Lithium oxalyldifluoroborate/carbonate electrolytes for LiFePO₄/artificial graphite lithium-ion cells

Jie Li, Keyu Xie, Yanqing Lai *, Zhi'an Zhang, Fanqun Li, Xin Hao, Xujie Chen, Yexiang Liu

School of Metallurgical Science and Engineering, Central South University, Changsha Hunan 410083, China

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ABSTRACT

The electrolytes based on lithium oxalyldifluoroborate (LiODFB) and carbonates have been systematically investigated for LiFePO₄/artificial graphite (AG) cells, by ionic conductivity test and various electrochemical tests, such as cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), and charge–discharge test. The conductivity of nine electrolytes as a function of solvent composition and LiODFB salt concentration has been studied. The coulombic efficiency of LiFePO₄/Li and AG/Li half cells with these electrolytes have also been compared. The results show that 1 M LiODFB EC/PC/DMC (1:1:3, v/v) electrolyte has a relatively higher conductivity (8.25 mS cm^{-1}) at 25 °C, with high coulombic efficiency, good kinetics characteristics and low interface resistance. With 1 M LiODFB EC/PC/DMC (1:1:3, v/v) electrolyte, LiFePO₄/AG cells exhibit excellent capacity retention ~92% and ~88% after 100 cycles at 25 °C and at elevated temperatures up to 65 °C, respectively; The LiFePO₄/AG cells also have good rate capability, the discharge capacity is 324.8 mAh at 4 C, which is about 89% of the discharge capacity at 0.5 C. However, at −10 °C, the capacity is relatively lower. Compared with 1 M LiPF₆ EC/PC/DMC (1:1:3, v/v), LiFePO₄/AG cells with 1 M LiODFB EC/PC/DMC (1:1:3, v/v) exhibited better capacity utilization at both room temperature and 65 °C. The capacity retention of the cells with LiODFB-based electrolyte was much higher than that of LiPF₆-based electrolyte at 65 °C, while the capacity retention and the rate capacity of the cells is closed to that of LiPF₆-based electrolyte at 25 °C. In summary, 1 M LiODFB EC/PC/DMC (1:1:3, v/v) is a promising electrolyte for LiFePO₄/AG cells.

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1. Introduction

Since the first commercialization of lithium-ion batteries by Sony in 1990, lithium hexafluorophosphate (LiPF₆) is the commercially dominant electrolyte salt in the state-of-the-art lithium-ion battery [1]. Its solvents in alkyl carbonate mixtures can reach a high specific conductivity, and it is not aggressive toward Al current collectors. There are, however, several drawbacks of LiPF₆ solutions as lithium-ion battery electrolyte [2]. First, the thermal stability of LiPF₆ with lithiated graphite or delithiated transition metal oxides is worse, which restricts its use in large-scale lithium-ion batteries for hybrid electric vehicle (HEV) and electric vehicle (EV) applications. In addition, LiPF₆ will decompose and generate HF, which will dissolve transition metal ions from cathode, resulting in fading of the capacity during the cycles, especially at elevated temperatures.

The above drawbacks of LiPF₆ have promoted great efforts to find replacements for this salt. And significant efforts have been made in the past to develop alternative salts, such as LiAsF₆ [3], LiClO₄ [4], LiBF₄ [5], LiN(SO₂CF₃)₂ [6], LiN(SO₂F)₂ [7], etc., in order

to improve the cycle performance and the thermal stability of lithium-ion batteries. However, none of these salts were able to replace LiPF₆ used in commercial lithium-ion batteries presently due to their toxicity, explosive nature, high reactivity with the electrodes or corrosion of the current collector [8]. Recently, lithium bis(oxalato)borate (LiBOB) has attracted much interest due to its favourable characteristics, such as good thermally stability, excellent ability to passivate Al and form steady SEI on the surface of both electrodes [9,10]. However, due to low salt solubility and high interface resistance formed with LiBOB, which consequently reduces power and rate capability of the cell, especially at low temperatures, this excellent salt could just find an application niche in high-temperature and low power applications [11].

In 2006, lithium oxalyldifluoroborate (LiODFB) is first reported by Zhang [12] as a unique lithium salt for improved electrolyte of lithium-ion battery. Since LiODFB has a part of similar structure as LiBOB, it is found that LiODFB has the most of the unique characteristics of LiBOB in lithium-ion batteries. Furthermore, LiODFB is superior to LiBOB in the properties as below [13]: (1) it is more soluble in linear carbonate solvents that are essential to lower viscosity and provide the probability to optimize the solvents for the higher conductivity over a wide temperature range; (2) it helps to form steady SEI with less resistance, so the lithium-ion cell using it has a

* Corresponding author. Tel.: +86 731 88830649; fax: +86 731 88830649.
  E-mail address: csulightmetals@126.com (Y. Lai).

Table 1
Composition of electrolytes.

