



LiPF₆ and lithium oxalyldifluoroborate blend salts electrolyte for LiFePO₄/artificial graphite lithium-ion cells

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ABSTRACT

The electrochemical behaviors of LiPF₆ and lithium oxalyldifluoroborate (LiODFB) blend salts in ethylene carbonate + propylene carbonate + dimethyl carbonate (EC + PC + DMC, 1:1:3, v/v/v) for LiFePO₄/artificial graphite (AG) lithium-ion cells have been investigated in this work. It is demonstrated by conductivity test that LiPF₆ and LiODFB blend salts electrolytes have superior conductivity to pure LiODFB-based electrolyte. The results show that the performances of LiFePO₄/Li half cells with LiPF₆ and LiODFB blend salts electrolytes are inferior to pure LiPF₆-based electrolyte, the capacity and cycling efficiency of Li/AG half cells are distinctly improved by blend salts electrolytes, and the optimum LiODFB/LiPF₆ molar ratio is around 4:1. A reduction peak is observed around 1.5 V in LiODFB containing electrolyte systems by means of CV tests for Li/AG cells. Excellent capacity and cycling performance are obtained on LiFePO₄/AG 063048-type cells tests with blend salts electrolytes. A plateau near 1.7–2.0 V is shown in electrolytes containing LiODFB salt, and extends with increasing LiODFB concentration in charge curve of LiFePO₄/AG cells. At 1C discharge current rate, the initial discharge capacity of 063048-type cell with the optimum electrolyte is 376.0 mAh, and the capacity retention is 90.8% after 100 cycles at 25 °C. When at 65 °C, the capacity and capacity retention after 100 cycles are 351.3 mAh and 88.7%, respectively. The performances of LiFePO₄/AG cells are remarkably improved by blending LiODFB and LiPF₆ salts compared to those of pure LiPF₆-based electrolyte system, especially at elevated temperature to 65 °C.

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1. Introduction

The olivine structure LiFePO₄ has been widely investigated as a promising cathode to substitute LiCoO₂ for its cheapness, rich source and environmental benignancy [1,2], these advantages make it attractive for developing advanced lithium-ion cells for transportation applications, such as electric vehicles (EV), hybrid electric vehicles (HEV) and plug-in hybrid electric vehicles (PHEV). However, an obvious obstacle of the application of LiFePO₄ is its low intrinsic electrical conductivity and slow Li⁺ diffusivity across the LiFePO₄/FePO₄ interface [3,4]. Previous researchers have found that LiFePO₄/graphite batteries exhibited poor cycle performance and much attention still focused on improving the conductivity of the material by coating [5–8], doping [9–12] and particle size minimization [13–15]. However, it is widely known that electrolyte plays an important role in the performance of batteries, and yet LiPF₆/carbonate-based electrolytes are still the most commonly used electrolytes in LiFePO₄/graphite batteries, only few papers are concerned with the improvement of LiFePO₄/graphite batteries by choosing suitable electrolytes [16,17].

Lithium oxalyldifluoroborate (LiODFB) is a novel lithium salt which has been drawn much attention since it was first reported by Zhang in 2006 [18]. Since LiODFB has a part of similar structure as LiBOB, a stable solid electrolyte interface (SEI) film can be formed on the graphite anode, protecting it from being eroded by solvent, such as propylene carbonate (PC). Compared to LiBOB, it is more soluble in linear carbonate solvents, and the SEI film is less resistive [19]. However, the study systems of this new salt mostly related to LiNi_{0.8}Co_{0.15}Al_{0.05}O₂/graphite [20], LiNi_{1/3}Mn_{1/3}Co_{1/3}O₂/graphite [19] and Li_{1.1}[Ni_{1/3}Co_{1/3}Mn_{1/3}]_{0.9}O₂/graphite [21] cells. Moreover, lower conductivity of the LiODFB-based electrolyte is an issue [18]. Using blend salts is an alternative approach to combine the advantages of different salts and to maximize the performances of lithium-ion cells [22–24]. LiPF₆ is the commercially dominant electrolyte salt with high conductivity in carbonate-based electrolytes. Liu et al. [25] have found that adding LiODFB to LiPF₆-based electrolyte can significantly improve both the capacity retention and the power retention of mesocarbon microbeads (MCMB)/Li_{1.1}Mn_{1/3}Ni_{1/3}Co_{1/30.9}O₂ lithium-ion batteries. Nevertheless, the use of LiODFB and LiPF₆ blend salts in LiFePO₄/graphite batteries has not been shown in any paper yet.

