



## Short communication

Rechargeable lithium/sulfur battery with liquid electrolytes containing toluene as additive<sup>☆</sup>Jae-Won Choi<sup>a</sup>, Gouri Cheruvally<sup>a</sup>, Dul-Sun Kim<sup>a</sup>, Jou-Hyeon Ahn<sup>a,\*</sup>, Ki-Won Kim<sup>b</sup>, Hyo-Jun Ahn<sup>b</sup><sup>a</sup> Department of Chemical and Biological Engineering and Engineering Research Institute, Gyeongsang National University, 900 Gajwa-dong, Jinju 660-701, Republic of Korea<sup>b</sup> School of Nano and Advanced Materials Engineering and Engineering Research Institute, Gyeongsang National University, 900 Gajwa-dong, Jinju 660-701, Republic of Korea

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## ABSTRACT

The effect of incorporating varying amounts (in the range 2.5–10 vol%) of toluene additive in 1 M LiCF<sub>3</sub>SO<sub>3</sub> in tetra(ethylene glycol) dimethyl ether (TEGDME) liquid electrolyte on the electrochemical performance of Li/S cell at room temperature was studied. Cyclic voltammetry measurements showed the same active voltage range for the electrolytes with and without toluene, consisting of an oxidation peak at 2.5 V and a pair of reduction peaks at 2.35 and 1.9 V. Electrolytes with toluene have higher redox currents resulting from increased ion mobility and ionic conductivity. Toluene addition enhanced initial discharge capacity; a maximum of 750 mAh g<sup>−1</sup> (45% of the theoretical specific capacity of sulfur) was obtained with 5% of toluene, which was 1.8 times that of the cell without toluene. The electrolyte with 5% toluene exhibited a stable cycle performance with the highest discharge capacity and charge–discharge efficiency. AC impedance analyses of Li/S cells with the electrolytes showed that toluene addition resulted in a lower initial interfacial resistance and a fast stabilization of electrode/electrolyte interfaces in the cell. Addition of toluene in low amounts is thus an effective means to enhance the electrochemical performance of 1 M LiCF<sub>3</sub>SO<sub>3</sub> in TEGDME electrolyte in Li/S cells at room temperature.

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## 1. Introduction

There is an ever increasing demand for the development of rechargeable lithium batteries with high energy density and long cycle life suitable for a variety of applications ranging from micro-batteries of miniature electronic devices to large power sources of electric vehicles. Elemental sulfur has gained considerable attention as a cathode-active material in the search to replace the presently used expensive and toxic transition metal-oxide-based cathode materials. The battery based on lithium/sulfur redox couple has a high theoretical specific capacity of 1672 mAh g<sup>−1</sup> of active material and a high theoretical specific energy of 2600 Wh kg<sup>−1</sup> [1]. In addition, sulfur is a cheap, easily available, and non-toxic material.

Electrochemical utilization of sulfur in Li/S cells is based on the reduction of elemental sulfur with lithium in more than one step. The discharge process in Li/S cells has been investigated by many researchers [2–5]. It has been identified that in the first discharge region at 2.4–2.1 V, elemental sulfur gets reduced to soluble polysulfides and in the second discharge region at 2.1–1.5 V, the

polysulfides get reduced to the solid products Li<sub>2</sub>S<sub>2</sub> and Li<sub>2</sub>S that deposit on the carbon matrix of the cathode [4]. Because of the insulating nature of sulfur and the solubility of polysulfide intermediates in many of the organic electrolytes, it is generally difficult to achieve 100% utilization of the active material at room temperature in Li/S cells. Moreover, the solid compounds Li<sub>2</sub>S<sub>2</sub> and Li<sub>2</sub>S getting deposited on the cathode matrix adversely affect the penetration of electrolyte into the cathode and the availability of active sulfur for further reaction. The earlier studies thus reported low sulfur utilization and cycle property for room temperature Li/S cells [1,6,7].

