Automotive Science and Engineering, Vol. 14, No. 2, (2024), 4360-4368

Iran University of Science and Technology

Automotive Science and Engineering

Journal Homepage: ase.iust.ac.ir

ISSN: 2008-9899

Evaluating Electrolyte Solvent Effects on Low-Temperature Performance of Lithium-ion Batteries Using Electrochemical Impedance Spectroscopy

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ARTICLE INFO

Article history:

Received : 19 Jan 2024

Accepted: 30 Mar 2024

Published: 21 Apr 2024

Keywords:

Li-ion battery

Electrolyte

Solvent

Low-temperature performance

Impedance spectroscopy

A B S T R A C T

The global transition towards renewable energy necessitates efficient energy storage solutions to address the intermittency of renewable sources like solar and wind power. Lithium-ion batteries (LIBs), widely utilized in electric vehicles (EVs) for their high energy density and efficiency, have emerged as a leading technology, yet their performance at low temperatures remains a challenge. This study investigates the influence of electrolyte solvent composition on LIB performance under low-temperature conditions using electrochemical impedance spectroscopy (EIS). Three electrolytes were studied: a standard electrolyte (STDE) comprising 1 M $LiPF_6$ in ethylene carbonate (EC) and diethyl carbonate (DEC), a low-temperature electrolyte (LTE) consisting of 1 M $LiPF_6$ in EC, ethyl methyl carbonate (EMC), and ethyl acetate (EA), and a long-cycle-life electrolyte (LCLE) containing 1 M $LiPF_6$ in EC/EMC. The EIS results revealed significant differences in resistance values among the electrolytes at varying temperatures. Specifically, at 0 °C, the STDE exhibited a charge transfer resistance (R_{ct}) of 1055.3 Ω and a solid electrolyte interface resistance (R_{SEI}) of 803.4 Ω, whereas the LTE showed a substantially lower R_{ct} of 507.4 Ω and R_{SEI} of 64.2 Ω, indicating superior low-temperature performance. Similarly, at -20 °C, the R_{ct} values for STDE, LTE, and LCLE were 8878.6 Ω, 854.2 Ω, and 15622 Ω, respectively, with corresponding R_{SEI} values of 172.1 Ω, 92.4 Ω, and 2364 Ω. These findings underscore the importance of solvent composition in mitigating resistance increases at lower temperatures and highlight the superior performance of the LTE formulation in maintaining lower R_{ct} and R_{SEI} values. Notably, the addition of ethyl acetate (EA) in the LTE formulation contributed to enhanced low-temperature performance, likely by lowering the overall viscosity of the electrolyte mixture and improving ionic mobility. This study demonstrates the critical role of solvent composition, particularly EA, in optimizing LIB performance for cold climate applications.

1. Introduction

The global energy landscape is undergoing a transformative shift from fossil fuels to renewable energy sources, driven by the urgent need to combat climate change and ensure sustainable energy security [1-4]. Renewable energy sources like solar, wind, hydro, and geothermal are abundant, sustainable, and emit little to no greenhouse gases. However, these sources face inherent challenges due to their intermittent and variable nature. Solar and wind power generation, for instance, are dependent on weather conditions

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https://doi.org/10.22068/ase.2024.672

[DOI: 10.22068/ase.2024.672]

and time of day, leading to fluctuations in energy supply. This variability necessitates reliable and efficient energy storage solutions to ensure a stable and continuous power supply. Energy storage systems (ESS) are pivotal in balancing supply and demand, enhancing grid stability, and optimizing the utilization of renewable energy [5-8].

Batteries, particularly lithium-ion batteries (LIBs), have emerged as a leading technology in energy storage due to their high energy density, long cycle life, low self-discharge rates, and high efficiency [9-11]. LIBs have revolutionized portable electronics and electric vehicles (EVs) and are increasingly being integrated into grid storage systems [12-17]. Despite their advantages, LIBs face significant performance challenges at low temperatures. Cold environments, especially sub-zero temperatures, can drastically impair the performance of LIBs by reducing their capacity, power output, and overall efficiency. This limitation is critical for applications in cold climates and high-altitude regions, as well as for certain aerospace applications where reliable battery performance is essential regardless of the ambient temperature [18-22].