In our previous work [26], we studied the performance of LiFePO₄/AG cells with electrolytes of 1 M LiODFB salt in different binary, ternary and quaternary solvents. It was found that

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cells with electrolyte of 1 M LiODFB in EC + PC + DMC (1:1:3, v/v/v) have the best electrochemical performance at room temperature. In this paper, we selected this ternary solution combined merits of the three solvents, a systematic study on the characteristics of LiFePO₄/Li half cells, Li/AG half cells and LiFePO₄/AG 063048-type cells was conducted with LiPF₆ and LiODFB/EC + PC + DMC electrolytes. We optimized the LiODFB/LiPF₆ ratio and focused on investigating the compatibility of the electrolytes with both cathode and anode materials.

2. Experimental

2.1. Electrolyte preparation

LiPF₆ (battery degree) was purchased from Ferro Corporation (China). LiODFB (battery degree) was provided by Lai-en Green Energy Research Institute. PC, EC and DMC were obtained from Shenzhen CAPCHEM Technology Co., Ltd., China. The detailed composition of the electrolytes used is listed in Table 1. All electrolytes were prepared in an argon-filled glove box (Universal 2440/750, Mikrouna Mech. Tech. Co., Ltd., Water content: <1 ppm, oxygen content: <1 ppm). Electric conductivities of electrolyte solutions were measured from 0 °C to 40 °C by use of a conductometer (SG3, METTLER TOLEDO). Water and free acid contents of the electrolytes were controlled below 20 ppm, which were determined by Mettler Toledo DL32 titrator and Karl-Fisher 798 MPT Titrimo, respectively.

2.2. Characterization with half cells

The LiFePO₄/Li half cells were assembled in the argon-filled glove box using Celgard 2400 as separator and an appropriate amount of electrolyte listed in Table 1. The working electrode was composed of 84% (mass fraction) LiFePO₄, 8% carbon black and 8% PVDF binder. The electrodes were coated on Al foils. The counter and reference electrodes were lithium foils. The initial charge/discharge performance of LiFePO₄/Li half cells was evaluated using CR2025 type coin cells on a Land charge/discharge instrument, China. The cells were cycled 3 times between 2.5 V and 4.2 V at a constant current of 0.1C at room temperature.

The Li/AG half cells were also fabricated as CR2025 type coin cells in the argon glove box using Celgard 2400 as separator and an appropriate amount of electrolyte. The working electrode was composed of 92% (mass fraction) AG, 2% carbon black and 6% PVDF binder. The electrodes were coated on copper foils. The counter and reference electrodes were lithium foils. The cells were initially cycled 3 times between 0.01 V and 2 V at a constant current of 0.1C at room temperature.

For LiFePO₄/Li half cells, the charge process was defined as the de-intercalation of Li⁺ from the LiFePO₄ cathode, and the discharge process was defined as the intercalation of Li⁺ into the LiFePO₄ cathode. For Li/AG half cell, the charge process was defined as the intercalation of Li⁺ into the graphite, and the discharge process was defined as the de-intercalation of Li⁺.

Table 1
Composition of electrolytes investigated.

No.	Electrolyte salts (molar ratio)		Solvent
	LiODFB	LiPF ₆	
1	1 M	0	EC + PC + DMC (1:1:3)
2	0.8 M	0.2 M	
3	0.5 M	0.5 M	
4	0.2 M	0.8 M	
5	0	1 M	

2.3. Characterization with LiFePO₄/AG 063048-type cells

LiFePO₄ and AG were cathode and anode for 063048-type cells, and also Celgard 2400 as the separator. The electrochemical performances of the 063048-type cells were also tested by Land charge/discharge instrument. The initial charge–discharge test was carried by CC–CV mode, first cycled with a constant current of 0.1C to 3.8 V, followed by a constant voltage of 3.8 V until the current decreased to 0.01C, then discharged at 0.2C current rate to 2.2 V after kept still for 30 min.

