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DESIGN OF ORGANIC MATERIALS FOR ELECTROCHEMICAL ENERGY STORAGE

Oleg V. Levin,

Saint Petersburg State University, 2019

Just so easy, with help of simple tools, one can turn white (or gray) BREAD into a TROLLEYBUS…

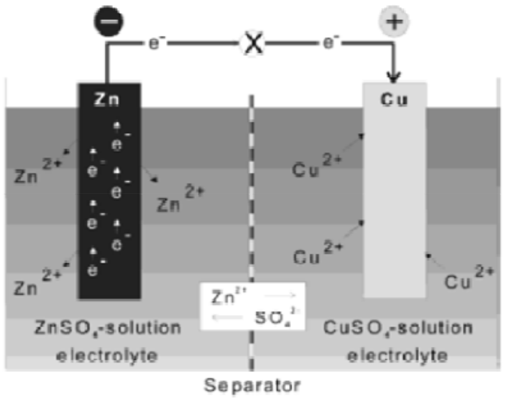
BUT WHAT FOR?!

Electrochemical power sources: formal requirements

$$\nu_1 \mathrm{Red}_1 + \nu_2 \mathrm{Ox}_2 \rightarrow \nu'_1 \mathrm{Ox}_1 + \nu'_2 \mathrm{Red}_2,\ \Delta G^0$$

$\nu_1 \mathrm{Red}_1 - ne^- \rightarrow \nu'_1 \mathrm{Ox}_1$ $\qquad$ $\nu_2 \mathrm{Ox}_2 + ne^- \rightarrow \nu'_2 \mathrm{Red}_2$

$$E = -\frac{\Delta G}{nF}$$



	Anode	Electrolyte Separator	Cathode
Requirements on electron conduction:	must	no	must
ion conduction:	can	must	can

Formal: any redox-pair

Limitation:

1) Fundamental (kinetics, reversibility, reliability)

2) Practical (Cycle and shell life, Form-factor)

Numbers:

Cell:

Energy density $W_{max} = nFU/\Sigma m$ or $W_{max} = nFU/\Sigma V$

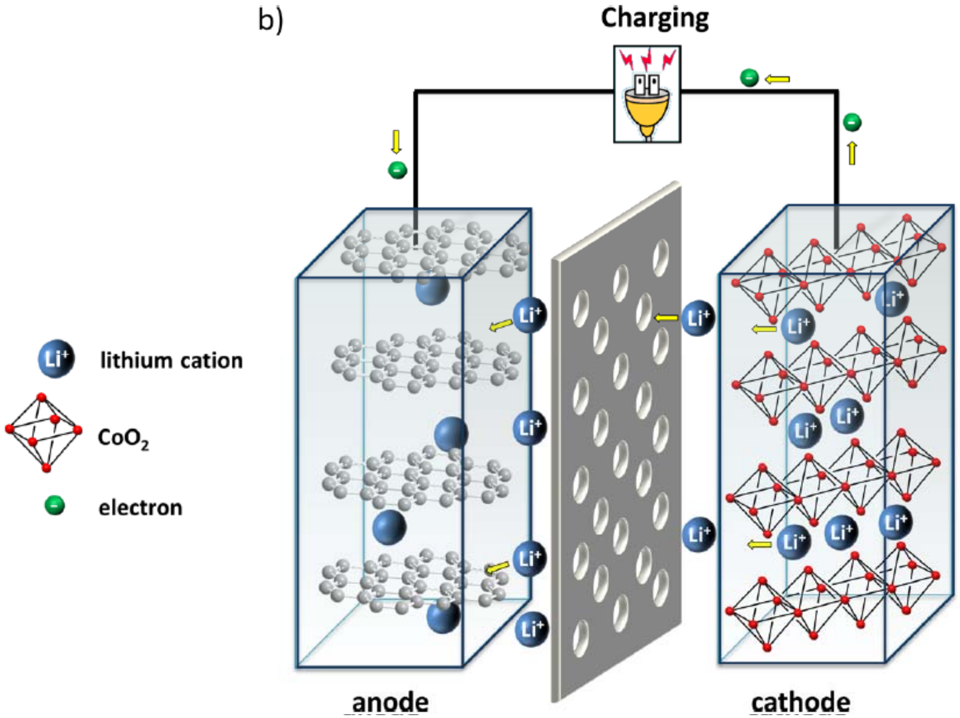
Power density $P = UI/\Sigma m$ or $P = UI/\Sigma V$

Electrode:

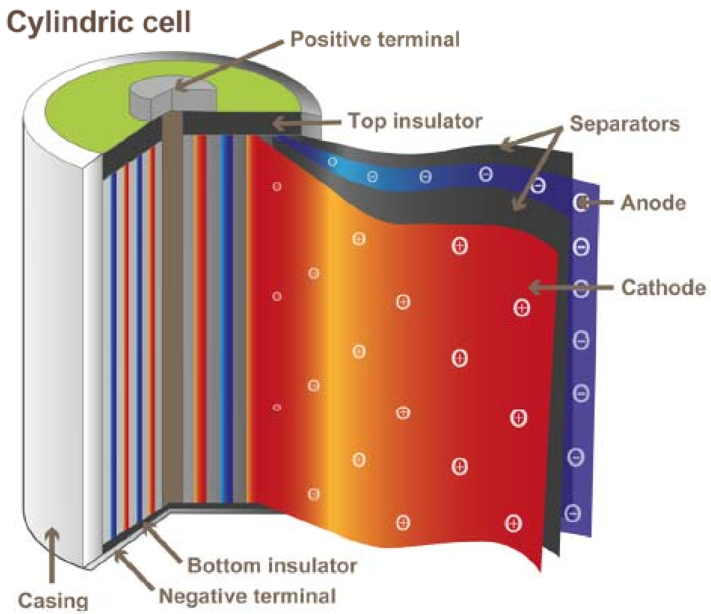
Capacity $\qquad Q_{max} = nF/\Sigma m$

EMF $\qquad E = -\Delta G/nF$

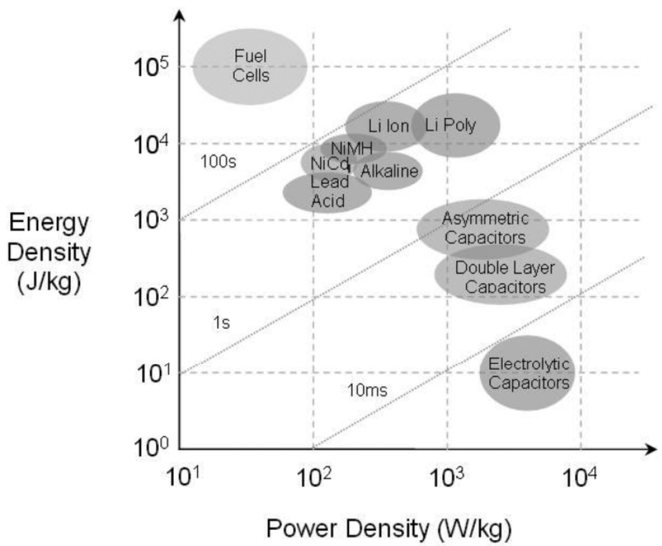
Current status



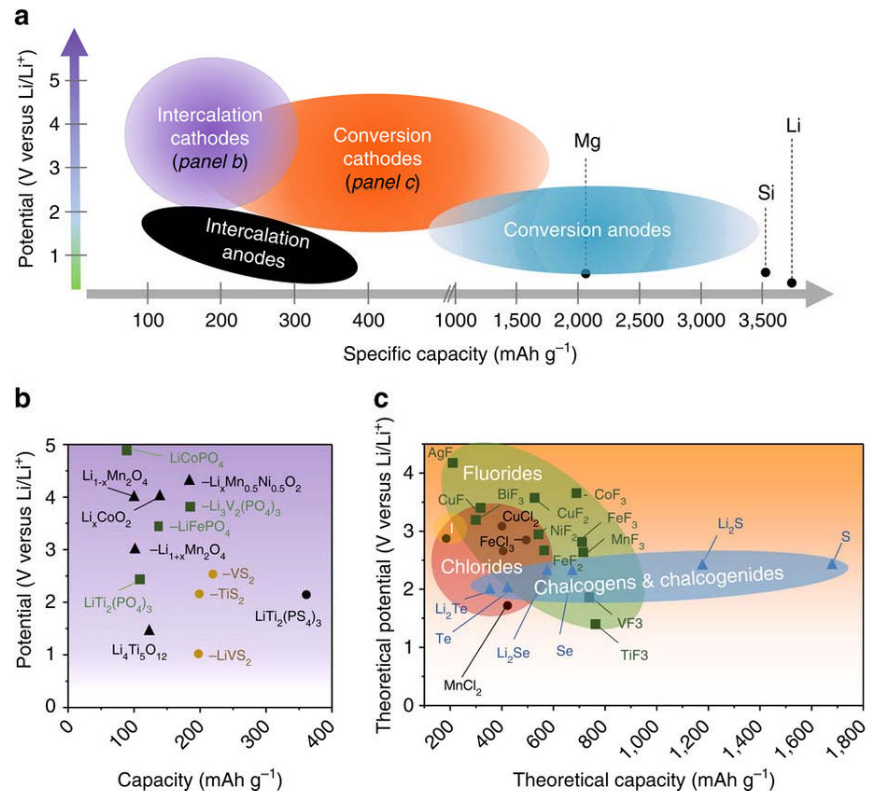
Metal-ion battery: low electrolyte content -> W!



Current status



Ragone chart for capacitors, supercapacitors, batteries and fuel cells

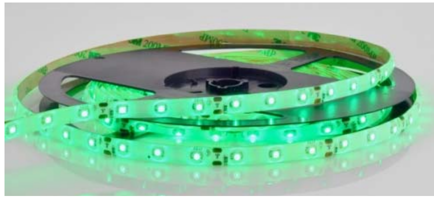


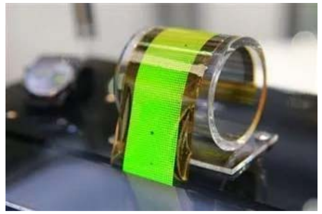
Sensors **2008**, *8*(12), 8037-8066; doi:10.3390/s8128037

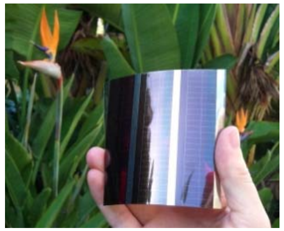
Nature Communications **7**, Article number: 12647 (2016)

Technology trends

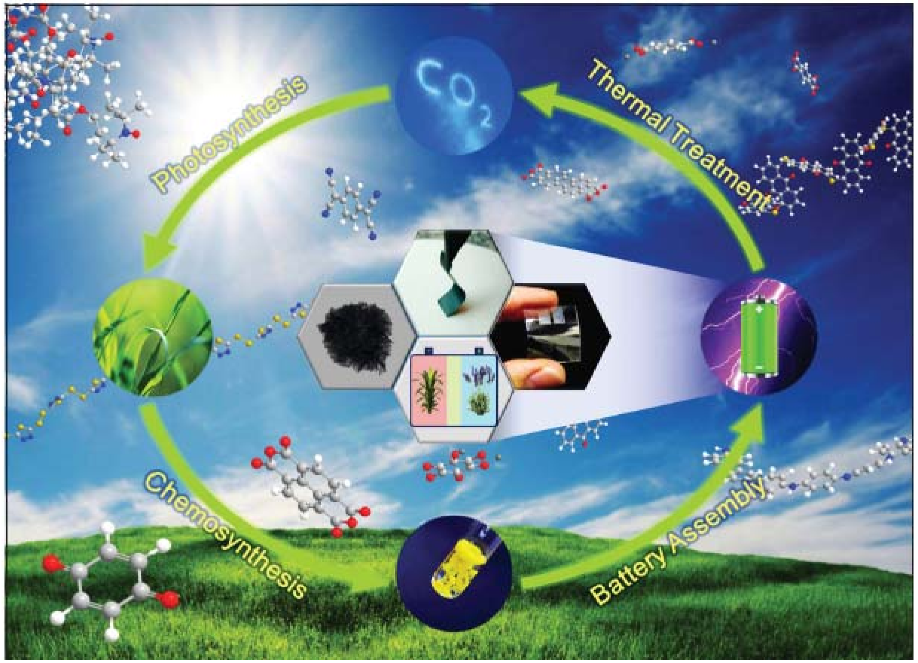
- **Inorganic → Organic**
  - ✓steel→ plastic
  - ✓LED → OLED
  - ➢Si → DSSC, Organic PV
- Batteries?







Organic material advantages



- Ltght·welght, flexIble, thin-film processable
- Less energy - consuming wet fabrication process
- Less-limited organic resources
- Easy disposability. Burnable away without toxic gas and ash formation
- Less-toxic, no-Ignition non-fuming ..... Safe & environmentally benign

Song & Zhou, *Energy Environ. Sci., 2013,* ***6, 2280.***

BATTERIES – PRESENT AND FUTURE CHALLENGES

Emerging battery technologies towards 2025

Helena Berg, AB Libergreen
Aleksandar Matic, Chalmers
Patrik Johansson, Chalmers
Goteborg, May 2015.

Table 7. Cost trend estimates (cost/storage capacity) for emerging battery technologies, compared to the improved Li-ion technology (- refers to relative cost reduction, + refers to relative cost increase).

Technology	Cost – cell	Cost – pack*	Cost driver
Solid Li-metal	- 8%	- 6%	Anode cost 1/3 of Li-ion, no Cu used
Na-ion	- 13%	- 10%	20% lower cell material cost
Mg	± 10%	+ 75%	Low cell voltage
Li-S	- 40%	> 100%	Low-cost raw materials. High pack cost due to low cell voltage and poor rate capabilities
Li-O_2	± 0%	+ 250%	Low electrode cost, high electrolyte cost, low cell voltage and poor rate capabilities, extra components for air/oxygen handling not included.
Organic	- 50%	- 35%	Low cell voltage
Asymmetric super capacitors**	± 0%	± 0%	High rate capabilities, low energy density

*The same cost for electronics, control, and management are assumed for all technologies. **HEV application only.

