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Benzimidazole-derived anion for lithium-conducting electrolytes

Leszek Niedzicki^{a,*}, Piotr Oledzki^a, Anna Bitner^a, Maria Bukowska^a, Przemysław Szczecinski^a ^aFaculty of Chemistry, Warsaw University of Technology, Noakowskiego 3, 00664 Warsaw, Poland * Corresponding author. E-mail: lniedzicki@ch.pw.edu.pl

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Abstract

lithium In this work we announce new salt of 5,6-dicyano-2-(trifluoromethyl)benzimidazolide (LiTDBI) designed for application in lithium conductive electrolytes. It was synthesized and completely characterized by NMR techniques. Studies show salt's thermal stability up to 270°C and electrochemical stability in liquid solvents up to +4.7 V vs. metallic lithium anode. Basic characterization of electrolytes made with this salt show conductivity over 1 mS cm⁻¹ and unusually high transference number at high concentrations (0.74 in EC:DMC 1:2 ratio mixture) along with low onset of conductivity peak. As a final proof of concept, cycling in half-cell was performed and electrolyte based on LiTDBI showed perfect capacity retention. Such properties show remarkable progress in creating efficient lithium-conducting electrolytes with use of weakly-coordinating anions.

1. Introduction

Although present in the market for a long time already, lithium-ion cell is still the energy storage technology of the fastest advancement nowadays. The momentum of that advancement is development of better and better electrode materials, which increases energy density of cells. Electrolyte and processes that are consequence of its contents are the main factors limiting applications of newly developed electrodes. Despite that, new anions for electrolytes in lithium-ion cells are rarely covered by scientific research. Most of the works is focusing on investigation of well-known salts, when their disadvantages are many. LiPF₆ – the most widely used salt in Li-ion batteries – is known to be subject to hydrolysis, forming caustic HF and toxic POF₃ [1] as well as to have poor thermal stability [2-3]. Other salts, notably: LiClO₄, LiAsF₆, LiCF₂SO₃ (LiTf), LiN(SO₂CF₃)₂ (LiTFSI), LiN(SO₂C₂F₅)₂ (LiBETI), LiPF₃(C₂F₅)₃ (LiFAP), LiB(C₂O₄)₂ (LiBOB), LiBF₂(C₂O₄) (LiDFOB), LiBF₄, even mixtures of those [4] and other borates, including oxyethylene derivatives [5], were tried as lithium-ion cell components. Unfortunately, all failed to enter the wide application for various reasons, including, but not limited to: low conductivity (LiTf) [6], explosiveness (LiClO₄) [7], corrosiveness towards other cell components (LiFSI, LiBETI) [8], forming too thick/blocking SEI (solid electrolyte interface) (LiBF₄) [9], obstructive toxicity (LiAsF₆) and too high manufacturing cost (LiFAP) [10-11]. Many salts have been synthesized and tested, but never got to commercial availability.

In the recent past, our group have proposed new anions for lithium salts for electrolytes' applications [12]. TDI. PDI and HDI (4,5-dicyano-2-(perfluoroalkyl)imidazoles) were designed for unification of charge distribution by symmetry and possibility of numerous tautomeric forms occurrence. These designed structures benefitted also from lack of bulky anions disadvantages, such as higher viscosity of their solutions. They also displayed smaller affinity to form agglomerates, which influence ionic conductivity of electrolyte [13]. TDI anion design is beneficial in lithium-ion cell applications, as proven by independent groups [14-15]. High lithium cation conductivity (product of conductivity and lithium cation transference number) [16] and high thermal and electrochemical stability (up to 260°C and 4.7 V vs Li, respectively) are especially worth mentioning [12].

