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Protected bis(hydroxyorganyl) polysulfides as modifiers of Li/S battery electrolyte

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ABSTRACT

The protected bis(hydroxyorganyl) polysulfides synthesized have been tested as modifiers of electrolyte of lithium-sulfur rechargeable batteries. The best result (35% increase of the battery capacity at the 50th cycle) was attained using 5 wt.% of 2,2,12,12-tetramethyl-4,10-diphenyl-3,11-dioxa-6,7,8-trithia-2,12-disilatridecane. Bis(hydroxyorganyl) polysulfides protected at the hydroxyl group have been synthesized for the first time by the reaction of oxiranes with sodium polysulfide (ethanol, $NaHCO_3$, 12 h, 20–25 °C) in yield 21–87%. For the hydroxyl protection in the hydroxy polysulfides, the acetal, tri(methyl)silyloxy or tri(ethoxy)silyl protecting groups were employed.

1. Introduction

Li-metal batteries are considered to be very attractive due to their high energy density [1]. Elemental sulfur seems to be the best cathode material for these batteries because of its high theoretical capacity (1672 mAh g^{-1}) which allows one to have a specific energy of 2600 Wh kg^{-1}, assuming the complete reaction of lithium with sulfur to Li_2S [2–4].

The development of Li/S batteries is now carried out by a number of companies and scientific centers and a commercially acceptable design of these power sources is under way [5–9]. The unique feature of Li/S battery is "liquescent cathode": during the first discharge cycle, almost the whole sulfur turns to higher lithium polysulfides.

$$S_8 + 2Li^0 \rightarrow Li^{+-}S_8{}^{-}Li^{+}$$

The polysulfides are soluble in electrolyte and can diffuse to the anode area. The reaction between lithium anode and higher polysulfides results in self-discharge of the battery, for example:

$$S_8{}^{2-} + 2Li \rightarrow S_7{}^{2-} + Li_2S$$

Finally, these reactions result in formation of insoluble lithium sulfides and disulfides (Li_2S and Li_2S_2) which just reluctantly or not at all participate in the redox processes thereby decreasing the content of active sulfur in the battery and deteriorating the surface of lithium anode. The accumulation of insoluble sulfides over the electrodes is commonly considered as a reason of poor cycle-life of Li/S batteries [2–4,10]. According to the patent [11], lithium sulfide and disulfide can produce a passivating layer during deposition onto the surface of a metal lithium electrode. This layer slows down or completely prevents further interaction of metal lithium with components of the electrolyte system. Generally it is well known that diorganyl polysulfides react with alkaline metal sulfides to give soluble longer-chain alkaline metal polysulfides [12–14].

This work deals with the synthesis of protected bis (hydroxyorganyl) polysulfides, which can react with lithium sulfide and disulfide with the formation of soluble and more mobile lithium compounds (organolithium polysulfides or mercaptides), thus improving the Li/S battery performance.

These species solubilize lithium sulfide and disulfide and move to the cathode to participate in electrochemical reactions with regeneration of soluble polysulfide molecules. So, these polysul-

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fides might act as a kind of catalytic carriers for insoluble Li_2S and Li_2S_2. Compounds described for the first time in this paper belong to the class of organic polysulfides $R–S_n–R$. Their efficiency as lithium sulfide and disulfide incorporating agents follows from the known property of inorganic sulfides to be inserted into the S–S bonds [15], therefore the following reactions are expected:

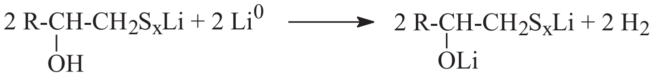
$$Li_2S + R_2S_n \rightarrow RS_xLi + RS_yLi$$

where $n \geq 2$, $x + y = n + 1$.

Generally, lithium organylsulfides (mercaptides) thus formed should be soluble in the battery electrolyte and their oxidation at cathode recovers organic polysulfides:

$$RS_xLi + RS_yLi \xrightarrow{-2e^-} R_2S_{x+y} + 2Li^+$$

In the patent [16], this approach was partially exemplified using some hydroxyorganyl polysulfides. However, these polysulfides should irreversibly consume Li-anode due to the reaction between Li-metal and hydroxy group with undesirable evolution of hydrogen.



$$2\ \underset{\displaystyle OH}{R\text{-}CH}\text{-}CH_2S_xLi + 2\ Li^0 \longrightarrow 2\ \underset{\displaystyle OLi}{R\text{-}CH}\text{-}CH_2S_xLi + 2\ H_2$$

To avoid this drawback, here we have synthesized and tested as electrolyte modifiers of Li/S battery a series of hitherto unknown protected hydroxyorganyl polysulfides, in which hydroxy groups are converted to the acetal, tri(methyl)silyloxy or tri(ethoxy)silyloxy functions.

