



Novel electrolytes based on aliphatic oligoether dendrons

Jinbao Zhao^{a,*}, Yuzo Ishigaki^b, Motoshi Yamanaka^b,
Hiroyuki Fukuda^b, Keigo Aoi^c

^a Battery Development Center, Hitachi Maxell, Ltd., 1-1-88, Ushitora, Ibaraki-shi, Osaka 567-8567, Japan

^b Nagoya Municipal Industrial Research Institute, 3-4-41 Rokuban, Atsuta-ku, Nagoya 456-0058, Japan

^c Graduate School of Biogricultural Sciences, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8601, Japan

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ABSTRACT

A novel electrolyte system having well structurally controlled aliphatic oligoether dendrons with a carbonate core is developed for lithium ion batteries. The synthetic dendrons have high boiling point, much higher dielectric constant up to 7.4–8.7 and better stability against oxidation than the conventional linear carbonates such as DEC. The ionic conductivities of the electrolyte with 80 wt% of dendrons and 20 wt% of LiTFSI are 0.11–0.61 mS cm⁻¹ at 20 °C. A 463443-type prismatic battery having the electrolyte solution with 20 wt% dendrons was prepared and its battery performance such as capacity and cycleability was investigated. The prismatic battery with dendron-based electrolyte has the same level of capacity to that with the conventional carbonate-based electrolyte, and shows good cycleability, suggesting a high possibility to use as a kind of cosolvents for lithium ion batteries.

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1. Introduction

Lithium ion batteries are widely used as power sources for various devices because of their high energy density, high power density, long cycle life and low self-discharge properties compared with the other rechargeable batteries [1]. Most commercial lithium ion batteries adopt liquid electrolytes composed of lithium salts and a mixture of cyclic carbonates such as ethylene carbonate (EC) with linear carbonates such as dimethyl carbonate (DMC), ethylmethyl carbonate (EMC) and diethyl carbonate (DEC). In the electrolyte formulation, lithium hexafluorophosphate (LiPF₆) is used as supporting salt because of its most well-balanced chemical and electrochemical properties among the numerous salts for lithium/lithium ion batteries. EC has large dielectric constant to give high solvation power to lithium salts leading to high ion transport although it has high melting point above room temperature and high viscosity. By contrast, linear carbonates usually are of small dielectric constant, but have low viscosity to promote lithium ion transport. The resultant electrolyte solutions with the above-mentioned mixed-organic carbonates and LiPF₆ salt benefit not only from the melting-temperature suppression of EC but also from the low viscosity of linear carbonates, and offer high ionic conduc-

tivity, stable solid interface with graphite and good voltage stability [2]. This is also the reason why the formulation of the liquid electrolytes based on a mixture of EC with linear carbonates set the main theme for the state-of-the-art lithium ion battery electrolytes [3–5].

However, linear carbonates usually have low boiling point and low flash point and are highly flammable, which are different from cyclic carbonates. The flammable organic solvents are recognized commonly as one of main causes of leakage, fire and explosion of lithium ion batteries, in case of internal short circuit or in other abuse conditions. The efforts aiming to improve the safety properties of the carbonate-based electrolytes for lithium ion batteries under abuse conditions have intensified in recent years. For example, new electrolyte systems with flame-retardant additives were developed with some success to lower the flammability of the liquid electrolytes based on carbonate solvents [6]. Phosphazene derivatives such as hexamethoxycyclotriphosphazene and hexaethoxycyclotriphosphazene [7], and organophosphates such as trimethyl phosphate [8] were investigated to evaluate their thermal stability and performance in Li-ion batteries. Many kinds of novel non-aqueous solvents have also been developed to use as cosolvents in the electrolyte to lower inflammability of electrolytes and to increase battery safety under thermal abuse conditions. Especially fluorinated organic compounds such as fluorinated carbonates [9], fluorinated esters [10] and fluorinated ethers [11], and ionic liquids [12] were extensively studied as potential sol-

* Corresponding author. Tel.: +81 726238140; fax: +81 726239231.