Few years ago, new modeling studies were published that were focusing on speculation over new proposals on anions for lithium-ion cells electrolytes. As far as simulations can predict, imidazole and benzimidazole derivatives should be of the special interest for such purpose [17]. Weakly coordinating anions need to comprise electronwithdrawing groups, as well as possess stable skeleton. Such skeleton have to distribute anion's charge uniformly. As a result, lithium cation should have possibly low dissociation energy. Benzimidazole and imidazole derivatives fulfill all of these requirements. Imidazole derivatives path has been already pursued by us. They were synthesized and investigated as electrolytes. This previous research of our own leading to the LiTDI success confirmed that such direction of investigation into weakly coordinating anions has a great potential. Likewise, recent work of independent group has shown applicability of weakly coordinating anions concept in Li-ion batteries with LiTDI as the best example [18,19]. As a conclusion to a few of these modeling works [17,20], benzimidazoles are of the special interest and deserve more attention. Thus, in this paper we follow this modelling suggestion, of the first anion of this family, 5,6-dicyano-2presenting properties (trifluoromethyl)benzimidazole.

New TDBI anion presented in this paper is the conceptual continuation of the idea which brought TDI to life. Benzimidazole skeleton with electron-withdrawing groups provide even more mesomeric forms stabilizing the anion. Better uniformity of the charge distribution takes place in such moiety. On the other hand, size of the anion is not big enough to influence viscosity to a large extent and as such, does not substantially diminish electrolyte's conductivity. Theoretical studies by Scheers *et al.* [17] have shown that such structure should manifest low ion pair dissociation energy – almost identical to quite successful LiTDI. In this paper we describe synthesis of the lithium salt of TDBI anion. Furthermore, salt's physicochemical and electrolytes' basic electrochemical characterization is provided. To show the proof of concept, we employed model battery-like solvent, like propylene carbonate (PC), and a typical battery solvent mixture, namely ethylene carbonate and dimethyl carbonate (EC:DMC) in a 1:2 ratio. We also test the salt for its thermal and electrochemical stability as well as cycle the salt in a half-cell.

2. Experimental

2.1. Experimental techniques

Nuclear magnetic resonance (NMR) spectra were recorded on Varian Gemini 500. Samples for NMR experiments were dissolved in deuterated dimethyl sulfoxide (DMSO-d₆, 99.96 atom % D, Aldrich). ¹H and ¹³C chemical shifts are reported relative to DMSO-d₆.

Thermogravimetric Analysis (TGA) was carried out under argon atmosphere on TA Instruments Q50 thermogravimetric analyzer. Heating rate was equal to 10 K min⁻¹.

All samples for measurements were assembled in the argon-filled drybox with moisture level below 1ppm. Prior to the assembly, the salt was vacuum-dried for 48 hours at 120°C. Solvents (propylene carbonate (PC), ethylene carbonate (EC), dimethyl carbonate (DMC)) were anhydrous and used as provided by Sigma-Aldrich (water content <20 ppm for PC and DMC, <50 ppm for EC).

Linear sweep voltammetry (LSV), cyclic voltammetry (CV), transference number, ionic conductivity and half-cell cycling measurements were carried out on VMP3 multichannel potentiostat (Bio-Logic Science Instruments) with frequency response analyzer option. All electrochemical experiments except for the conductivity measurements were performed at ambient temperature.

In case of LSV and CV measurements were carried out in three-electrode Li | electrolyte | Pt system (with lithium metal as reference electrode). LSV scan rate was 10 mV s^{-1} . CV scan rate was 5 mV s^{-1} .

For ionic conductivity measurements electrochemical impedance spectroscopy (EIS) was employed and samples were thermostated for at least one hour at each temperature in Haake D50 cryostat in the -20 to $+50^{\circ}$ C temperature range with the 10° C interval and a precision of 0.05° C.