2. Experimental

2.1. Measurements

IR spectra were recorded on a Bruker IFS-25 spectrometer. NMR spectra were run on a Bruker DPX 400 instrument [400.13 (1H) MHz] in $CDCl_3$ using HMDS as an internal standard.

Two electrode electrochemical cells (model of button cell) were used for electrochemical testing of the synthesized compounds. A lithium anode and an aluminum cathode coated with carbon and a composition based on elemental sulfur, activated carbon, and polyethylene oxide in a ratio of 65:30:5 (wt.%) were employed. The sulfur density on the cathode was 1.11 mg cm^{-2}. The anode was an Li metal foil of 0.2 mm thick. A 2 M solution of $(CF_3SO_2)_2NLi$ in a 1:1 mixture of 1,2-dimethoxyethane and 1,3-dioxolane was served as standard electrolyte. The Li/S cell cycling was performed on a Maccor battery tester at a discharge–charge current density of 0.25 mA cm^{-2} in a potential range from 1.25 V to 2.80 V at an ambient temperature.

2.2. Materials

All solvents were dried by standard methods and distilled before use. Chemically pure sodium sulfide $Na_2S·9H_2O$, $NaHCO_3$, $MgSO_4$, K_2CO_3 and elemental sulfur were used.

2-Phenyloxirane (**1**), *N*-butoxyethene, 2-[(allyloxy) methyl]oxirane (**2**) ("Aldrich") were distilled before use.

The purity of 2-{[2-(vinyloxy)ethoxy]methyl}oxirane (**3**) was 99.99% (GLC control). Physical constants of 2-{[2-(vinyloxy)ethoxy] methyl}oxirane corresponded to the literature (bp 77 °C/6 Torr, n_D^{20} 1.4480, d_4^{20} 1.0333 [17].

The sodium polysulfide was synthesized from $Na_2S·9H_2O$ and elemental sulfur [18]: to a solution of $Na_2S·9H_2O$ (6.0 g, 25.0 mmol) in 5 ml of water and 1 ml of ethanol, 3.2 g (100 mmol) of elemental sulfur was added at 50–60 °C over 20 min by portions (0.1–0.2 g). The mixture was heated at 60 °C for 1 h and cooled to room temperature.

1-(2-oxiranylmethyl)-1H-pyrrole (**4**) was synthesized in a yield of 39% from 1*H*-pyrrole and 2-(chloromethyl)-oxirane by the protocol developed for 1-oxiranylmethyl-4,5,6,7-tetrahydroindole [19]. The process was realized through the reaction of the K salt of 1*H*-pyrrole, prepared from KOH and 1*H*-pyrrole in toluene with azeotropic removal of water followed by the interaction of the salt obtained with 2-(chloromethyl)-oxirane (60 °C, 3 h).

1-(2-oxiranylmethyl)-1H-indole (**5**) was synthesized in a yield of 36% from 1*H*-indole and 2-(chloromethyl)-oxirane (45 °C, 3 h) by the protocol developed for 1-oxiranylmethyl-4,5,6,7-tetrahydroindole [19].

Mixed 1,3-dioxolan-4-ylmethyl 2-oxiranylmethyl ether and 1,3-dioxan-5-yl 2-oxiranylmethyl ether (**6**) (3:1) was synthesized in a yield of 21% from the mixture of 1,3-dioxolan-4-ylmethanol and 1,3-dioxan-5-ol (35:65%), powdered NaOH and 2-(chloromethyl) oxirane for 3 h at room temperature.

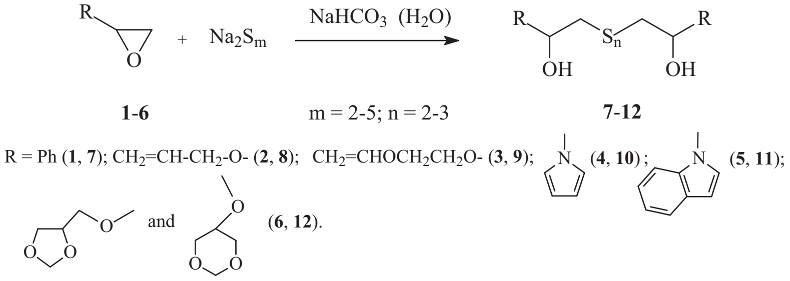
2.3. Synthesis of the additives

The typical procedures of the syntheses were as follows.