Electrolytes	Solvents	Ratio (v/v)
A	EC/DMC	1:1
B	EC/DEC	1:1
C	EC/EMC	1:1
D	EC/PC/DMC	1:1:3
E	EC/PC/DEC	1:1:3
F	EC/PC/EMC	1:1:3
G	EC/PC/DMC/DEC	2:2:3:3
H	EC/PC/DMC/EMC	2:2:3:3
I	EC/PC/DEC/EMC	2:2:3:3
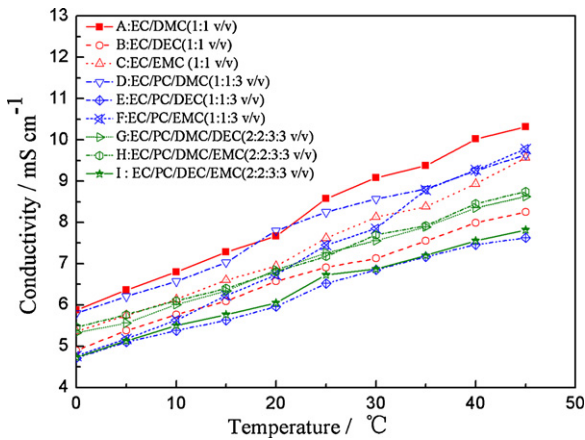
Fig. 1. Conductivities of 1 M LiODFB salt in various solvent systems (0–45 °C).

Fig. 2. Conductivities of LiODFB salt of different concentrations in EC/PC/DMC (1:1:3, v/v).

better power capability and low temperature cyclic performance. These unique characteristics make LiODFB as a promising salt for high-power applications [14].

Up to date, only the electrochemical performance of LiODFB in ethylene carbonate (EC)/propylene carbonate (PC)/ethyl methyl carbonate (EMC) or EC/dimethyl carbonate(DMC) solvent systems with some kinds of high-power lithium-ion cells were studied, such as $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$/graphite [12], $LiNi_{1/3}Mn_{1/3}Co_{1/3}O_2$/graphite [13,15] and $Li_{1.1}[Ni_{1/3}Co_{1/3}Mn_{1/3}]_{0.9}O_2$/graphite cells [16]. However, to the best of our knowledge, the electrochemical performances of LiODFB based electrolytes with $LiFePO_4$/graphite battery, which is now popularly considered as the most promising one for using in large-scale lithium-ion batteries for HEV and EV applications, because of its low cost, safer performance and environmental benign nature, has not been shown in any document yet.

Based on its unique characteristics as mentioned above, LiODFB would assist in improving the performance of $LiFePO_4$/graphite batteries, which is seriously restricted by commercialized electrolytes as following aspects: on one hand, $LiFePO_4$/graphite batteries are known to exhibit fast capacity fading, especially at elevated temperatures; on the other hand, low-temperature performance is
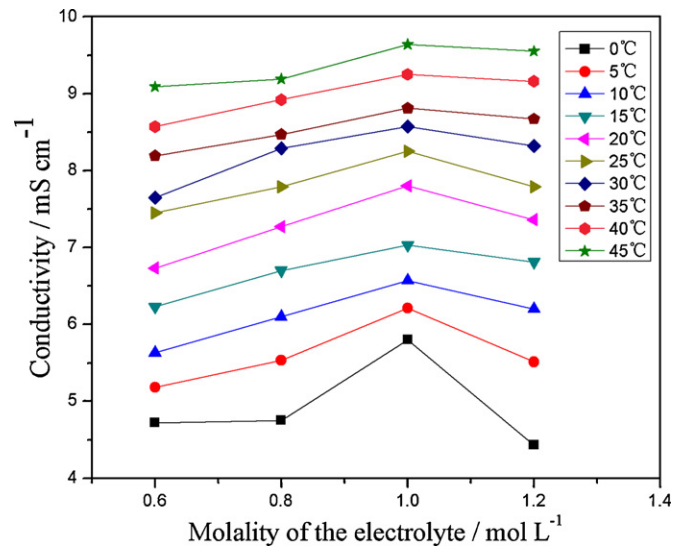
still challenging requirement for $LiFePO_4$/graphite batteries. It was for this purpose that we first systematically investigated LiODFB, with the focus placed on investigating solvent systems for this salt with carbonates and evaluating the resultant electrolytes in $LiFePO_4$/artificial graphite (AG) cells, compared with $LiPF_6$-based electrolyte, using various electrochemical techniques.

2. Experimental

Battery-grade LiODFB and $LiPF_6$ were obtained from Lai-en Green Energy Research Institute and Ferro Performance Materials Company. Battery-grade EC, PC, DMC, EMC, ethylmethyl carbonate (DEC), purchased from Shenzhen Capchem Chemicals Co., Ltd., were dried in sequence using 4 Å molecular sieves and neutral alumina. In an argon-filled glove box (Universal 2440/750, Mikrouna Mech. Tech. Co., Ltd.) with both oxygen and water contents less than 1 ppm, nine electrolytes with different solvent ratio were prepared as show in Table 1. Water and free acid contents in these electrolytes were controlled below 20 ppm, which were determined by Karl-Fisher (DL32/DL39) and Karl-Fisher 798 MPT Titrino, respectively. Ionic conductivities of these electrolytes were determined from 0 °C to 45 °C by a conductometer (SG3, METTLER TOLEDO).

The $LiFePO_4$ cathodes consisted of 84 wt% $LiFePO_4$, 8 wt% carbon black and 8 wt% PVDF, were fabricated by coating the slurry of $LiFePO_4$ active material, carbon black, and PVDF on aluminum foil collector. The graphite anodes, consisted of 92 wt% AG, 2 wt% carbon black and 6 wt% PVDF, were fabricated by coating the mixing slurry of AG, carbon black, and PVDF, on copper foil collector.