Lithium cation transference number was determined using standard Bruce-Vincent-Evans method [21] using the following equation: $T_+ = (I_s (\Delta V - I_0 R_0)) / (I_0 (\Delta V - I_s R_s))$, where ΔV is the polarization voltage equal to 20 mV; I_0 and I_s are the initial and the steady-state current during said polarization, respectively; R_0 and R_s are resistances of the solid electrolyte interface (SEI) immediately before and after the polarization, respectively. The Li | electrolyte | Li cells were used for transference number experiments. Electrochemical impedance spectroscopy (EIS) used to obtain R_0 and R_s has been performed with 5 mV amplitude over the 500 kHz-100 mHz frequency range with 10 points per decade. At least three samples were measured for each electrolyte composition for more consistent data. Detailed description of this method can be found in other papers [22].

Charge-discharge half-cell cycling used the Li | electrolyte | graphite cell. Standard commercial single-coated graphite electrode from MTI-XTL was used in that experiment. Custom-made coin cell-type was used in which electrodes and separator of disk shape are sandwiched in the polypropylene tube between stainless steel punches. Cycling voltage was set in the 0.1-2.6 V range. Current was chosen in such a way that both discharge and charge processes would take 5 hours (C/5 rate). Half-cell contained 0.7 mol kg⁻¹ LiTDBI in EC:DMC (1:2 weight ratio) electrolyte composition. Additional measurement with the same conditions and the same cell arrangement was made for investigation of rate capability of an electrolyte.

2.2. Synthesis of lithium 5,6-dicyano-2-(trifluoromethyl)benzimidazolide

Synthesis scheme is presented in Figure 1.



Fig. 1. Synthesis scheme for the lithium 5,6-dicyano-2-(trifluoromethyl)benzimidazolide (LiTDBI) salt described in the text.

25 g (0.22 mol) of trifluoroacetic acid (>99%, Sigma-Aldrich) precooled with waterice mixture was mixed with 2 g (0.0127 mol) of 1,2-diamino-4,5-dicyanobenzene (>97%, Chemical-Block) under the argon atmosphere and kept under reflux for 8 hours (reaction progress controlled by TLC). Upon vacuum evaporation to dry mass, the dark residue was recrystallized from acetonitrile. 2.2 g of 5,6-dicyano-2-(trifluoromethyl)benzimidazole were obtained (74% yield). T_m 297-299°C – lit [23] (different synthesis route).

¹H NMR (DMSO-d₆): 8.65 (s, 2H, 2x C**H**), 15.14 (bs, 1H, N**H**).

¹³C NMR (DMSO-d₆): 145.6 (q, C-CF₃, J(C,F) = 40.1 Hz), 139.8 (s, 2C, C=C shared by both rings), 124.5 (s, 2C, C-CN), 118.3 (q, CF₃, J = 271.6 Hz), 116.4 (s, 2C, CH), 108.8 (s, 2C, CN).

¹⁹F NMR (DMSO-d₆): one peak (not calibrated).

1.7 g (7.2 mmol) of 5,6-dicyano-2-trifluoromethylbenzimidazole was mixed with 0.4 g of LiOH·1H₂O (30% excess) in 5 ml of water. Subsequently, activated carbon was added to this solution which was stirred together for ca. 10 minutes. Activated carbon was removed by filtering and resulting clear solution was evaporated under vacuum (water removal) to obtain dry mass. Residue was dissolved in dry acetonitrile, the resulting solution was filtered and concentrated to small volume. Afterwards, a small amount of benzene was added and solution was left in refrigerator for crystallization. 1.24 g of lithium salt (71% yield) were obtained after vacuum drying.

¹H NMR (DMSO-d₆): 8.22 (s, 2H, C**H**).

¹³C NMR (DMSO-d₆): 156.9 (q, C-CF₃, J(C,F) = 34.2 Hz), 147,9 (s, 2C, C=C shared by both rings), 125.1 (s, 2C, C-CN), 121.7 (q, CF₃, J = 271.2 Hz), 118.7 (s, 2C, CH), 102.5 (s, 2C, CN).

¹⁹F NMR (DMSO-d₆): one peak (not calibrated).

¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of LiTDBI can be obtained as a supporting information to this paper.