State of art Li-ion 2032 cell

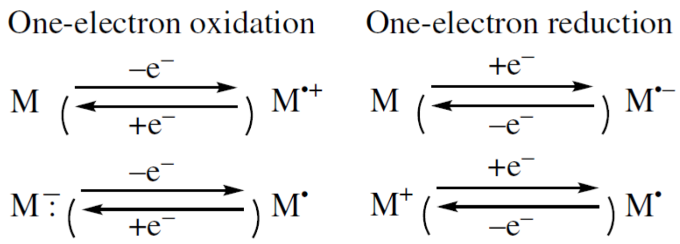
Feature	LIR2032 battery can maintain 80% capacity after 500 cycles
Model	LIR2032
Nominal Voltage	3.6V
Nominal Capacity	40mAh
Max discharge current	12mA
Max pulse discharge current	75mA
Dimensions(Dia x H)	20mm (0.8") x 3.2mm(0.1")
Weight	3.1 grams (0.1Oz)

TABLE 1. Specifications of the Al–Li/Polyaniline Coin-Type Cells

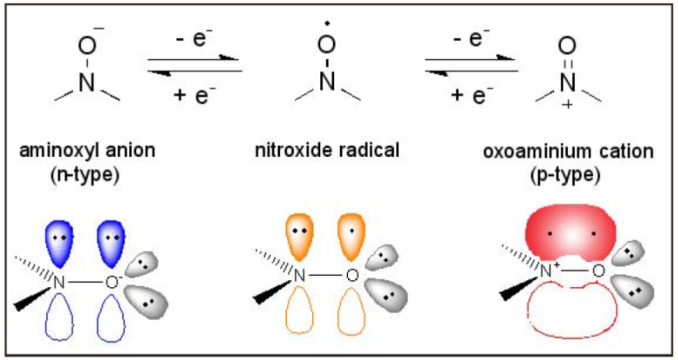
	AL 2016	AL 2032	AL 920
Dimension Diameter (mm)	20	20	9
Dimension Thickness (mm)	1. 6	3. 2	2. 0
Weight (g)	1. 7	2. 6	0. 4
Nominal Voltage (v)	3		
Nominal Operating Voltage (V)	3 ～ 2		
Nominal Capacity (mAh)	3	8	0. 5
Standard Current (A)	1μ ～ 5m	1μ ～ 5m	1μ ～ 1m
Cycle Life depth (mAh)	1	3	0. 1
Cycle Life life (cycles)	more than 1,000	more than 1,000	more than 1,000
Operating Temperature (℃)	−20 ～ +60		
Recommended Charging Method	Constant Voltage Charge		

J.S. Miller, *Advanced Materials*, 5 (**1993**) 671-676
T. Matsunaga, H. Daifuku, T. Nakajima, T. Kawagoe, *Polymers for Advanced Technologies*, 1 (**1990**) 33-39

Charge balance in organic electrode materials



Example:



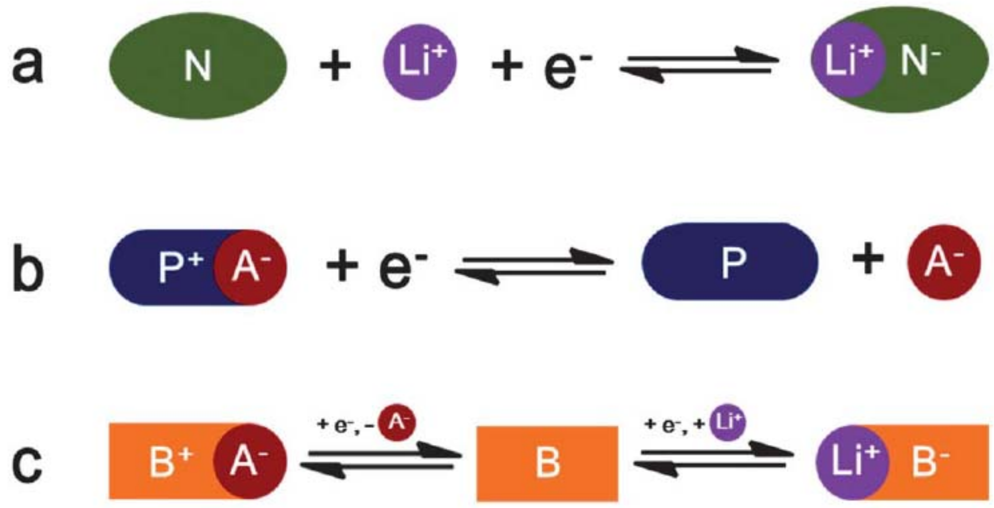
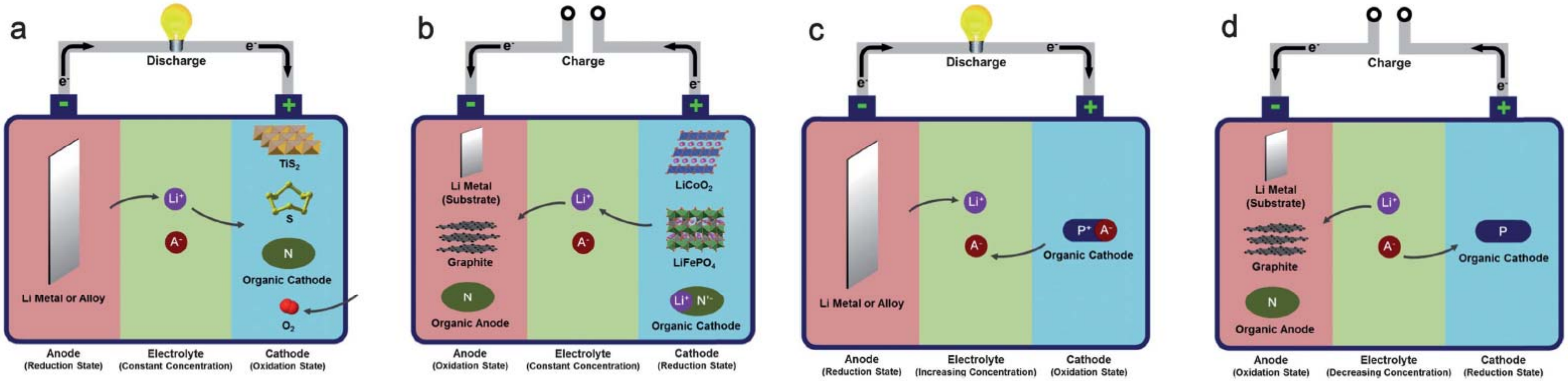


Fig. 1 The redox reaction of three types of electroactive organics: (a) n-type; (b) p-type; (c) bipolar. A^- means anion of the electrolyte and Li^+ can be replaced by other alkali ions.

Energy Environ. Sci., 2013, 6, 2280–2301

Cell configurations



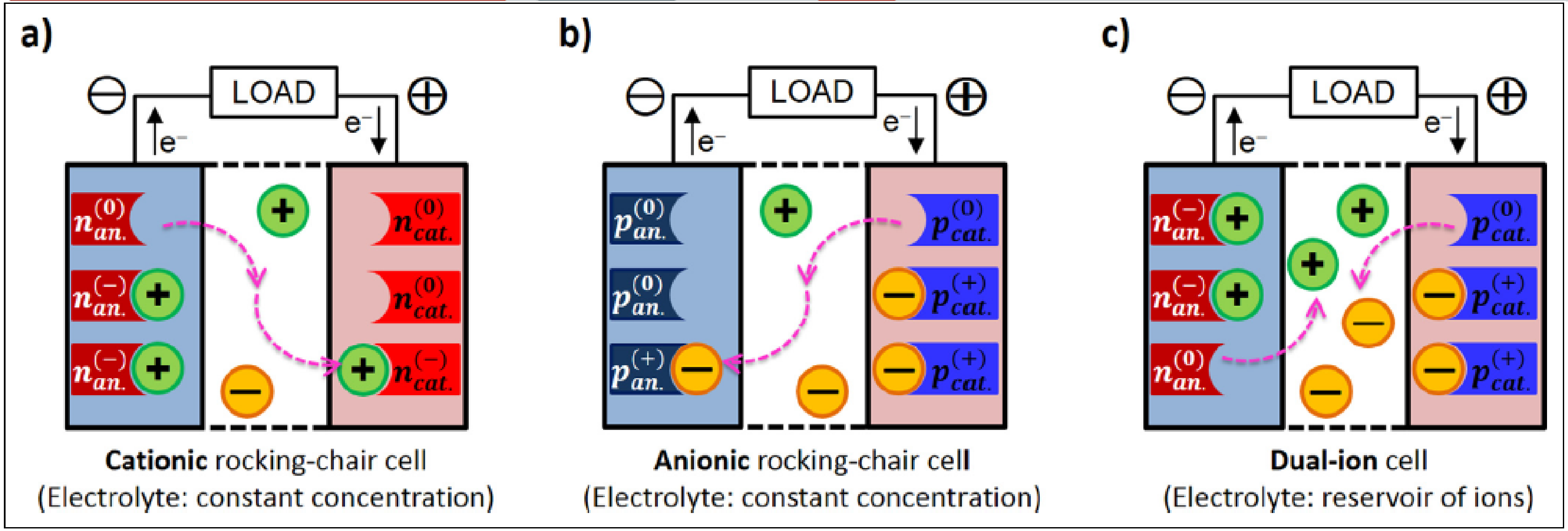
•Cathode and anode balance

•Attention to initial states of electrodes

•Attention to doping mechanisms

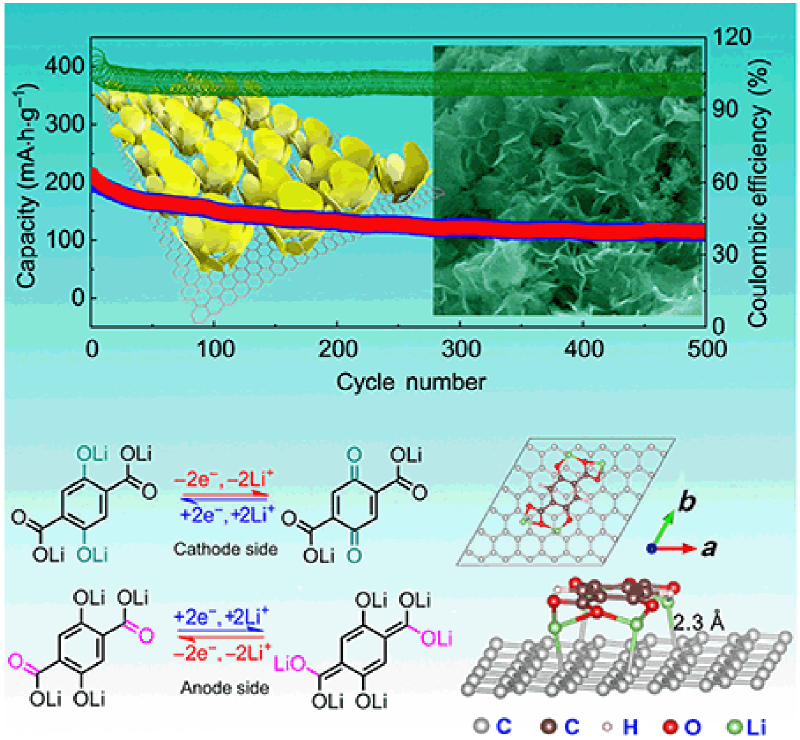
Energy Environ. Sci., 2013, 6, 2280–2301

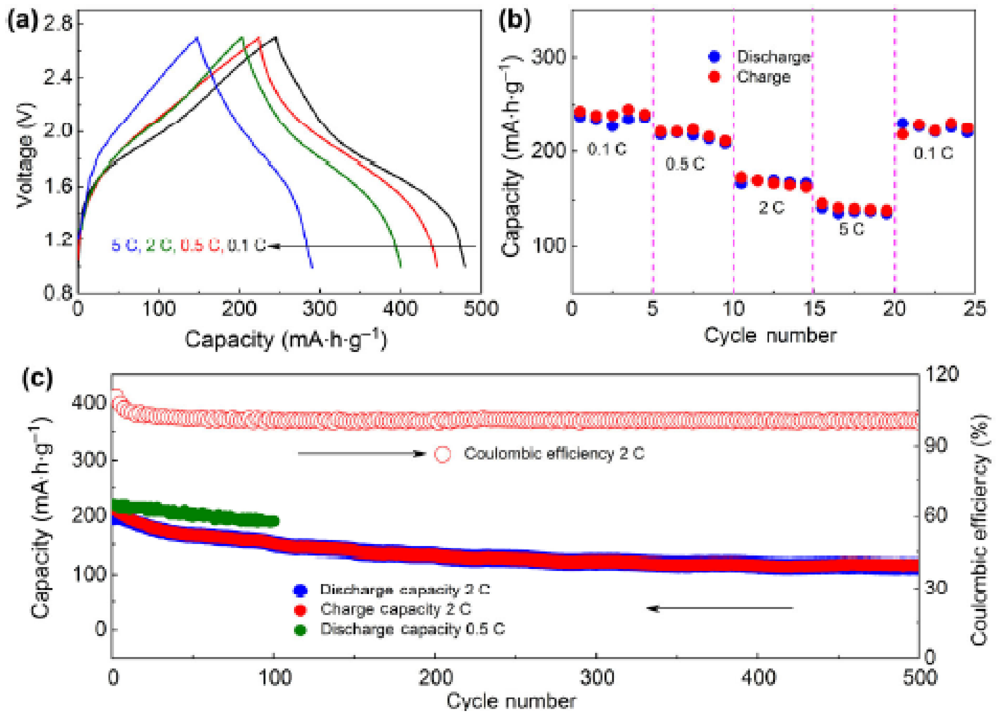
Organic materials – no selectivity to metal ion



Indeed, the shuttling ion can be either protons or ammonium-type cations but also anions.

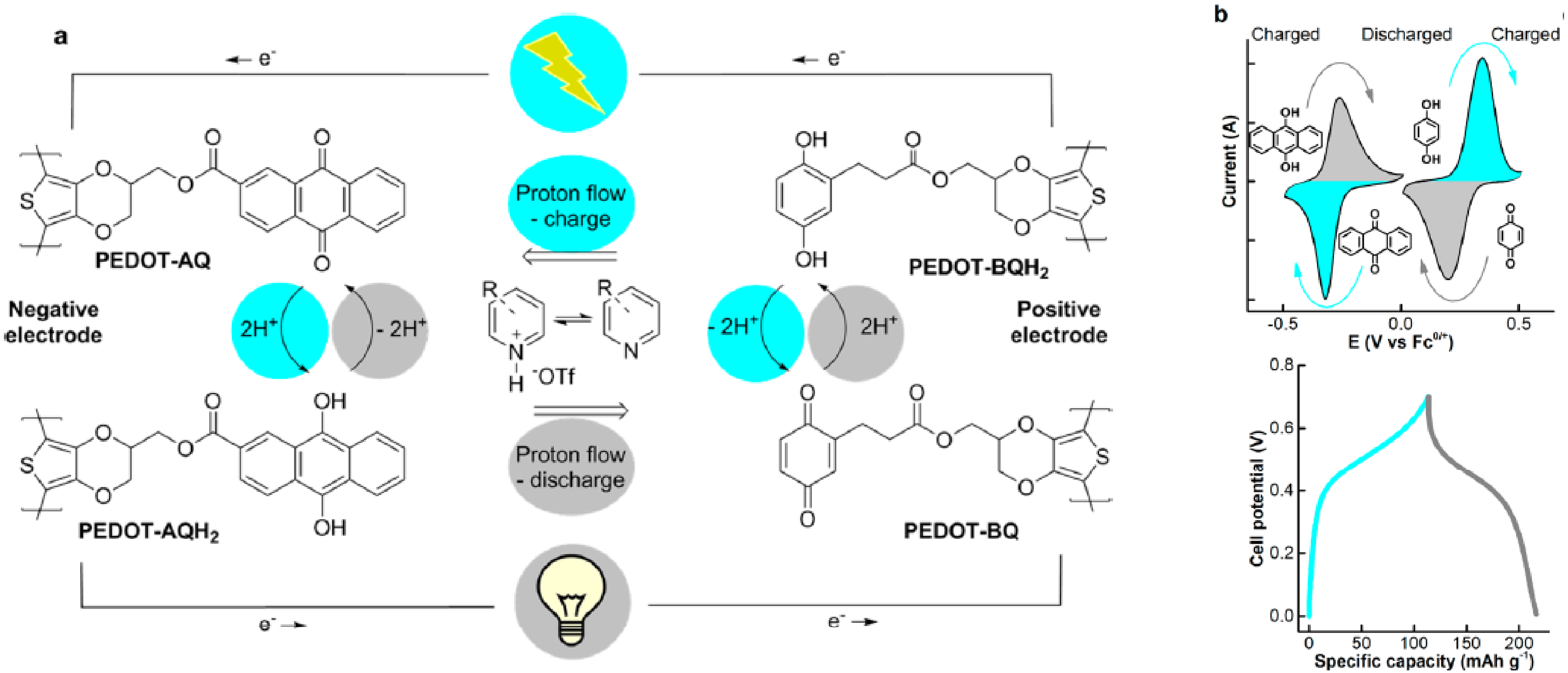
Remarkable example: Rocking-chair Li-ion cell