To evaluate the electrochemical behavior of these electrolytes in $LiFePO_4$/Li and AG/Li half cells, 2025-coin type cells with lithium

Table 2
Charge and discharge performances of $LiFePO_4$/Li half cells with different electrolytes.

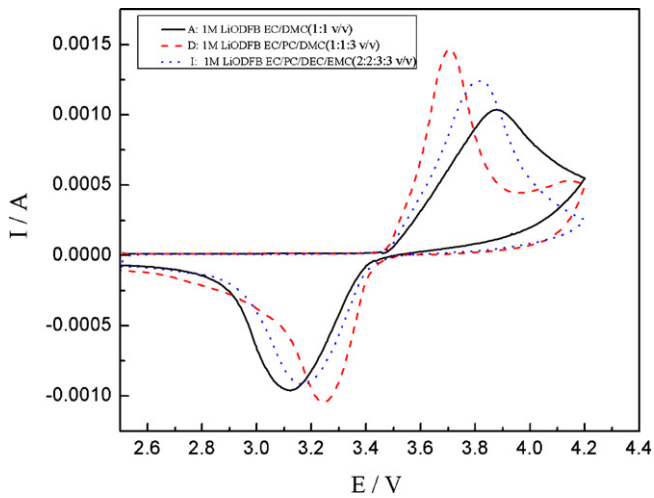| Electrolytes	1st Charge (mAh g⁻¹)	η_1 (%)	2nd Charge (mAh g⁻¹)	η_2 (%)	3rd Charge (mAh g⁻¹)	η_3 (%)
A	144.5	93.2	139.5	97.4	135.8	97.8
B	139.4	91.2	137.4	98.4	133.4	97.0
C	141.3	92.6	138.1	97.8	133.6	96.2
D	144.6	91.6	142.5	99.3	140.8	98.2
E	144.0	91.3	142.2	99.7	140.0	97.9
F	142.7	91.9	141.3	99.7	140.0	98.3
G	141.2	92.0	135.9	98.8	129.2	98.7
H	141.8	92.3	137.0	99.0	130.4	99.0
I	145.2	92.7	140.8	99.1	137.3	97.3

Fig. 3. CV of LiFePO$_4$/Li cells with different electrolytes between 2.5 V and 4.2 V at 1 mV s^{-1} in the first cycle at 25 °C.

foil as counter and reference electrode, and Celgard 2400 as the separator were used. The initial charge/discharge performance of LiFePO$_4$/Li half cells were evaluated on Land CT2001A tester between 2.5 V and 4.2 V at a constant current of 0.1 C; While AG/Li half cells were evaluated between 0.01 V and 2 V at 0.1 C. Cyclic voltammetry (CV) and AC impedance for LiFePO$_4$/Li and AG/Li 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 1 mV s^{-1} and 2 mV s^{-1} for LiFePO$_4$/Li and AG/Li half cells, respectively. 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.

The rate capacity and cycle performance at different temperature were evaluated with 063048-type cells using LiFePO$_4$ as cathode, AG as anode, and Celgard 2400 as the separator on Land CT2001A tester. The 063048-type cells with nominal capacity of 380 mAh were charged to 3.8 V at 0.1 C rate followed by a constant voltage charging to a cut-off current of 0.01 C rate (CC–CV). Then, cells were discharged to 2.2 V at 0.5 C rate after keeping still for 30 min. The cells were charged and discharged between 2.2 V and 3.8 V with 1 C current rate for cycle performance. The cycle per-
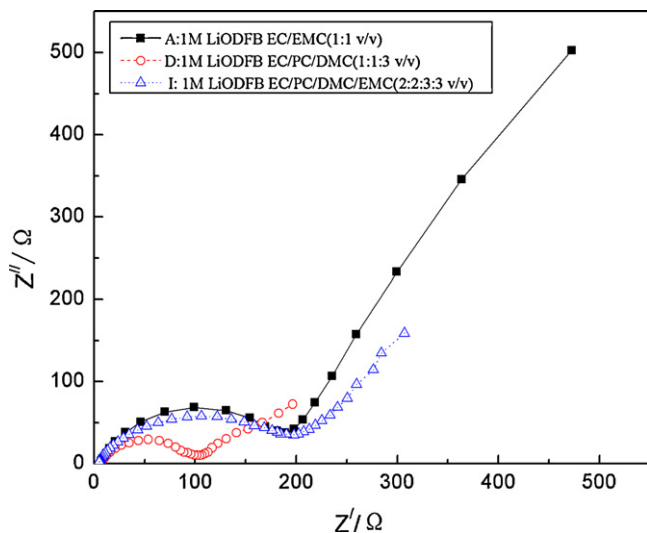

