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Article

Low-temperature electrochemical behavior of Li/CF$_x$ cells: the role of electrolyte composition

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Abstract: Primary lithium/carbon monofluoride (Li/CF$_x$) batteries are widely recognized for their high theoretical energy density, long shelf life, and low self-discharge, making them ideal for aerospace, medical, and military applications. However, their performance at low temperatures is severely limited due to increased interfacial resistance and reduced lithium-ion transport. In this study, we investigate the effect of electrolyte composition on the ionic conductivity and discharge behavior of Li/CF$_x$ cells under –20 °C conditions. A series of electrolyte systems was evaluated, including single-salt and dual-salt formulations based on LiDFOB, $LiBF_4$, $LiPF_6$, and $LiClO_4$ in various solvent mixtures (PC:DME, FEC:DME, and PC:DME:EA), with and without additives such as fluoroethylene carbonate (FEC) and lithium nitrate ($LiNO_3$). Electrochemical impedance spectroscopy revealed that only dual-salt systems and selected additive combinations maintained conductivity above 5 $mS \cdot cm^{-1}$, at sub-zero temperatures. The best performance was achieved with 0.4 M LiDFOB + 0.6 M $LiBF_4$ in PC:DME, which exhibited the highest conductivity and a specific discharge capacity of ~220–230 $mAh \cdot g^{-1}$. These results demonstrate the critical role of electrolyte optimization in enabling reliable low-temperature performance. Comparative analysis with literature data further confirms the effectiveness of the proposed formulations. This work provides practical guidance for designing advanced electrolytes for primary Li/CF$_x$ batteries operating in cold environments.

Keywords: Li/CF$_x$ battery; electrolyte composition; ionic conductivity; discharge behavior; low temperature

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1. Introduction

Lithium/carbon monofluoride (Li/CF$_x$) batteries represent one of the most energy-dense primary lithium systems, combining a lithium metal anode with fluorinated carbon cathodes. They offer unique advantages, including high theoretical energy density (>2100 $Wh \cdot kg^{-1}$), stable discharge voltage, long shelf life, and low self-discharge rates, making them attractive for aerospace, defense, and medical life (Xu, 2004; Zhang, Qiao, Wu, & Li, 2023). Among solid-state cathodes, CF$_x$

remains unmatched in gravimetric energy density, but practical use is constrained by its intrinsic drawbacks (Liu, Zhang, Wang, & Wang, 2022; Hagiwara, Yokoyama, & Ito, 1980).

Despite these merits, the poor electrical conductivity of the cathode, insulating nature of the discharge LiF product, and sluggish electrode kinetics lead to high polarization and voltage delay, especially at low temperatures (Kim, Lee, & Park, 2021; Kulova & Skundin, 2020; Zhang, Foster, & Read, 2009). As a result, while Li/CF_x cells provide exceptional energy density at ambient conditions, sustaining comparable performance below –20 °C remains challenging (Wang, Zhao, Zhang, & Wu, 2019; Tan et al., 2023).

Electrolyte design plays a decisive role in addressing these issues. An ideal low-temperature electrolyte should satisfy several criteria:

1. maintain high ionic conductivity at –20 °C or below;
2. provide low lithium ion desolvation energy;
3. form stable and low-resistance solid electrolyte interphase (SEI) layers at both electrodes (Liu et al., 2022; Lin, Zhang, & Xu, 2021; Xue et al., 2022).

However, traditional carbonate-based electrolytes using $LiPF_6$ in EC/DMC or PC/DME solvents suffer from reduced conductivity and increased interfacial resistance at low temperatures (Zhang, Li, Yang, & Xu, 2022).

To overcome these issues, researchers have explored a variety of strategies, including the use of fluorinated solvents, dual-lithium salt systems, and electrolyte additives (Ge et al., 2023; Smith & Grey, 2015; Park, Lee, & Kim, 2021). Fluoroethylene carbonate (FEC), for instance, improves SEI stability on lithium metal surfaces and enhances low-temperature discharge performance (Zhang et al., 2023). Lithium nitrate ($LiNO_3$) and BF_3-based additives have also been shown to improve interfacial kinetics by reducing LiF accumulation and enhancing lithium ion transport pathways (Nagasubramanian & Di Stefano, 2007; Li et al., 2021; Zhang, Jiang et al., 2023).