The selection of a suitable electrolyte is very important in the successful utilization of the active material in Li/S cells because of the multi-step reduction reactions of sulfur and the variations in the solubility of the different polysulfides in the electrolyte. Different types of electrolytes have been evaluated for their suitability in Li/S cells. These include systems based on organic solvents such as the carbonate systems [8], tetrahydrofuran (THF) [3], dimethoxy ethane (DME) [9], 1,3-dioxolane (DIOX) [2,9–12], tri(ethylene glycol) dimethyl ether [7], tetra(ethylene glycol) dimethyl ether (TEGDME) [2,3–6,10,12–15], solid polymer electrolytes based on poly(ethylene oxide) (PEO) [1,16,17], gel polymer electrolytes based on PEO [6], PAN [18] and poly(vinylidene difluoride) (PVdF) [3,8,13]. Most often, a single organic solvent does not meet all the requirements of an electrolyte in a Li/S cell and optimization based on a

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mixture of solvents such as TEGDME/DIOX [2,10], TEGDME/THF [3] and DME/DIOX [9,11] have been found to yield better performance. Previous studies have shown that TEGDME is an attractive organic solvent for Li/S cell at room temperature providing high initial discharge capacity of over  $1200 \text{ mAh g}^{-1}$ , both in the form of liquid and gel/polymer electrolytes [5,12,13]; however, the capacity fade rate has not so far been satisfactory.

Addition of small amounts of organic additives has been reported to improve discharge characteristics of Li/S cells by forming a better electrode/electrolyte interface [12]. Thus, adding 5 vol% of methyl acetate enhanced the performance of Li/S cells at low temperatures. In an earlier study, we also found that toluene added to TEGDME could improve the performance of Li/S cells at room temperature [14]; however, the quantity of toluene in the composition was not optimized. Here, we report the effect of adding varying amounts of toluene in TEGDME solvent on the discharge properties, cycle performance and impedance behaviour of Li/S cells at room temperature.

## 2. Experimental

### 2.1. Materials

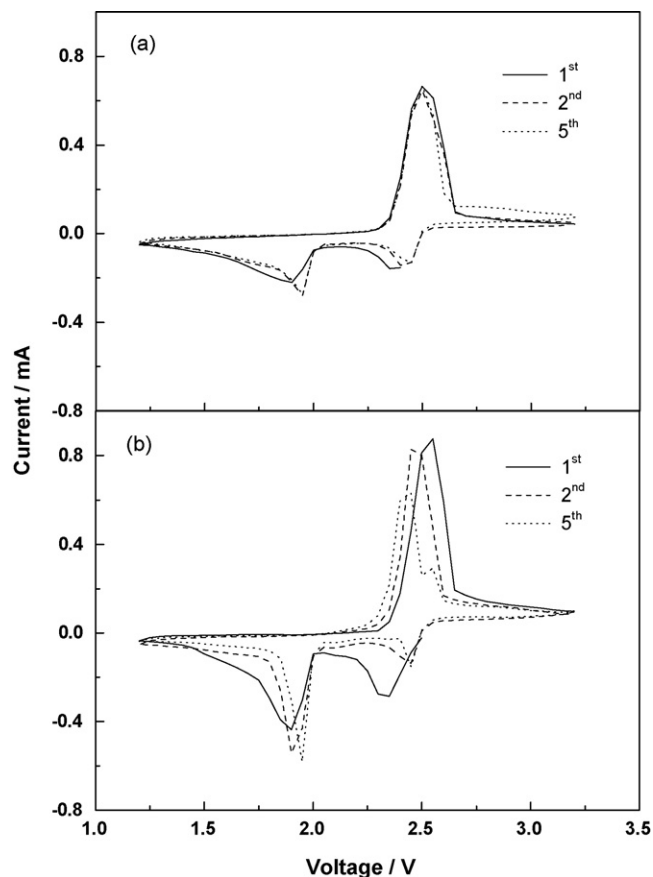
Active cathode material sulfur (99.98%, Aldrich) and the conducting agent acetylene black (Alfa) were dried at 50 and  $120^\circ\text{C}$  respectively under vacuum for 24 h before use. For preparing sulfur cathode, 60 parts by weight (pbw) of sulfur, 20 pbw of acetylene black and 20 pbw of PVdF binder were taken in NMP solvent and mixed in a spex ball mill at room temperature for 1 h at 1080 rpm. The slurry was cast on an aluminium current collector, dried for 48 h at room temperature and further at  $60^\circ\text{C}$  under vacuum for 12 h. The sulfur cathode film so obtained was uniformly pressed to  $20 \mu\text{m}$  thickness using a roll press. The film was cut into circular discs of area  $0.95 \text{ cm}^2$  and mass  $\sim 2.0 \text{ mg}$  for use as cathode. Toluene (Merck) was used as received.  $1 \text{ M LiCF}_3\text{SO}_3$  (Aldrich) solution in TEGDME without any additive and with 2.5, 5, 7.5 and 10 vol% of toluene as additive were used as the liquid electrolytes in the cells.