Fig. 4. AC impedance of LiFePO$_4$/Li cells at first charged state with different electrolytes at 3.8 V (25 °C).
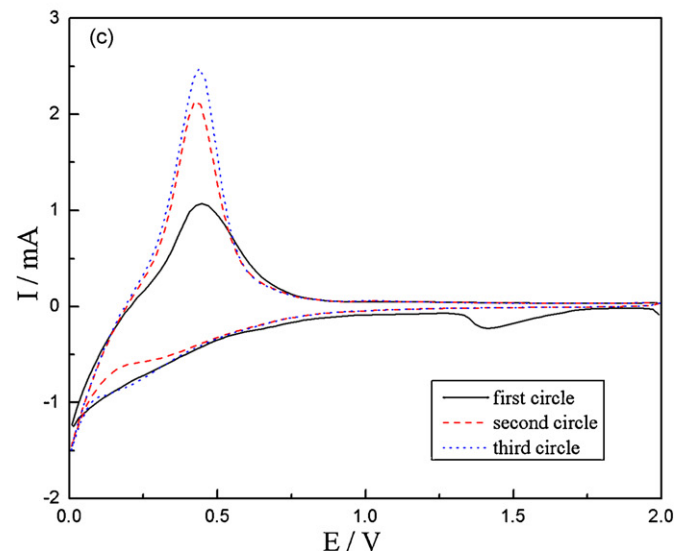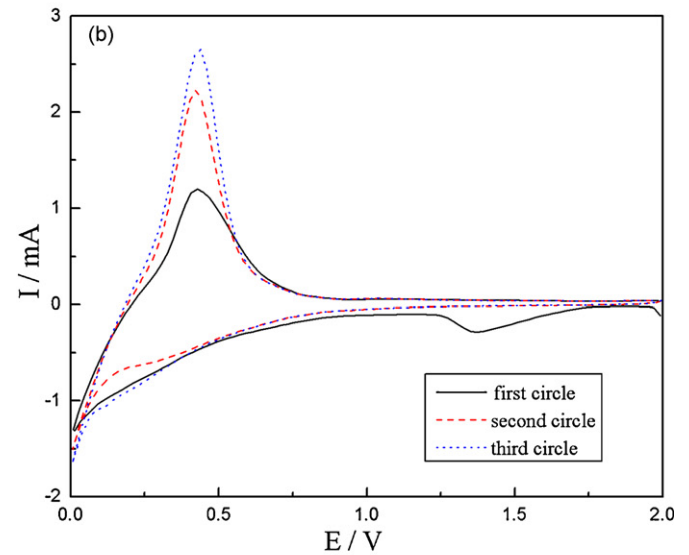

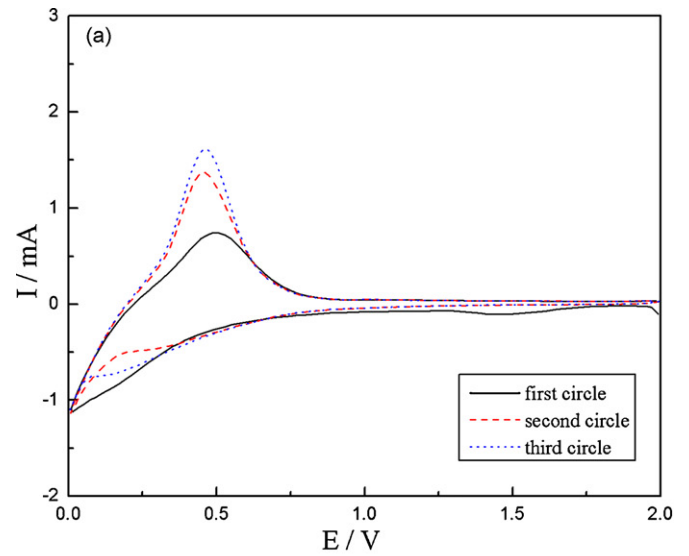**Fig. 5.** CV of AG/Li cells with different electrolytes between 0 V and 2 V at 2 mV s^{-1} in the first three cycles at 25 °C: (a) 1 M LiODFB EC/EMC (1:1, v/v), (b) 1 M LiODFB EC/PC/DMC (1:1:3, v/v), and (c) 1 M LiODFB EC/PC/DMC/EMC (2:2:3:3, v/v).

Table 3
Charge and discharge performances of AG/Li half cells in different electrolytes.

Electrolytes	1st Charge (mAh g^{-1})	η_1 (%)	2nd Charge (mAh g^{-1})	η_2 (%)	3rd Charge (mAh g^{-1})	η_3 (%)
A	340.1	83.8	310.7	97.8	300.4	98.6
B	344.2	93.9	323.8	98.9	324.3	99.1
C	365.8	93.9	346.1	99.0	342.0	99.2
D	351.9	93.7	335.3	98.8	336.3	99.0
E	347.5	93.2	327.3	98.9	330.0	99.1
F	346.5	94.0	327.4	99.0	330.1	99.2
G	347.1	89.2	316.4	97.5	326.2	98.1
H	352.1	88.7	319.3	97.9	328.0	98.2
I	337.7	88.2	319.0	99.0	317.9	99.3

formance at low temperature (−10 °C) was carried in a high-low temperature test-chamber (GDH-2005C).

3. Results and discussion

3.1. Ionic conductivity of LiODFB-based electrolyte

The conductivities of 1 M LiODFB based electrolytes in different binary, ternary and quaternary solvent mixtures at different ratios between 0 °C and 45 °C are shown in Fig. 1. The conductivities of these electrolytes are increased in the order of A > D > C > F > G > H > B > I > E at 25 °C. Among the binary solvent mixtures, LiODFB based electrolytes show a maximum conductivity (8.58 mS cm^{-1}) in EC/DMC binary mixtures. This behavior is attributed to the combined effect of the viscosity and dielectric constant of the solvents [17], because DMC has the highest dielectric constant and lowest viscosity among DMC, EMC and DEC [18]. The similar situation has also been seen in the ternary and quaternary solvent mixtures. In addition, in the whole temperature range studied, the conductivities of these electrolytes increases as temperature increasing. The reason is that viscosity becomes lower and ion becomes active along with the rise of temperature [19].