3. Results and discussion

First, the salt was tested for its stability. In order to do that, TGA (thermogravimetric analysis) was employed. Obtained results show (available as a supporting information to this paper) decomposition onset as high as 272° C. This is much better result than for the industry standard, LiPF₆ [2]. It is also similar to its predecessor, LiTDI (256°C). Contrary to LiPF₆, LiTDBI is stable as water solute – it is not a subject to hydrolysis. This means lower requirements for handling and storage of LiTDBI comparing to LiPF₆, which is susceptible to moisture even in trace amounts. Thus, toxic products of hydrolysis, as is in case of LiPF₆, are omitted as well.

Stability of the electrolyte is one of the key important properties in terms of applicability of these salts in lithium-conducting electrolytes. LSV plot of 0.1 mol kg⁻¹ LiTDBI solution in PC is pictured on Figure 2. As it can be seen, LiTDBI salt exhibits stability up to 4.75 V vs. Li. This is more than enough for all kinds of contemporary electrode materials. CV plot of 0.1 mol kg⁻¹ LiTDBI solution in EC:DMC is available as a supporting information to this paper.



Fig. 2. Linear sweep voltammetry of the Li | 0.1 M LiTDBI-PC | Pt system with a lithium metal as the reference electrode.



Fig. 3. Dependence of ionic conductivity on temperature for wide range of the LiTDBI concentration in a propylene carbonate: 0.3 (\Box), 0.2 (\Diamond), 0.1 (Δ), 0.05 (\circ), 0.02 (\times), 0.01 (+), 0.005 (**n**), 0.002 (**\diamond**), and 0.001 (**\Delta**) mol kg⁻¹.

LiTDBI ionic conductivity investigation was performed in model solvent (PC) and standard battery solvent mixture (EC:DMC in 1:2 weight ratio). Results presented in Figure 3 for propylene carbonate solutions show the temperature dependence of ionic conductivity of PC-based solutions of LiTDBI. Solutions were prepared in wide range of concentrations – from 0.001 mol kg⁻¹ to 0.3 mol kg⁻¹ which was the maximum solubility of LiTDBI in propylene carbonate. Conductivity increases monotonically with the concentration in the whole investigated range. The highest conductivity at 20°C is in the order of 10^{-3} S cm⁻¹ (0.81 mS cm⁻¹) and is shown by the 0.3 mol kg⁻¹ solution.

Figure 4 shows temperature dependence of ionic conductivity of LiTDBI solution in EC:DMC mixture (1:2 weight ratio). Investigated solutions were in the range from 0.001 mol kg⁻¹ to 0.7 mol kg⁻¹ (maximum LiTDBI concentration). Ionic conductivity at 20°C is the highest in case of 0.2 mol kg⁻¹ solution, although electrolyte of 0.7 mol kg⁻¹ concentration exhibited almost the same conductivity value. Conductivity of the former is equal to 1.35 mS cm⁻¹ and in the case of latter 1.27 mS cm⁻¹. Concentration range



Fig. 4. Dependence of ionic conductivity on temperature for wide range of the LiTDBI concentration in a mixture of ethylene carbonate and dimethyl carbonate (1:2 weight ratio): 0.7 (\Box), 0.5 (\Diamond), 0.2 (Δ), 0.1 (\circ), 0.05 (\times), 0.02 (+), 0.01 (\blacksquare), 0.005 (\diamond), 0.002 (\blacktriangle) and 0.001 (\bullet) mol kg⁻¹.

of electrolytes showing conductivities above 1 mS cm⁻¹ at the room temperature starts at the 0.2 mol kg⁻¹.