The properties of the electrolyte heavily influence the performance of LIBs in low-temperature conditions. The electrolyte in a LIB serves as a medium for ion transport between the cathode and anode during the charge and discharge processes. It typically consists of a lithium salt dissolved in a solvent or a mixture of solvents and may also include co-solvents and additives to enhance performance [23, 24]. Lithium salts are a crucial component of the electrolyte, providing the necessary lithium ions for conduction. Due to its good ionic conductivity and stable electrochemical performance, $LiPF_6$ salt finds widespread use, despite its susceptibility to moisture and potential for decomposition at high temperatures.

Lithium bis(trifluoromethane)sulfonimide (LiTFSI) salt is more expensive and can damage aluminum current collectors, but it is better at low temperatures and has better thermal and chemical stability than $LiPF_6$. It also conducts ions more efficiently [25, 26].

The choice of solvent significantly affects the electrolyte's properties, including its viscosity, dielectric constant, and electrochemical stability. Common solvents used in LIB electrolytes include ethylene carbonate (EC), dimethyl carbonate (DMC), and propylene carbonate (PC). Co-solvents are often added to the electrolyte to optimize performance by balancing the properties of the primary solvents. For instance, ethyl methyl carbonate (EMC) is often used alongside EC and DMC to improve low-temperature performance and reduce viscosity. Diethyl carbonate (DEC) helps to lower the freezing point of the electrolyte and reduce its viscosity [27].

Additives play a crucial role in enhancing the performance and safety of LIB electrolytes. They can improve the formation and stability of the solid electrolyte interphase (SEI) layer, enhance ionic conductivity, and increase the overall electrochemical stability of the electrolyte. For example, vinylene carbonate (VC) is known for improving the stability of the SEI layer and enhancing battery life [28]. Fluoroethylene carbonate (FEC) enhances low-temperature performance and the stability of the SEI layer, especially for silicon-based anodes [29, 30]. Lithium difluoro(oxalato)borate (LiDFOB) is used to improve thermal stability and reduce the risk of thermal runaway [31, 32].

In the context of low-temperature performance, electrochemical impedance spectroscopy (EIS) can reveal critical information about the increased resistance and reduced ion mobility that affect battery performance. By systematically analyzing different electrolyte formulations through EIS, researchers can develop strategies to optimize electrolyte composition, enhancing the low-temperature performance of LIBs [33, 34]. This approach is essential for advancing battery technologies that can reliably operate in diverse and extreme environmental conditions, supporting the broader integration of renewable energy systems across various geographic and climatic regions.

The present study investigates the effect of electrolyte solvent composition on the electrochemical performance of lithium-ion batteries under low temperature conditions, utilizing the EIS technique. The three investigated electrolytes consist of $LiPF_6$ salt dissolved in various solvents, including EC/DEC, EC/EMC, and EC/EMC/EA.

2. Experimental

2.1. Cathode electrode preparation

The cathode electrode was fabricated utilizing the active constituent of nickel manganese cobalt oxide (NMC, Targray group). The conductive agent employed in the cathodes was carbon black

[DOI: 10.22068/ase.2024.672]

C45, manufactured by Imerys Company. The binder solution was produced by incorporating N-Methyl-Pyrrolidone (NMP, Scharlau Company), a solvent, and polyvinylidene fluoride (PVDF) from the Arkema group. The process of surface modification of aluminum foil was carried out with cold plasma. Subsequently, the aluminum foil was appropriately dimensioned. The cathode slurries were applied to aluminum current collectors using a blade gap of 80 µm at a speed of 10 m min^{-1}. Afterwards, the electrodes underwent a drying process within a vacuum oven for a duration of 20 hours, keeping a temperature of 60 °C.