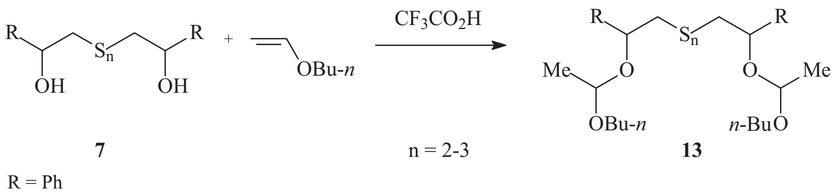
2.3.1. Synthesis of hydroxyorganyl polysulfides

2-[3-(2-hydroxy-2-phenylethyl)trisulfanyl]-1-phenyl-1-ethanol (**7**) was synthesized according to Scheme 1 (see Section 3).

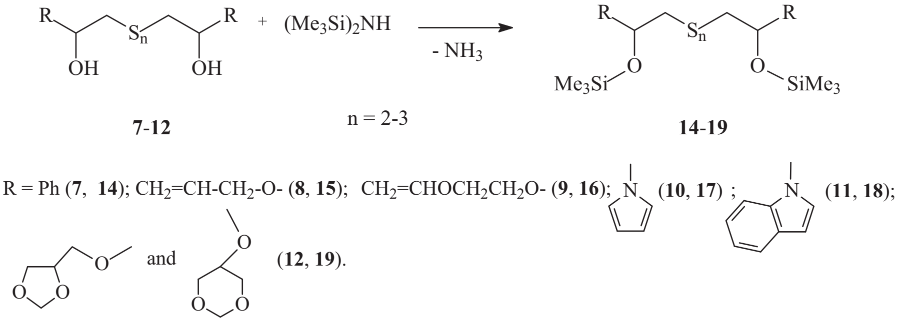
To a mixture of 2-phenyloxirane **1** (10.0 g, 83.2 mmol), ethanol (5 ml) and $NaHCO_3$ (5.0 g), a solution of sodium polysulfide (see above) was added portion-wise (~0.2 ml) under stirring over 1.5 h at 20–25 °C. The resultant mixture was stirred for additional 2 h, allowed to stay for 12 h and filtered off. The filtrate was diluted with water (10 ml) and extracted with diethyl ether (5 × 10 ml). The ether extract was dried over $MgSO_4$ (4.0 g, 12 h) and distilled off. The residue was distilled under vacuum (2 Torr, 70–100 °C), and 2.8 g of 2-phenyloxirane **1** was recovered. The residue is hydroxyorganyl polysulfide **7**, a yellow resin-like substance (yield 5.0 g, 49% based on the reacted oxirane), soluble in diethyl ether, ethanol, benzene, DMSO, etc. Molecular mass (cryoscopy in dioxane): 338.7. Calcd: 338.5.



Scheme 1. Synthesis of the starting hydroxyorganyl polysulfides.



Scheme 2. Synthesis of the polysulfide **13**.



Scheme 3. Synthesis of the polysulfide **14–19**.

Hydroxyorganyl polysulfides **8–12** were prepared by analogical methodic.

2.3.2. The reaction of hydroxyorganyl polysulfide with N-butoxyethene

2-{3-[2-(1-Butoxyethoxy)-2-phenylethyl]trisulfanyl}-1-phenylethyl 1-butoxyethyl ether **13** was prepared according to Scheme 2 (see Section 3).

The hydroxyorganyl polysulfide **7** (3.5 g, 10.3 mmol) was dissolved in 1,2-dimethoxyethane (2 ml), then *N*-butoxyethene (2.00 g, 20.0 mmol) and trifluoroacetic acid (0.04 ml) were added. The solution was stirred for 12 h at 20 °C. Then, K_2CO_3 (0.2 g) was added to the solution, the resultant mixture was allowed to stay for 1 h at room temperature and filtered off. Excess of *N*-butoxyethene and the solvent (1,2-dimethoxyethane) were removed by vacuum distillation to give 5.57 g (100% yield) of polysulfide **13**, a yellow viscous liquid, soluble in diethyl ether, 1,2-dimethoxyethane and insoluble in water.