Similarity of both conductivity plots (for PC and EC:DMC) can be seen in similar leaps in conductivity values (shown on the Figure 5). Both have this sudden changes (visibly bigger increase in conductivity than for other intervals) in conductivity between 0.02 and 0.05 mol kg⁻¹ concentrations as well as between 0.002 and 0.005 mol kg⁻¹. Monotonically increasing activation energy of the conductivity process from 0.02 mol kg⁻¹ to the maximum conductivity in each solvent is another notable similarity. The difference is that in case of PC solutions energy activation of conductivity for lower concentrations is on the similar level as for the 0.02 mol kg⁻¹ and in case of EC:DMC mixture it is the minimum and it is increasing towards lower concentration. Such change in conductivity and activation energy might be connected to change of conductivity mechanism. It is possible, that at this concentration level associates (triplets) presence starts to be non-negligible, giving its input to the higher conductivity, but also increasing activation energy of the conductivity.



Fig. 5. Dependence of ionic conductivity on LiTDBI concentration at 20°C in a mixture of ethylene carbonate and dimethyl carbonate (1:2 weight ratio) (\circ) and in a propylene carbonate (\blacktriangle).

Notable difference between the solvents conductivity plots is that the highest conductivity in case of the EC:DMC mixture is at the level of the maximum concentration of PC solutions (0.3 mol kg⁻¹) and then it is decreasing just to increase again at the highest concentration for EC:DMC mixture (0.7 mol kg⁻¹). At the highest concentration in EC:DMC mixture the activation energy is also increasing much above that of lower concentrations. It is probably due to the conductivity mechanism change at the highest concentrations, as associates (triplets) take over the bulk of the conductivity. That also explains the difference in solubility between PC and EC:DMC mixture, as different solvation of the lithium cation affect the associates' formation constants. As a result of weaker shielding of solvation layer around lithium cation formation. It is visible by the rapid changes in conductivity activation energy, and resulting in the overall drop in conductivity due to lower molar conductivity, but the molar conductivity due to the higher fraction of associates is visibly lower.



Fig. 6. Dependence of lithium cation transference number dependence on LiTDBI concentration in a mixture of ethylene carbonate and dimethyl carbonate (1:2 weight ratio) (\circ) and in a propylene carbonate (\blacktriangle).

Figure 6 presents values of lithium cation transference numbers for all solutions investigated for ionic conductivity, *i.e.* PC and EC:DMC (1:2 weight ratio) based solutions of LiTDBI in concentration range from 0.001 mol kg⁻¹ to the maximum solubility. Plot for electrolytes based on both solvents show similar shape, although values clearly differ. Both plots have their global minimum for 0.002 mol kg⁻¹ concentration, with slightly higher value at 0.001 mol kg⁻¹ concentration and with higher (in case of PC much higher) values at the higher concentrations. PC solutions exhibit high, but similar values of lithium cation transference number in the plateau in 0.005-0.2 mol kg⁻¹ range. The values in case of EC:DMC based electrolytes are increasing toward 0.02 mol kg⁻¹ and for concentrations above 0.05 mol kg⁻¹ are decreasing. Then, for the highest (one but the highest for EC:DMC) concentration the lithium cation transference number drops. The only qualitative difference is that in case of the highest concentration for EC:DMC (0.7 mol kg⁻¹), which is unavailable for PC solutions, the transference number is increasing again. The highest transference number at the 0.02-0.05 concentration range might be connected to the lowest conductivity activation energy in this range. Also, it might be a synergy (golden mean) between increasing

concentration and decreasing fraction of free ions – not involved in ion pairs or associates formation. That would be an analogous behavior to other weakly-coordinating anions with a similar structure [22].