2.2. Coin cell assembly

The CR2032 coin-type half-cells, consisting of two electrodes, were manufactured in a glove box with controlled moisture and oxygen levels, both kept below 0.5 ppm. A porous polypropylene film served as the separator for all cells. Three distinct cathode half-cells were produced, with each one using a different electrolyte. An NMC cathode electrode was used as the working electrode (positive side). Lithium metal foil, with a diameter of 16 mm and a thickness of 0.6 mm, served as both the counter and reference electrode (negative side). Three different kinds of electrolytes were employed: a standard electrolyte (STDE), a low-temperature electrolyte (LTE), and a long-cycle-life electrolyte (LCLE). The STDE, obtained from MTI Corporation, was 1 M $LiPF_6$ in EC/DEC (1:1). The LTE, provided by Dongguan Shanshan Battery Material Co., Ltd., consisted of 1 M $LiPF_6$ in EC/EMC (3:7) with Ethyl Acetate (EA). The LCLE, sourced from Gelon, consisted of 1 M $LiPF_6$ in EC/EMC (1:1).

An electric coin cell crimping machine was used to crimp the coin cells, ensuring a strong seal. Following the assembly process, a 24-hour resting period was given for the cells. The resting time ensured that the electrolyte percolated thoroughly across the separator's pores, which is critical for the cell's effective operation.

2.3. Characterization

The formation of coin cells was carried out through five charge-discharge cycles within the voltage range of 2.8 to 4.2 V. The charging procedure was conducted using the constant current-constant voltage (CC-CV) method, with a current of 20 mA g^{-1} (about 0.1C) and a cut-off current of 4 mA g^{-1}. The cells were discharged with a current of 20 mA g^{-1}. The battery test system (BTS 8000, Neware Ltd.) was used to perform the formation charge and discharge cycles. The formation data were obtained at ambient pressure and also at a temperature of 25 °C set by a temperature control chamber. Following the formation process, the electrochemical impedance spectrometry (EIS) tests were conducted in a fully charged state. The experiments were carried out within a frequency range of 100 kHz to 10 mHz at temperatures of 25, 0, -20, and -40 °C. The Autolab PGSTAT302N, which is galvanostat and potentiostat equipment, was employed for conducting EIS experiments.

3. Results and discussion

Figure 1(a)–(c) and Figure 1(d) show the charge-discharge profiles and discharge capacity diagrams during the five-cycle electrochemical formation of coin cells, respectively. For all three types of electrolytes, the overall appearance of the charge-discharge profiles is the same. The curve of the first charge had a higher voltage level than the subsequent charge curves that overlapped during the next 4 cycles. Most of the electrochemical formation process takes place during the first charge. For the case of LTE, the charging voltage was started above 4 V in the first cycle, while for the other two cases, it was below 4 V. The LTE exhibited a distinct behavior in the first charge due to the presence of EA in its formulation. The inclusion of EA likely reduced the overall viscosity of the electrolyte, enhancing ionic mobility. This improvement in ionic transport could explain why the LTE started with a higher voltage above 4 V during the first charge, compared to the other electrolytes. The lower viscosity and better ionic conductivity facilitated a more efficient electrochemical formation process, leading to a different charging profile.

According to the discharge curves, the average discharge voltage during 5 cycles for all three cases was close to each other, so it was 3.85 V for the STDE and 3.84 V for the other ones. According to Figure 1(d), the discharge capacity increased gradually from the first to the fifth cycle of formation for STDE and LCLE, while for the LTE sample, it first increased and then stabilized. Based on the averaging of the discharge capacities obtained during 5 formation cycles, the LTE sample provided the highest value (184.9 mAh g^{-1}) and the STDE sample had the lowest capacity (181.6 mAh g^{-1}). Higher capacity in LTE compared to STDE and LCLE can be attributed to the superior ionic conductivity provided by

[DOI: 10.22068/ase.2024.672]

the EA in the electrolyte, which helps maintain lower resistance and stable electrochemical reactions during discharge.