It can be seen from Fig. 2 that the electrolyte conductivity increases as the salt concentration is increased from 0.6 M to 1.0 M, and then falls with the salt concentration is 1.2 M. These may be attributed to the interactions between the free ionic number and the viscosity of the electrolyte. That is, higher salt concentration in the electrolyte helps by enhancing free ionic number, but also increasing the viscosity. The maximum of conductivity (8.25 mS cm^{-1}) of LiODFB based electrolyte in EC/PC/DMC (1:1:3, v/v) is obtained for a molar concentration in salt of approximately 1 M at 25 °C.

3.2. Electrochemical performance of LiFePO$_4$/Li half-cell in LiODFB-based electrolyte

Table 2 shows the first three charge–discharge dates of LiFePO$_4$/Li cells with nine electrolytes. At first cycle, all the cells can be activated to deliver almost their full capacity with charge–discharge efficiency over 90%. The irreversible capacities of the first cycle are related to the formation of SEI on the surface of LiFePO$_4$ materials as other cathode materials [20]. In the following two cycles, all the cells exhibited high coulombic efficiency (η) about 99% and stable charge–discharge capacity. In general, all the LiFePO$_4$/Li cells with LiODFB based electrolytes show very good charge–discharge performances and cycling stability.

The CV of the LiFePO$_4$/Li cells with LiODFB salt in binary, ternary and quaternary solvent systems was measured (Fig. 3). It is clear that using electrolyte D, the potential separation between the anodic and cathodic peaks was the smallest, furthermore, the peaks were the narrowest and the peak intensity was the strongest. This indicates that LiFePO$_4$/Li cells with electrolyte D have the best kinetics characteristics.

Fig. 4 shows the AC impedance of LiFePO$_4$/Li cells at first charged state in electrolytes A, D, I. 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 °C from the real axis in the low frequency region, which reflects the solid-state Li ion diffusion into the active mass. It is obvious that LiFePO$_4$/Li cells with electrolyte D showed the least R_{ct}. This means that, with electrolyte D, LiFePO$_4$/Li cells has better kinetics characteristics than using electrolytes A and I. This is in good accord with the results of CV.

3.3. Electrochemical performance of AG/Li half-cell in LiODFB-based electrolyte

The first three charge–discharge dates of these AG/Li cells with nine electrolytes are shown in Table 3, from which η were between 83.8% and 94.0% for the first cycle which is similar to the LiPF$_6$ electrolyte [21]. In the following two cycles, η was about 99% and charge–discharge capacity was very stable. With high η, electrolytes C, D, H show the highest discharge capacity among the binary, ternary and quaternary solvent systems, respectively.

Fig. 5 shows the CV of AG/Li cells in electrolytes C, D, H for the first three cycles. As seen in this figure, the anodic currents measured at potentials about 1.5 V in the three electrolytes is attributed to the reduction of −OCOCOO− pieces as a result of the chemical equilibrium of oxalatoborate ring-opening; Furthermore, the reduced products participate in the formation of a preliminary SEI to support graphite cycling in high PC-containing solutions for improved low temperature performances [13]. It is also very significant that all the cells show better kinetics in the following cycles because of the narrow gap between the anodic and cathodic peaks.

Fig. 6 shows the AC impedance of AG/Li cells using different electrolytes. The AC impedance of the AG/Li cells after two cycles are mainly composed of a semicircle at high frequency and medium frequency. It can be observed that the SEI resistance (R_{SEI}), which is related to the first semicircle at high frequency, is decreased in the order of C > H > D. This indicates that with the electrolyte D, AG/Li cells has better kinetics characteristics than using electrolytes C and H.

3.4. Electrochemical performance of LiFePO$_4$/AG cells in LiODFB-based electrolyte

All the cells have a very short voltage plateau at 1.7 V as shown in inset of Fig. 7. This short plateau is related to the reduce of −OCOCOO− pieces in the first cycle on the graphite anode as discussed above. As a result, the cells with the 1 M LiODFB EC/PC/DMC (1:1:3, v/v) electrolyte shows the highest discharge capacity with the coulombic efficiency of 74% in the first cycle.

Fig. 8(a) shows the cycling performances at 25 °C for LiFePO$_4$/AG cells with electrolytes A, D and H. After the first 100 cycles, there
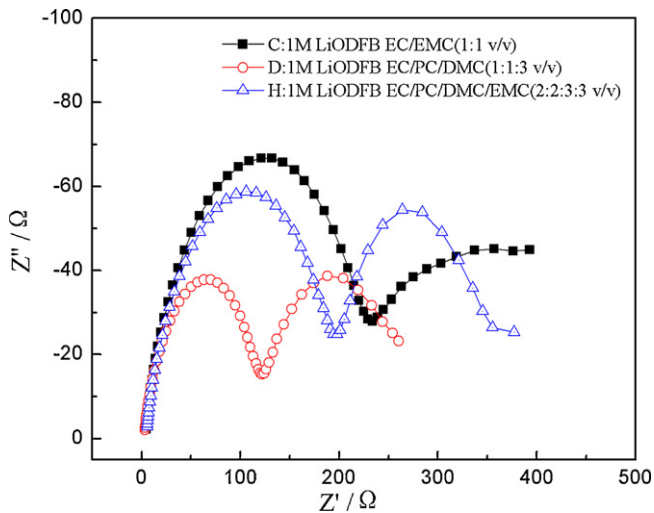
Fig. 6. AC impedance of AG/Li cells after the first cycle with different electrolytes at 2.0 V (25 °C).