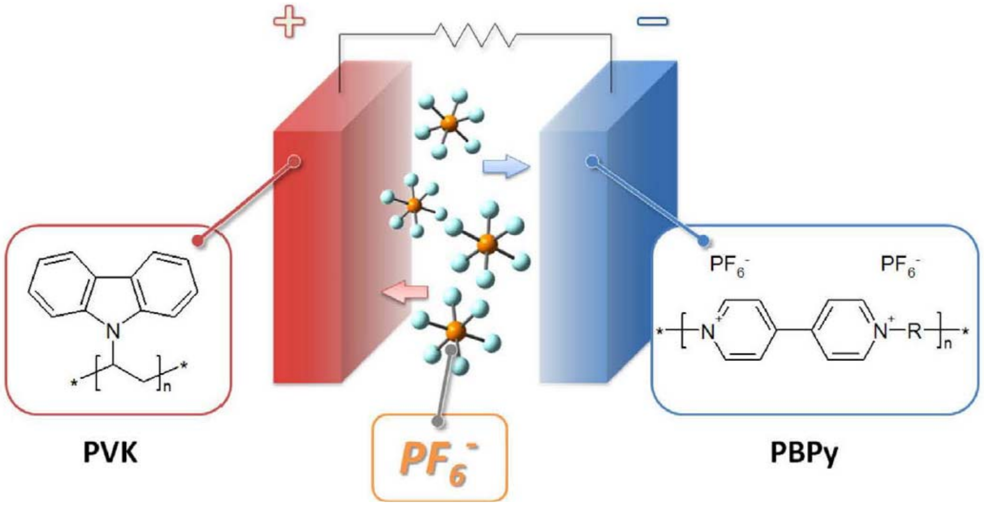
Zhao, Q., Wang, J., Chen, C. et al. Nano Res. (2017) 10: 4245. https://doi.org/10.1007/s12274-017-1580-9

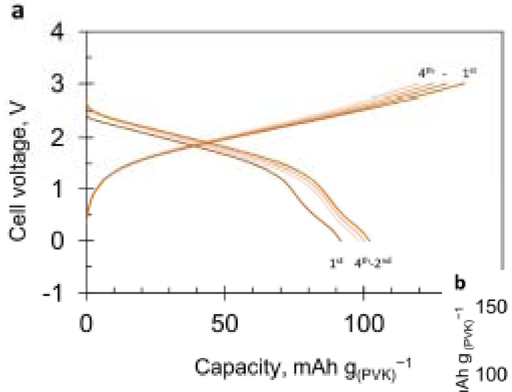
Remarkable example: Rocking-chair organic H-ion cell

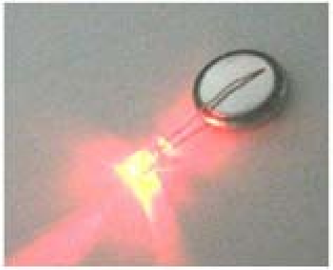


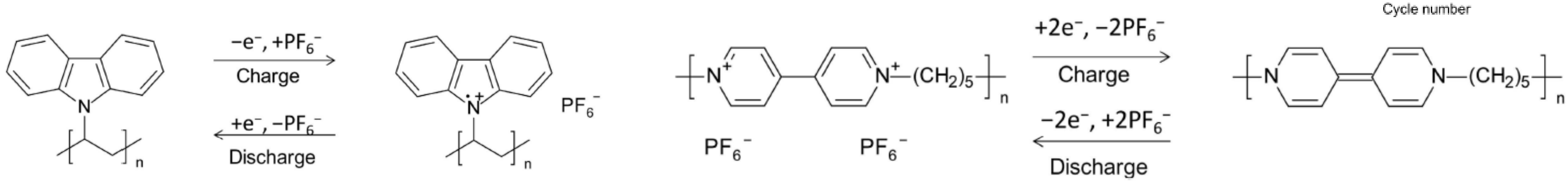
 *Rikard Emanuelsson, Mia Sterby, Maria Strømme, Martin Sjödin J. Am. Chem. Soc.*2017139134828-4834

Remarkable example: Rocking-chair anion cell









 Masaru Yao, Hikaru Sano, Hisanori Ando & Tetsu Kiyobayashi Scientific **Reports, 2015 | 5:10962 | DOI: 10.1038/srep10962**

Modern organic electrode materials – low temperature

Yonggang Wang, Yongyao Xia and their colleagues at Fudan University in Shanghai, China: first discussion of this feature (2018)

At −70 °C, the team's battery retained 70% of the capacity it had at room temperature (at 0.5 C) and 20% (at 5 C).

Why?

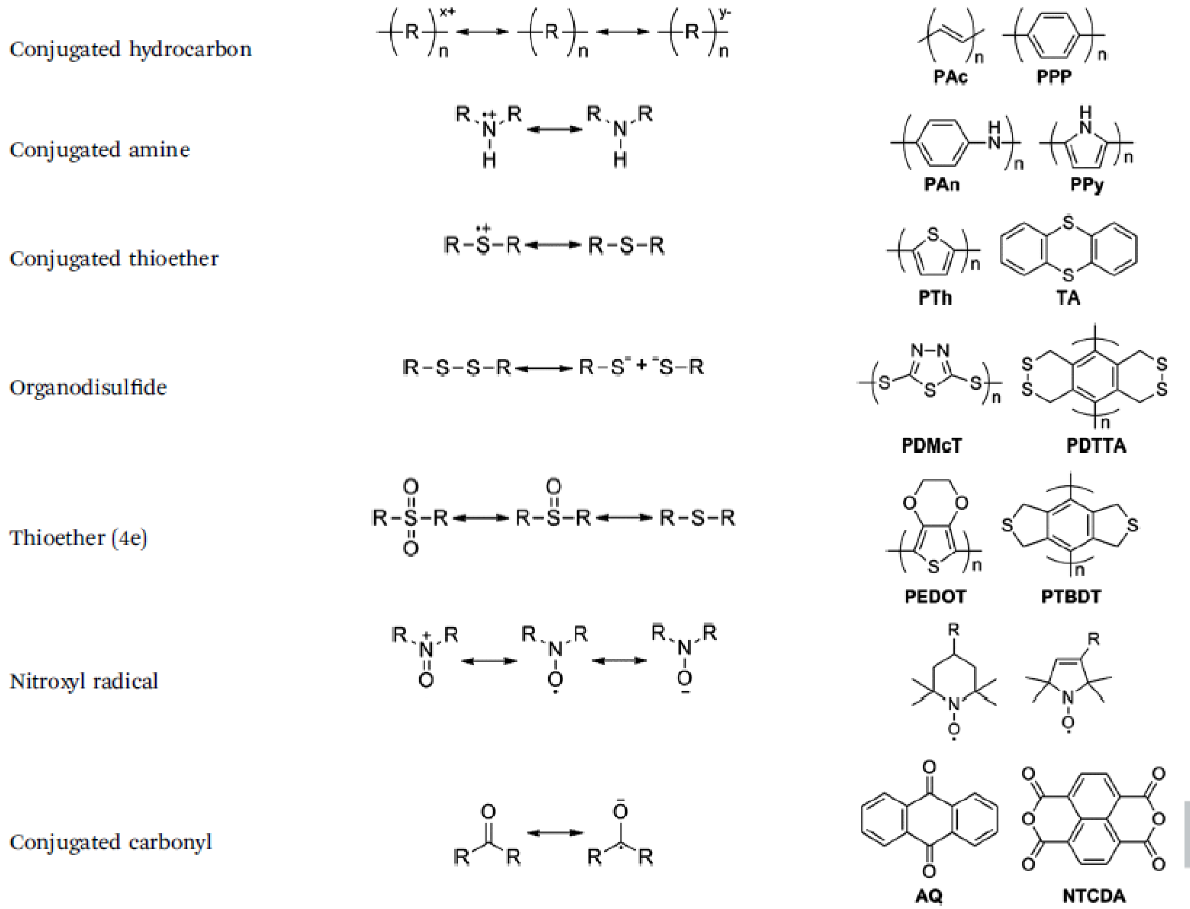
- An ethyl acetate-based electrolyte
- No need of sluggish desolvation of Li^+, which limits low-temperature operation of the inorganic batteries

But…

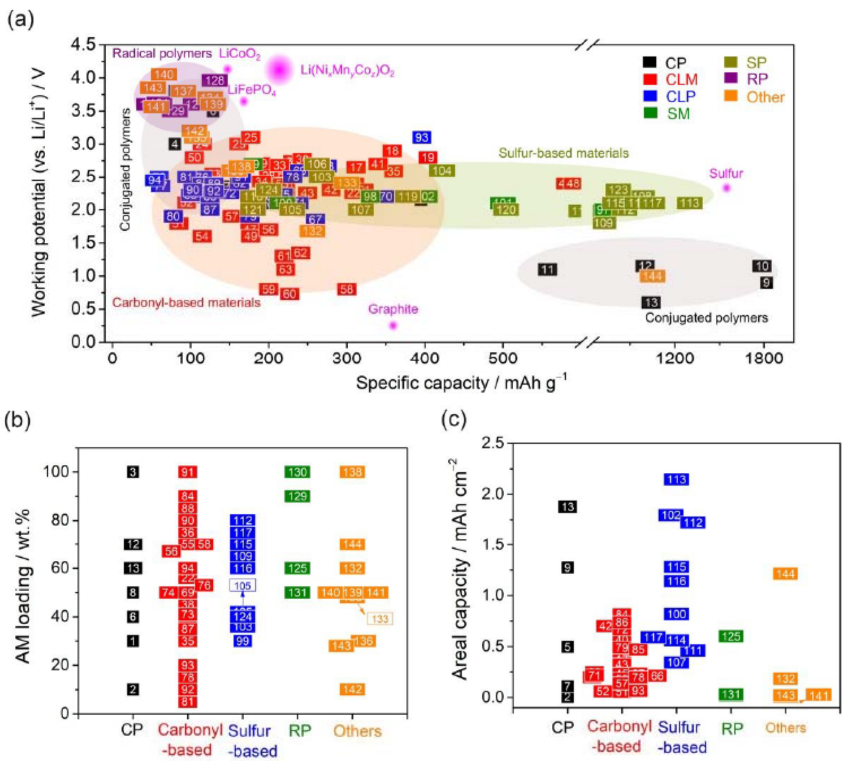
- Energy density only 33 Wh kg^{-1}

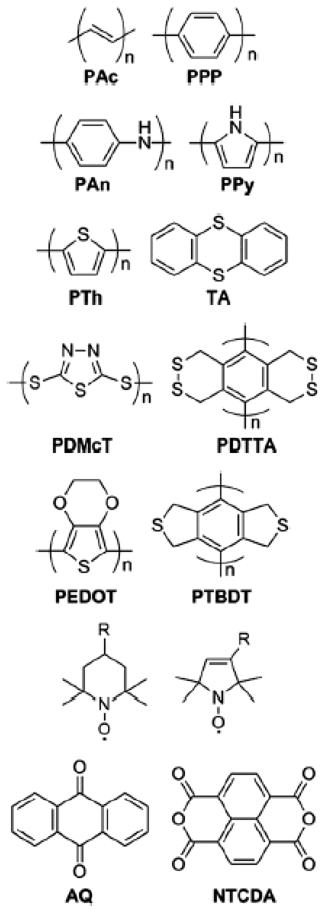
Xiaoli Dong, Zhaowei Guo, Ziyang Guo, Yonggang Wang, Yongyao Xia. **Organic Batteries Operated at −70°C**. *Joule*, 2018; DOI: 10.1016/j.joule.2018.01.017

Electrode material classes



Modern organic electrode materials - numbers





CP
π- conjugated systems, «conducting polymers»

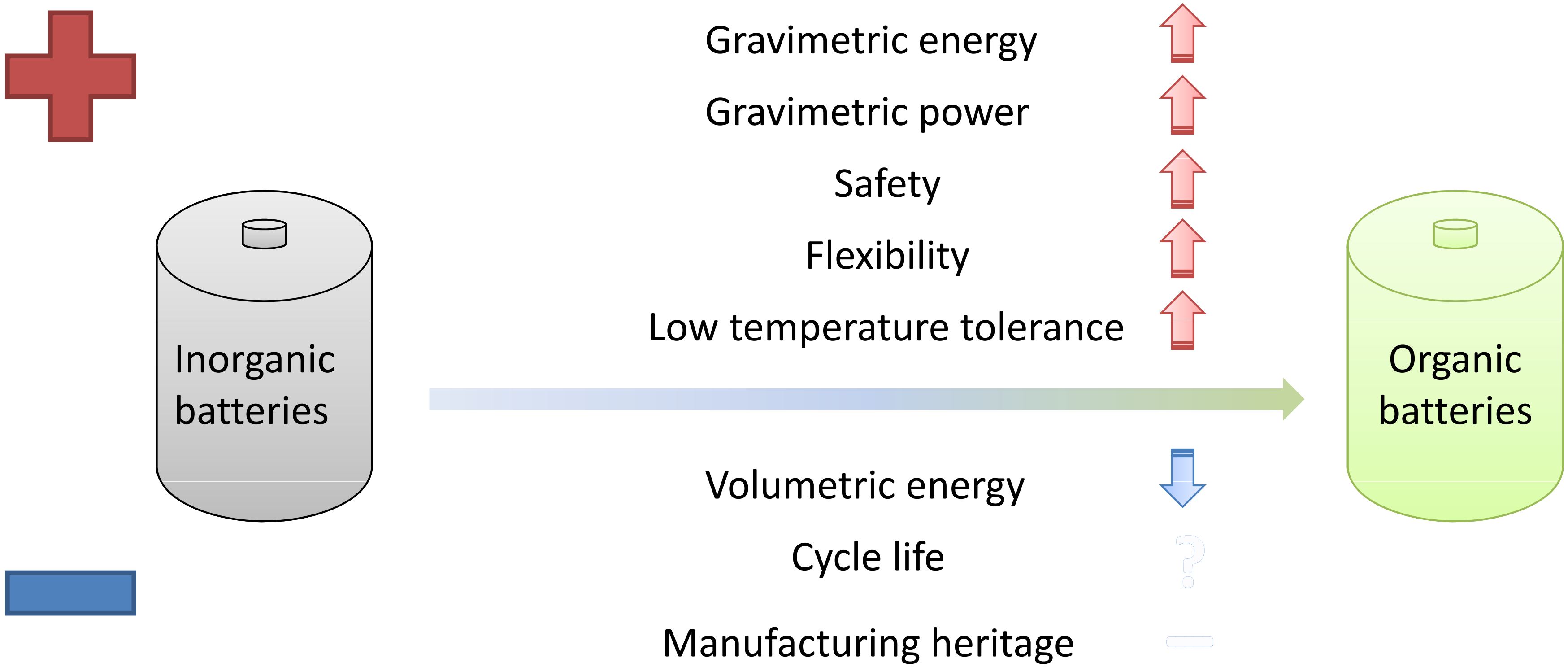
SP
Sulfur based materials

RP
Radical polymers

CP
Carbonyl-based

Energy Density Assessment of Organic Batteries Xabier Judez, Lixin Qiao, Michel Armand, and Heng Zhang *ACS Appl. Energy Mater.*, ***2019***