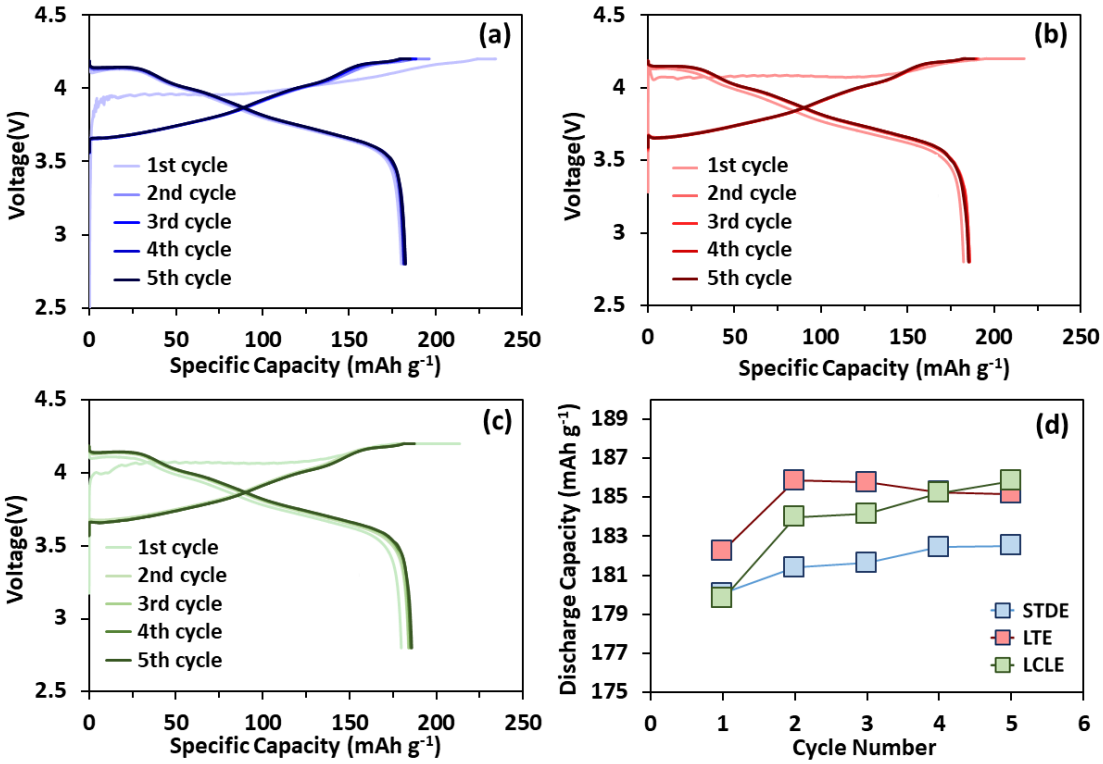

Figure 1: Charge-discharge profiles of (a) STDE, (b) LTE, and (c) LCLE; and (d) discharge capacity diagrams during the five-cycle electrochemical formation.

The Nyquist diagrams at different temperatures of 25, 0, -20, and -40 °C for the half-cells assigned STDE, LTE, and LCLE are depicted in Figure 2. The corresponding equivalent circuit is illustrated as well in Figure 3(a). The R_o represents the resistance of the bulk electrolyte, whereas the parallel circuit of $R_{cc}||CPE_{cc}$ is responsible for the current conduction among the electrode and current collector. The subsequent parallel circuit ($R_{SEI}||CPE_{SEI}$) indicates the influence of the SEI film on the impedance's middle frequency range. The $R_{ct}||CPE_{ct}$ parallel circuit represents the charge transfer resistance during the intercalation process across the electrodes and electrolyte. The slope of the line at low frequencies refers to the Warburg impedance, which denotes the diffusion of Li^+ ions at the boundary between the electrode and electrolyte. Table 1 and Figure 3 show the results of simulations derived from EIS data for different electrolytes at various temperatures.

