance of the LiDFOB + DOL electrolyte can be attributed to the synergistic effects of the borate salt, which promotes the formation of a stable SEI enriched with LiF and boron species, and DOL, which enhances the mechanical integrity of the interphase. Together, these factors enable more uniform lithium deposition and reduce charge-transfer resistance, thereby improving voltage stability under dynamic conditions.

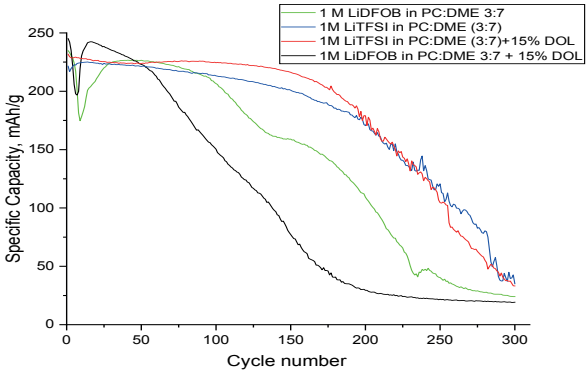

Figure 2 - Cycling performance of Li—V_2O_5 cells with different electrolyte compositions: variation of specific discharge capacity versus cycle number

Figure 2 displays the long-term cycling performance up to 300 cycles. The results clearly show that DOL-containing electrolytes exhibited superior capacity retention. The LiTFSI + DOL system retained ~85% of its initial capacity after 300 cycles, while LiDFOB + DOL retained ~75%. In contrast, systems without DOL showed significant capacity degradation: LiDFOB retained less than 50%, and LiTFSI about 60%. Figure 2 presents the long-term cycling performance of Li– V_2O_5 cells with different electrolyte compositions over 300 cycles. Among the four tested systems, the 1 M LiTFSI in PC: DME (3:7) with 15% DOL exhibited the most stable cycling behavior, retaining approximately 75% of its initial specific capacity after 300 cycles. This result indicates that the combination of LiTFSI with DOL effectively stabilizes the lithium metal anode, likely due to the formation of a uniform, elastic, and ion-conductive SEI layer supported by the synergistic effects of the high-ionic-conductivity TFSI anion and DOL-derived polymeric interface.

Surprisingly, the electrolyte with 1 M LiDFOB + 15% DOL, which had demonstrated excellent rate capability in Figure 1, showed the most rapid capacity fading during extended cycling. The capacity dropped sharply after ~50 cycles and approached zero by the 300th cycle. This suggests that while the initial SEI formed by LiDFOB and DOL may be effective, it lacks long-term mechanical or chemical stability**,** potentially leading to SEI breakdown, increased resistance, or parasitic reactions over time.

The electrolyte 1 M LiDFOB without DOL maintained moderate cycling performance, retaining about 60% of its initial capacity, suggesting that LiDFOB alone forms a somewhat stable SEI, albeit less effective under extended stress. The electrolyte 1 M LiTFSI without DOL, showed around 50% capacity retention**,** highlighting that while TFSI provides good ionic conductivity, the absence of a co-solvent like DOL results in unstable interphase growth and gradual degradation.

Overall, these results emphasize that the combination of TFSI□ with DOL is more favorable for long-term cycling performance than DFOB□ with DOL, despite the latter showing better initial rate capability. This finding underlines the importance of compatibility between the lithium salt and co-solvent in designing electrolytes for high-energy lithium metal batteries.