2.3.3. The reactions of hydroxyorganyl polysulfides with hexamethyldisilazane

2,2,12,12-Tetramethyl-4,10-diphenyl-3,11-dioxa-6,7,8-trithia-2, 12-disilatridecane **14** was obtained according to Scheme 3 (see Section 3).

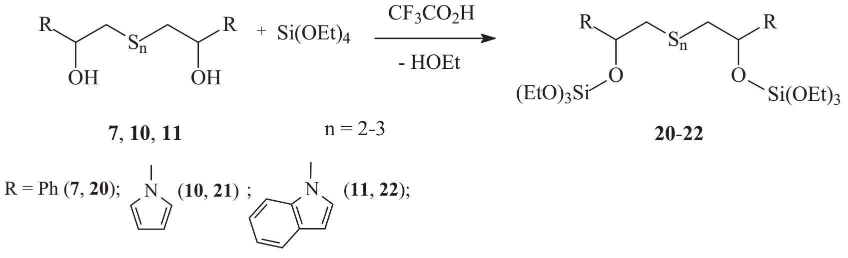
The hydroxyorganyl polysulfide **7** (2.0 g, 5.9 mmol) was mixed with hexamethyldisilazane (2.0 g, 12.3 mmol), and the mixture was stirred for 48 h at room temperature. Excess of hexamethyldisilazane was removed by vacuum distillation to produce 2.85 g (100% yield) of polysulfide **14**, a light-yellow viscous liquid, soluble in diethyl ether, 1,2-dimethoxyethane and insoluble in water.

^{1}H NMR (acetone-D$_6$), δ, ppm: 0.08s (18H, CH_3), 2.57–4.18m (4H, CH_2S); 4.20–5.09m (2H, CH–CH_2S), 7.36 (7.20–7.40)m (10H, Ph); IR (neat, cm^{-1}): 3085, 3061, 3029 (ν, =CH, Ph); 2955, 2896, 2864 (ν, CH_3, CH_2, CH); 1946, 1873, 1803, 1723 (overtones, Ph); 1601, 1584 (ν, C=C, Ph); 1493, 1453, 1204, (ν, C–C; δ, C–C–S; δ, CH_2); 1250 (δ, Si–CH_3); 1025–1170 (ν, C–O–C, Si–O–C); 918, 875, 616 (ν, C–S); 841 (ν, Si–C); 749, 697 (δ, =CH, Ph); 525 (ν, S–S); elemental analysis calcd (%) for $C_{22}H_{34}O_2S_3Si_2$: C, 54.72; H, 7.10; S, 19.92; Si, 11.63; found: C, 54.46; H, 7.22; S, 20.01; Si, 11.45.

Allyl 3-(3-{3-(allyloxy)-2-[(trimethylsilyl)oxy]propyl}trisulfanyl)-2[(trimethylsilyl)oxy]propyl ether **15** was synthesized from 2-[(allyloxy)methyl]oxirane **2** and hexamethyldisilazane according to Schemes 1 and 3 in 81% yield as light-yellow mobile liquid, soluble in diethyl ether, acetone, dioxane, 1,2-dimethoxyethane and insoluble in water.

2,2-Dimethyl-10-[(trimethylsilyl)oxy]-4-{[2-(vinyloxy)ethoxy] methyl}-3,12,15-trioxa-6,7,8-trithia-2-sila-16-heptadecene **16** was synthesized from 2-{[2-(vinyloxy)ethoxy]methyl}oxirane **3** and hexamethyldisilazane according to Schemes 1 and 3, in 85% yield as light-yellow liquid, soluble in diethyl ether, 1,2-dimethoxyethane and insoluble in water.