E-mail address: kimbao-cho@maxell.co.jp (J. Zhao).

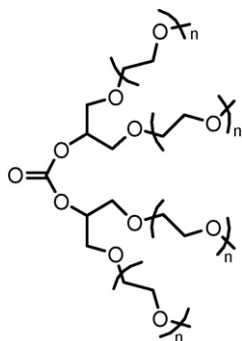


Fig. 1. Molecular structures of dendrons. $n=0-3$ responds to Dendrons A–D.

vents for lithium ion batteries. Like the linear carbonates, none of these solvents were used as single solvents but rather as cosolvents with cyclic carbonates including EC. Furthermore, gel electrolytes are also reported to provide improved safety and alleviate leakage problems compared to the liquid counterpart [13,14]. However, the reduction in flammability with flame-retardant additives, novel solvents and gel polymers has only been achieved at the expenses of the indexes of cell performances such as capacity, rate and reversibility of the cell. It is still necessary to develop novel electrolyte systems with high flash point to reduce the possibility of thermal runaway without degradation of cell performances.

In the present work, well structurally controlled aliphatic oligoether dendrons having a carbonate group were synthesized and examined to use as solvents of electrolyte for lithium ion batteries. Its electrochemical properties such as ionic conductivity and potential window were studied. The battery performance of a 463443-type prismatic battery having the dendron-based electrolyte solutions was also evaluated.

2. Experimental

Aliphatic oligoether dendrons having a carbonate group, showed in Fig. 1, were synthesized via a convergent approach [15,16]. The precursors, they are, biantennary secondary alcohols were prepared from oligoethylene glycol monomethyl ethers and epichlorohydrin. Dendrons A–D were synthesized by reaction of the corresponding secondary alcohol and 1,1'-carbonyldiimidazole followed by Kugel–Rohr distillation or gel filtration process. The structures of dendrons were determined by IR, ^1H NMR and ^{13}C NMR measurements.

Differential scanning calorimetry (DSC) measurement was carried out with a WS-002N (Mac Science Ltd.) under a nitrogen atmosphere. In advance of the measurement, the sample was heated to 120°C , and the data were recorded in the range from 120 to -120°C at a rate of $10^\circ\text{C min}^{-1}$ during cooling process.

The viscosity of dendrons and their electrolytes was measured by SVISCOSTICK (Maruyasu Industries Co., Ltd.) in the temperature range from 10 to 60°C with a 1°C interval of temperature under a nitrogen atmosphere.

Ionic conductivity of the electrolytes was investigated by a conventional complex impedance method. The impedance measurement was carried out with the Solartron SI1287 electrochemical interface (Scribner Associate, Inc.) with 1255B frequency response analyzer in the frequency range from 0.1 Hz to 100 kHz. The ionic conductivity was measured in the temperature range from -20 to 70°C with a 10°C interval of temperature during storing the cell at the every measured temperature for 3 h. The sample cells for ionic-conductivity measurement were constructed with a pair of parallel platinum plate electrodes with a 0.50 -mm Teflon spacer.

Moreover, a three-electrode model cell with a platinum plate or SUS plate as a working electrode and metallic Li as counter and reference electrode was applied for cyclic voltammetry measurements. The experiments were also performed on the above-mentioned Solartron SI1287 electrochemical interface. All of sample cells were assembled in a glove box filled with dry Ar gas.