The standard electrolyte (STDE), which consists of 1 M LiPF6 in EC/DEC (1:1), exhibited significant increases in various resistances as the temperature decreased. At 25 °C, the R_{ct} was 96.4 Ω and the R_{SEI} was 116.1 Ω, indicating reasonable performance under standard conditions. However, at 0 °C, the R_{ct} increased to 1055.3 Ω and R_{SEI} to 803.4 Ω, reflecting a significant drop in performance. At -20°C, these values surged to 8878.6 Ω and 172.1 Ω, respectively, and at -40°C, the R_{ct} reached a staggering 20755 Ω with the R_{SEI} at 1799 Ω. These results indicate that the STDE's performance severely deteriorates at lower temperatures due to the increased viscosity of the EC/DEC mixture, which hinders ionic mobility and increases resistance.

[DOI: 10.22068/ase.2024.672]

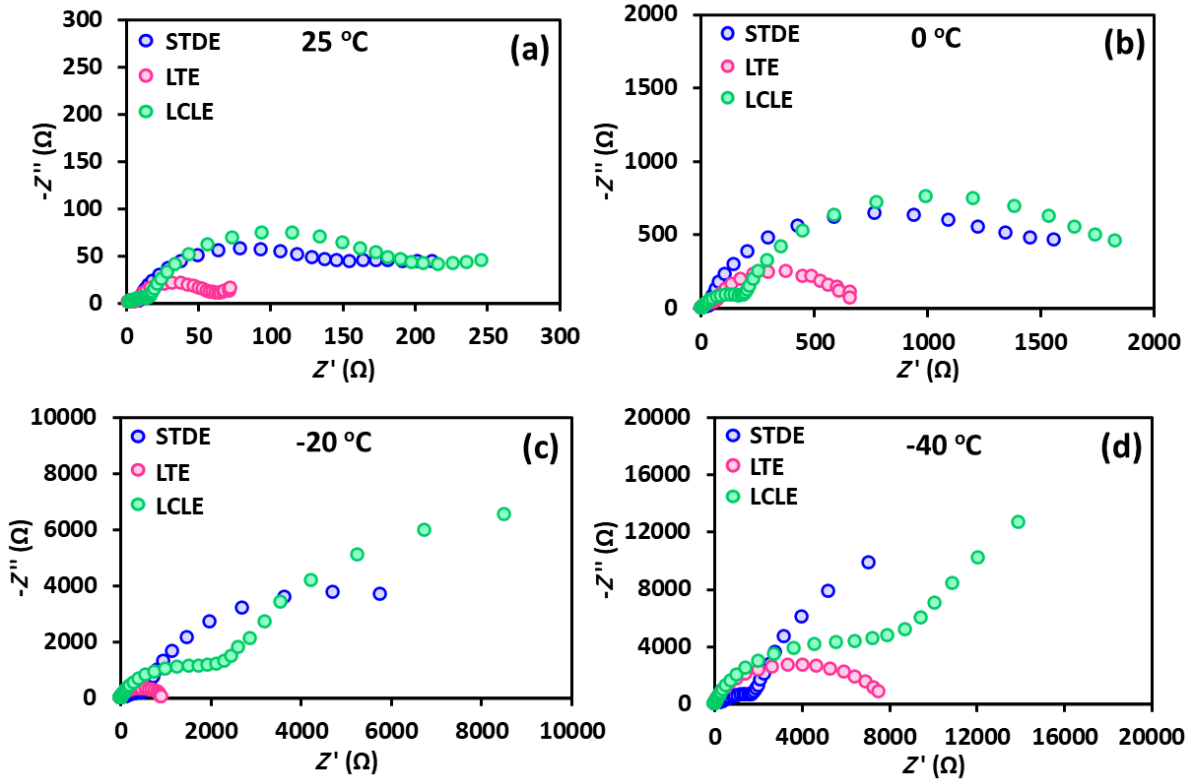

Figure 2: The Nyquist diagrams at different temperatures of 25, 0, -20, and -40 °C for the half-cells assigned STDE, LTE, and LCLE.