^{1}H NMR ($CDCl_3$), δ, ppm: 0.14 s (18H, CH_3), 2.70–3.22m (4H, CH_2–S), 3.45–3.58m (4H, O–CH_2–CH–OSi), 3.67–3.76 and 3.82–3.86m (8H, CH_2CH_2O), 3.95–4.01dd and 4.13–4.20dd (4H, CH_2=), 4.25–4.41m (2H, CH–OSi), 6.42–6.51q (2H, CH=); IR (neat, cm^{-1}): 3117, 3046 (ν,=C–H); 2924, 2900, 2873 (ν, CH, CH_2); 1636–1617 (ν, C=C); 1250 (δ, CH_3); 1040–1180 (ν, C–O–C); 976 (ω, –CH=CH_2); 841 (ν, Si–C); 690, 615 (ν, S–C); 477 (ν, S–S); elemental analysis calcd (%) for $C_{20}H_{42}O_6S_3Si_2$.: C, 45.25; H, 7.97; S, 18.12; Si, 10.58; found: C, 46.00; H, 7.62; S, 17.88; Si, 9.84.



Scheme 4. Synthesis of the polysulfide **20–22**.

Bis{3-(1H-pyrrol-1-yl)-2-[(trimethylsilyl)oxy]propyl}disulfide **17** was synthesized from 1-(2-oxiranylmethyl)-1*H*-pyrrole **4** and hexamethyldisilazane according to Schemes 1 and 3, in 61% yield as brown viscous liquid, soluble in diethyl ether, acetone, 1,2-dimethoxyethane.

2-(1H-indol-1-yl)-1-[(3-{3-(1H-indol-1-yl)-2-[(trimethylsilyl)oxy]propyl}trisulfanyl)methyl]ethyl trimethylsilyl ether **18** was synthesized from 1-(2-oxiranylmethyl)-1*H*-indole **5** and hexamethyldisilazane according to Schemes 1 and 3 in 87% yield as brown liquid, soluble in diethyl ether, acetone, 1,2-dimethoxyethane.

^{1}H NMR ($CDCl_3$), δ, ppm: 0.23 s (18H, CH_3), 2.84–3.17m (4H, CH_2–S), 4.23m (4H, CH_2N), 4.47m (2H, CH–O), 6.64–7.76 (12H, indole ring); IR (neat, cm^{-1}): 3080, 3040, 2945 (ν, =C–H, CH_3); 2920, 2890, 2840 (ν, CH, CH_2); 1605 (ν, C=C), 1498 (ν, N–C); 1060–1080 (ν, Si–O–C; δ, indole ring); 820 (ν, Si–C); 720 (δ, indole ring); 680 (ν, S–C); 460, 440 (ν, S–S); elemental analysis calcd (%) for $C_{28}H_{40}N_2O_2S_3Si_2$: C, 57.10; H, 6.85; N, 4.76; S, 16.33; Si, 9.54; found: C, 58.54; H, 7.23; N, 4.89; S 16.41; Si 8.00.

Mixed of 4,9-bis[(1,3-dioxolan-4-ylmethoxy)methyl]-2,2,11,11-tetramethyl-3,10-dioxa-6,7-dithia-2,11-disiladodecane and 4,9-bis[(1,3-dioxan-5-yloxy)methyl]-2,2,11,11-tetramethyl-3,10-dioxa-6,7-dithia-2,11-disiladodecane **19** was synthesized from the mixture of 1,3-dioxolan- and 1,3-dioxanoxiranes **6** (3:1) according to Schemes 1 and 3 in 21% yield as brown viscous liquid, soluble in diethyl ether, acetone, 1,2-dimethoxyethane.

The product contains 73% of dioxolane and 27% of dioxane moieties, respectively.

2.3.4. The reactions of hydroxyorganyl polysulfides with tetraethyl orthosilicate

9,9-Diethoxy-1,7-diphenyl-8,10-dioxa-3,4,5-trithia-9-siladodecyl triethyl orthosilicate **20** was synthesized according to Scheme 3 (see Section 3).

The hydroxyorganyl polysulfide **7** (2.00 g, 5.9 mmol) was dissolved in 3 ml of 1,2-dimethoxyethane, then 6.50 g (31.2 mmol) of tetraethyl orthosilicate and 0.02 ml of trifluoroacetic acid were added. The solution was stirred for 24 h at 60 °C. Then, K_2CO_3 (0.10 g) was added to the solution, the resultant mixture was stirred for 1 h at room temperature and filtered off. Excess of tetraethyl orthosilicate was removed by vacuum distillation to produce 3.92 g (100% yield) of polysulfide **20**, a brown mobile liquid, soluble in diethyl ether, 1,2-dimethoxyethane and insoluble in water.