A 463443-type prismatic battery having an external size of 4.6 mm depth (thick), 34 mm width and 43 mm height was employed to check the battery performance. The prismatic battery with a LiCoO_2 positive electrode and a natural-graphite negative electrode was prepared as follows. A positive electrode with 94 wt% LiCoO_2 , 3 wt% carbon black and 3 wt% polyvinylidene fluoride, and a negative electrode with 98 wt% natural-graphite and 2 wt% binder were coated in advance on the 15 μm aluminum foil and 8 μm copper foil, respectively, and were dried at 120°C under vacuum for 11 h and then the resulting electrodes were pressed into the required sizes for battery preparation. A pair of the above-mentioned positive electrode and negative electrode was wound up in spiral via 20 μm microporous polyethylene separator. The resultant spiral electrode assembly was inserted in a rectangular battery case of aluminum base alloy having an external size of 4.6 mm depth (thick), 34 mm width and 43 mm height, and then a non-aqueous electrolytic solution was poured into the cell and sealed by laser system. The cell was charged for formation and was stored at 60°C overnight before use for evaluation of its battery performance. With respect to the non-aqueous secondary battery, when charging was terminated upon reaching to 4.2 V, its nominal capacity is 700 mAh. In the prismatic battery, 1.0 mol dm^{-3} (1.0 M) LiPF_6 electrolyte solution of EC/Dendron A/DEC ($1:1:3$, w/w/w) with 2.5 wt% vinylene carbonate was applied. For comparison, the electrolyte solution with a tertiary mixture of EC/EMC/DEC ($1:1:3$, w/w/w) was used, where Dendron A in the electrolyte was replaced by EMC with low flash point.

3. Results and discussion

3.1. Properties of dendrons

The synthetic aliphatic oligoether Dendrons A–D shown in Fig. 1 have a well-controlled molecular structure with a carbonate core and four oligoether branches like a dendrite. They are unimolecular organic compounds in spite of high molecular weight, different from polymers and conventional oligoethers that are mixture consisting of congeners with various molecular weight. All of the dendrons synthesized in the present work are liquid at ambient temperature.

The properties of Dendrons A–D were measured and the results were shown in Table 1. It was found that no boiling point was observed for Dendrons A–D at atmospheric pressure under their decomposition temperature, indicating their boiling point be much higher than that of DEC (127°C) and poly(ethylene glycol) dimethyl ether with an average molecular weight $M_n = 500$ (PEG500). Even in high vacuum condition, the boiling point of dendrons was still high. For example, the boiling point of Dendron C was up to 270°C at 0.10 Torr. Purification of Dendron D by Kugel–Rohr distillation was attempted under reduced pressure over 280°C in vain and thus purified by gel filtration chromatography. The higher boiling point may result in safer occurrence for lithium ion batteries.

Specific dielectric constant of Dendrons A–D was in the range from 7.4 to 8.7 , much higher than 2.8 of DEC and very close to 8.9 of PEG500, which was expected that dendrons have high solvation power to lithium salts and was necessary to for a solvent to use in the electrolyte for lithium ion batteries. In fact, we found the dendrons had excellent miscibility with LiPF_6 and other lithium

Table 1
Properties of aliphatic oligoether dendrons

Dendron	Molecular weight	Density ^a (g cm ⁻³)	Boiling point (°C mmHg ⁻¹)	Dielectric constant ^a
A	266.29	1.10	79/0.03	7.4
B	442.50	0.10	195/0.03	8.4
C	618.71	0.10	270/0.1	8.7
D	794.92	1.11	ND ^b	8.7
PEO500	500 ^{c,d}	1.05 ^c	ND ^b	8.9
DEC	118.13	0.98	127/760	2.8

^a 23 °C.

^b Unmeasurable.

^c Reagent catalog value.

^d Number average molecular weight.

salts, and were miscible with organic non-polar materials, especially with carbonate-based solvents at any ratio.

The viscosity of the dendrons was over 40 mPa s at 23 °C, much higher than that of the current linear carbonate. Their viscosity was largely depended on molecular weight and the larger the molecular weight was, the higher the viscosity of dendrons was, indicating that high viscosity of dendrons was due to their high molecular weight. DSC analysis showed that the dendrons only have a glass transition point in the range of -107 to -86 °C without freezing point, whereas PEO500 indicated an exothermic peak derived from its freezing at 5 °C. It is suggested that the dendrons are liquid in a wider temperature range (Fig. 2). On the other hand, the temperature dependence of viscosity of dendrons was also investigated and the results were shown in Fig. 3. It was apparent from Fig. 3 that the viscosity of dendrons was strongly depended on temperature, especially the increase of viscosity became large below 30 °C.