In contrast, the LTE, formulated with 1 M $LiPF_6$ in EC/EMC (3:7) and ethyl acetate (EA), demonstrated superior low-temperature performance. At 25 °C, the R_{ct} was a mere 3.6 Ω, and the R_{SEI} was 48.8 Ω, which are significantly lower than those of the STDE, indicating excellent ionic conductivity. Even at 0°C, the LTE maintained a R_{ct} of 507.4 Ω and an R_{SEI} of 64.2 Ω. At -20 °C, the R_{ct} remained manageable at 854.2 Ω and the R_{SEI} at 92.4 Ω. Finally, at -40 °C, although the resistances increased to 5709 Ω for R_{ct} and 1346 Ω for R_{SEI}, they were still substantially lower than those of the STDE. The superior performance of the LTE at low temperatures can be attributed to the presence of EA, which helps to lower the overall viscosity of the electrolyte mixture and enhance ionic mobility even at sub-zero temperatures.

The LCLE, composed of 1 M $LiPF_6$ in EC/EMC (1:1), showed the highest R_{ct} values across all temperatures, indicating poor performance in low-temperature conditions. At 25°C, R_{ct} was 140.8 Ω and R_{SEI} was 136 Ω, already higher than the other electrolytes. As the temperature dropped to 0 °C, R_{ct} increased to 1369.1 Ω and R_{SEI} to 172.1 Ω. At -20 °C, these values soared to 15622 Ω for R_{ct} and 2364 Ω for R_{SEI}. At -40 °C, the LCLE displayed the highest resistances among all tested electrolytes, with R_{ct} at 24486 Ω and R_{SEI} at 7530 Ω. The poor performance of the LCLE at low temperatures can be explained by the higher viscosity of the EC/EMC (1:1) mixture, which significantly impedes ionic transport and increases resistance.

A comparative analysis of the solvents EMC and DEC based on the EIS results reveals that EMC, particularly when combined with EA, offers superior low-temperature performance compared to DEC. The LTE with EMC and EA showed consistently lower R_{ct} and R_{SEI} values across all temperatures tested, indicating better ionic conductivity and lower impedance. The DEC-containing STDE, on the other hand, exhibited a dramatic increase in resistance as the temperature dropped, highlighting the limitations of DEC in low-temperature applications.

[DOI: 10.22068/ase.2024.672]