### 2.2. Characterization

The morphological features of the sulfur cathodes were observed using scanning electron microscope (SEM) (JEOL JSM 5600). Ionic conductivities of the electrolytes at  $25^\circ\text{C}$  were measured with a stainless steel (SS) Swagelok cell using IM6 frequency analyzer by the AC impedance method over the frequency range 2 MHz–100 mHz. Li/S cells were assembled by stacking the separator (Celgard® 2500) containing liquid electrolyte between sulfur cathode and lithium metal anode ( $300 \mu\text{m}$  thickness, Cyprus Foote Mineral Co.) in SS Swagelok® type, circular cells of 23 mm diameter.  $12 \mu\text{l}$  of electrolyte was accurately added into the cell using a micro-syringe. The charge–discharge and cycling tests were performed between 1.5 and 2.8 V at a constant current density of  $1/16\text{C}$  at room temperature using a WBCS3000 potentiostat/galvanostat (WonA Tech. Co.). Cyclic voltammetry (CV) of the Li/S cells with the electrolytes was measured at a scan rate of  $0.1 \text{ mV s}^{-1}$  between 1.2 and 3.2 V. Impedance of Li/S cell was measured using IM6 frequency analyzer over a frequency range of 100 mHz to 2 MHz.

## 3. Results and discussion

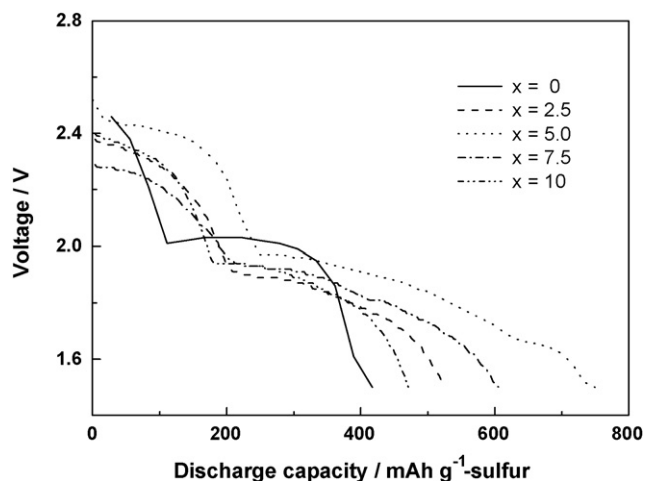
In an earlier study on Li/S cells with liquid electrolytes, we found that the variation in the amount of liquid electrolyte contained in the cell could influence the discharge capacity and cycle performance of the cell [14]. Based on those results, the optimum