The cells were charged and discharged between 2.2 V and 3.8 V with 1C current rate for capacity evaluation. For room temperature and high temperature performance determination, cycle tests were carried with a charge current of 0.5C and a discharge current of 1C. The cycle performance at 65 °C was carried in a high-low temperature test-chamber (GDH-2005C).

2.4. Cyclic voltammetry and AC impedance measurements

To evaluate the electrochemical behavior of LiFePO₄/Li and Li/AG half cells in various electrolyte systems, 2025-coin type cells with lithium foil as counter and reference electrodes, and Celgard 2400 as the separator were used. The initial charge/discharge performance of LiFePO₄/Li half cells was evaluated on Land CT2001A tester between 2.5 V and 4.2 V at a constant current of 0.1C; while Li/AG half cells were evaluated between 0.01 V and 2 V at 0.1C. Cyclic voltammetry (CV) and AC impedance for LiFePO₄/Li and Li/AG half cells were measured using 2025-coin type cells. The tests were measured using PARSTAT 2273 electrochemical measurement system (PerkinElmer Instrument, USA). CV test was performed with a scan rate of 0.1 mV s⁻¹ for both LiFePO₄/Li and Li/AG half cells. For the AC impedance, the cells were measured in the frequency range between 20 mHz and 100 kHz with a perturbation amplitude of 5 mV.

3. Results and discussion

3.1. Electric conductivities

Conductivity is of practical importance which determines the internal resistance, rate performance, and capacity of lithium-ion battery at some extent. Fig. 1 shows the conductivity of electrolytes with different LiODFB/LiPF₆ ratio at 0–40 °C. As it can be seen the conductivity improved with the increase of temperature for all electrolytes. The pure LiPF₆-based electrolyte showed a

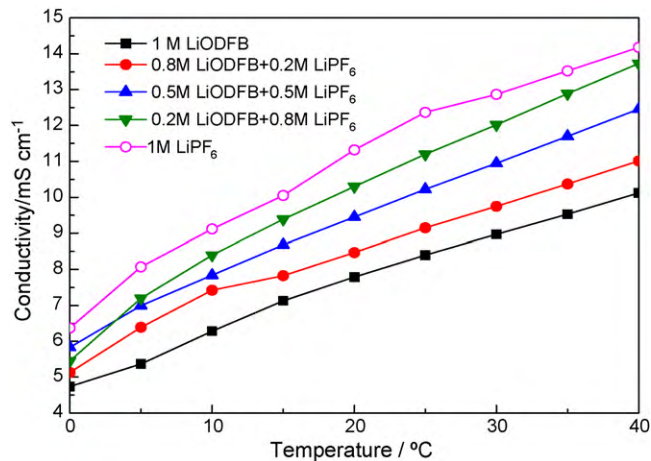


Fig. 1. Electric conductivity (κ) of electrolytes with different LiODFB/LiPF₆ ratio in EC + PC + DMC (1:1:3) solution (0–40 °C).

Table 2
Charge and discharge performance of LiFePO₄/Li half cells with different electrolytes.

No.	1st charge (mAh g ⁻¹)	η_1 (%)	2nd charge (mAh g ⁻¹)	η_2 (%)	3rd charge (mAh g ⁻¹)	η_3 (%)
1	154.9	91.2	141.9	96.6	135.3	96.6
2	157.1	91.3	142.3	96.3	134.9	96.1
3	152.4	90.5	138.9	96.3	132.1	96.2
4	153.2	89.8	138.3	96.6	131.8	96.3
5	159.9	90.2	147.2	98.4	147.8	98.2