8,8-Diethoxy-1,6-bis(1H-pyrrol-1-ylmethyl)-7,9-dioxa-3,4-dithia-8-silaundecyl triethyl orthosilicate **21** was synthesized from 1-(2-oxiranylmethyl)-1*H*-pyrrole **4** and tetraethyl orthosilicate according to Schemes 1 and 4 in 65% yield as brown, highly viscous oil, soluble in diethyl ether, 1,2-dimethoxyethane and insoluble in water.

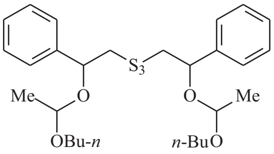
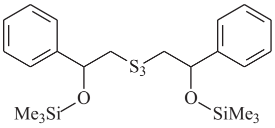
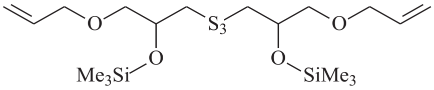
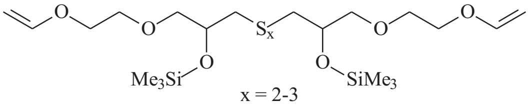
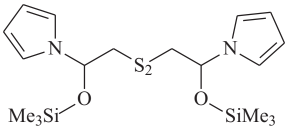
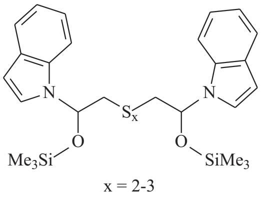
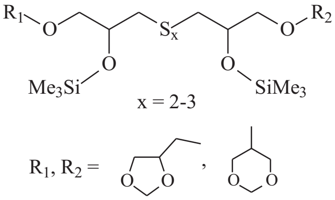
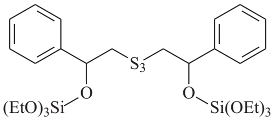
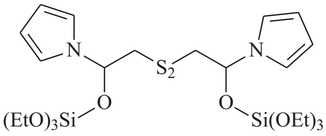
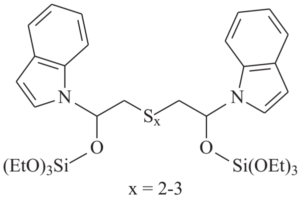
9,9-Diethoxy-1,7-bis(1H-indol-1-ylmethyl)-8,10-dioxa-3,4,5-trithia-9-siladodecyl triethyl orthosilicate **22** was synthesized from 1-(2-oxiranylmethyl)-1*H*-indole **5** and tetraethyl orthosilicate according to Schemes 1 and 3 in 49% yield as brown, highly viscous oil, soluble in diethyl ether, 1,2-dimethoxyethane and insoluble in water.

3. Results and discussion

The reaction of oxiranes with sodium polysulfide in the presence of sodium hydrocarbonate as a buffer was used for the synthesis of the starting hydroxyorganyl polysulfides (Scheme 1). The hydroxyorganyl polysulfides have been protected at the hydroxyl group by the reactions with *N*-butoxyethene (Scheme 2), hexamethyldisilazane (Scheme 3) and tetraethyl orthosilicate (Scheme 4).

The protected functionalized polysulfides synthesized are listed in Table 1.

Table 1
Protected bis(hydroxyorganyl)polysulfides.