**Fig. 1.** CVs of Li/S cells using electrolytes (a)  $1 \text{ M LiCF}_3\text{SO}_3$  in TEGDME and (b)  $1 \text{ M LiCF}_3\text{SO}_3$  in TEGDME with 5% toluene. Scan rate:  $0.1 \text{ mV s}^{-1}$ , voltage range: 1.2–3.2 V.

electrolyte content of  $12 \mu\text{l}$  was used in the present study. CV was carried out to analyze the active voltage range of the Li/S cells being studied. Fig. 1 shows the comparison of CVs of Li/S cells with (a)  $1 \text{ M LiCF}_3\text{SO}_3$  in TEGDME and (b)  $1 \text{ M LiCF}_3\text{SO}_3$  in TEGDME with 5% toluene additive. No major variation in the active voltage range was observed with toluene addition. A pair of well-defined reduction peaks at 2.35 and 1.9 V and an oxidation peak at 2.5 V are observed for both systems. The oxidation peak is attributed to the conversion of polysulfide into sulfur and lithium. The reduction of sulfur occurs in two steps: the first at 2.35 V represents reduction of sulfur to soluble lithium polysulfides ( $\text{Li}_2\text{S}_n$ ,  $2 < n < 8$ ) and the second at 1.9 V represents further reduction of polysulfides to solid lithium sulfides  $\text{Li}_2\text{S}_2$  and  $\text{Li}_2\text{S}$  [6,16]. The intermediate polysulfides may be dissolved in the electrolyte and move within the coating layer over the cathode if the solubility is low or diffuse away from the cathode if the solubility is high. Compared to the electrolyte with toluene, good reversibility of redox reactions upon cycling is observed for the electrolyte without toluene. For the system with toluene additive, the oxidation peak is shifted to lower potential and reduction peaks to higher potential with increase in cycle number, indicating an improvement of reversibility of the cell with cycling.

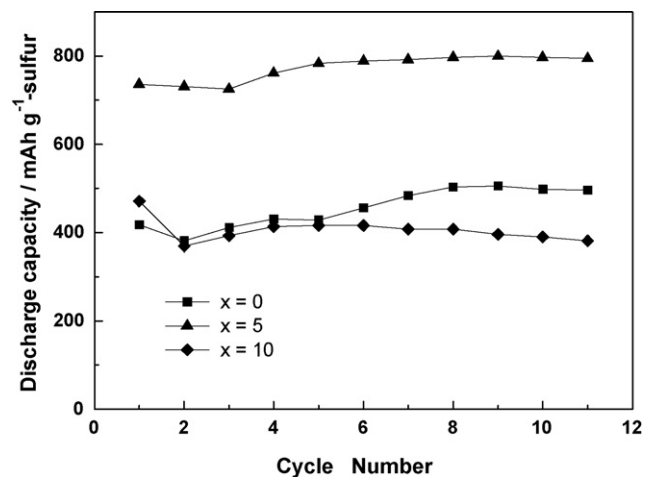
As the cycle number increased, it is observed that with toluene additive, the reduction peak at 2.35 V becomes less significant, while the one at 1.9 V grows higher in intensity. This could imply that the polysulfides generated during the first step reduction of sulfur has low solubility in TEGDME containing toluene when compared to TEGDME alone and are further available for the subsequent second discharge process. With toluene additive, there is an increase in peak currents: oxidation peak current of 0.88 mA vs. 0.66 mA and reduction peak current at 1.9 V of 0.44 mA vs. 0.22 mA;