3.2. Ionic conductivities of the electrolyte with dendrons

It has been known that the ionic conductivity of electrolyte is mainly governed by the number of carrier ions and their mobility. The former was influenced by dielectric constant of electrolyte solutions and the dissociation constant of lithium salt. And the latter was related to viscosity of the electrolyte. An electrolyte solution with 80 wt% of dendrons and 20 wt% of lithium bis(trifluoromethanesulfonyl) imide (LiTFSI) was prepared to investigate the ionic conductivity of dendron-based electrolytes. The reason why LiTFSI was used in the dendron-based electrolyte is that LiTFSI has high thermal stability and low sensitivity toward hydrolysis by trace amount of moisture in non-aqueous solvents, which is unlike LiPF₆. The salt also has highly delocalized anionic charge and the relatively high ionic conductivity, reflecting its higher dissociation constant [17].

The temperature dependence of the bulk ionic conductivity for dendron-based electrolyte was measured by a complex impedance method and was calculated from IR drop (Fig. 4). The ionic conductivities of the electrolyte with dendrons were 0.11–0.61 mS cm⁻¹ at 20 °C, much lower than that of DEC and PEG500. Low ionic conductivity of the dendron-based electrolyte was related to its high viscosity. Fig. 4 showed that the ionic conductivity decreased in the sequence of Dendron **A**, Dendron **B**, Dendron **C** and Dendron **D**, suggesting the lower the molecular weight of the dendrons was, the higher the ionic conductivity of the electrolyte was. The dependence of ionic conductivity on molecular weight corresponded to the dependence of electrolyte viscosity on molecular weight of the dendrons. It was reasonable that the temperature dependence of viscosity and ionic conductivity of the dendron-based electrolyte was derived from the relatively high molecular weight, dendritic molecular structure, the interaction between salt and dendrons, and the intermolecular and intramolecular interactions of dendrons. Furthermore, the observed curvature in Fig. 4 showed that the electrolyte with dendron obeyed Vogel–Tamman–Fulcher theory, an empirical equation concerning the temperature dependence of ionic conductivity for amorphous electrolyte [18].

DSC analysis showed that the dendron-based electrolytes only have a glass transition point below -60 °C without freezing point, whereas PEO500-based electrolyte had an exothermic peak derived from its freezing at -3.4 °C. It was apparent that the dendron-based electrolytes were liquid in a wider temperature range from -60 to 120 °C, alike to dendrons without lithium salt (Fig. 5). There are little differences between the electrolytes with Dendrons **A–D** in DSC profiles.

The dissociation behavior of lithium salt in the dendron-based electrolyte and the reductive behavior of dendrons were examined by cyclic voltammetry within the voltage range of -0.5 to 3.0 V versus lithium. Fig. 6 showed cyclic voltammograms of SUS/lithium

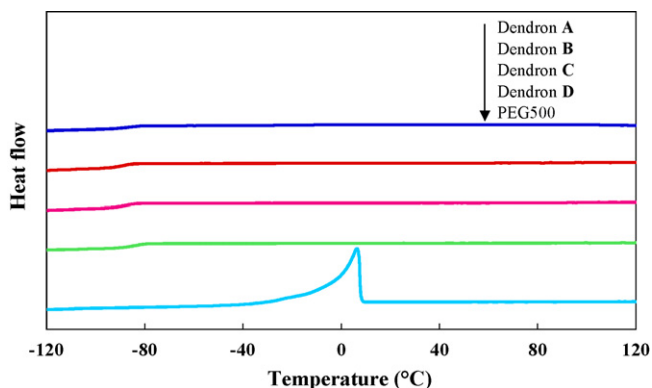


Fig. 2. DSC profiles of Dendrons **A–D** and PEG500 (from upper to lower). All data were taken in the cooling process from 120 °C at a scan of 10 °C min⁻¹.