is no obvious difference in the capacity fading of these electrolytes, and all the cells still retained about 92% of the initial capacity. Fig. 8(b) shows that all the LiFePO$_4$/AG cells have a slightly higher capacity at 65 °C compared to that at room temperature, mainly because at this temperature, the electrolyte conductivity increases and the electrode wetting characteristics are improved. The cell also cycles well with a limited capacity fade after 100 cycles, especially for 1 M LiODFB EC/PC/DMC (1:1:3, v/v) electrolyte with the highest retention about 88%. Charging and discharging capacities of the lithium-ion cell at −10 °C are plotted as a function of cycle number in Fig. 8(c). While the others cycled well with a high remained capacity, the cells failed to cycle with electrolyte A. Because electrolyte A contains too much EC (50%, v/v), which has a high freezing point. However, the capacity utilization is low. Further improvement should be done by optimizing the solvents of electrolyte for low temperature applications.

The rate capability of LiFePO$_4$/AG cells with different electrolytes at room temperature are shown in Fig. 9. The specific discharge capacities gradually decreased with increasing the applied current densities for all the cells. It is obviously that LiFePO$_4$/AG cell with 1 M LiODFB EC/PC/DMC (1:1:3, v/v) has an
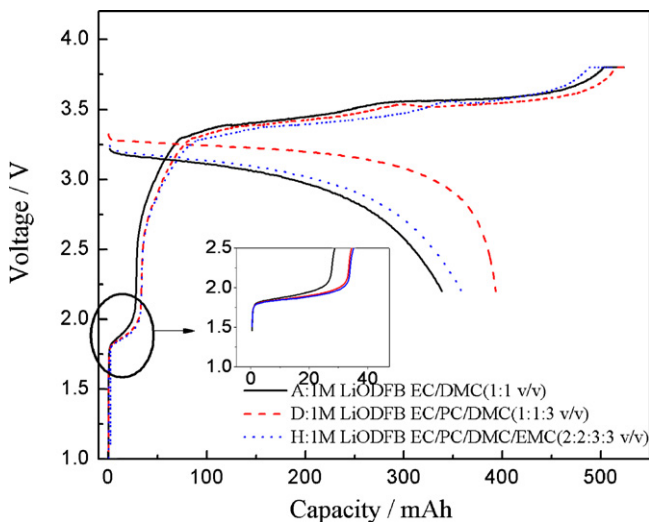

Fig. 7. The first charge–discharge curve of LiFePO$_4$/AG cells with different electrolytes at 25 °C.
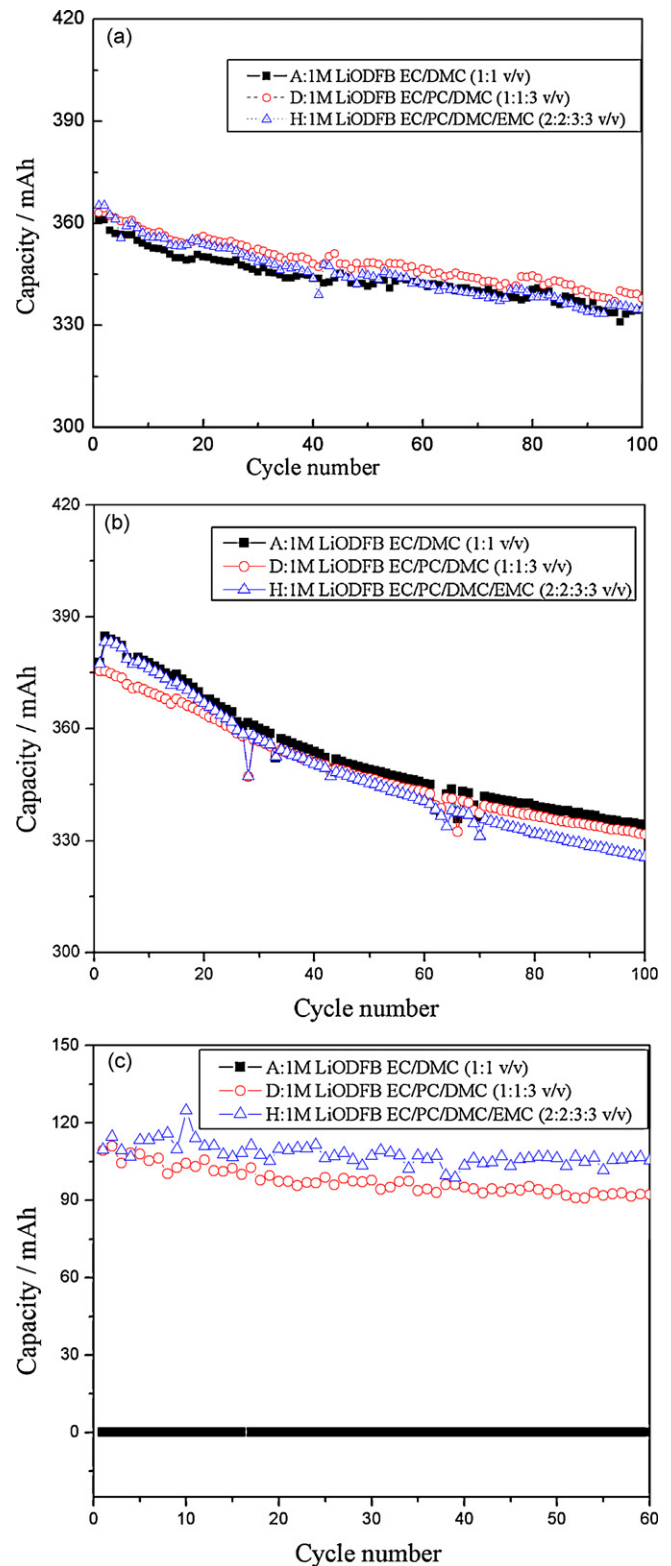