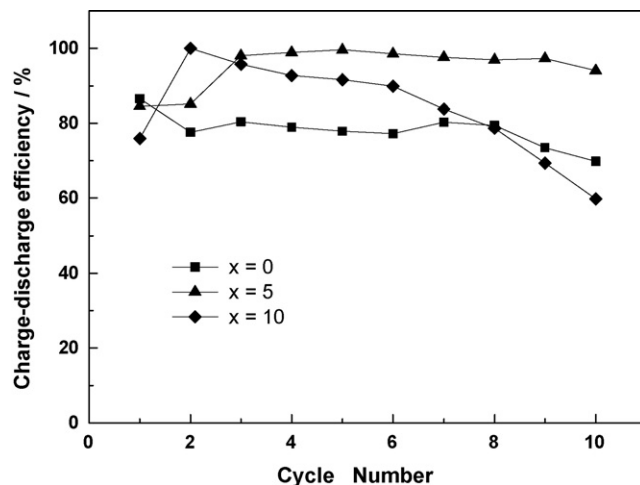
**Fig. 2.** Initial discharge capacities of Li/S cells at room temperature with 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME electrolyte containing  $x\%$  of toluene additive. Current density:  $1/16$  C, voltage range: 1.5–2.8 V.

this suggests a more effective ion transport in the electrolyte with toluene. The ionic conductivity comparison also showed an increasing trend with the addition of toluene (ionic conductivity of electrolyte without toluene:  $1.0 \times 10^{-2} \text{ S cm}^{-1}$ ; range of ionic conductivity of electrolytes with 2.5 to 10% toluene:  $1.1 \times 10^{-2}$  to  $2.6 \times 10^{-2} \text{ S cm}^{-1}$ ). This could be resultant from the lower viscosity of toluene compared to TEGDME (0.7 cPs vs. 4.05 cPs at 25 °C), facilitating easy migration of ions in the electrolyte.

Fig. 2 presents the variation in initial discharge capacity of Li/S cells with varying amounts of toluene additive in the electrolyte of 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME. The discharge behaviour of the cells containing toluene additive is similar to that of the cell without the additive. Two voltage plateau regions are observed in the discharge process, one at the higher voltage  $\sim 2.4$  V is less predominant compared to the lower one at  $\sim 1.9$  V (as was observed in the CV also). With increase in cycle number, the higher voltage plateau disappeared, whereas the position of lower plateau was unaffected (not shown here). These observations are typical of Li/S discharge process at room temperature [1,2,5,12,13,15]. Addition of toluene improves the initial discharge capacity of the cell, and the maximum of  $750 \text{ mAh g}^{-1}$  (corresponding to 45% of theoretical capacity of  $1675 \text{ mAh g}^{-1}$  of sulfur) was observed with 5% toluene addi-