Moving to organic materials

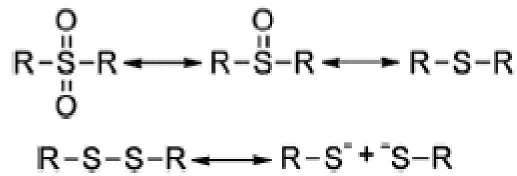


Energy Density Assessment of Organic Batteries Xabier Judez, Lixin Qiao, Michel Armand, and Heng Zhang *ACS Appl. Energy Mater.*, ***2019***

Wishlist for organic electrode materials

- Chemical and thermodinamical reversibility

High activation barrier



- Redox-potential

cathode: 2-4 V vs. Li/Li^{+}

anode: 0-2 V vs. Li/Li^{+}

p-type compounds have higher potential;
Potential is tuned by donating
(–OH, $–NH_2$, $–OCH_3$)
or acceptor groups (–Cl, –F, –CN, $–NO_2$)

- Higher capacity

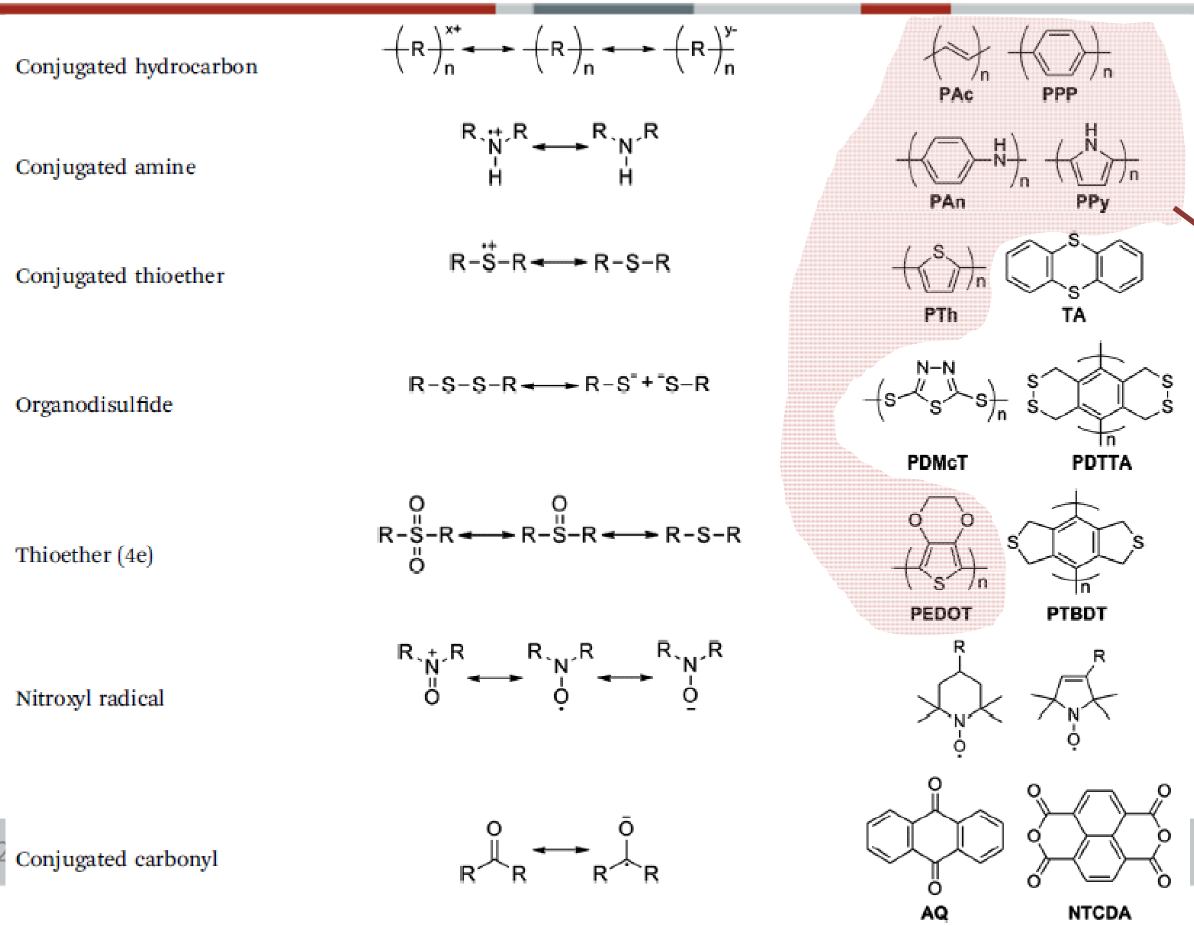
Multielectrone reactions;
Lower molar mass

- Low solubility

Small molecules are soluble in electrolytes
To avoid solubility: polymerization, cross-linking

- Price, synthetic scalability

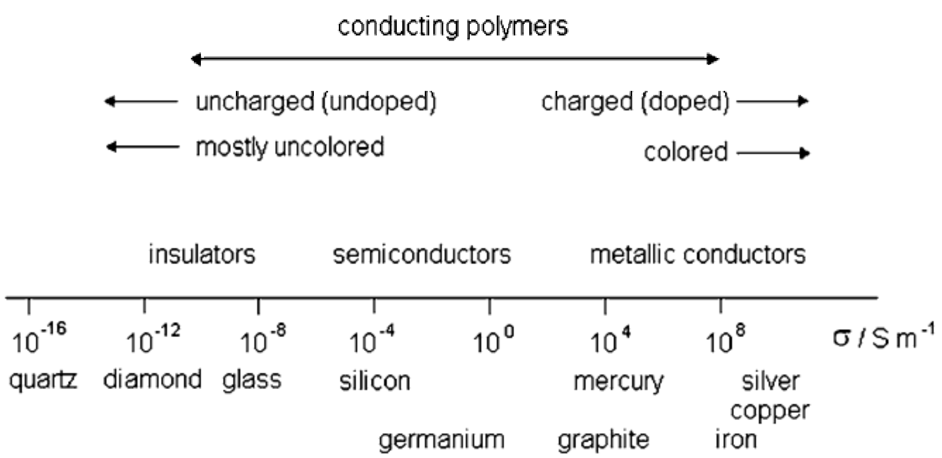
Typical materials and their features



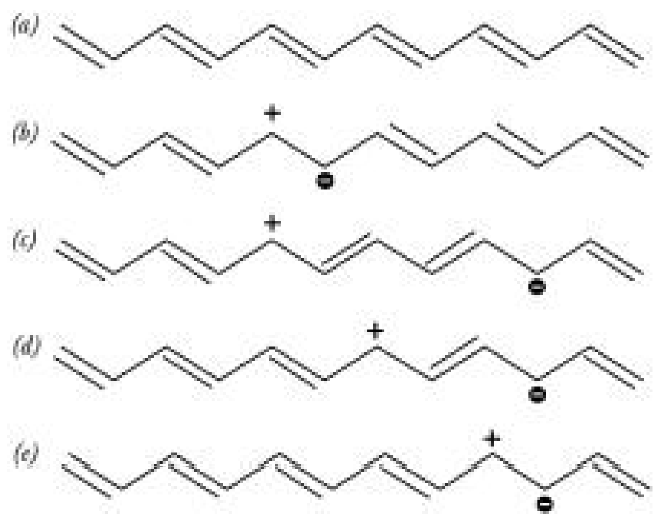
π-conjugated, «conducting polymers»

π-conjugated, «conducting polymers»

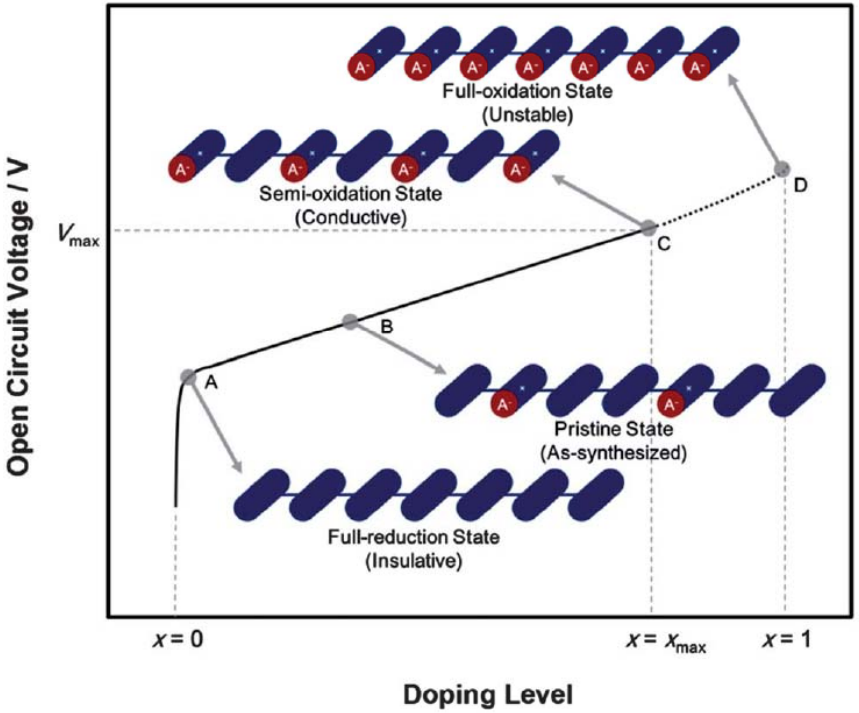
- High conductance



- Charge transfer through the conjugated bond system



π-conjugated, «conducting polymers»



+ High conductance and ionic conductivity
+ High power
+ May be used as a basis for composites

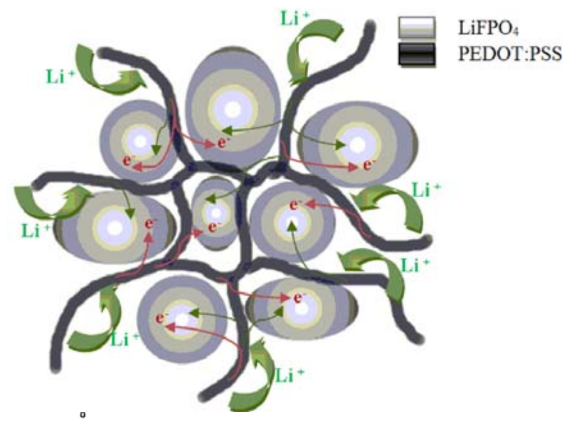
- Capacity is limited by doping level
-Overoxidation and stability problems
-Low processability (neither soluble, nor melting)

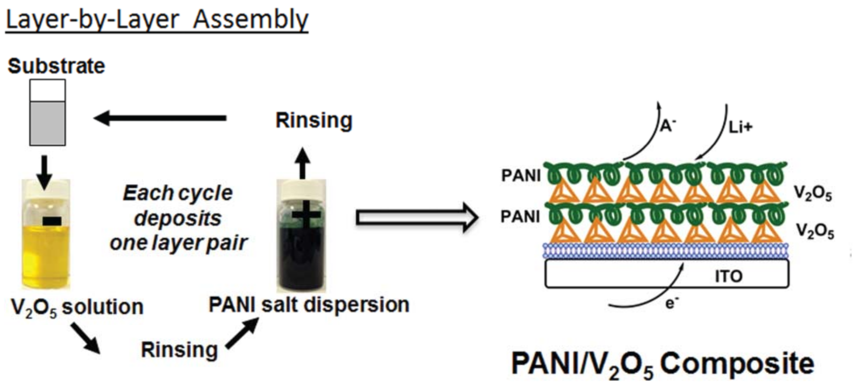
➢**p- and n- doping possible,**

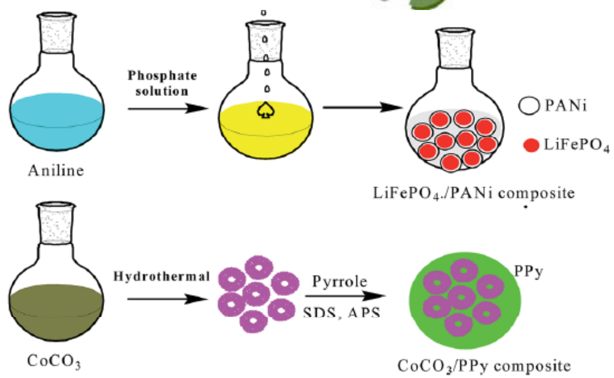
➢**p- doping is more frequent**

Tasks for «conducting polymers»

- Applying them as a conductive backbone of composites
- Processability enhancing
- Grafting by functional groups



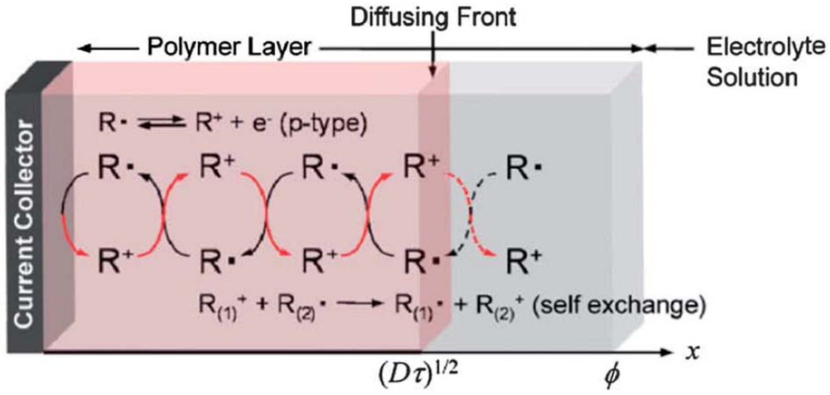




JOURNAL OF POLYMER SCIENCE, PART B: POLYMER PHYSICS 2013, 51, 468–480

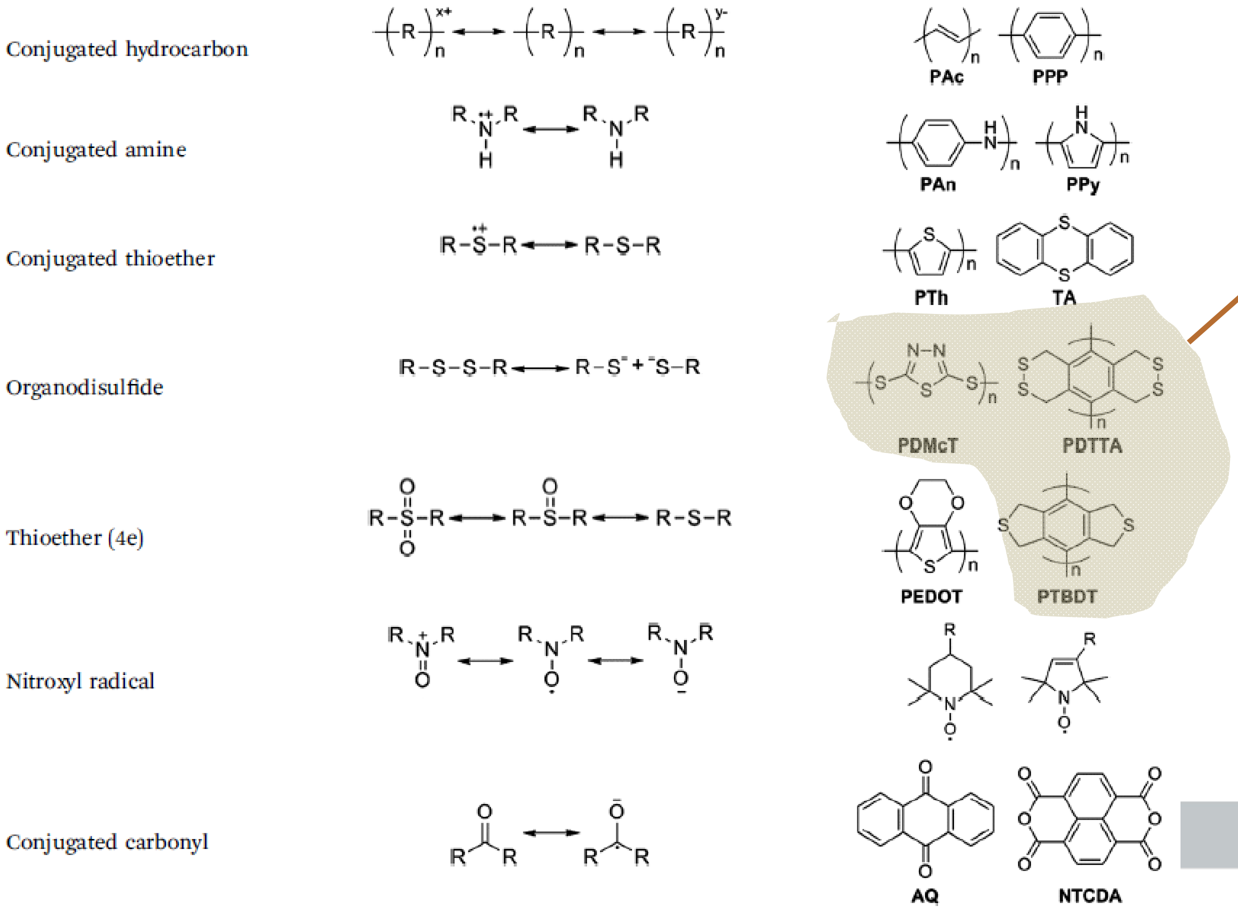
RSC Adv., 2015, 5, 42109–42130

Nonconjugated (redox) polymers



- Hopping mechanism of charge transfer
  - Low electronic conductivity
  - "Diffusing front" kinetics
- All type of doping is possible
- Variety of structures
- "flat" discharge curve