Polysulfide	Structural formula
13	
14	
15	
16	
17	
18	
19	
20	
21	
22	

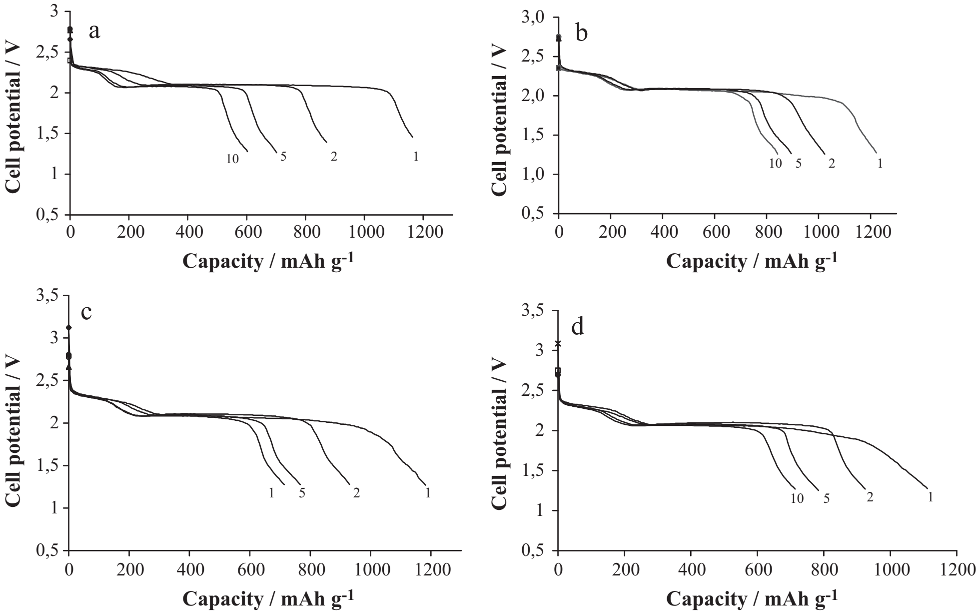


Fig. 1. Discharge curves for Li/S cells containing three most effective additives (5 wt.%) **14** (b), **18** (c), **16** (d) in comparison with that of additive-free cell with the standard electrolyte (a) (1st, 2nd, 5th and 10th cycles).

The addition of 5 wt.% of organic polysulfides to the standard electrolyte (see Section 2) does not change the shape of discharge curves in the Li/S electrochemical cell. Fig. 1 shows the discharge curves of Li/S cells containing the three most effective additives **14**, **18**, **16** in comparison with that of additive-free cell: the surplus of the capacity on the 5th cycles equals 27, 9 and 12% for the additives **14**, **18** and **16**, respectively and the relative gain of the capacity of the additives keeps retaining and even augmenting during 50th cycles (25–35% for the same additives) and further on (Fig. 2). On the discharge curves, there are two regions at 2.35 and 2.06 V corresponding to a two-stage mechanism of the electrochemical reduction of sulfur that is in agreement with literature data [3,5,8].

Fig. 2 illustrates the cycling behavior of the cell with the standard electrolyte and the cells with electrolytes modified by 5 wt.% polysulfides **14** and **18**. It is seen that these polysulfides remain active at least up to 50th cycles and sustain

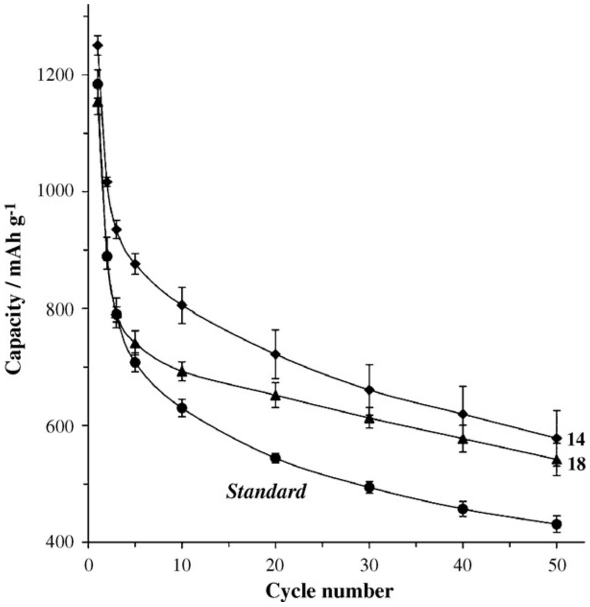


Fig. 2. The effect of 5 wt.% of polysulfides **14** and **18** added to the standard electrolyte on the discharge capacity of Li/S cells during cycling.