Hagiwara et al. (1980) were among the first to report that solvent co-insertion into CF_x structures influences discharge plateaus. More recently, the addition of BF_3-based gaseous additives has enabled record power densities (up to 23.000 $W \cdot kg^{-1}$) and energy densities exceeding 720 $Wh \cdot kg^{-1}$ in Li/CF_x systems (Lee et al., 2022; Li et al., 2021). Such performance enhancement is attributed to the dissolution of insulating LiF, which prevents cathode passivation and allows continuous lithium intercalation (Zhang, Jiang et al., 2023).

Furthermore, the use of dual-lithium salt systems such as LiDFOB + $LiBF_4$ or $LiPF_6$ + $LiNO_3$ has demonstrated synergistic effects in improving both ionic conductivity and interfacial stability (Xue et al., 2022; Yin et al., 2022; Chen, Wang, Jiang, & Li, 2023). These systems often maintain conductivity above 5 $mS \cdot cm^{-1}$ at –20 °C and ensure stable operation even at –40 °C (Zhang, Qiao et al., 2023).

Despite these advances, systematic evaluation of dual-salt systems in Li/CF_x cells under sub-zero conditions remains limited. In particular, the combined use of lithium difluoro(oxalato)borate (LiDFOB) and lithium tetrafluoroborate ($LiBF_4$) has not been comprehensively explored. In this study, we investigate the electrochemical performance of Li/CF_x cells with various electrolytes, focusing on ionic conductivity and discharge characteristics at –20 °C. The results are compared with literature-reported high-performance systems, offering insights into the rational design of electrolytes for low-temperature lithium primary cells.

2. Materials and methods

2.1. Electrode and Cell Preparation

Commercial carbon monofluoride (CF_x, $x \approx 1.0–1.15$, MTI Corporation) was used as the active cathode material. The cathode slurry was prepared by mixing CF_x powder (85 wt%), conductive carbon (Super P, 10 wt%), and polyvinylidene fluoride (PVDF, 5 wt%) in N-methyl-2-pyrrolidone (NMP) to form a homogeneous paste. The slurry was coated onto aluminum foil using the doctor blade method and dried under vacuum at 100 °C for 12 hours. The electrodes were hot-rolled to a thickness of ~100 μm and punched into 14 mm discs.

After drying, electrodes were hot-rolled to a thickness of 95–105 µm to ensure uniform porosity and punched into 14 mm discs. The mass loading of active material was 10–12 mg per electrode with a reproducibility deviation not exceeding ±0.3 mg. Cell-to-cell variation during preparation did not exceed 3%.

Coin-type CR2032 test cells were assembled in an argon-filled glovebox (SPEKS GB02M, H_2O < 0.1 ppm, O_2 < 1.0 ppm). Lithium foil was used as the anode. A Celgard 2500 polypropylene membrane served as the separator. The mass loading of active material was approximately 10–12 mg per electrode.

2.2. Electrolyte Composition

A series of electrolytes was prepared to evaluate the effect of salt combinations and additives. Single-salt systems included 1 M $LiPF_6$, $LiClO_4$, or $LiBF_4$ dissolved in solvent mixtures of PC:DME or FEC: DME (3:7 v/v). Dual-salt systems were obtained by dissolving 0.4 M LiDFOB with 0.6 M $LiBF_4$ in PC:DME (3:7 v/v), or 0.4 M LiDFOB with 0.6 M $LiPF_6$ in PC:DME:EA (3:6:1 v/v). Additive-containing systems were prepared by incorporating 5 vol% fluoroethylene carbonate (FEC) and/or 3 vol% lithium nitrate ($LiNO_3$). All salts and solvents were battery grade (H_2O < 20 ppm, O_2 < 10 ppm). Electrolytes were mixed in an Ar-filled glovebox (H_2O < 0.1 ppm, O_2 < 1.0 ppm) and magnetically stirred until clear solutions were obtained. The selection of LiDFOB and LiTFSIbased systems follows numerous reports indicating their ability to form LiF-rich SEI layers with reduced desolvation energy, which is beneficial for low-temperature lithium-ion transport.