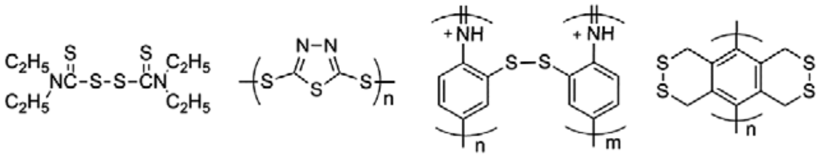
Typical materials and their features



Sulfides – they posses bond cleavage at charge

Organic sulfides

$$-(SRS)-_n + 2ne^- + 2nLi^+ \leftrightarrow nLiSRSLi$$



+ Two electron reaction – higher capacity

+ Doped by metal ions

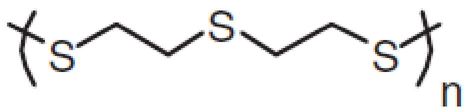
- Sluggish kinetics

- Often soluble in electrolyte

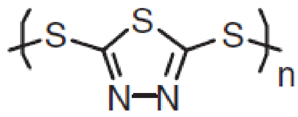
- Low electronic conductivity

Tasks for «organic sulfides»

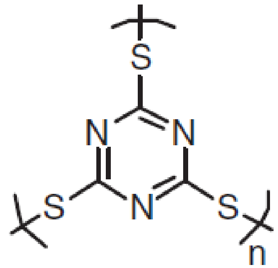
Potential tuning by functionalization



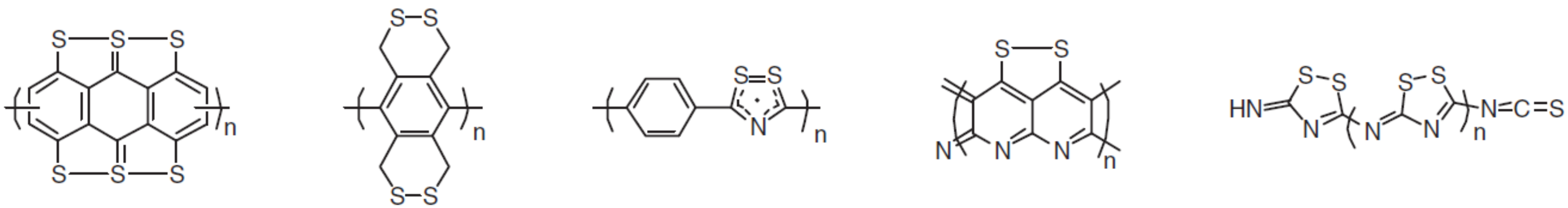
2.3 B



3 B

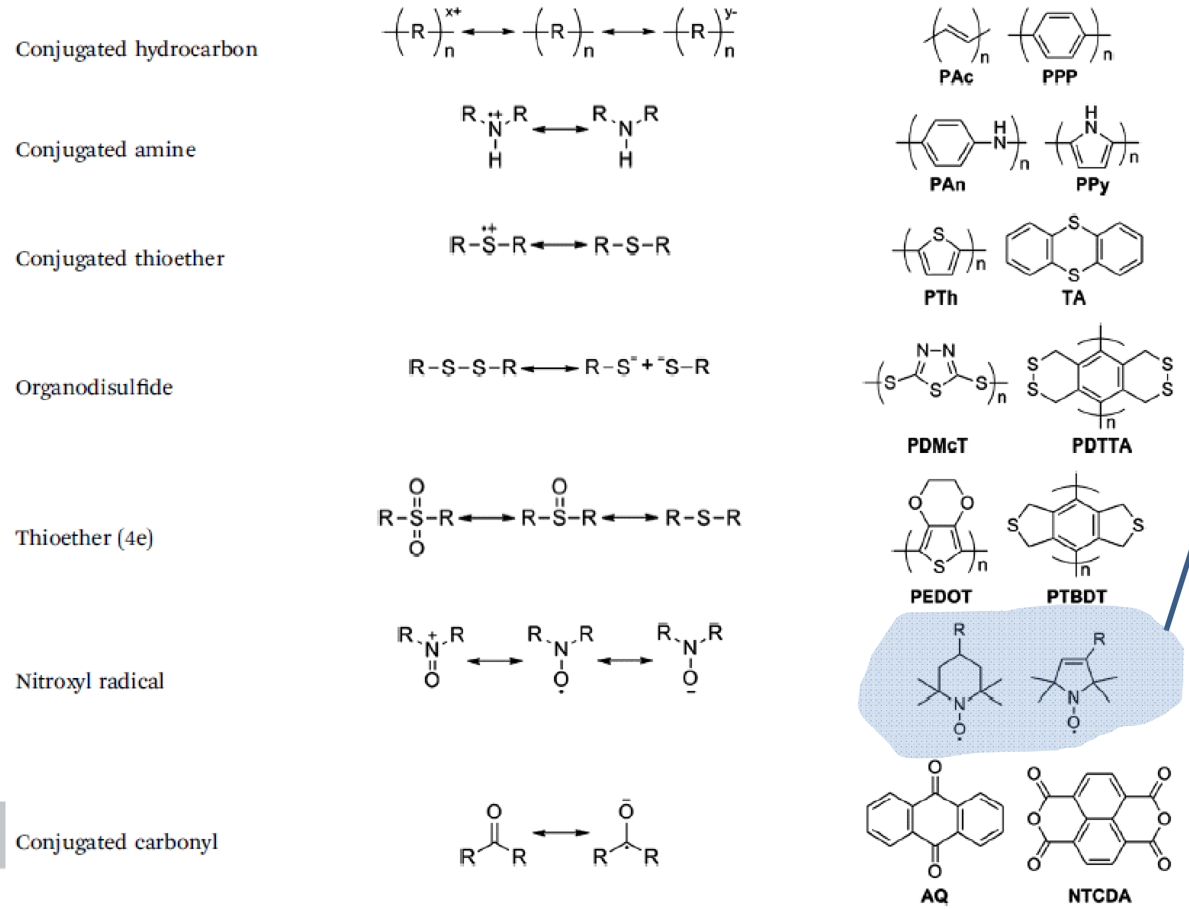


Lowering of activation energy and solubility – using the side chains



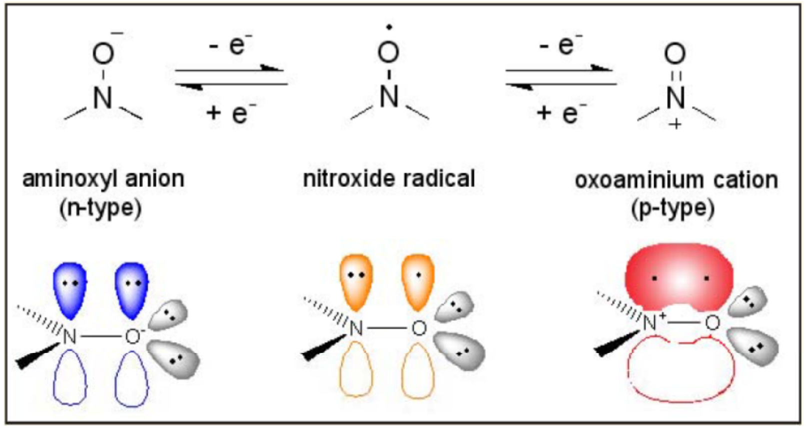
Adv. Energy Mater. ***2011, 2, 742–769***

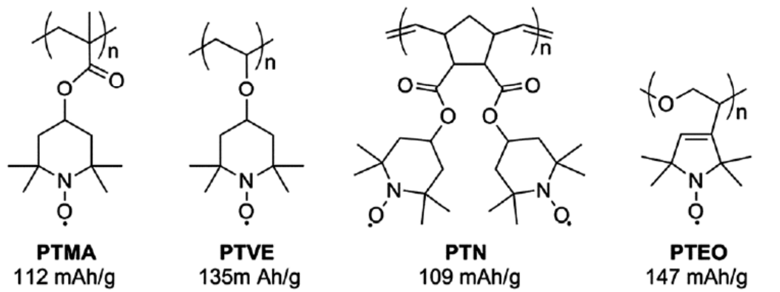
Typical materials and their features



Nitroxyl radical polymers: very fast and reversible

Nitroxyl radical polymers





+ Reversible process, high stability

+ Fast kinetics

- High molecular weight – low capacity

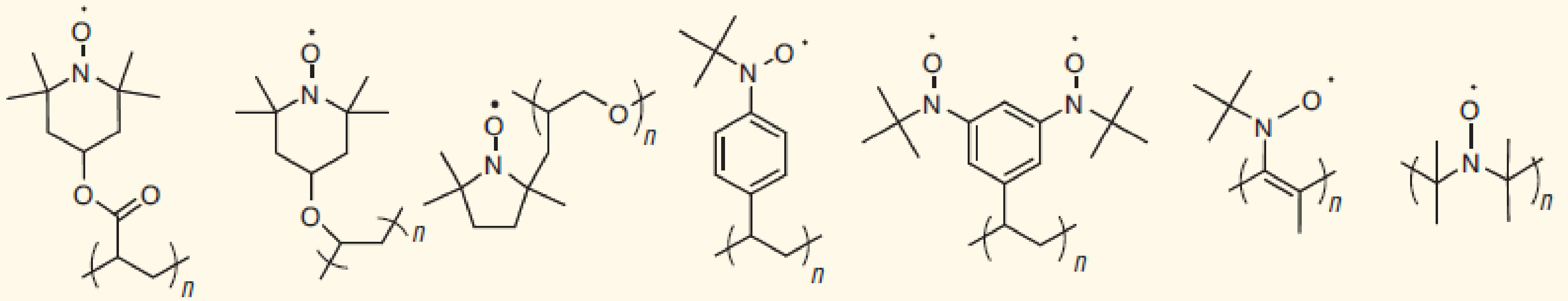
- Low electronic conductance

➢**Mostly p-doped**

Tasks for “Nitroxyl radical polymers”

- Increase the capacity – decrease the mass

(p-type)

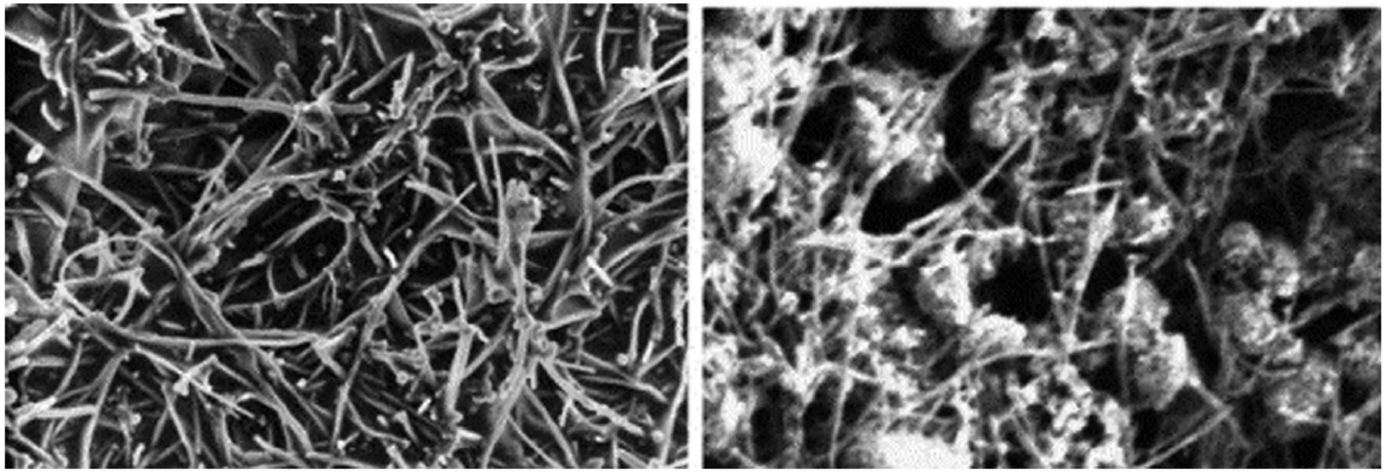


	1	2	3	4	5	6	7
Mw	226	198	184	196	138	127	114
Theoretical capacity (Ah/kg)	118	135	145	141	194	211	224

Nishide and Suga, The Electrochemical Society *Interface* • *Winter 2005*

Tasks for “Nitroxyl radical polymers”

- Increase the conductance – make composites



SEM images of PTMA−carbon composite electrode made by (a) liquid−solid and (b) solid−solid mixing methods.

Up to 70 % of inert components!