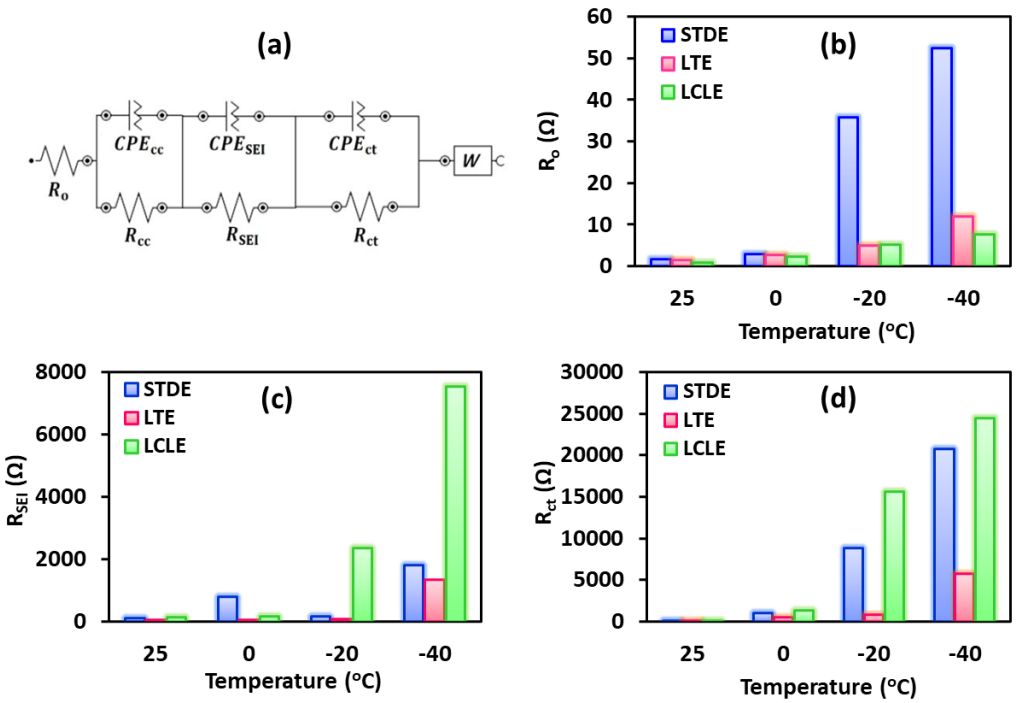

Figure 3: The corresponding equivalent circuit (a) and the results of simulations derived from EIS data for STDE, LTE, and LCLE half-cells at various temperatures (b, c, and d).

Table 1: The results of simulations derived from EIS data for different electrolytes at various temperatures.

Temperature (°C)	Electrolyte	R_o (Ω)	R_{cc} (Ω)	R_{SEI} (Ω)	R_{ct} (Ω)
25	STDE	1.65	7.57	116.07	96.46
	LTE	1.47	3.36	48.83	3.62
	LCLE	0.86	21.39	136.04	140.81
0	STDE	2.81	47.72	803.45	1055.32
	LTE	2.78	4.09	64.18	507.38
	LCLE	2.23	11.69	172.07	1369.11
-20	STDE	35.74	341.42	172.11	8878.61
	LTE	4.88	5.51	92.43	854.17
	LCLE	5.08	28.75	2364.03	15622.25
-40	STDE	52.44	801.07	1799.21	20755.36
	LTE	11.86	14.29	1346.91	5709.73
	LCLE	7.63	29.11	7530.62	24486.47

[DOI: 10.22068/ase.2024.672]

4. Conclusions

This study elucidates the pivotal role of electrolyte solvent composition in shaping the low-temperature performance of LIBs. Three electrolytes were studied: a standard electrolyte comprising 1 M $LiPF_6$ in EC/DEC, a low-temperature electrolyte consisting of 1 M $LiPF_6$ in EC/EMC/EA, and a long-cycle-life electrolyte containing 1 M $LiPF_6$ in EC/EMC. The results underscore significant differences in resistance values among the electrolytes at varying temperatures, highlighting the critical influence of solvent composition on LIB performance. At 0 °C, the STDE exhibited a R_{ct} of 1055.3 Ω and a R_{SEI} of 803.4 Ω, while the LTE demonstrated substantially lower R_{ct} (507.4 Ω) and R_{SEI} (64.2 Ω), indicative of superior low-temperature performance. Similarly, at -20 °C, the R_{ct} values for STDE, LTE, and LCLE were 8878.6 Ω, 854.2 Ω, and 15622 Ω, respectively, with corresponding R_{SEI} values of 172.1 Ω, 92.4 Ω, and 2364 Ω. Furthermore, at -40 °C, the resistance values followed a similar trend, emphasizing the superior performance of the LTE formulation. The addition of ethyl acetate (EA) in the LTE formulation played a crucial role in enhancing low-temperature performance by potentially reducing viscosity and improving ionic mobility. This finding shows the importance of solvent composition, particularly the inclusion of EA, in optimizing LIB performance for cold climates and extreme environmental conditions. The results have significant implications for the application of LIBs in battery electric vehicles (BEVs) and hybrid electric vehicles (HEVs). By maintaining lower resistance and higher ionic mobility at sub-zero temperatures, the LTE formulation can enhance the efficiency and reliability of LIBs in BEVs and HEVs, especially in regions with harsh winters. This improvement aligns with the global transition towards renewable energy and electric mobility, as discussed in the introduction. Optimizing electrolyte formulations for low-temperature performance is crucial for extending the range, lifespan, and overall performance of electric vehicles, thereby supporting broader adoption and integration of renewable energy solutions.

Declaration of Conflicting Interests

The authors declared no potential conflicts of interest with respect to the research, authorship, and publication of this article.

Acknowledgements

The authors would like to express their gratitude to the Institute of Mechanics for financial support.

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[DOI: 10.22068/ase.2024.672]