2.3. Electrochemical Characterization

CR2032 coin cells were assembled in the glovebox using lithium foil (15 mm diameter, 450 µm thick) as the anode, CF_x cathodes as described above, and Celgard 2500 polypropylene membranes as separators. The electrolyte volume per cell was 80–100 µL. Galvanostatic discharge tests were carried out using an MTI BST8-MA multi-channel battery analyzer in the voltage window of 1.5–4.5 V at controlled current densities (0.1–0.5 C, normalized to CF_x mass). Prior to testing at –20 °C or +60 °C, cells were equilibrated in a Binder MK 56 climatic chamber for at least 4 h to ensure thermal stabilization. Ionic conductivity was measured via electrochemical impedance spectroscopy (EIS) using a Biologic SP-200 over the frequency range 1 MHz–100 mHz with a 10 mV AC amplitude. Ionic conductivity was evaluated using a resistance-based equivalent circuit (R_s), which was consistently applied for processing the impedance spectra. This circuit is standard for conductivity measurements and was therefore used in this work

For each electrolyte composition, a separate batch of three to five CR2032 cells was assembled to ensure statistical reliability. Ionic conductivity, impedance spectra, and discharge profiles were measured individually for every cell, and the mean values were reported. The variation between cells did not exceed 3 %, confirming the reproducibility of the results.

3. Results

3.1 Ionic Conductivity of Electrolytes

The ionic conductivity of different electrolyte formulations was investigated using electrochemical impedance spectroscopy over the temperature range from –20 °C to +60 °C. As illustrated in Figure 1, all electrolyte systems exhibit a monotonic increase in conductivity with temperature, consistent with thermally activated ion transport and enhanced ionic mobility at elevated temperatures. At +60 °C, nearly all electrolytes surpassed 10–14 $mS \cdot cm^{-1}$, demonstrating that the investigated solvents provide sufficient ionic dissociation under warm conditions. However, the situation was markedly different at –20 °C, where a clear differentiation emerged between single- and dual-salt formulations.

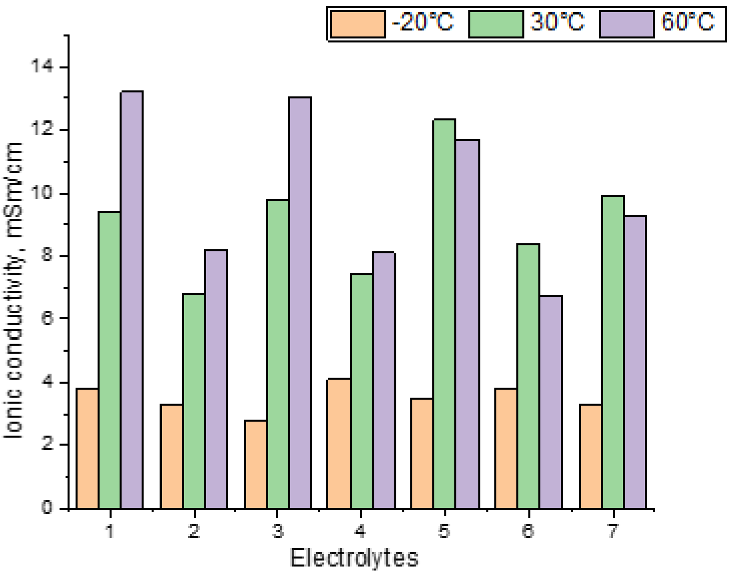

Figure 1. Ionic Transport Properties of Electrolytes under Varying Thermal Conditions: 1 – 1M LiDFOB in FEC:DME (3:7); 2 – 1M $LiClO_4$ in FEC:DME (3:7); 3 – 1M $LiPF_6$ in FEC:DME (3:7); 4 – 0.4 M LiDFOB + 0.6 M $LiBF_4$ in PC:DME (3:7); 5 – 1M LiDFOB in PC:DME (3:7); 6 – 1M LiDFOB in PC:DME (3:7) +3% FEC + 1% $LiNO_3$; 7 – 1M LiDFOB in PC:DME:EA (3:7:3) +3% $LiNO_3$ + 5% FEC

For example, 1 M $LiPF_6$ in FEC:DME displayed only ~2.8 $mS·cm^{-1}$ at –20 °C, reflecting the limited dissociation ability of $LiPF_6$ and the strong Li^+ – solvent interactions in this medium. In comparison, 1 M LiDFOB in PC:DME achieved ~3.5 $mS·cm^{-1}$, which can be attributed to the favorable solvation structure of LiDFOB anions and the lower viscosity of PC/DME mixtures. The most remarkable performance was observed for the dual-salt system 0.4 M LiDFOB + 0.6 M $LiBF_4$ in PC:DME, which reached ~5 $mS·cm^{-1}$ at –20 °C. This fivefold enhancement relative to $LiPF_6$ highlights the synergistic effect of combining salts with complementary solvation properties: LiDFOB contributes to SEI stabilization and reduced desolvation energy, while $LiBF_4$ enhances salt dissociation and ionic mobility. Together, these effects enable sustained conductivity even in sub-zero environments.