Chem. Rev. 2016, 116, 9438−9484

Typical materials and their features

Conjugated hydrocarbon

Conjugated amine

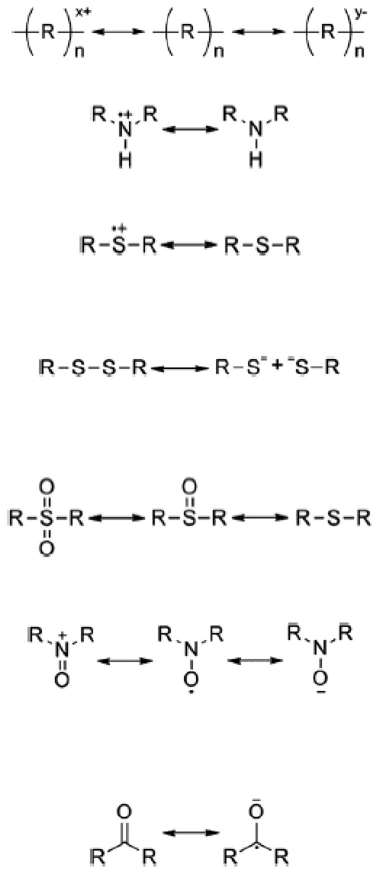
Conjugated thioether

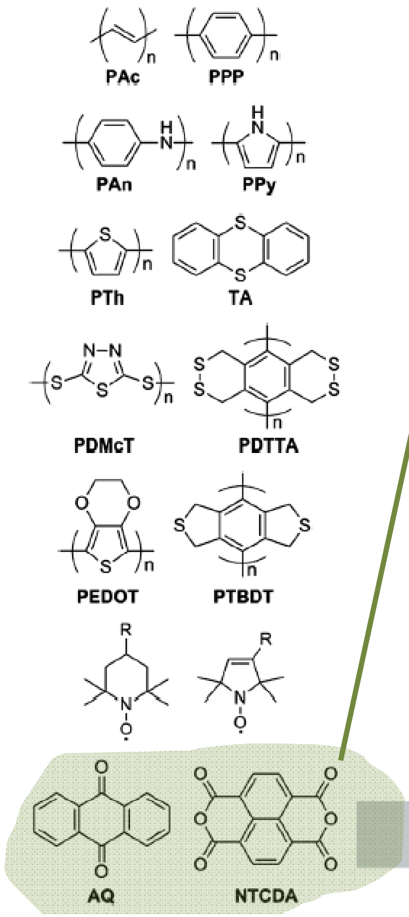
Organodisulfide

Thioether (4e)

Nitroxyl radical

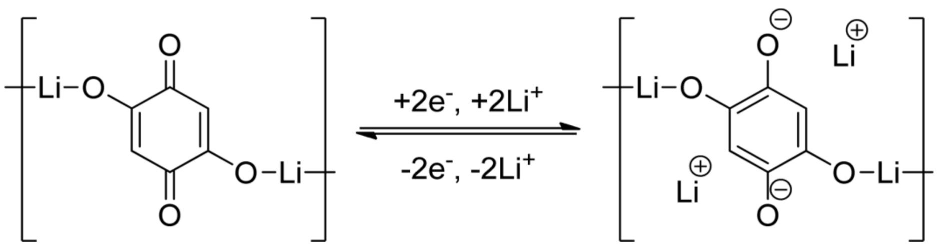
Conjugated carbonyl





Quinoid polymers: high capacity (in theory)

Quinoid polymers



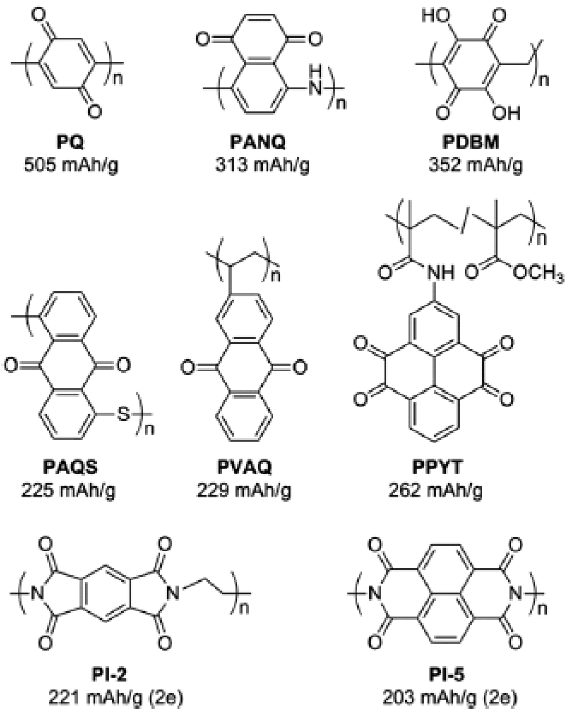
+ May be doped by metal ions (compatible with Me-ion technology)

+ May have high capacity due to multielectrone transitions

- Low redox potential (1.5 – 2 V vs. Li/Li^+)

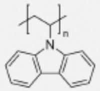
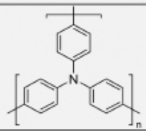
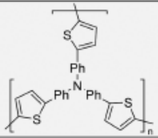
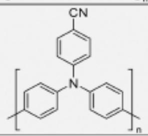
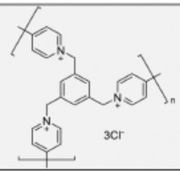
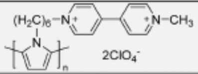
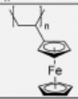
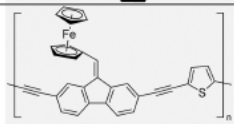
- Low electronic conductance

Tasks for quinoid materials



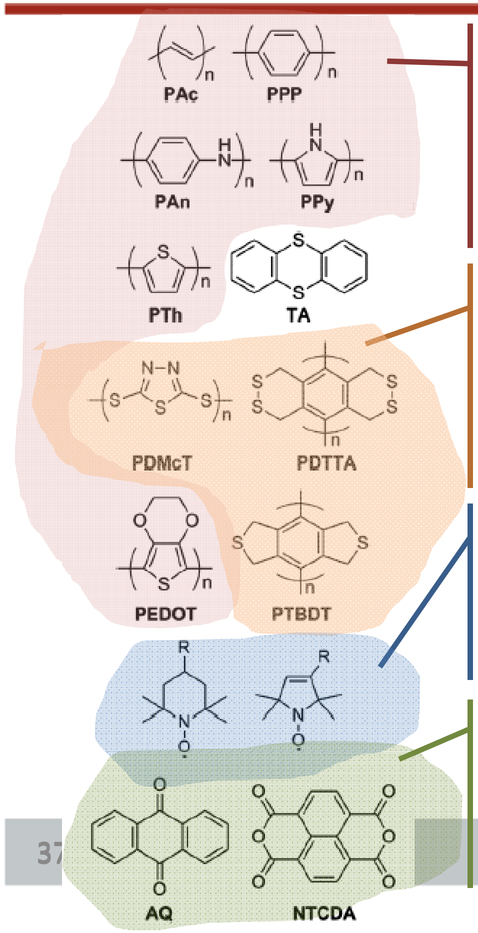
- Immobilization on the polymer backbone
- Redox potential tuning
- Conductivity enhancement by creating composite materials

Other electrode materials

	backbone	structure	polymerization method		discharge voltage [V]; [a] counter electrode	initial discharge capacity [Ah kg^{-1}]	current (density)	cycling stability (loss)	cycle no.
Carbazoles	poly(*N*-vinyl carbazole)		electropolym.	s	3.5-1.0; Li	115 (20th cycle)	10 A kg^{-1}	–	–
			purchased	s	4.6-3.4; Li	ca. 120 (125 highest) [b]	20 A kg^{-1}	6%	50
Triphenylamines	poly(triphenylamine)		oxid. polym. ($FeCl_3$)	s	4.2-3.6; Li	91	20C	8%	1000
	poly(tris(4-(2-thienyl) phenyl)amine)		oxid. polym. ($FeCl_3$)	s	4.2-3.5; Li	129	20 A kg^{-1}	9%	50
	poly(4-cyano triphenylamine)		oxid. polym. ($FeCl_3$)	s	4.0-3.7; Li	75 (80 highest) [b]	40 A kg^{-1}	0%	150
Viologens	poly (tripyridinio-mesitylene)		electropolym.	pp	1.5/1.1; TEMPO	165	60C	20%	2000
	poly(viologen pyrrol)		electropolym.	p	1.0; PPy[ABTS]	16	1 A m^{-2}	30%	100
Ferrocenes	poly(vinylferrocene)		FRP (AIBN)	p	3.2; Li	105	200 A kg^{-1}	5%	300
	poly (fluorenylethynylene ferrocene)		Sonogashira cross-coupling	p	3.4; Li	52	5C	10%	100

Chem. Rev. 2016, 116, 9438–9484

Specific features of different types of materials



π- conjugated systems, «conducting polymers»

+ High electric and ionic conductivity; High power output; May be used as conductive component in composite material

- Low capacity, limited by doping level; Overoxidation and stability problems; Processing problems

Organic sulfides – bond cleavage reactions

+ Two electrone process – high capacity; Metal ion doping

- Slow kinetics; Solubility in electrolyte; Low electronic conductivity

Nitroxyle radical grafted polymers: capacity and reversibility

+ High reversibility and stability; fast charge/discharge

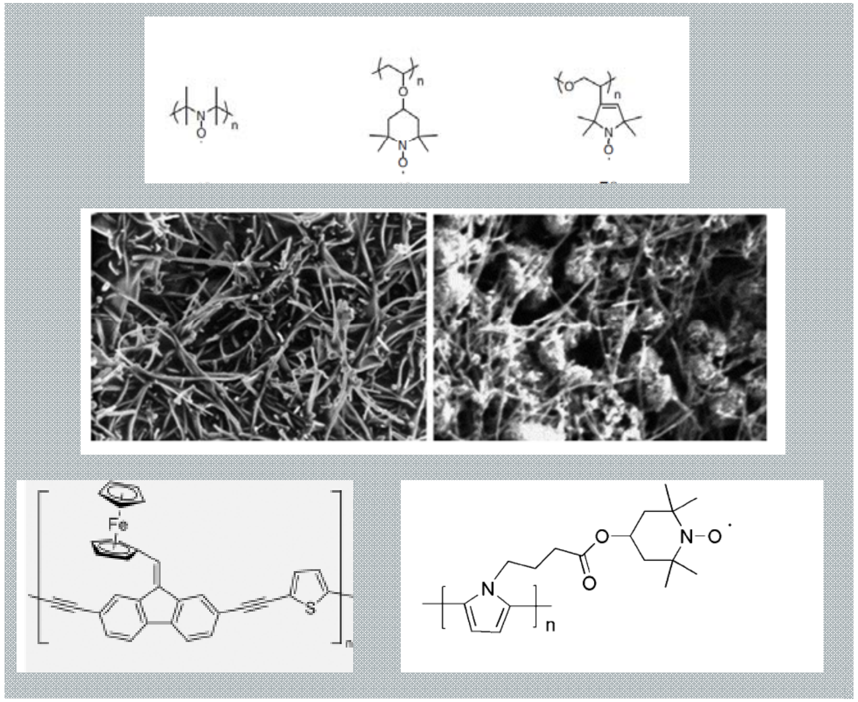
- High molecular weight – low capacity; Low electronic conductivity

Quinone and its derivatives : high capacity

+ May be doped by metal cations; Multielectrone processes (high capacity); fast charge/discharge

- Low redox potential; Low electronic conductivity

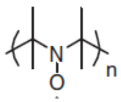
Research directions



- Capacity increase -reducing the molecular weight, increase in the number of electrons
- Conductivity improvement- creation of composite materials, use of conductive additives
- Increase the rate of charge/discharge process - kinetics control, ensuring ion transport
- Energy boost – tuning of redox potential by introduction of substituents
- Stability increase -control of polymerization and crosslinking to suppress the dissolution in electrolyte

Research directions

• Molar mass manipulation



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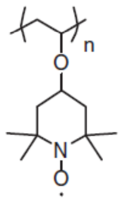

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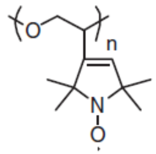

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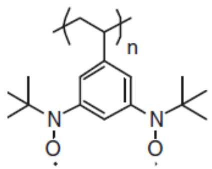

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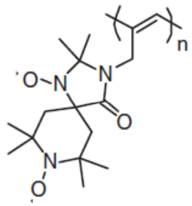

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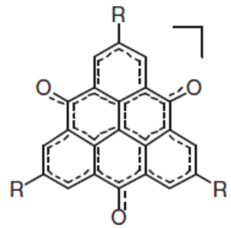
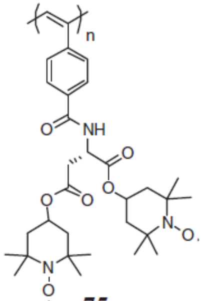


73 R = *t*Bu
74 R = Br

Adv. Energy Mater. 2012, 2, 742–769

Research directions

•Charge/discharge rate increse



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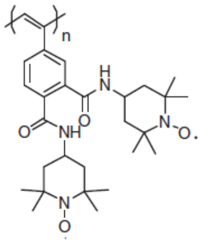

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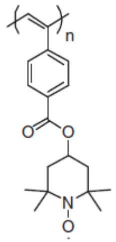

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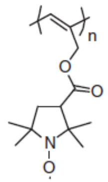

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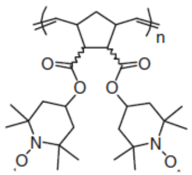
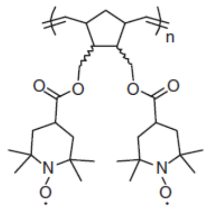
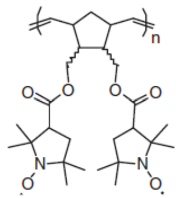


79 (2,3-*endo*, *exo*)
80 (2,3-*endo*, *endo*)

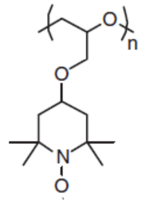


81 (2,3-*endo*, *endo*)
82 (2,3-*exo*, *exo*)



83 (2,3-*endo*, *endo*)
84 (2,3-*exo*, *exo*)

Electron exchange rate



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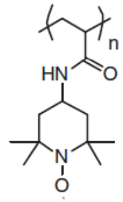

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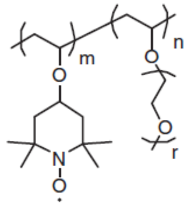
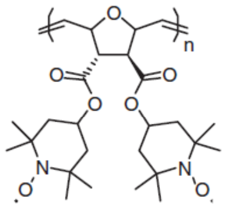


87 r = 1, 3



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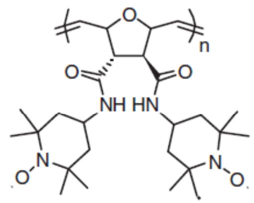