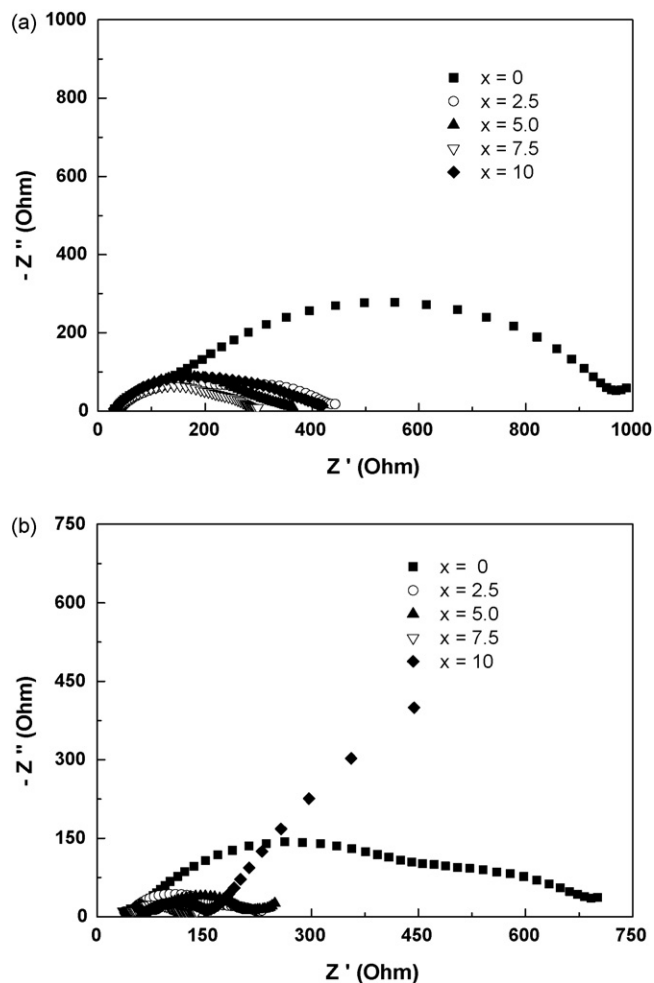


**Fig. 3.** Cycle performance of Li/S cells with 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME electrolyte containing  $x\%$  of toluene additive. Current density:  $1/16$  C, voltage range: 1.5–2.8 V.



**Fig. 4.** Variation of charge–discharge capacity efficiency with cycle number of Li/S cells with 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME electrolyte containing  $x\%$  of toluene additive.

tive, which is 1.8 times higher compared to the electrolyte without toluene ( $418 \text{ mAh g}^{-1}$ ). Adding small amounts of organic additives such as methyl acetate has been reported to improve discharge characteristics of Li/S cells by forming a better electrode/electrolyte



**Fig. 5.** Electrode/electrolyte interfacial resistance of Li/S cells with 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME electrolyte containing  $x\%$  of toluene additive. (a) Initial and (b) after 10 cycles. Frequency range: 2 MHz–100 mHz.



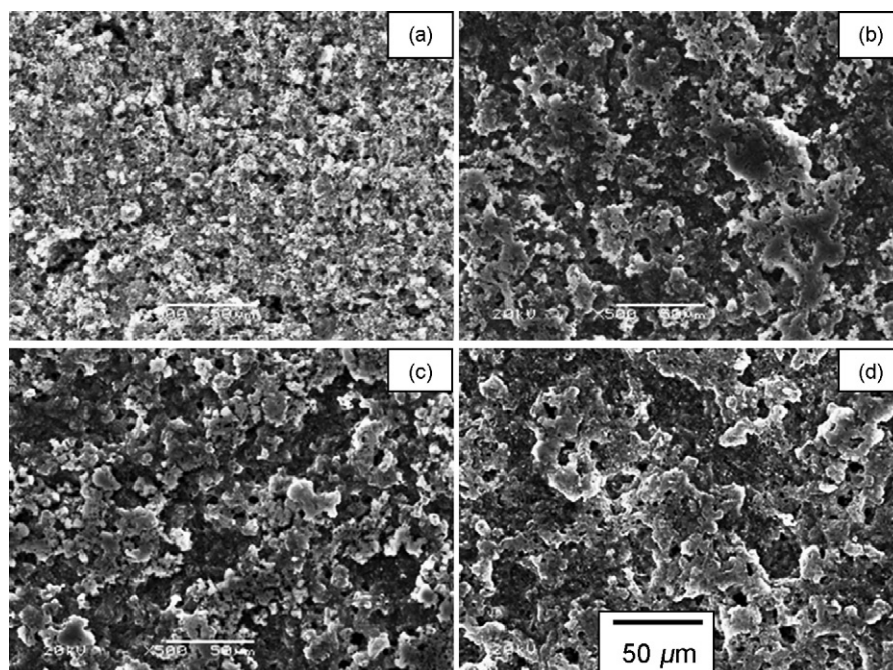
interface [12]. Since toluene has a lower viscosity compared to TEGDME, addition of toluene is expected to facilitate the easy migration of ions in the cell between the electrodes and also provide better wetting on the surface making it easy for the ions to penetrate into the electrode. Thus, toluene can enhance the availability of lithium ions at the sulfur cathode and this in turn leads to higher utilization of sulfur. Toluene in higher amounts is found to cause a lowering of discharge capacity, probably because of its inert, non-conductive nature and lower dielectric constant compared to TEGDME (2.38 vs. 7.9) that would adversely affect the effective dissociation of lithium salt.