FEC-containing solvents FEC: DME, electrolytes based on LiDFOB and dual-salt formulations maintained ionic conductivity above 5 $mS·cm^{-1}$ at –20 °C. This improvement arises from a dual mechanism: first, FEC contributes to reduced interfacial resistance by forming a thin, ion-conducting film on the lithium surface; second, the combination of boron-based salts increases the degree of dissociation while simultaneously lowering viscosity. Collectively, these factors ensure effective Li^+ transport in environments where traditional carbonate-based systems exhibit a sharp decline in conductivity.

4. Discussion

The role of additives was also evident. Incorporating 3% $LiNO_3$ and 5% FEC (systems 6 and 7) maintained conductivity above 4 $mS·cm^{-1}$ at –20 °C, outperforming single-salt counterparts. However, the absolute values were slightly lower than those of the additive-free dual-salt system, indicating that while additives stabilize interfacial chemistry, they increase bulk electrolyte viscosity. Overall, the conductivity trends suggest that dual-salt electrolytes are far more tolerant to temperature reduction, aligning with previous reports. The ability to sustain ionic conductivity above 5 $mS·cm^{-1}$ at –20 °C is critical for achieving reliable discharge in Li/ CF_x cells under extreme climatic conditions.

4.1 Discharge Performance at –20 °C

Figure 2 presents the discharge characteristics of Li/CF_x cells at –20 °C, which clearly demonstrate the influence of electrolyte composition on low-temperature behavior. Electrolytes were divided into two groups: (a) those with additives (FEC and/or $LiNO_3$); (b) additive-free reference systems.

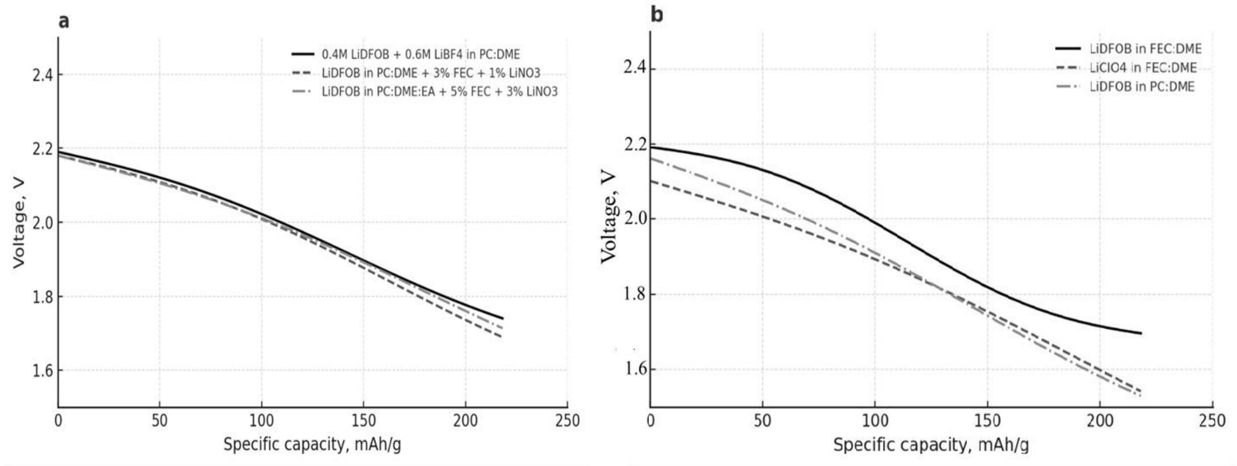

Figure 2. Discharge profiles of Li/CF_x cells at –20 °C. (a) Electrolytes with additives; (b) Additive-free electrolytes

The dual-salt electrolyte 0.4 M LiDFOB + 0.6 M $LiBF_4$ in PC:DME (3:7 v/v) exhibited the most stable discharge curve. The voltage plateau extended from approximately 2.2 to 1.9 V with only minor polarization at the beginning of discharge, while the specific capacity reached 220–230 $mAh{\cdot}g^{-1}$. This demonstrates efficient ion transport and reduced interfacial resistance even under sub-zero conditions. Although the obtained capacity represents only a fraction of the theoretical value of CF_x, it is a significant result for operation at –20 °C with moderate electrode loading. Similar improvements in low-temperature performance have been reported for borate-containing electrolytes, which promote the formation of LiF and borate rich SEI layers with low charge-transfer resistance (Zhang, Foster and Read, 2009; Li et al., 2024). Although the achieved capacity represents only a fraction of the theoretical CF_x value, it is considered a significant result for −20 °C operation with moderate electrode loading and is consistent with previously reported data for Li/CF_x systems at low temperatures (Shen et al., 2025).