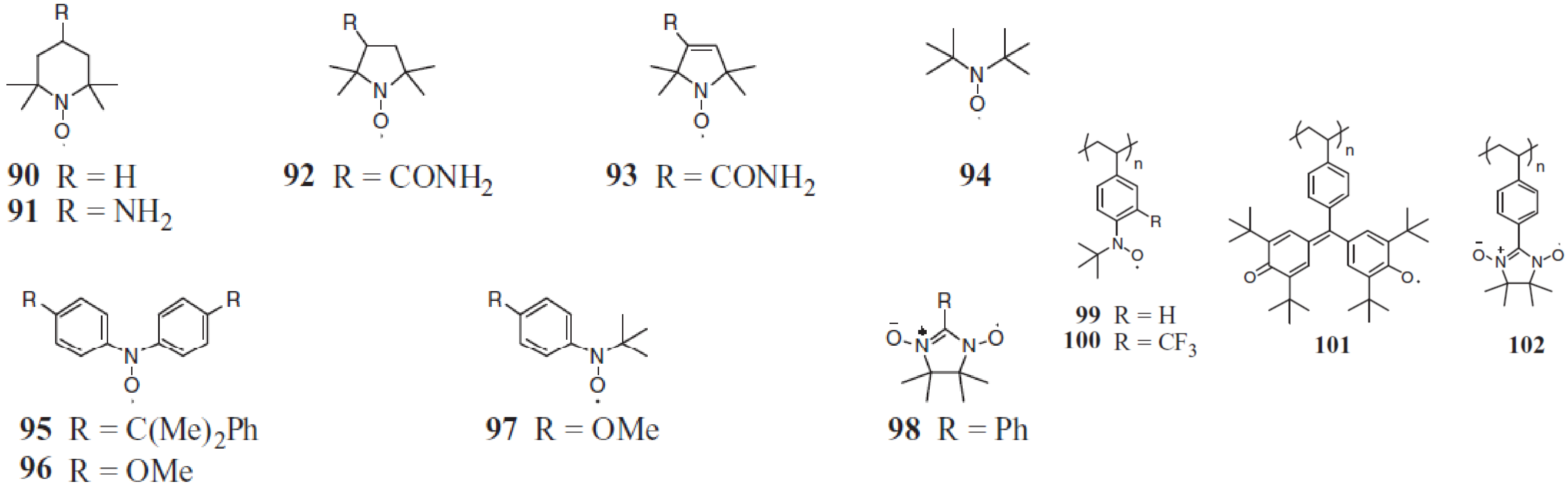
89

Ionic transport

Adv. Energy Mater. 2012, 2, 742–769

Research directions

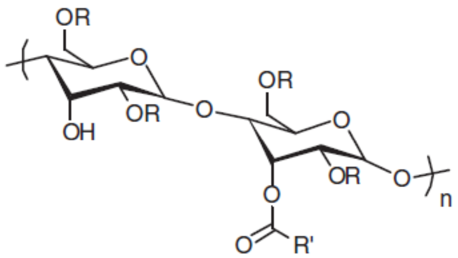
• Redox-potential tuning



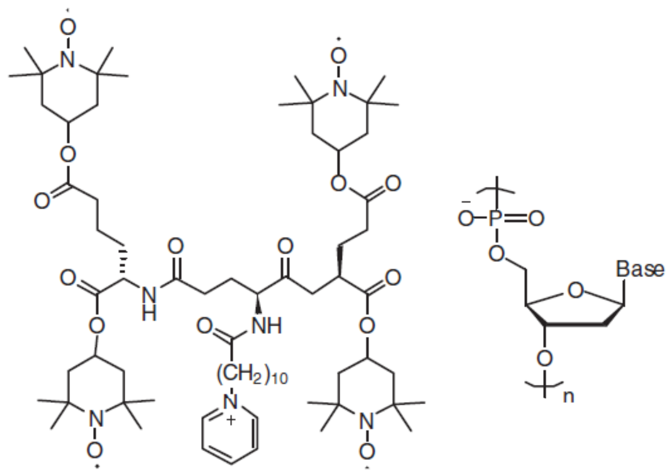
Adv. Energy Mater. 2012, 2, 742–769

Research directions

- Enhancing stability, decreasing solubility



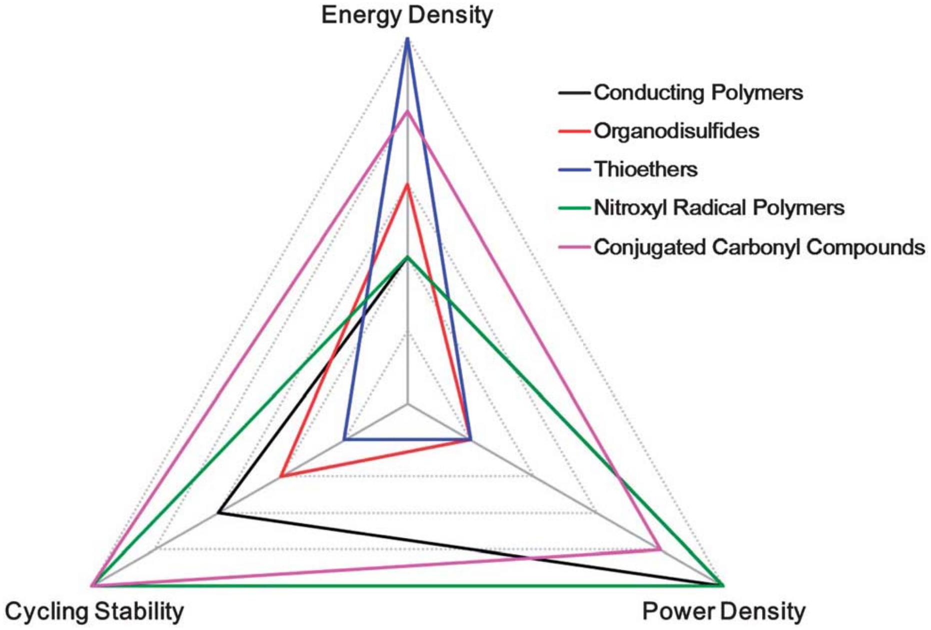
103 R = Et or Ac, R' = TEMPO or PROXY



104

Adv. Energy Mater. 2012, 2, 742–769

Demand for hybrid systems

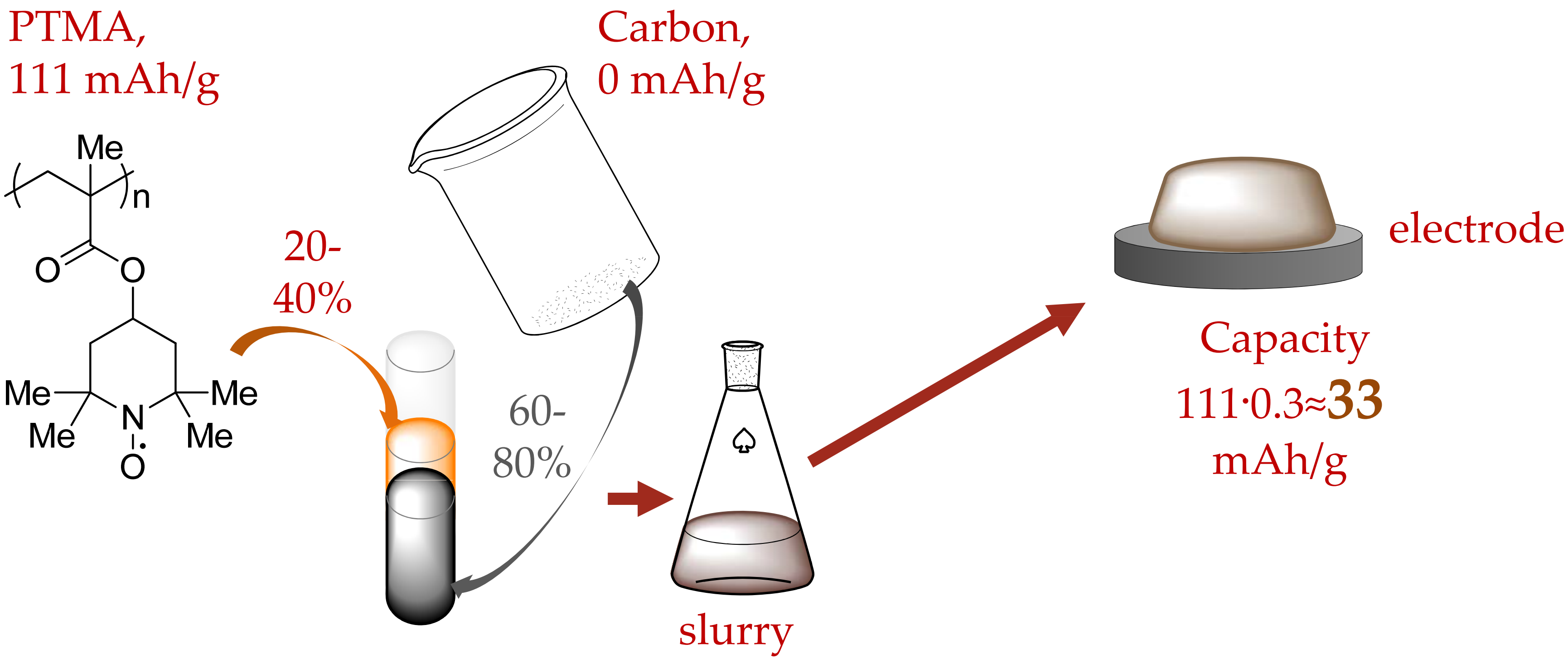


Comparison of the comprehensive electrochemical performance between different types of organic electrode materials.

- A lot of information on redox-active functional moieties is collected
- No known organic electrode material type can be considered as the best one, taking into account all practical parameters
- Organic materials may be combined

 Energy Environ. Sci., 2013, 6, 2280–2301

Conductivity problem

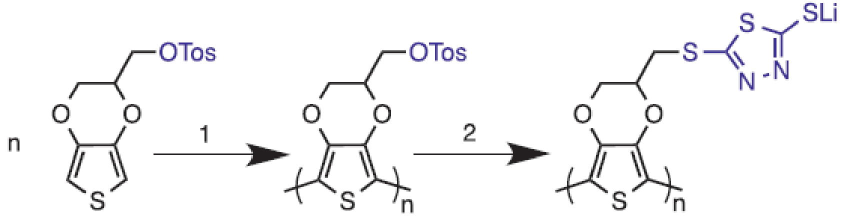


All together?

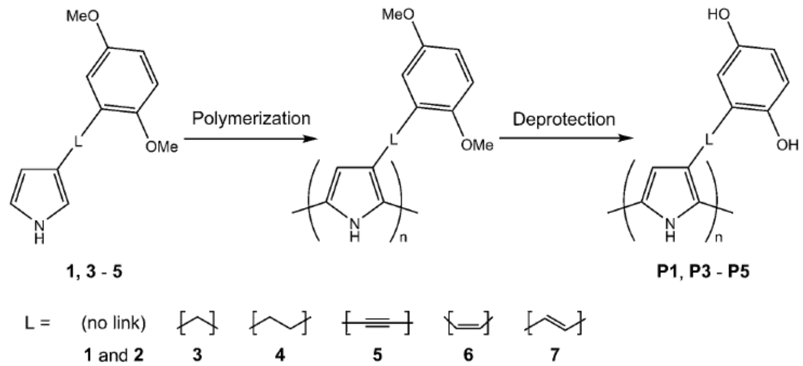
- Use of active building blocks

Resurrection of conducting polymers?