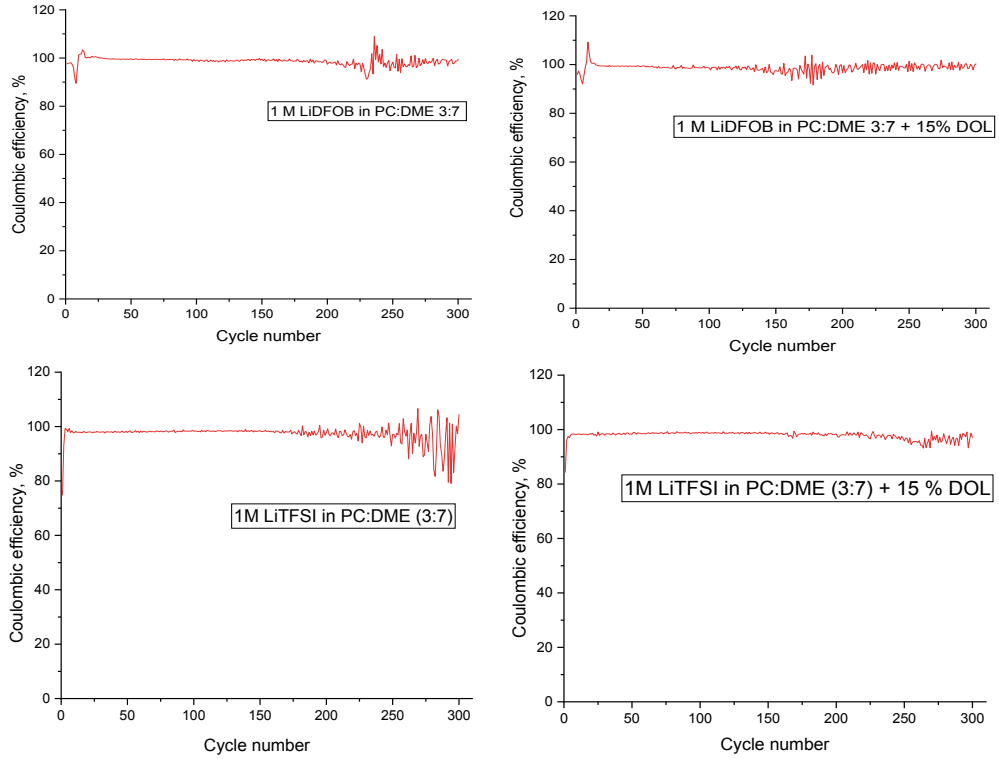

Figure 3 - Coulombic efficiency of Li—V_2O_5 cells over cycling for different electrolyte compositions: (a) 1 M LiDFOB in PC: DME (3:7); (b) 1 M LiDFOB in PC: DME (3:7) + 15% DOL; (c) 1 M LiTFSI in PC: DME (3:7) + 15% DOL; (d) 1 M LiTFSI in PC: DME (3:7)

Figure 3 presents the evolution of Coulombic efficiency (CE) in Li–V_2O_5 cells with different electrolytes over 300 cycles. All systems achieved CE values close to or above 98%, but important differences in stability and fluctuation patterns were observed.

Electrolytes containing DOL initially reached higher CE values (approaching 99—100%**)** within the first few cycles. However, over prolonged cycling, both DOL-containing systems exhibited increased fluctuations in CE, especially after 200 cycles. This behavior may indicate evolving interfacial instability or progressive degradation of the SEI formed in the presence of DOL, possibly due to mechanical stress or electrolyte depletion.

In contrast, DOL-free electrolytes demonstrated lower absolute CE values, particularly in the early cycles, but maintained more stable and consistent CE behavior throughout the entire test. This suggests that although the SEI formed without DOL may be less protective or conductive, it remains chemically more uniform and structurally stable over time, minimizing cycle-to-cycle variability.

Thus, the use of DOL improves initial CE and SEI formation, but its long-term benefits may be limited by mechanical or chemical degradation of the polymer-rich interfacial layer. The lower fluctuation in CE observed in DOL-free systems highlights the trade-off between initial interfacial activation and long-term electrochemical stability**.**