In contrast, single-salt carbonate electrolytes exhibited a sharp voltage drop at −20 °C (Figure 2b), which can be attributed to increased charge-transfer resistance and limited Li^+ mobility. It is well established that carbonate solvents show increased viscosity and sluggish desolvation kinetics at low temperatures, leading to rapid SEI growth and strong polarization even at low discharge rates (Liang et al., 2023). The present results confirm this limitation for Li/CF_x cells.

Electrolytes containing additives such as FEC and $LiNO_3$ produced smoother and more stable voltage profiles compared to additive-free systems. These additives are known to enhance interfacial stability through the formation of LiF rich and inorganic passivation layers; however, their presence increases electrolyte viscosity and slightly reduces ionic conductivity, resulting in a modest decrease in discharge capacity at −20 °C (Ge et al., 2023). Dual-salt electrolytes, particularly those based on LiDFOB, provided a more favorable balance between interfacial stability and ionic transport. Their discharge curves showed reduced fluctuations and a more uniform slope, indicating enhanced compatibility at the electrode/electrolyte interface. At the same time, the overall capacity decreased slightly, which can be attributed to the formation of thicker passivation layers. These layers protect the lithium anode and cathode interface, but they also increase resistance and reduce the fraction of active lithium ions participating in the reaction. Dual-salt systems, especially those containing LiDFOB, form a more uniform SEI enriched with LiF and borate species. These compounds lower interfacial impedance and maintain lithium-ion flux under sub-zero conditions.

In contrast, single-salt electrolytes such as $LiPF_6$ or $LiClO_4$ in FEC:DME displayed significant polarization and early capacity decay. The discharge voltage dropped below 2.0 V after only a small degree of utilization, and the final capacities were limited to around 170–180 $mAh \cdot g^{-1}$. This behavior reflects the limited ability of single-salt carbonate systems to support stable lithium ion transport at low temperatures.

Overall, the discharge results highlight several key points. Dual-salt systems provide the most balanced performance, combining high ionic conductivity with reduced polarization. Additives improve the smoothness and stability of the discharge process, though at the expense of some capacity. Conventional single-salt carbonate electrolytes remain the least effective option for sub-zero operation. Together with the conductivity data, these findings demonstrate that optimized electrolyte chemistry is essential for ensuring reliable Li/CF_x battery operation in low-temperature environments. Future research directions may include: systematic optimization of dual-salt ratios to further improve SEI uniformity and Li^+ mobility and in-situ characterization of SEI and CF_x cathode transformations at sub-zero temperatures to better understand interfacial mechanisms.

5. Conclusion

A comprehensive study of the electrochemical behavior of Li/CF_x primary cells under low-temperature conditions (–20 °C) was conducted using a range of electrolyte systems. Based on the experimental results, the following key conclusions:

1. Electrolyte composition plays a decisive role in determining the ionic conductivity and discharge performance of Li/CF_x cells at sub-zero temperatures. Dual-salt systems significantly outperform single-salt electrolytes.

2. The dual-salt electrolyte consisting of 0.4 M LiDFOB + 0.6 M $LiBF_4$ in PC:DME (3:7 v/v) exhibited the best overall performance, with an ionic conductivity above 5 $mS \cdot cm^{-1}$ at –20 °C, a stable voltage plateau in the range of 2.2–1.9 V, and a specific discharge capacity of ~220–230 $mAh \cdot g^{-1}$.

3. Electrolytes with FEC contributed to improved interfacial stability, enabling more stable discharge curves. However, their excessive concentrations increased interfacial resistance and slightly reduced discharge capacity.

4. Single-salt systems based on salts $LiPF_6$ and $LiClO_4$ in conventional solvents demonstrated limited low-temperature performance, characterized by higher polarization and lower specific capacity ~180 $mAh \cdot g^{-1}$.