The variation of discharge capacities with cycle number of Li/S cells containing TEGDME electrolytes without and with 5% and 10% toluene was studied up to 10 cycles and the results are shown in Fig. 3. TEGDME has already been identified as a good electrolyte solvent for Li/S cells and in this study also we observed that a rather stable cycle performance is provided by the electrolyte. The cell with the medium amount of additive, i.e., 5% toluene performed better than the one without additive. This cell, after an initial marginal decrease in capacity during the first few cycles, got stabilized fast and after 10 cycles, the cell showed discharge capacity of  $790 \text{ mAh g}^{-1}$ , almost maintaining its initial discharge capacity. At the higher toluene content of 10%, the cycle performance was inferior.

The charge–discharge efficiency of the cells during cycling is compared in Fig. 4. The cell without additive has an efficiency of  $\sim 80\%$  and it maintains that efficiency during cycling, thus showing good cycle performance, as was observed in Fig. 3. The cell with 5% toluene shows higher efficiency, nearly 100% during cycling. Higher efficiency is attributed to the better reversibility of the redox reactions taking place in the cell. TEGDME being a polar solvent has good solubility for the polysulfides, which are ionic compounds. Thus, the discharge products (polysulfides) might be carried away from the cathode by getting dissolved in the electrolyte and their availability for further redox process is reduced causing a decrease in charge–discharge efficiency. When non-polar toluene is added to TEGDME, the solubility of polysulfides in the electrolyte gets

slightly reduced, enhancing the probability of retaining them at the cathode during subsequent cycles of charge–discharge; this results in increased efficiency. These advantageous effects of toluene addition are outweighed by its inertness at higher amounts ( $\geq 10\%$ ) and lead to lower charge–discharge efficiency.

In order to have a better understanding of the effect of toluene addition on the electrochemical performance of the electrolyte, the impedance properties of Li/S cells with the electrolytes were studied. The impedance spectra of Li/S cells with TEGDME alone and with different amounts of toluene as additive were evaluated initially and after 10 cycles of charge–discharge and are shown in Fig. 5(a) and (b) respectively. Addition of toluene does not cause any dramatic change in the bulk resistance of the electrolyte ( $R_e$ ) measured at the high frequency end corresponding to 2 MHz; the values ranged between  $32\text{--}56 \Omega$  initially and  $43\text{--}66 \Omega$  after 10 cycles. However, there is a wide variation in the interfacial resistance ( $R_f$ ) of the electrolytes. Initial  $R_f$  is determined by the properties of the passivation layers spontaneously formed on the surface of electrodes in contact with the electrolytes. It is observed that the electrolytes containing toluene have much lower initial  $R_f$  (in the range  $260\text{--}400 \Omega$ ) compared to TEGDME alone ( $935 \Omega$ ). The lowest initial  $R_f$  of  $260 \Omega$  was obtained for the electrolyte containing 7.5% toluene. Although the interfacial contributions from both electrodes could be contributing to the measured property, the effects from lithium metal interface could be more critical than the sulfur interface. For understanding this, we analyzed the impedance of Li/electrolyte/Li cells, and obtained initial  $R_f$  values of  $1338 \Omega$  for TEGDME without toluene and  $749 \Omega$  for TEGDME with 5% toluene. Thus, we observe that presence of toluene contributes remarkably to a lower initial  $R_f$  on the lithium metal interface, which could be the result of the lowered viscosity of the electrolyte and hence an easy transport of ions across the interface. Addition of low amounts of benzene in  $\text{LiClO}_4/\text{PC}$  electrolyte has been reported to influence the impedance profiles on lithium metal and decrease the  $R_f$  [19]. This decrease was attributed to the accumulation of the non-polar and lyophobic benzene at the electrode/electrolyte interface leading to its adsorp-



**Fig. 6.** SEM of sulfur cathodes of Li/S cells: (a) before discharge and (b)–(d) after 10 cycles of charge–discharge with the electrolytes; (b) 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME; (c) 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME with 5% toluene and (d) 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME with 10% toluene.

tion, which effectively prevents the non-conductive passivation layer growth at the surface. Toluene, a non-polar and lyophobic solvent similar to benzene, is also expected to form a thin adsorbed layer on the electrodes that are conducive for conduction of lithium ions and this effectively reduces the possibility of reactions between the electrolyte components and electrodes that could form thick and impermeable protective layers on the electrode surface.