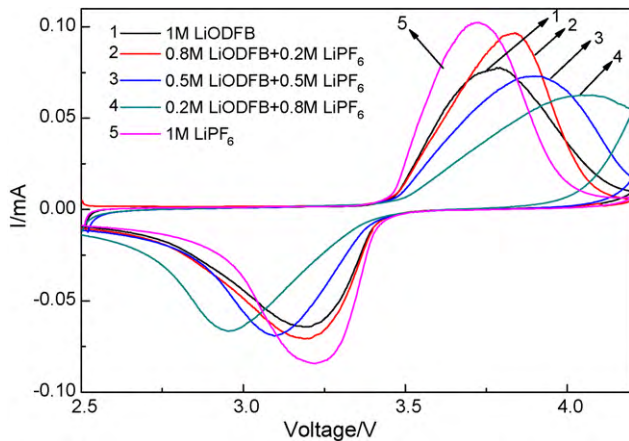


Fig. 2. CV curves of LiFePO₄/Li half cells with different salts cycled between 2.5 V and 4.2 V (vs. Li/Li⁺) at 0.1 mV s⁻¹.

maximum conductivity (12.37 mS cm⁻¹) at room temperature. The conductivity decreased with the increase of LiODFB content. This phenomenon can be explained that the van der Waals volume of the ODFB⁻ is larger than that of PF₆⁻, and LiODFB has lower ionic dissociation ability than LiPF₆, when LiPF₆ salt added, the number of free ions increased. What is more, electrolyte viscosity is proportional to anion size [27], the larger size of ODFB⁻ than PF₆⁻ increases the viscosity of electrolyte, which also results in the decreasing of electrolyte conductivity. Therefore, adding LiPF₆ can enhance the conductivity of LiODFB containing electrolytes.

3.2. Electrochemical performance of the LiFePO₄/Li half cell

Table 2 shows the first three charge and discharge performances for cells with different electrolytes at room temperature. It can be seen that the specific capacity and capacity efficiency of LiFePO₄/Li cells with LiODFB containing electrolytes had no obvious differences at the first three cycles. The LiFePO₄/Li cells with pure LiPF₆-based electrolyte showed superior performance in specific capacity and capacity efficiency. It can be concluded that LiODFB and LiPF₆ blend salts systems may have little superiority compared to commercial LiPF₆-based electrolyte in LiFePO₄/Li cells.

The CV curves of LiFePO₄/Li cells with different electrolytes were measured (Fig. 2). It can be seen that there is only one peak pair, consisting of one anodic peak and one cathodic peak, which corresponds to the two-phase charge/discharge reaction of the Fe³⁺/Fe²⁺ redox couple. For LiODFB containing electrolytes, the voltage separation got somewhat larger with the increase of LiPF₆ content.

Table 3
Charge and discharge performance of Li/AG half cells with different electrolytes.

No.	1st charge (mAh g ⁻¹)	η_1 (%)	2nd charge (mAh g ⁻¹)	η_2 (%)	3rd charge (mAh g ⁻¹)	η_3 (%)
1	351.9	93.7	335.3	98.8	336.3	99.0
2	355.5	89.1	337.8	97.5	339.9	98.2
3	347.4	88.9	331.7	97.4	330.0	98.0
4	323.7	87.5	345.2	96.3	341.6	97.6
5	527.0	50.3	323.9	90.6	314.0	94.2

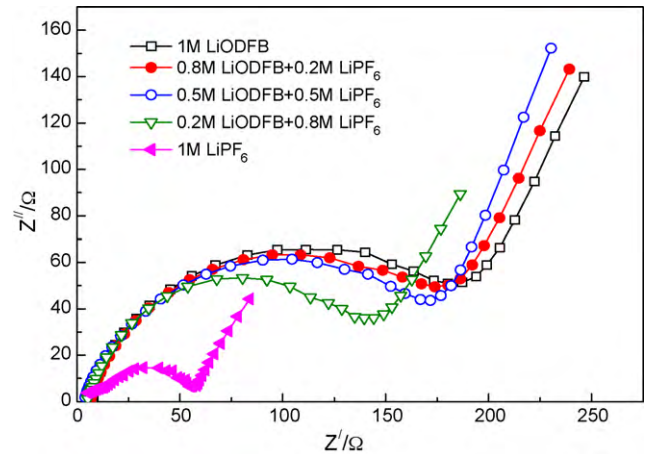


Fig. 3. AC impedance of LiFePO₄/Li half cells in different electrolyte systems at first charged state of 3.8 V vs. Li/Li⁺.