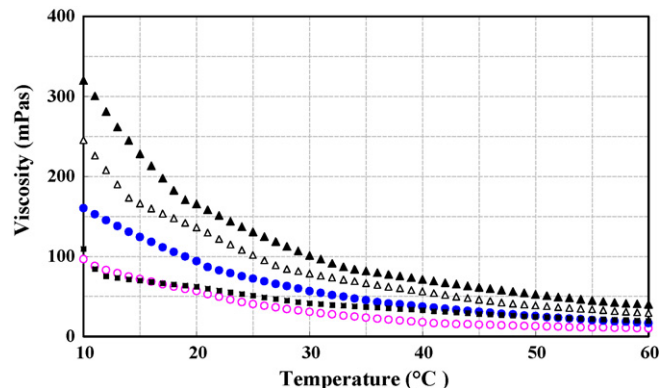


Fig. 3. Temperature dependence of viscosity of Dendron **A** (○), Dendron **B** (●), Dendron **C** (△), Dendron **D** (▲) and PEG500 (■).

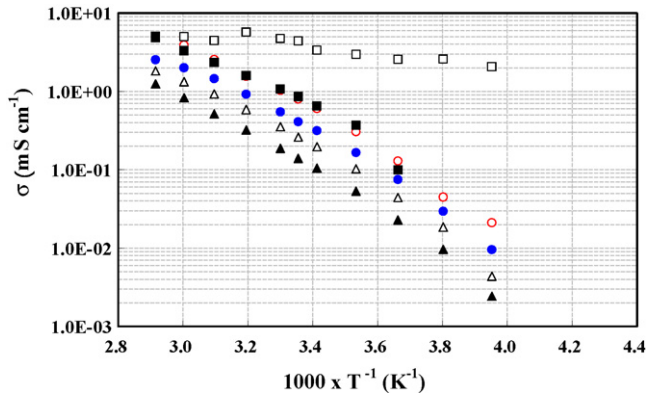


Fig. 4. Arrhenius plots of the ionic conductivities for the electrolytes composed of 20 wt% LiTFSI and 80 wt% Dendron A (○), Dendron B (●), Dendron C (△), Dendron D (▲), DEC (□) and PEG500 (■).

metal having the electrolyte with 80 wt% of Dendron A or DEC and 20 wt% of LiTFSI, where DEC as a single solvent was used for comparison. It was found that there was a pair of strong reduction peak below 0V and strong oxidation peak at approximately +0.51 V existed. The reversible peaks corresponded to reductive deposition and oxidative dissolution of lithium, respectively, reflecting the reduction and oxidation of lithium easily occurred in the dendron-based electrolyte during potential scan. No significant differences between Dendron A and DEC were found in the cyclic voltammograms, suggesting Dendron A has good stability against reductive decomposition.

The possibility of the oxidation decomposition of the LiTFSI-based electrolyte solution with single solvent was also confirmed by cyclic voltammetry. As cyclic voltammograms from Fig. 7 showed, on the platinum surface, the electrolyte with DEC was found to be oxidized from 4.0 V versus lithium, while the electrolyte with Dendron A remained stable up to 5.3 V at a potential scan of 5 mV s^{-1} , suggesting Dendron A has excellent stability against oxidation. The other dendrons showed the same electrochemical behaviors as Dendron A.

3.3. Battery performance with dendron-based electrolyte

In a practical lithium ion battery, electrolyte (solvents and salts), facing the challenges from both cathode and anode, are easier to be oxidized and reduced to result in decomposition, because of highly catalytic surface of lithium metal oxide and highly crystalline graphite. In order to investigate the possibility of the dendron