The lithium cation transference number values show the real potential of weakly coordinating anions. Although the highest concentrations are exhibiting smaller transference numbers than in case of the maximum in 0.02-0.05 mol kg⁻¹ range, they are truly impressive. In case of EC:DMC mixture, maximum is around 1, which is the theoretical maximum. In PC based solutions it is ca. 0.5. For more practical concentrations, at which conductivity is high, PC solutions are still ca. 0.5 (0.45 for the highest concentration). However, for EC:DMC mixture based electrolytes they are above 0.7. More importantly, high lithium cation transference number values overlap with the highest conductivity values – which is the case for 0.2 and 0.7 mol kg⁻¹ concentrations. For those two electrolytes, ionic conductivity is 1.35 and 1.27 mS cm⁻¹, respectively and lithium cation transference number is 0.74 and 0.71, respectively. That means lithium cation conductivity is as high as 1 mS cm⁻¹ and 0.9 mS cm⁻¹ for 0.2 and 0.7 mol kg⁻¹ LiTDBI in EC:DMC electrolytes, respectively. In case of PC based solutions, the highest lithium cation conductivity is shown by 0.3 mol kg⁻¹ LiTDBI solution and is equal to 0.37 mS cm⁻¹. To compare with the industry standard, LiPF₆ in PC solutions shows lithium cation transference numbers in 0.3 to 0.1 range, decreasing with concentration and conductivity between 1 and 5 mS cm⁻¹ in concentration range from 0.1 to 1 mol kg⁻¹ [24]. In LiPF₆-PC electrolytes the lithium cation conductivity does not exceed 0.7 mS cm⁻¹ value. Thus, despite low maximum conductivity, LiTDBI thanks to high transference number has high lithium cation conductivity and might be applicable to lithium-ion cells.

As a proof of concept, 0.7 mol kg⁻¹ LiTDBI-EC:DMC (1:2 weight ratio) electrolyte was used in a graphite | electrolyte | Li half-cell and cycled with C/5 rate. As shown on the Figure 7, starting discharge capacity of graphite electrode is 351 mAh g⁻¹ (94% of the theoretical limit for graphite - 372 mAh g⁻¹). After 50 cycles capacity retention is very good – discharge capacity at 50th cycle is 348 mAh g⁻¹ (over 99% of the 1st cycle). This means that LiTDBI is compatible with standard graphite electrode and might be applied to lithium-ion cell as an electrolyte.

Additional preliminary measurement to obtain the rate capability of the electrolyte was performed employing 0.7 mol kg⁻¹ LiTDBI-EC:DMC (1:2 weight ratio) as an electrolyte in a graphite | electrolyte | Li half-cell with C/10 to 2C rate. The result is exhibited on the Figure 8. As visible, discharge capacity is decreasing fast with the rate from 1C on.



Fig. 7. Discharge anodic capacities during cycling of the graphite | 0.7 mol kg⁻¹ LiTDBI-EC:DMC (1:2 weight ratio) | Li half-cell at C/5 rate.



Fig. 8. Discharge anodic capacities during rate capability cycling of the graphite | 0.7 mol kg⁻¹ LiTDBI-EC:DMC (1:2 weight ratio) | Li half-cell at C/10, C/5, 1C and 2C rates.

However, it shows some potential for use as an electrolyte in lithium-ion cells, given that it is non-optimized electrolyte and there is a vast area to improve this performance, as this is only a preliminary study.

4. Conclusions

Here we present a completely new, "tailored" anion for the purpose of electrolytes for lithium-ion cells. The aim was to obtain weakly-coordinating anion, which would enhance transport properties of lithium-conducting electrolyte without compromising other properties. As a result, the new lithium salt containing TDBI anion is stable both thermally (up to 272° C) and electrochemically (up to 4.75 V vs Li). It is also compatible with graphite anode. As for the transport properties, ionic conductivity is good enough to apply it in lithium-ion cells (above 1 mS cm⁻¹at 20°C). Also, lithium cation transference number is in the highest possible range, *i.e.* around 1. Even for the highest, practical concentrations, it is around 0.75, value that is not reachable for commercially available electrolytes (at least for those without special additives). Such high value of lithium cation transference number is the proof that properly designed weakly-coordinating anions can be in fact reaching the best transport performance without compromising other properties (neither stability nor electrochemical ones). Especially high (351 mAh g⁻¹) and retained capacity of the half-cell shows the potential for the future application of LiTDBI salt.

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