However, for pure LiPF₆ salt electrolyte system, the oxidation and reduction processes occur at 3.71 V and 3.23 V, respectively, with the smallest voltage separation of 480 mV between the anodic and cathodic peaks, which indicates that the reversibility of the reaction with blend salts electrolyte systems is no better than pure salt electrolyte systems. This result suggests that the blending of LiPF₆ and LiODFB has some effects on the Li⁺ transfer rate and the interfacial properties of LiFePO₄.

Fig. 3 shows the AC impedance of LiFePO₄/Li half cells at first charged state in various electrolytes. All the spectra contain a high frequency pronounced semicircle, which can be assigned to charge-transfer reaction resistance (R_{ct}), and a very short straight line with a slope of 45° from the real axis in the low frequency region, which reflects the solid-state Li⁺ diffusion into the active mass. It is obvious that LiFePO₄/Li half cells with electrolyte of pure LiPF₆ salt showed the least R_{ct} . The results indicate that LiODFB is the leading cause of increasing the reaction resistance for LiFePO₄/Li half cells.

3.3. Electrochemical performance of the Li/AG half cell

Table 3 shows the first three charge and discharge performances of Li/AG half cells with various electrolytes. Apparently, it can be seen that LiODFB containing electrolytes had superior initial cycle efficiency than that of pure LiPF₆-based electrolyte system, which maybe attribute to LiODFB salt participation in forming the SEI film [19], and perfect SEI film can be formed on the surface of AG with LiODFB containing electrolytes. However, some structural damages

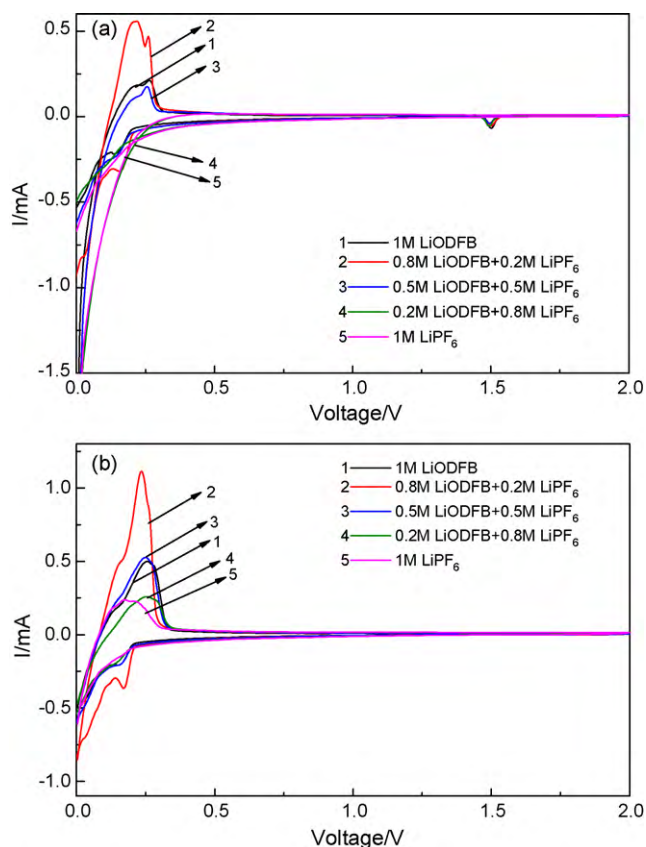


Fig. 4. CV curves of Li/AG half cells with different electrolytes cycled between 0 V and 2 V (vs. Li/Li⁺) at 0.1 mV s⁻¹ from (a) the first scan and (b) the second scan.

may occur in pure LiPF₆-based electrolyte system, which the initial cycle efficiency was only 50.3% (49.7% irreversible loss). It also can be seen that the electrolyte with 0.8 M LiODFB and 0.2 M LiPF₆ was near the optimum concentration for Li/AG half cell, with a reversible capacity of 339.9 mAh g⁻¹ and cycle efficiency of 98.2% after three cycles.