Fig. 8. Cycling performance of the LiFePO$_4$/AG cells with various electrolytes at different temperature: (a) 25 °C, (b) 65 °C, and (c) −10 °C.

excellent rate-capability. For example, at 4 C, the specific discharge capacity was 324.8 mAh, about 89% of the discharge capacity at 0.5 C. The possible reason might be the relatively low SEI resistance of LiFePO$_4$/AG cells with 1 M LiODFB EC/PC/DMC (1:1:3, v/v) electrolyte.
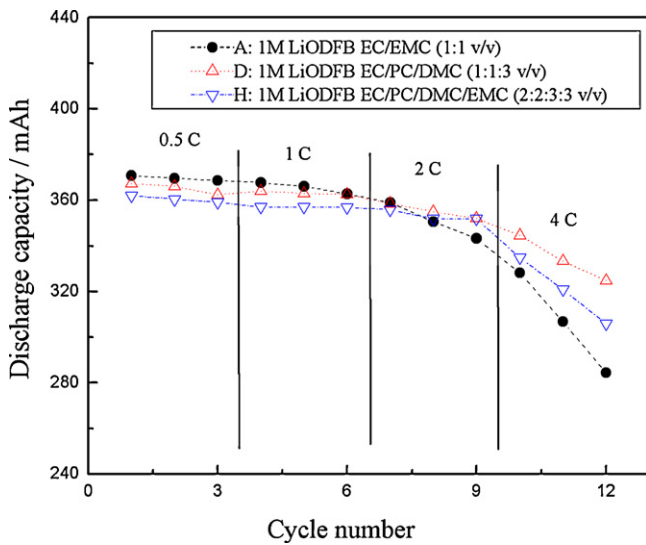
Fig. 9. Capacity retention as a function of C rate of the LiFePO$_4$/AG cells with different electrolytes at 25 °C. (The charge rate was equivalent to the discharge rate for rate capability measurements.)

3.5. Comparison of LiODFB and LiPF$_6$ in EC/PC/DMC (1:1:3, v/v) for LiFePO$_4$/AG cells

In order to further evaluate the LiODFB-based electrolytes for practical application, the electrochemical properties of LiFePO$_4$/AG cells with 1 M LiODFB EC/PC/DMC (1:1:3, v/v) is compared with that with 1 M LiPF$_6$ EC/PC/DMC (1:1:3, v/v).

Table 4 shows that, by contrast, the coulombic efficiency and discharge capacity of LiFePO$_4$/AG cells with LiODFB-based electrolyte are much higher than that of LiFePO$_4$/AG cells with LiPF$_6$-based electrolyte. This clear shows that LiODFB can effectively stabilize graphitic structure in electrolyte containing high concentration of PC, a solvent known to exfoliate the graphene sheets by cointercalation with the Li$^+$ ions [22].

Fig. 10(a) and (b) shows the cycling performances for LiFePO$_4$/AG cells with LiODFB-based electrolyte and LiPF$_6$-based electrolyte at 25 °C and 65 °C, respectively. At 25 °C, all the cells maintained excellent capacity retention against the prolonged cycling. However, when the ambient temperature is 65 °C, the capacity retention is very high, about 88% after 100 cycles, for LiFePO$_4$/AG cells using LiODFB-based electrolyte; while the cell's capacity faded quickly for LiFePO$_4$/AG cells with LiPF$_6$-based electrolyte, more than 50% capacity was lost after 100 cycles. This may be attributed to form more effective, compact and stable surface film in LiODFB-based electrolyte on the surface of both electrodes [23].