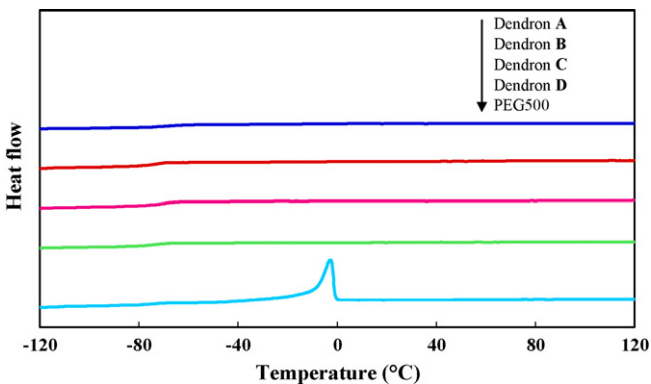


Fig. 5. DSC profiles of the electrolyte composed of 20 wt% LiTFSI and 80 wt% single solvent. Curves from upper to lower responded to Dendrons A–D and PEG500. All data were taken in the cooling process from 120°C at a scan of $10^\circ\text{C min}^{-1}$.

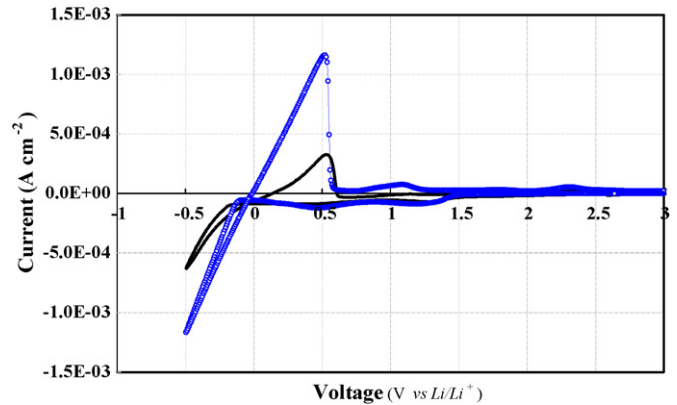


Fig. 6. Cyclic voltammograms of SUS/electrolyte/Li with Dendron A (○) and DEC (solid line). The electrolyte composed of 20 wt% LiTFSI and 80 wt% single solvent and the experiment was carried out at a potential scan of 5 mV s^{-1} from 3.0 to -0.5 V vs. Li at 23°C .

compounds as a solvent for lithium ion batteries, a 463443-type prismatic battery consisting of LiCoO_2 positive electrode, natural-graphite negative electrode was employed. Due to high viscosity and high dielectric constant of dendrons, the dendron-based electrolytes free of linear solvents with low viscosity were not able to wet well the polyolefin separator such as polyethylene porous film and not to soak into electrodes completely, which generally results in worse battery performance. Thus, dendrons as a single solvent or as a cosolvent in binary electrolyte with EC were difficult to be free of linear carbonates for lithium ion batteries at the present point. In this work, instead of entirely replacing linear carbonates, a tertiary electrolyte of EC/Dendron A/DEC (1:1:3, w/w/w) with 1.0 M LiPF_6 was used in the prismatic battery. In the electrolyte formulation, Dendron A was used to replaced EMC with low boiling point (109°C) and low flash point (ca. 20°C) in the commercial electrolyte with EC/EMC/DEC. To improve the properties of solid electrolyte interface on the graphite anode, 2.5 wt% of vinylene carbonate was added into the above electrolyte solutions.

463443-type prismatic battery with 20 wt% of Dendron A and 20 wt% of EMC was fully charged and then was discharged at 23°C for its capacity check. The fully charged regime was as follows, charging the cell at 1 C (700 mA) to 4.2 V and following a charge at 4.2 V. The total charge time was 2.5 h. A pair of discharge curves, typical of the performance of 463443-type prismatic battery at 23°C , was showed in Fig. 8. The prismatic battery with 20 wt% of Dendron A delivered the discharge capacity of 710 mAh, close to 716 mAh of the battery with the tertiary electrolyte with EMC