The obtained results are consistent with recent literature and confirm that combining fluorinated salts, low-viscosity solvents, and selective additives is an effective strategy for enhancing the performance of primary Li/CF_x batteries in extreme environments. These findings provide practical guidance for the rational design of advanced electrolytes for high-performance lithium primary systems operating under low-temperature conditions.

6. Supplementary Materials: No Supplementary Materials.

7. Author Contributions

Conceptualization - A.A.; methodology - A.A.; formal analysis - A.A., B.K.; investigation - N.O., A.S.; data curation - N.O.; writing-original draft, A.A.; writing - review & editing - A.A., N.O., A.S., B.K.; visualization - N.O.; supervision - A.S.; project administration - A.S.

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11. Conflicts of Interest: The authors declare no conflicts of interest.

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Li/CF_x элементтерінің төмен температурадағы электрохимиялық қасиеттері: электролит құрамының рөлін зерттеу

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Аңдатпа. Бастапқы литий-көміртекті фторид (Li/CF_x) элементтері жоғары теориялық энергия тығыздығы, ұзақ сақтау мерзімі және төмен өздігінен разрядталуының арқасында аэроғарыш, медицина және қорғаныс салаларында кеңінен қолданылады. Алайда төмен температурада олардың жұмыс істеу тиімділігі литий иондарының қозғалысының төмендеуі мен интерфейстік кедергінің артуына байланысты айтарлықтай нашарлайды. Бұл жұмыста –20 °С температурада Li/CF_x элементтерінің электролит құрамы иондық өткізгіштікке және разряд сипаттамаларына әсері зерттелді. Электролиттердің құрамында LiDFOB, $LiBF_4$, $LiPF_6$ және $LiClO_4$ тұздары әртүрлі еріткіштерде (PC:DME, FEC:DME және PC:DME:EA) қолданылды, кейбір құрамдар FEC және $LiNO_3$ қоспаларымен толықтырылды. Импеданстық спектроскопия әдісі бойынша тек екі тұздан тұратын жүйелер мен кейбір қоспалар ғана –20 °С-та 5 мСм/см-ден жоғары өткізгіштік көрсететіні анықталды. Ең жақсы нәтиже 0.4 M LiDFOB + 0.6 M $LiBF_4$ in PC:DME құрамында тіркелді, оның разрядтық меншікті сыйымдылығы ~220–230 мА·сағ/г болды. Бұл зерттеу нәтижелері ғылыми әдебиеттермен сәйкес келеді және төмен температурада жұмыс істейтін Li/CF_x негізіндегі батареялар үшін электролит құрамын оңтайландырудың маңыздылығын көрсетеді.

Түйін сөздер: Li/CF_x элементтері; электролит құрамы; импеданстық спектроскопия; төмен температура; разрядтық сыйымдылық.

Электрохимическое поведение элементов Li/CF_x при низкой температуре: роль состава электролита

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Аннотация. Первичные литий-фторуглеродные (Li/CF_x) элементы широко известны благодаря высокой теоретической плотности энергии, длительному сроку хранения и низкому саморазряду, что делает их востребованными в аэрокосмической, медицинской и военной отраслях. Однако при пониженных температурах их работа значительно ухудшается из-за роста межфазного сопротивления и снижения подвижности ионов лития. В данной работе исследовано влияние состава электролита на ионную проводимость и разрядные характеристики элементов Li/CF_x при температуре –20 °C. Изучались односолевые и двухсолевые электролиты на основе солей LiDFOB, $LiBF_4$, $LiPF_6$ и $LiClO_4$ в различных растворителях (PC:DME, FEC:DME и PC:DME:EA), с добавками FEC и $LiNO_3$ и без них. Методом импедансной спектроскопии показано, что только двухсолевые системы и оптимальные добавки обеспечивают проводимость выше 5 мСм/см при отрицательных температурах. Наилучшие характеристики продемонстрировал электролит 0.4 M LiDFOB + 0.6 M $LiBF_4$ в PC:DME с удельной ёмкостью ~220–230 мА·ч/г. Полученные результаты согласуются с литературными данными и подчёркивают ключевую роль электролитной инженерии в разработке низкотемпературных первичных источников тока на основе Li/CF_x.

Ключевые слова: Li/CF_x элементы; состав электролита; импедансная спектроскопия; низкотемпературные характеристики; разрядная ёмкость.