Fig. 4 shows the cyclic voltammograms of Li/AG half cells in different electrolyte systems. As shown in Fig. 4(a), during the first cathodic potential scan, a reduction peak appeared around 1.5 V, which attributed to a series of complicated exchanging reactions of LiODFB [19]. However, there was no such a peak in pure LiPF₆-based electrolyte system. A broad and large reduction band which was observed between 0 V and 0.6 V centered at about 0.15 V could be corresponding to the Li⁺ insertion process of graphite and the formation of SEI film. At the anodic process, there was a peak around 0.25 V, which ascribed to Li⁺ extraction from the graphite electrode. The anodic response current of high LiODFB containing electrolytes (i.e. 1–3) was much higher than that of high LiPF₆ containing electrolytes (i.e. 4 and 5). This result indicates that LiODFB significantly influences the Li⁺ de-intercalation behavior on graphite surface. For high LiPF₆ containing electrolytes 4 and 5, Li⁺ co-intercalated to graphite material with PC, the graphite material suffered some damage, this leads to the difficulty of Li⁺ de-intercalation process, and which further affects the capacity and cycle efficiency. During the second scan (Fig. 4(b)), the reduction peak around 1.5 V disappeared, this phenomenon may attribute to the reaction of LiODFB mainly occurred at the first scan. For the second scan, the anodic peak area rises obviously, which indicated the increase of cycle efficiency. The strongest reaction peak in electrolyte system 2 suggested the highest reversibility of intercalation and de-intercalation for Li⁺, and a suitable SEI film was formed with this electrolyte.

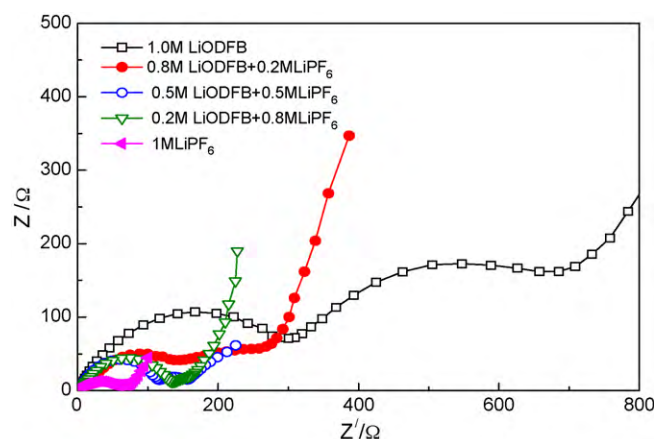


Fig. 5. AC impedance of Li/AG half cells after three cycles in different electrolyte systems at 2.0 V vs. Li/Li⁺.

AC impedance was used to investigate the kinetic characteristics of AG anode in different electrolyte systems (Fig. 5). Cells were first cycled between 0.01 V and 2 V for 3 times at charged state. As reported [28], the semicircle in high frequency region corresponds to Li⁺ migration through the SEI film. The enlargement of the high frequency semicircle represents the increase of the SEI film resistance (R_{SEI}). Apparently, with the adding of LiPF₆, R_{SEI} became smaller, which further proved LiODFB participated in forming the SEI film. That the LiODFB/LiPF₆ ratio of 4/1 was the optimum for Li/AG half cell may attribute to the higher conductivity of the electrolyte compared to pure LiODFB-based electrolyte, and much more suitable SEI film can be formed on the AG surface with electrolyte of this ratio compared to pure LiPF₆-based electrolyte.

3.4. Electrochemical performance of the LiFePO₄/AG 063048-type cells

It can be seen from Fig. 6(a) that there was a small potential plateau near 1.7–2.0 V in electrolyte systems containing LiODFB salt at the first charge cycle, and it extended with the LiODFB concentration increasing. However, there was no such a plateau in pure LiPF₆-based electrolyte system. This is possible that some reduction reaction took place on the interface of electrode and electrolyte, commonly regarded as the reduction of –OCOCOO–. Fig. 6(b) shows that the cell displayed the highest discharge platform and capacity in blend salts system when LiODFB/LiPF₆ ratio was 4:1, which were similar to Li/AG half cell tests.

The capacities of 063048-type cells in different electrolyte systems are compared in Fig. 7. The results suggested that the cells with LiPF₆ and LiODFB blend salts electrolytes had higher discharge capacity than that of pure LiPF₆-based electrolyte system. The plateau at the charge process shown in Fig. 6(a) disappeared, which further indicated that the reduction reaction only occurred at the first cycle. The average capacity decreased in the following order: 2 > 1 > 3 > 4 > 5. This result suggested that a small amount of LiPF₆ can compensate for the poor conductivity of LiODFB electrolyte, and maybe play a role in increasing the capacity value.