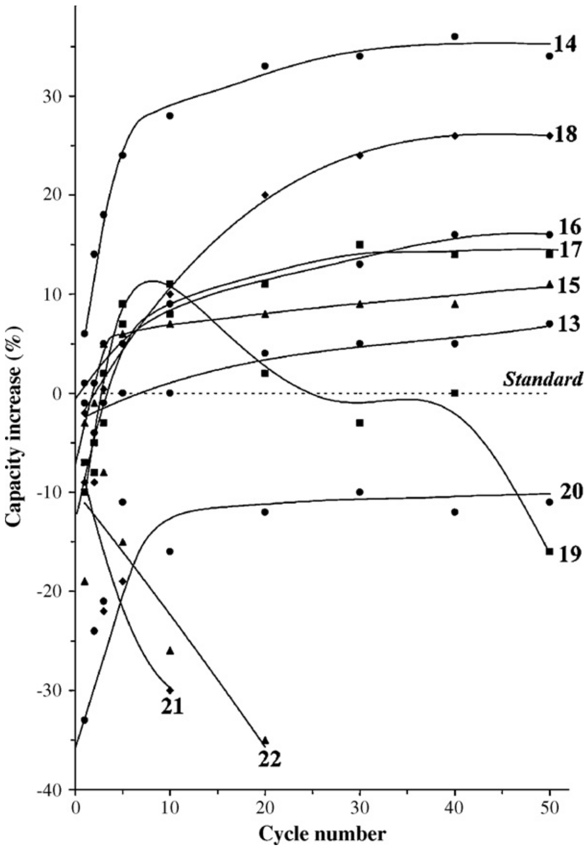


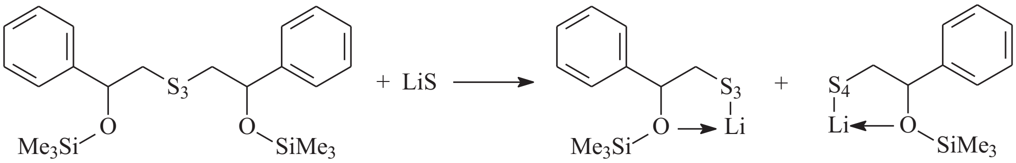
Fig. 3. The effect of polysulfides **13–22** structure on the discharge capacity of Li/S cells (5 wt.% in the standard electrolyte). The curve numbers correspond to polysulfide numbers.

the discharge capacity always higher than that of the standard cell.

Analysis of the relationship between the polysulfides structure and their effect on the battery performance (Fig. 3) allows one to divide the compounds into the three groups:

1. Active additives which increase the discharge capacity up to 25–35% relative to the standard electrolyte. These are polysulfides **14** and **18** with trimethylsilyl protection and aromatic or heteroaromatic (benzene or indole) moieties.
2. Additives of moderate activity giving 5–15% capacity increase (polysulfides **13**, **15–17**).
3. Additives with negative effect on the battery capacity (polysulfides **19–22**).

The higher activity of polysulfides **14** and **18** is likely caused by a better “solubility” of the lithium mercaptides (lithium organyl polysulfides) resulted from the insertion of Li_2S and Li_2S_2 into the polysulfide chain (see Section 1) and also by their ability to coordinate the lithium cation by the neighboring oxygen atom of trimethylsilyloxy group:

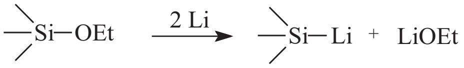


Upon charging, they release the inserted extra sulfur atoms while depositing elemental sulfur on the cathode to regenerate the starting additives (protected (bis)organyl)hydroxy polysulfides).

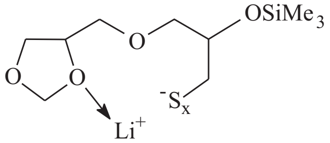
Insoluble short-chain lithium sulfides is known to react with long-chain lithium polysulfides to produce soluble polysulfides [20].

Acetal-protected (**13**) and some other trimethylsilyl protected hydroxypolysulfides (**15–17**) generally demonstrate moderate positive effect on the standard electrolyte performance.

Triethoxysilyl group located near the polysulfide bond (polysulfides **20–22**) diminishes the battery capacity probably due to the reduction of the Si–O bonds with Li metal:



The behavior of the batteries with additive **19** changes upon cycling: the capacity is higher than standard at 5–15th cycles, approximately equal to standard at 20–40th cycle and then drops (Fig. 3). The tentative explanation of this fact is based on the known ability of the oxygen-containing cycles (particularly, 1,3-dioxolanes and 1,3-dioxanes) to be polymerized under the action of alkali metal cations [21]. The first stage of this reaction is coordination of Li^+ with oxygen which may be assisted by the neighboring polysulfide moieties, e.g.:



Unsubstituted 1,3-dioxolane which is a component of the battery electrolyte can be involved in this polymerization resulting in deterioration of both electrolyte and lithium anode.

4. Conclusions

In conclusion, bis(hydroxyorganyl) polysulfides protected at hydroxyl group are promising for further systematic search of the modifiers of the Li/S battery electrolyte to improve the battery capacity and cycling life. The presence of tri(methyl)silyl group in the molecules of bis(hydroxyorganyl) polysulfides increases efficiency of the additives in terms of the battery capacity and its cycling life.

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