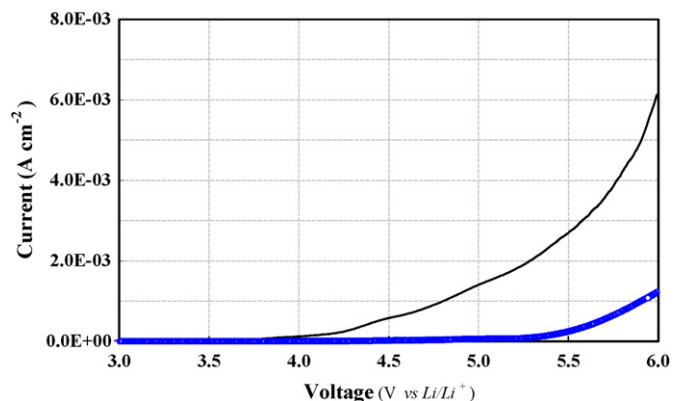


Fig. 7. Cyclic voltammograms of Pt/electrolyte/Li with Dendron A (○) and DEC (solid line). Test was carried out at the same condition as Fig. 6 from 3.0 to 6.0 V vs. Li.

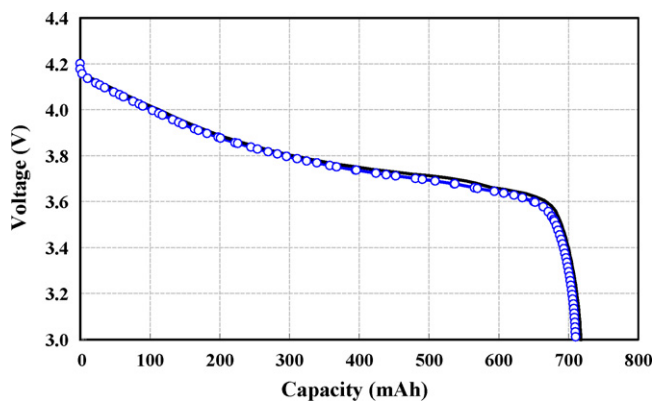


Fig. 8. Typical discharge curves of 463443-type prismatic battery with EC/Dendron A/DEC (1:1:3, w/w/w) (○) and EC/EMC/DEC (1:1:3, w/w/w) (solid line) at a discharge of 0.2 C (140 mA) to 3.0 V. The cell was charged at 700 mA to 4.2 V and then was charged at 4.2 V at 23 °C. The total charge time was 2.5 h.

when discharged to 3.0 V at a current of 0.2 C (140 mA). Its coulombic efficiency of the initial charge–discharge cycle was 89.4%, also the same level of that of the battery with the tertiary electrolyte with EMC (89.5%). The impedance of the prismatic battery at fully charged state was also measured and the resultant Nyquist plots of impedance spectra were shown in Fig. 9. The interceptions of Nyquist plots in Fig. 9, a kind of Ohmic polarization (IR drop), are related to the ion conductivity in the bulky electrolytes of the prismatic battery. It was observed that the IR drop of the cell with Dendron A was 57 mΩ, about 11 mΩ larger than that of EMC-based cell. It was apparent from Fig. 8, the low operation voltage of the cell with dendron-based electrolyte even at the low rate discharge was mainly related to the relatively high internal resistance. The inset of Fig. 9 showed the impedance spectra that were calculated by subtracting the interception (IR drop) from Nyquist plots, related to the ion transport in the surface film and the Faradaic process. It was found from the inset of Fig. 9, the resistances corresponding to the ion transport process in the surface film and the charge-transfer process between electrode and electrolyte were same in the cells with dendron-based electrolyte and EMC-based