The cycle performances of 063048-type cells with electrolytes of different LiODFB/LiPF₆ ratio are shown in Fig. 8. At room temperature (Fig. 8(a)), it can be seen that the cell capacity increased with increasing the LiODFB concentration. When the LiODFB/LiPF₆ ratio was 4:1, the cell capacity was 336.2 mAh after 100 cycles, which was much higher than that of pure LiPF₆-based electrolyte system. As shown in Fig. 8(b), at 65 °C, high LiODFB containing electrolytes had much superior capacity and cycle performance, whereas the cycle performance of cells with pure LiPF₆-based elec-

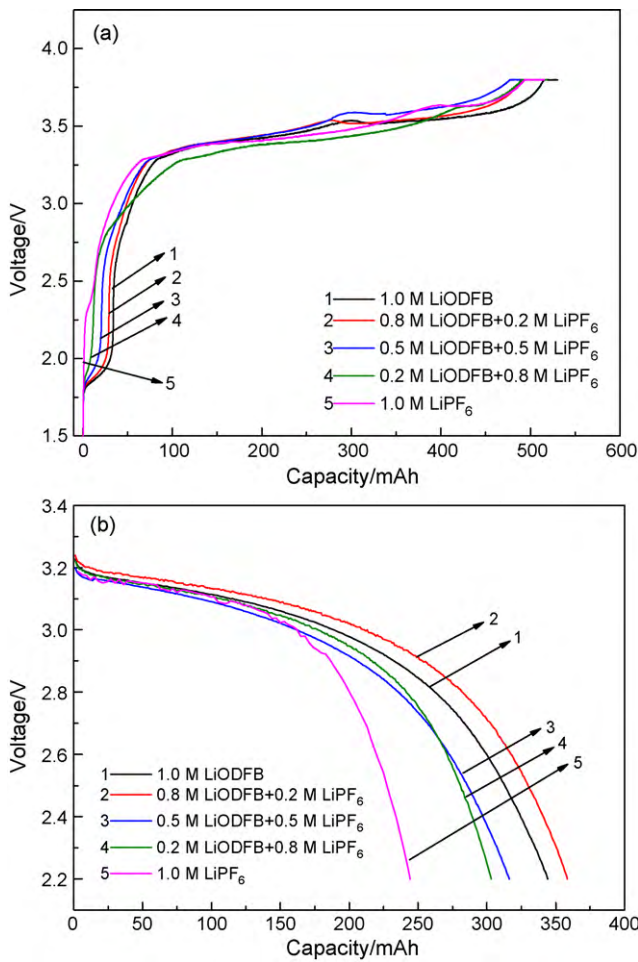


Fig. 6. The first charge (a) and discharge (b) curves of 063048-type cells with different electrolytes (charge rate: 0.1C; discharge rate: 0.2C, 25 °C).

trollyte deteriorated seriously after 100 cycles. The capacity and capacity retention were 351.3 mAh and 88.7%, respectively when the LiODFB/LiPF₆ ratio was 4:1 after 100 cycles. However, for pure LiPF₆-based electrolyte system, the capacity and capacity retention were only 121 mAh and 52%, respectively. This result can also be explained that high LiODFB concentration is beneficial to form dense and steady SEI film in rich PC containing electrolyte, particularly at elevated temperature. Furthermore, a small amount of LiPF₆

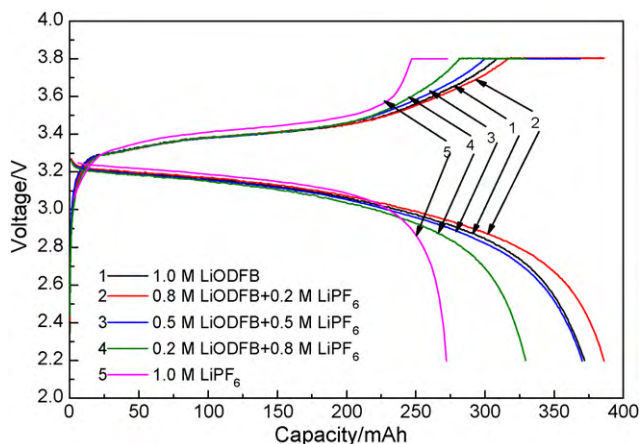


Fig. 7. Charge and discharging curves for capacity evaluation of 063048-type cells with different electrolytes.