After 10 cycles of charge–discharge (in Li/S cells), it is seen that (Fig. 5(b))  $R_f$  has decreased considerably from its initial value for all the electrolytes. For TEGDME without toluene,  $R_f$  decreased by 32% over 10 cycles and reached 638  $\Omega$ . This proves that TEGDME is an electrolyte of choice for Li/S cells that can provide compatible and stable electrode/electrolyte interface. The electrolytes with toluene additive showed further decrease in  $R_f$  (~55–75% decrease over 10 cycles). The lowest  $R_f$  of 89  $\Omega$  after 10 cycles is observed for the cell with the highest amount of toluene.

The surface morphologies of the sulfur cathode before discharge and after 10 cycles of charge–discharge were observed by SEM for understanding the interface structure between the electrode and electrolyte and are shown in Fig. 6. The cathode before discharge shows a homogenous morphology showing uniform distribution of the active material, binder and conducting agent. The micrographs taken after 10 cycles show the presence of insoluble reduction products of sulfur  $\text{Li}_2\text{S}_2$  and  $\text{Li}_2\text{S}$  getting deposited on the carbon matrix of the cathode, as has been reported in similar studies [2,15]. For TEGDME electrolyte without toluene, the surface of the cathode is passivated by the solid, non-conducting products over a larger area and in a non-uniform way, compared to that for the electrolytes with 5% toluene. This is attributed mainly to the high viscosity of TEGDME electrolyte restricting its deep penetration into the interior of cathode and hence redox reactions are greatly localized on the surface of the cathode. With 5% toluene as additive, the cathode morphology is more uniform, showing smaller particles of lithium sulfides on the surface. This results from the easy transport of ions into the interior of the cathode, ensuring a higher utilization of sulfur during cycling, as indeed was observed during cycle performance experiments. The morphology for TEGDME with 10% toluene showed the presence of large, sheet-like particles (passivated layer), as in the case of TEGDME alone. Although not clear, the reason could be the significantly lowered solubility of the polysulfides including the long chain ones, in the electrolyte containing a higher amount of toluene, thus retaining them on the cathode as such.

#### 4. Conclusions

The effect of adding toluene as an additive (2.5–10 vol%) to the liquid electrolyte 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME on the electrochemical properties of Li/S cells at room temperature was evaluated.

CV comparison showed the active voltage range to be the same with and without toluene. However, the redox current was higher with toluene additive, showing easy migration of ions resulting from the enhanced ionic conductivity. The initial discharge capacity increased with toluene incorporation and the maximum of 750  $\text{mAh g}^{-1}$  was obtained with 5% addition; this was nearly 1.8 times higher compared to the electrolyte without toluene. The cycle performance of the cell containing 5% toluene was better, this resulting from an improved charge–discharge efficiency of the cell. A comparison of the impedance behaviour of the Li/S cells showed that the initial  $R_f$  as well as the  $R_f$  after 10 charge–discharge cycles were lower for the electrolytes containing toluene. SEM observation revealed the formation of a better morphology with 5% toluene addition in the electrolyte. Thus, toluene as an additive in low amounts improves the performance of 1 M  $\text{LiCF}_3\text{SO}_3$  in TEGDME electrolyte by forming a stable, thin, porous and conducting electrode/electrolyte interface and also by reducing the solubility of polysulfides in the electrolytes preventing them from being carried away from the sulfur electrode.

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