Fig. 11 shows that, at high current densities (4 C), the discharge capacity with LiODFB-based electrolyte maintained about 89% of that at 0.5 C; while the discharge capacity of the cell with the LiPF$_6$-
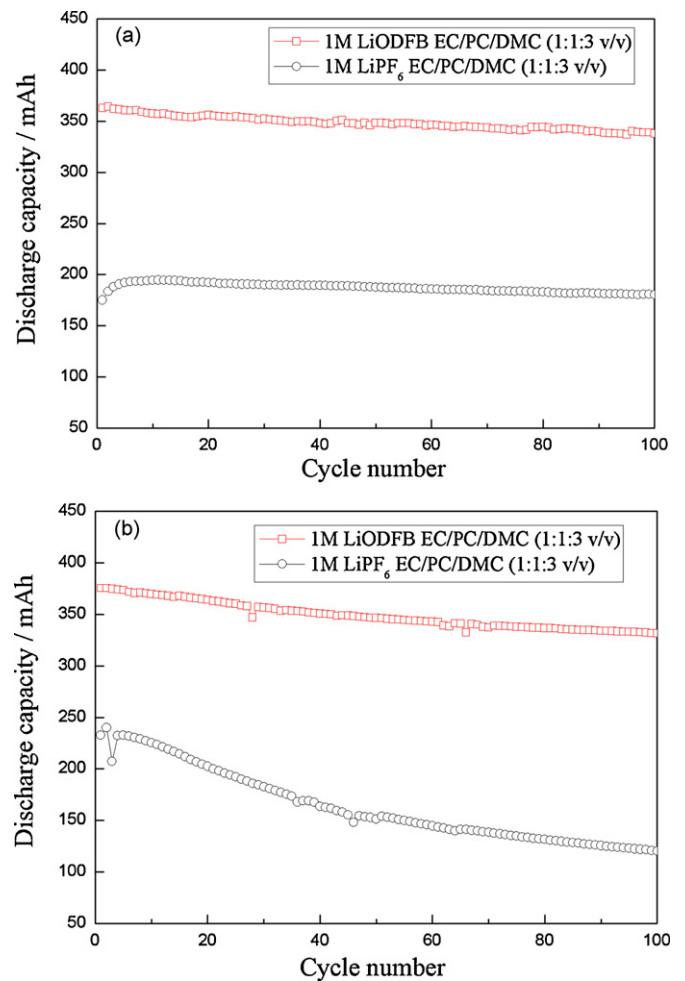

Fig. 10. Comparison of cycling performance in two different electrolytes for the LiFePO$_4$/AG cells at various temperature: (a) 25 °C and (b) 65 °C.

based electrolyte could also maintain well (about 92%). Thus, the rate performance of the cells using LiODFB as lithium salt in electrolyte is similar to the cells that using LiPF$_6$ as lithium salt.

Based on the merits mentioned above, 1 M LiODFB EC/PC/DMC (1:1:3, v/v) could be a promising electrolyte for LiFePO$_4$/AG cells.
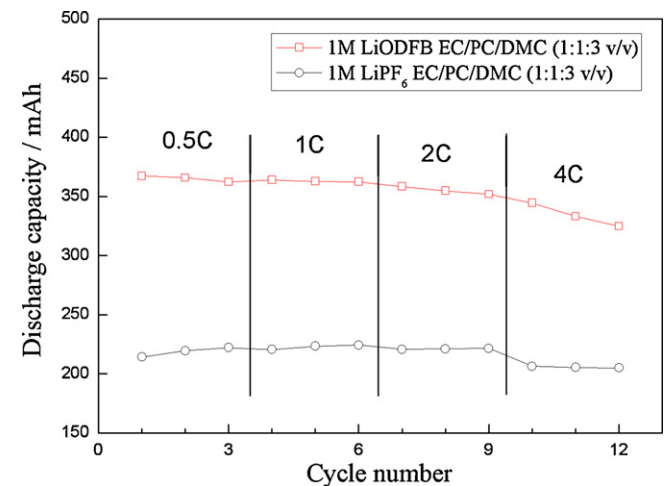

Fig. 11. Comparison of rate capability in two different electrolytes for the LiFePO$_4$/AG cells at 25 °C. (The charge rate was equivalent to the discharge rate for rate capability measurements.)

Table 4
First charge and discharge performances of LiFePO$_4$/AG cells in different electrolytes(charge at 0.1 C; discharge at 0.5 C).

Electrolytes	1st Charge (mAh g^{-1})	1st Discharge (mAh g^{-1})	η_1 (%)
1 M LiODFB EC/PC/DMC (1:1:3, v/v)	530.4	393.2	74
1 M LiPF$_6$ EC/PC/DMC (1:1:3, v/v)	487.5	244.3	50

4. Conclusions

LiODFB/carbonate electrolyte has been systematically studied as promising electrolytes for LiFePO$_4$/AG cells. At room temperature, when 1 M LiODFB EC/PC/DMC (1:1:3, v/v) electrolyte was used, both the LiFePO$_4$/Li and AG/Li half cells show high coulombic efficiency and lower interface resistance, due to its relatively higher conductivity (8.25 mS cm^{-1} at 25 °C) and better ability to form SEI film of low resistance. Further study found that the LiFePO$_4$/AG cells using 1 M LiODFB EC/PC/DMC (1:1:3, v/v) electrolyte have excellent capacity retention at −10 °C, 25 °C and even at elevated temperatures up to 65 °C, and it also has excellent C-rate capability for power applications. Compared with the LiFePO$_4$/AG cells with LiPF$_6$-based electrolyte, the cells in LiODFB-based electrolyte have much higher coulombic efficiency and discharge capacity in the first charge and discharge. At 25 °C, all the cells in these two electrolytes maintained excellent capacity retention against the prolonged cycling. However, at 65 °C, the capacity retention of the cells with LiODFB-based electrolyte was 88% much higher than that of LiPF$_6$-based electrolyte (50%). Furthermore, the rate performance of the cells using LiODFB as lithium salt in electrolyte is similar to the cells that with LiPF$_6$ as lithium salt. So 1 M LiODFB EC/PC/DMC (1:1:3, v/v) is a promising electrolyte for LiFePO$_4$/AG cells.

Acknowledgements

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