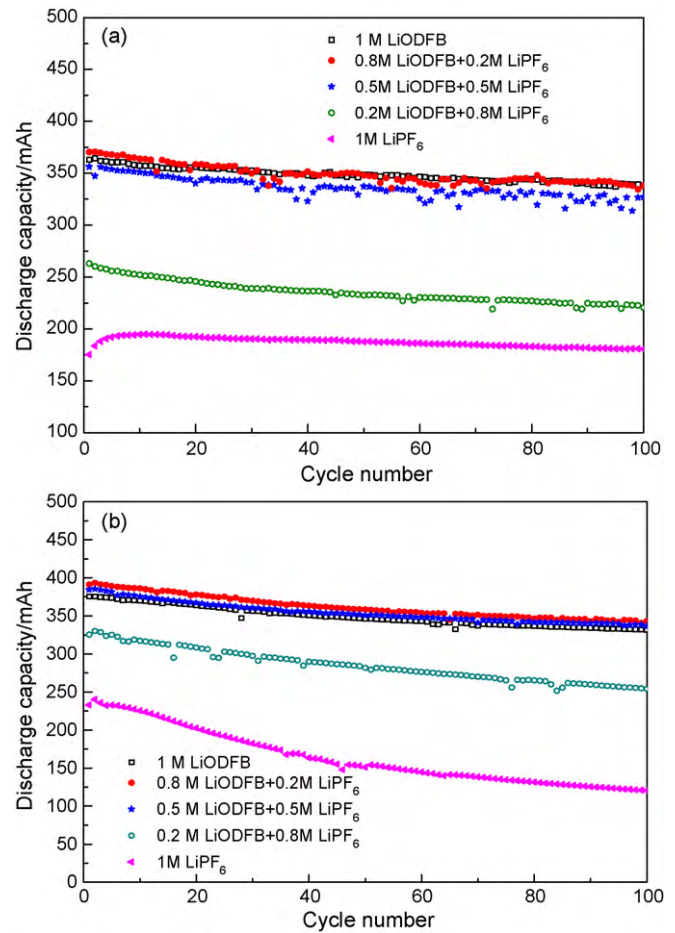


Fig. 8. Cycle performance of LiFePO₄/AG 063048-type cells with electrolytes of different LiODFB/LiPF₆ ratio at the charge rate 0.5C, and discharge rate 1C. (a) 25 °C and (b) 65 °C.

salt improved the electrolyte conductivity and increased in capacity. In all, compared to both pure LiODFB-based electrolyte and pure LiPF₆-based electrolyte, electrolyte with LiODFB/LiPF₆ ratio of 4:1 have better compatibility with LiFePO₄/AG cells.

4. Conclusions

LiPF₆ and LiODFB blend salts for LiFePO₄/AG lithium-ion cells have been investigated in EC + PC + DMC solution. The electrochemical performance shows that the blending of LiPF₆ and LiODFB was inferior to pure LiPF₆-based electrolyte system on the LiFePO₄/Li half cells, but LiPF₆ and LiODFB blend salts obviously affect the performances of Li/AG half cells and LiFePO₄/AG 063048-type cells. The electrochemical performance indicates that the LiODFB/LiPF₆ ratio of 4:1 (0.8 M:0.2 M) was around the best choice. For Li/AG half cells, the discharge capacity and current efficiency remarkably improved by the blending of LiODFB and LiPF₆. For the LiFePO₄/AG 063048-type cells, a small potential plateau can be observed near 1.7–2.0 V in electrolyte systems containing LiODFB salt at the first charge process. The capacity and the capacity retention were also markedly improved by increasing the LiODFB content, especially at elevated temperature. It can get a conclusion that the electrolyte with LiODFB/LiPF₆ ratio around 4:1 has the best compatibility with LiFePO₄/AG lithium-ion cells.

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