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Investigation of the interaction of separator materials and electrolyte solvents for Li-ion batteries

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Summary

- Setup of drop shape analysis under inert conditions (water reduced conditions)
- Measurement of contact angles and solvent uptake with commercial separators
- Correlation of wetting characteristics of separator materials and solvents

Motivation

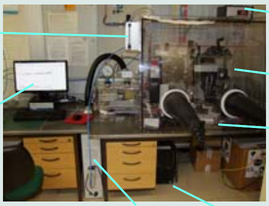
- Time consuming process of electrolyte filling during the cell preparation
- Wetting characteristics of separators with new electrolyte formulations are different
- Separators can be very hygroscopic based on nano- and micro-particles onto the separator surface
- Quantifying of the electrolyte-separator interaction
- Necessity for separator developing

Solvents and separators

- Ionic liquids
- Sulfur-containing solvents (sulfolane)
- Organic carbonates and carbonate mixtures
- EC/DMC (1:1 wt.-%) in comparison

- Glass fiber separators
- Polyolefine separators with and without ceramic particles
- PET-based separators with and without ceramic particles

Experimental setup



Solvent uptake by separators

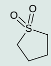
- Separator is fixed on microscope slides
- Double faced adhesive tape (3M, thickness: 50 mm, temperature stable up to 150 °C)
- Drop (2.75 ml) is put on the surface
- Contact angle after 10 seconds
- Extensive drying of the solvents and separators
- At least 10 drops per separator material

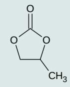
- Interaction of novel electrolyte solvents with commercial separators
- Chemical properties of solvents (conductivity, electrochemical window)
- Wetting of aluminum, glass, separators
- Time dependency of wetting characteristics:

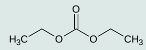
Electrochemical properties of solvents

solvent	δ [g cm^{-3}] 20 °C	η [mPa s] 20 °C	κ [mS cm^{-1}] 20 °C	Fp [°C]
Ionic liquid 1	1.5208	48.65	7.49	>250
Ionic liquid 2	1.4096	115.13	1.87	270
Sulfolane	1.2656	11.36	< 0.003	151
Mixture 4*	1.2673	14.47	< 0.02	> 140
Mixture 5*	1.3234	4.43	< 0.006	142
Propylene carbonate	1.2048	3.48	< 0.02	126
Diethylcarbonate	0.9750	0.752	< 0.003	32
EC/DMC	1.2028	1.68	< 0.002	24

* Mixture 4: Mixture of sulfolane derivatives; Mixture 5: Mixture of ethylene carbonate and sulfur containing solvent


Sulfolane

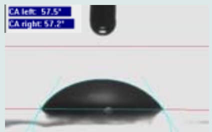

Propylene carbonate


Diethyl carbonate

- High flash points for safety enhancement of electrolytes
- Viscosity between 1 – 120 mPa·s at 20 °C

Measurement of the contact angles

- Contact angle of the reference (EC/DMC) after 10 s between 8±1° and 59±1° in dependence of separator material
- Contact angles of all solvents between 5° und 117° in dependence of solvent and separator material



- Significant improvement of the wetting characteristics by ceramic particles
- Dependence of the contact angle from separator porosity (the smaller the porosity the poorer the wetting characteristics)
- Small contact angles (13°) can be obtained also with ionic liquids

Solvent-uptake by separators: time dependency

Separator	Contact angle after dropping [°]	Contact angle after 5.6 h [°]	Porosity [%]	sorpivity [x10^{-2} mm min$^{-1/2}$]	R^2	specific surface area [m^2 g^{-1}]
S-2*	42.1	7.5	-	1.03	0.991	1.52
S-3	104.2	51.1	39	-	-	56.5
S-4	78.6	60.1	39	-	-	23.7
S-5	80.7	59.8	45	0.31	0.979	50.5
S-6	102.7	64.3	42	0.56	0.908	42.4
S-7*	95.5	5.0	-	1.59	0.978	23.7
S-8*	108.1	55.1	59	1.00	0.895	3.90
S-9*	105.9	34.0	59	2.00	0.991	1.55
S-10*	100.2	10.4	55	1.93	0.920	2.76
S-11	104.2	51.1	60	0.16	0.939	0.54

* Coated with ceramic particles

- Solvent mixture: Mixture 5
- Different solvent uptake in dependence of the separator
- Decrease of the contact angle with time
- Calculation of sorptivity $S = V \cdot A^{-1} \cdot t^{-1/2}$ as an estimation of the solvent uptake

Conclusions

- Solvents based on safety and high voltage applications
- Setup of drop shape analysis under inert conditions (water reduced conditions)
- Measurement of contact angles and solvent uptake with commercial separators
- Correlation of wetting characteristics of separator materials and solvents

Acknowledgements

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