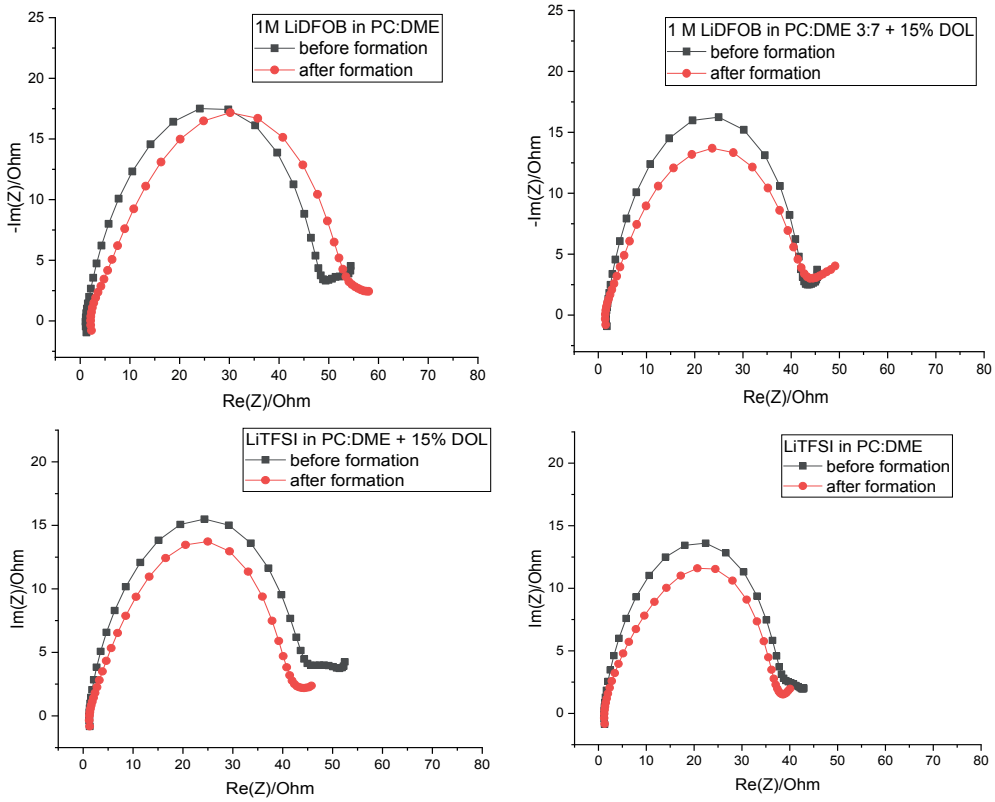

Figure 4 - Nyquist plots of Li–V_2O_5 cells before and after initial formation cycles for different electrolyte formulations: (a) 1 M LiDFOB in PC: DME (3:7); (b) 1 M LiDFOB in PC: DME (3:7) + 15% DOL; (c) 1 M LiTFSI in PC: DME (3:7) + 15% DOL; (d) 1 M LiTFSI in PC: DME (3:7)

Figure 4 presents the results of electrochemical impedance spectroscopy (EIS) for Li—V_2O_5 cells with different electrolyte compositions, measured before and after initial formation cycles. In all cases, the Nyquist plots exhibit characteristic semicircles in the high-to-mid frequency range, which correspond to charge-transfer resistance (R_ct) and interfacial impedance associated with the formation and properties of the solid electrolyte interphase (SEI) on the lithium metal anode. After formation, all systems demonstrate a decrease in semicircle diameter, indicating improved electrode—electrolyte contact due to SEI development. However, the extent and stability of this improvement differ markedly depending on the electrolyte formulation.

The electrolyte based on 1 M LiTFSI in PC: DME (3:7) + 15% DOL (Figure 4c) exhibited the lowest interfacial resistance and minimal increase in R_ct after formation, suggesting the formation of a highly conductive and mechanically robust SEI. These findings are consistent with earlier results shown in Figure 2 (superior capacity retention) and Figure 3 (high and stable Coulombic efficiency), confirming that the combination of LiTFSI and DOL leads to an interfacial structure favorable for long-term cycling and fast lithium-ion transport.

In contrast, the LiDFOB + DOL system (Figure 4b), while initially promising

in high-rate discharge tests (Figure 1), showed a pronounced increase in impedance after formation. This may be attributed to the gradual accumulation of decomposition products or instability of the polymer-rich SEI formed from DOL, leading to higher resistance and unstable performance in prolonged cycling (Figure 2) and fluctuating CE (Figure 3). Additionally, the LiTFSI-only electrolyte (Figure 4d) maintained moderately low impedance, further supporting the known electrochemical stability of the $TFSI^-$ anion. This agrees with prior, which emphasize the benefits of LiTFSI in high-performance lithium battery systems due to its weak coordination strength and favorable SEI chemistry.

In summary, this EIS investigation confirms that LiTFSI-based electrolytes, particularly in combination with DOL, yield more stable, conductive, and low-resistance interphases, which directly contribute to enhanced electrochemical performance and durability of Li— V_2O_5 cells.

Conclusion. The growing demand for high-energy and long-life lithium-metal batteries requires the development of advanced electrolyte systems capable of stabilizing the lithium anode and ensuring compatibility with high-voltage cathode materials. This study addresses this challenge by systematically evaluating the impact of different electrolyte compositions - based on LiDFOB and LiTFSI salts in PC: DME solvent mixtures, with and without the addition of 1,3-dioxolane (DOL) - on the electrochemical performance of Li— V_2O_5 cells.

The research confirmed that electrolyte composition plays a decisive role in governing the cycling stability, Coulombic efficiency, and interfacial resistance of lithium-metal batteries. Electrolytes containing DOL showed superior rate performance, particularly LiDFOB + DOL**,** due to improved ionic conductivity and reduced polarization. Notably, the LiDFOB + DOL electrolyte demonstrated excellent performance at high current densities, maintaining stable operation under increased rate conditions. However, long-term cycling tests revealed that LiTFSI + DOL offers the best combination of capacity retention (~75% after 300 cycles) and high Coulombic efficiency, which remained consistently close to 99%. Electrochemical impedance spectroscopy further demonstrated that this formulation yields the lowest charge-transfer resistance after formation, highlighting the formation of a stable and conductive SEI.

The use of LiTFSI, a salt known for its high ionic mobility and chemical stability, together with DOL, which enables the formation of an elastic polymer-rich SEI, proved to be particularly effective. The synergy between these two components led to improved interfacial stability, suppressed parasitic reactions, and reduced degradation over time.

By contrast, while the LiDFOB + DOL system provided excellent initial performance, its stability under extended cycling was limited. This underlines the importance of not only additive selection but also salt—solvent compatibility in tailoring electrolyte formulations for specific battery chemistries.

In conclusion, the 1 M LiTFSI in PC: DME (3:7) + 15% DOL electrolyte demonstrated the most promising overall performance and can be considered a viable candidate for further scaling and integration into advanced lithium-metal battery systems. Future work may focus on optimizing DOL concentration, incorporating additional additives, or extending the approach to other cathode materials to further enhance performance.

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