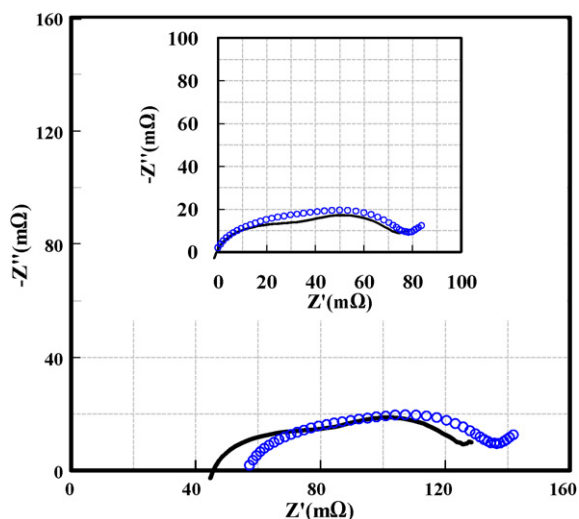


Fig. 9. Nyquist plots of impedance spectra for the fully charged 463443-type prismatic battery in the frequency range from 0.1 Hz to 100 kHz at 23 °C, in which the inset shows the impedance spectra that were calculated by subtracting the interception from the original Nyquist plots. Electrolytes are 1 M LiPF₆ with EC/Dendron A/DEC (1:1:3, w/w/w) (○) and EC/EMC/DEC (1:1:3, w/w/w) (solid line).

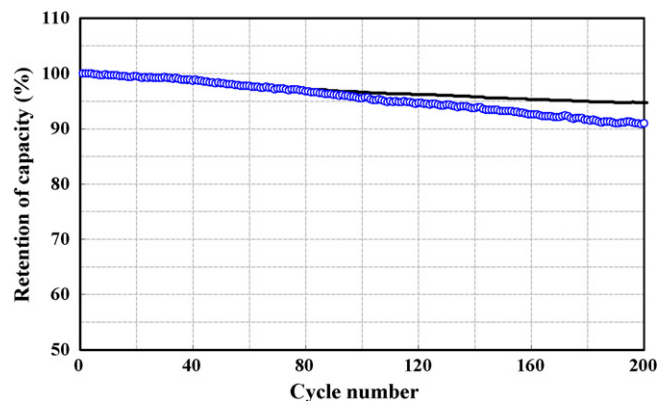


Fig. 10. Dependence of capacity retention on cycle number for the prismatic battery with EC/Dendron A/DEC (1:1:3, w/w/w) (○) and EC/EMC/DEC (1:1:3, w/w/w) (solid line). The cell was charged to 4.2 V at 350 mA for 2.5 h and discharged to 3.0 V at a 350 mA at 23 °C.

electrolyte. Since the resistances corresponding the charge-transfer process and the ion transport in the surface reflected the combination of these corresponding processes on both anode/electrolyte and cathode/electrolyte interfaces, it was difficult to interpret and characterized the impedance spectra clearly. But by comparing the impedance spectra of the prismatic battery, significant differences existed. It was suggested that Dendron A was also associated with the above-mentioned processes.

The cycleability of the prismatic battery was also measured at 23 °C. Fig. 10 showed the dependence of capacity retention on cycle number for the prismatic battery. In this case, the cell was charged to 4.2 V at 0.5 C (350 mA) for 2.5 h and discharged to 3.0 V at 0.5 C to give one cycle, and then the repetitive charge and discharge cycle was carried out. It was noted that the cycleability of the cell with Dendron A was good and its capacity retention was 91% at the 200th cycle, despite of less than 94% of the cell with EMC.

These results suggest that the Dendron A is not oxidized in the lithium ion battery with LiCoO₂ positive electrode and natural-graphite negative electrode, and that there is the high possibility to use as a kind of cosolvents for lithium ion batteries.

4. Conclusions

Four kinds of well structurally controlled aliphatic oligoether dendrons having a carbonate group were synthesized and examined to use as solvents of electrolyte for lithium ion batteries. The synthetic dendrons had high boiling point, much higher dielectric constant up to 7.4–8.7 and better stability against oxidation than linear carbonates such as DEC. Due to high viscosity, the ionic conductivities of the electrolyte with dendrons were 0.11–0.61 mS cm⁻¹ at 20 °C. The 463443-type prismatic battery having the electrolyte solution with 20 wt% of dendrons had the same level of capacity to that with the conventional carbonate-based electrolyte, and showed good cycleability, suggesting the high possibility to use as a kind of cosolvents for lithium ion batteries.

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