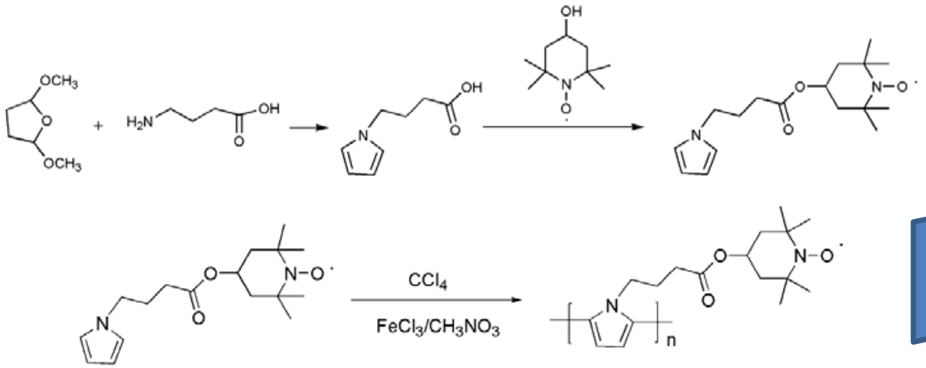
•Fictionalization by Red/Ox groups



Electrochimica Acta 167 (2015) 55–60

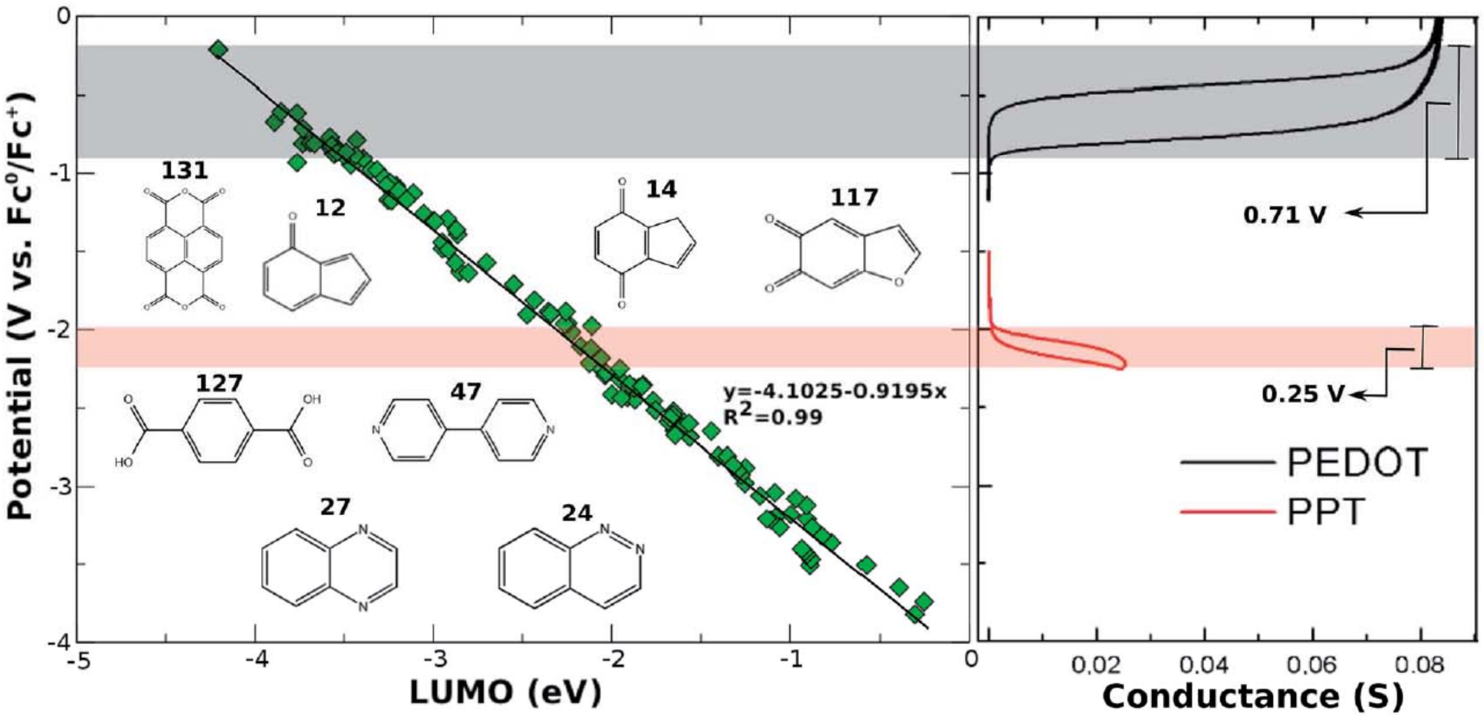


RSC Adv., 2015, 5, 11309–11316



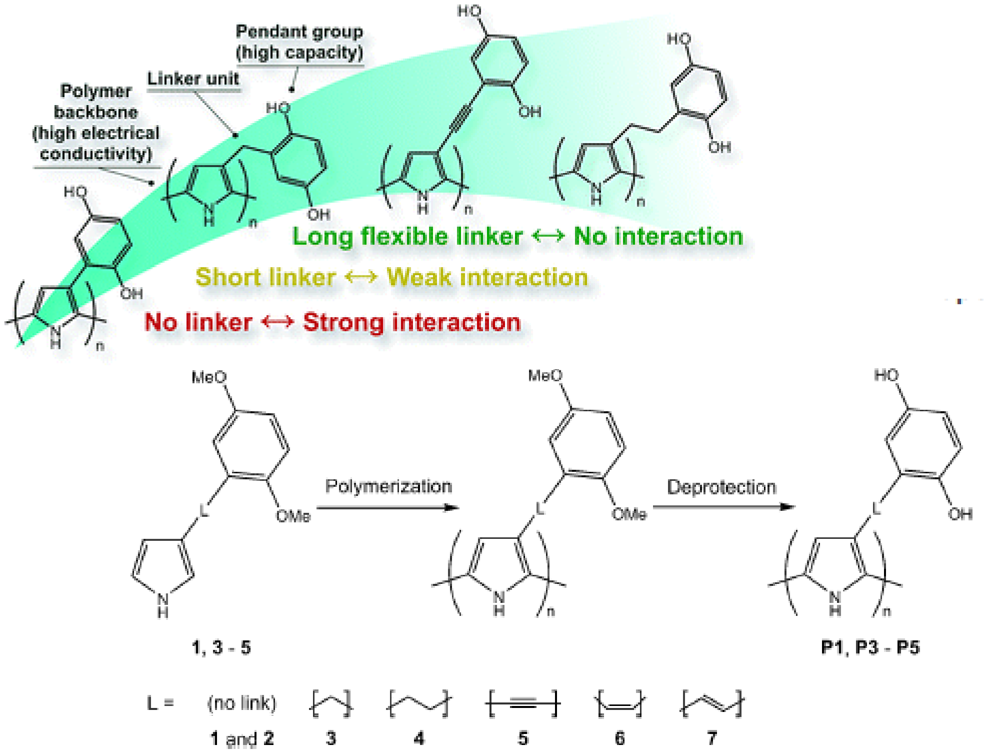
Electrochimica Acta 130 (2014) 148–155

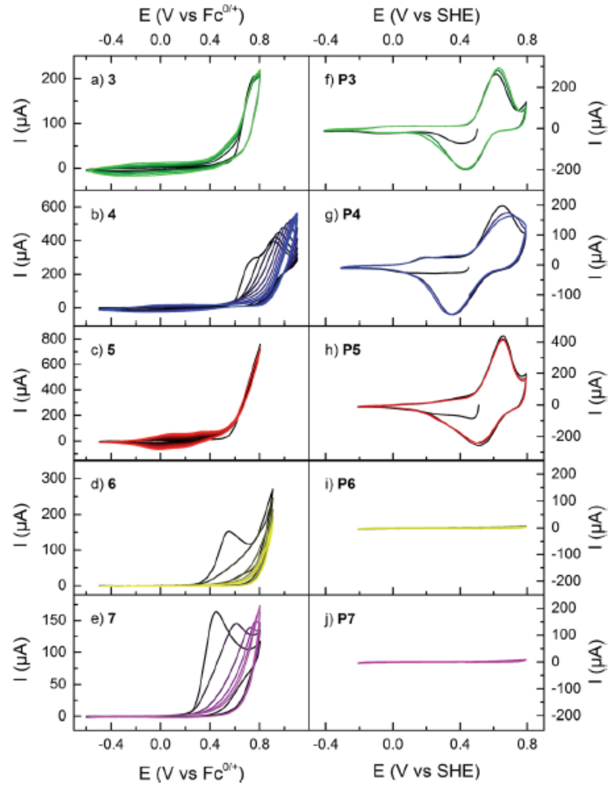
Task: redox-matching



R.B. Araujo, A. Banerjee, P. Panigrahi, L. Yang, M. Strømme, M. Sjödin, C.M. Araujo, R. Ahuja, *J. Mater. Chem. A*, 5 (**2017**) 4430-4454

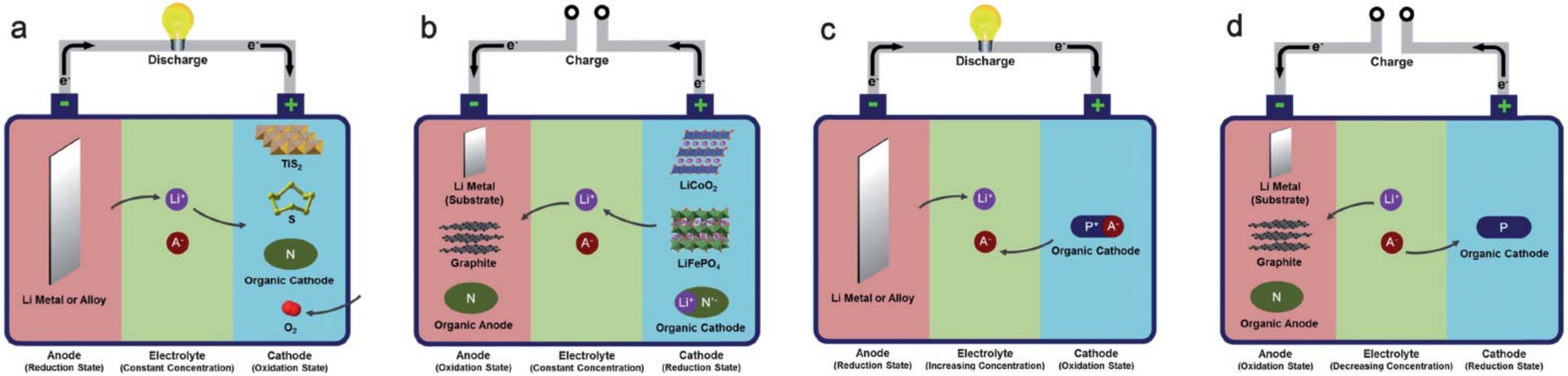
Task: backbone – side group interaction

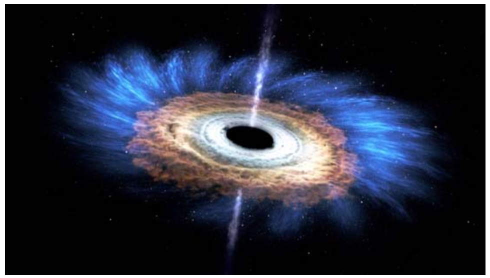




C. Karlsson, H. Huang, M. Strømme, A. Gogoll, M. Sjödin, *RSC Adv.*, 5 (**2015**) 11309-11316

Task – pairing of doping types



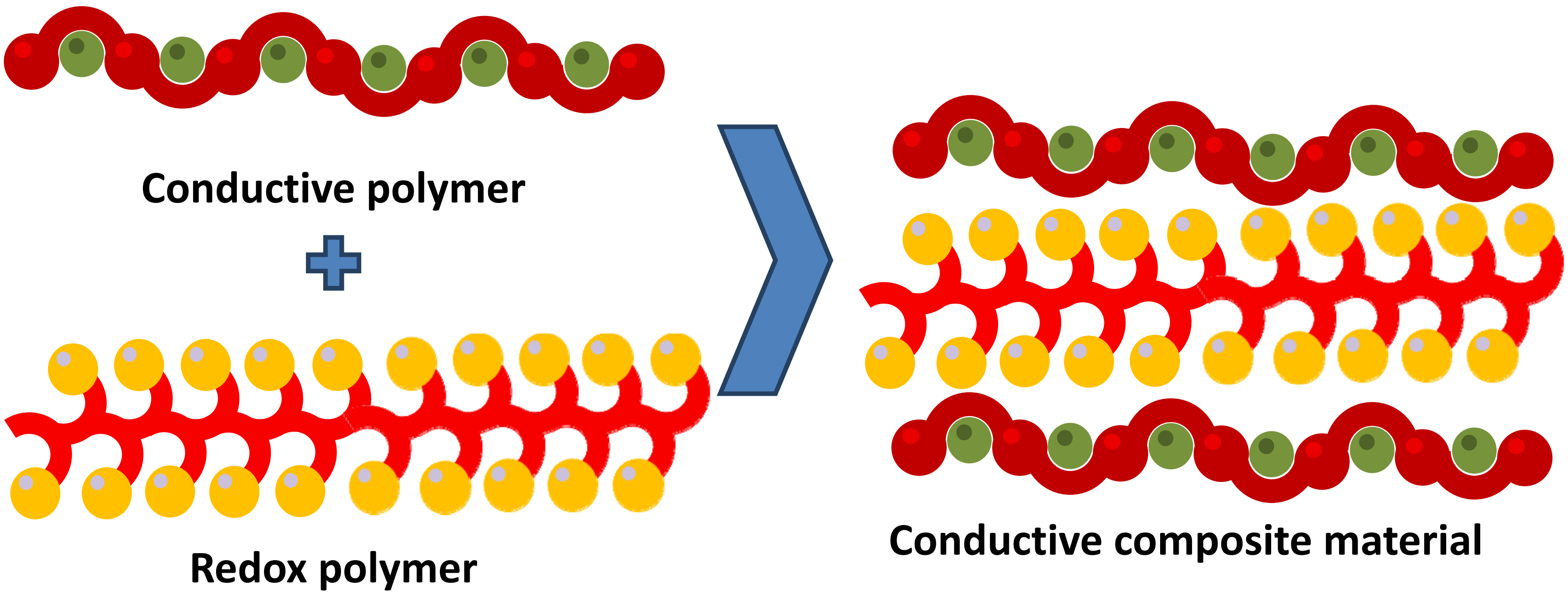


C. Karlsson, H. Huang, M. Strømme, A. Gogoll, M. Sjödin, *RSC Adv.*, 5 (**2015**) 11309-11316

St Petersburg University

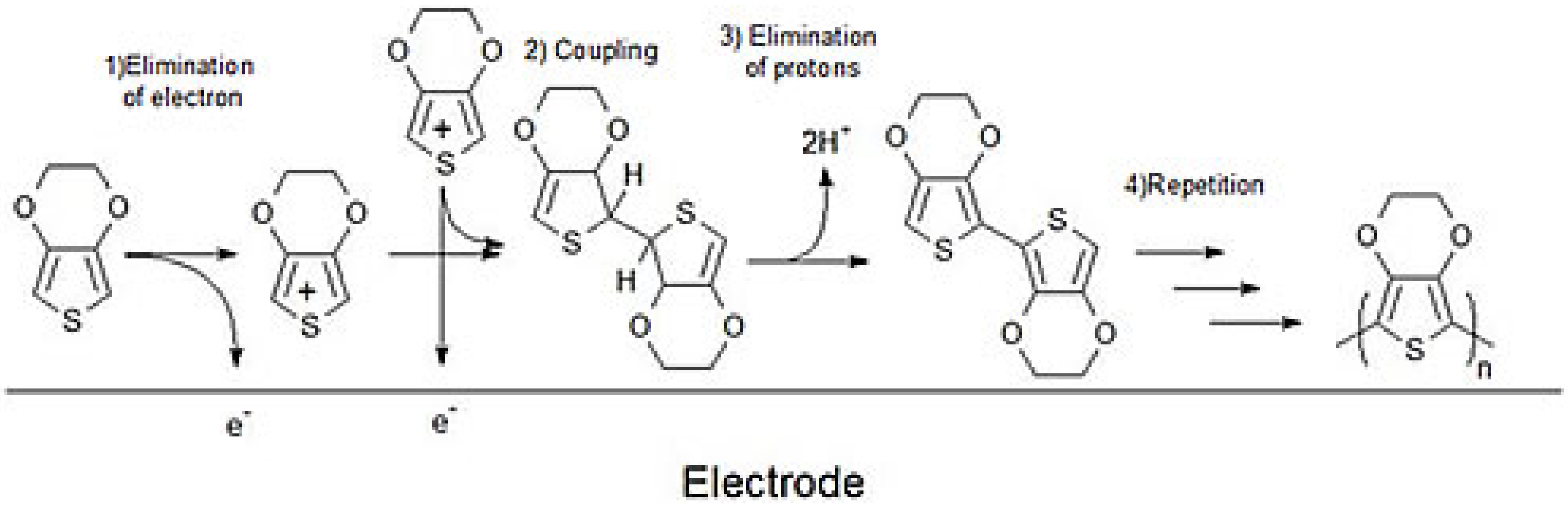
DESIGN OF ORGANIC MATERIALS FOR ELECTROCHEMICAL ENERGY STORAGE

Approach 1: conductive composite

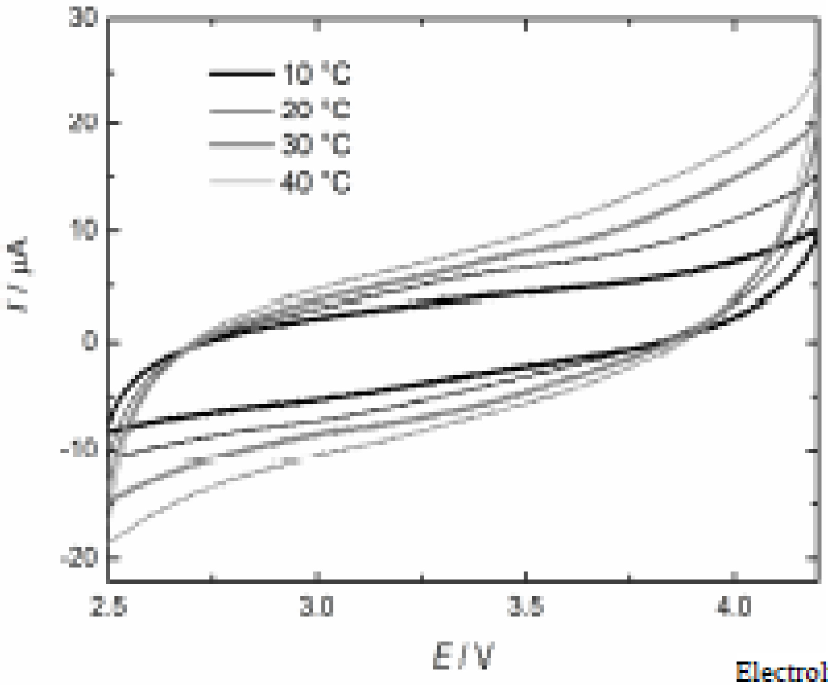


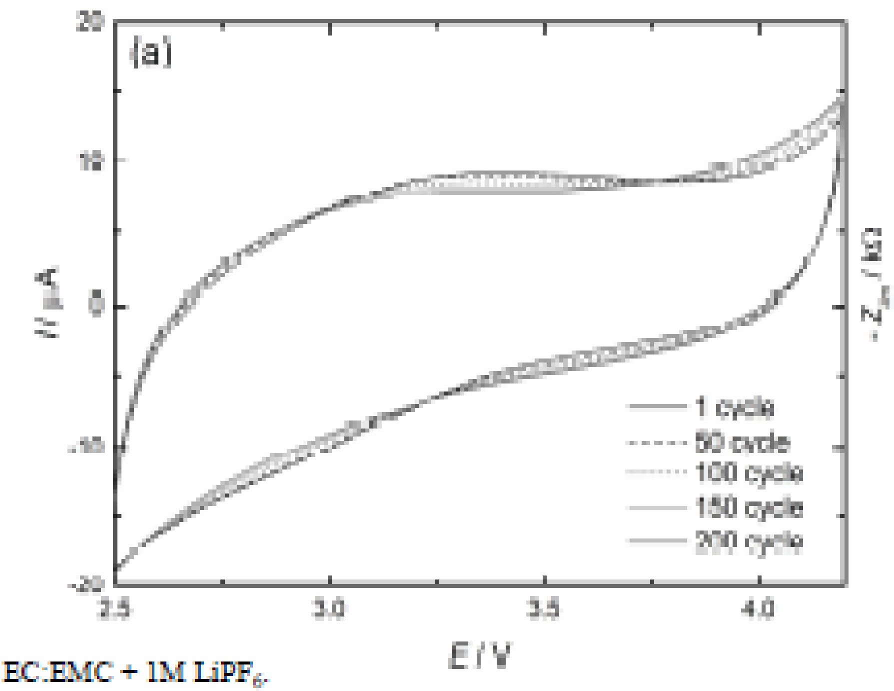
Conductive backbone

PEDOT



Electrochemistry of PEDOT



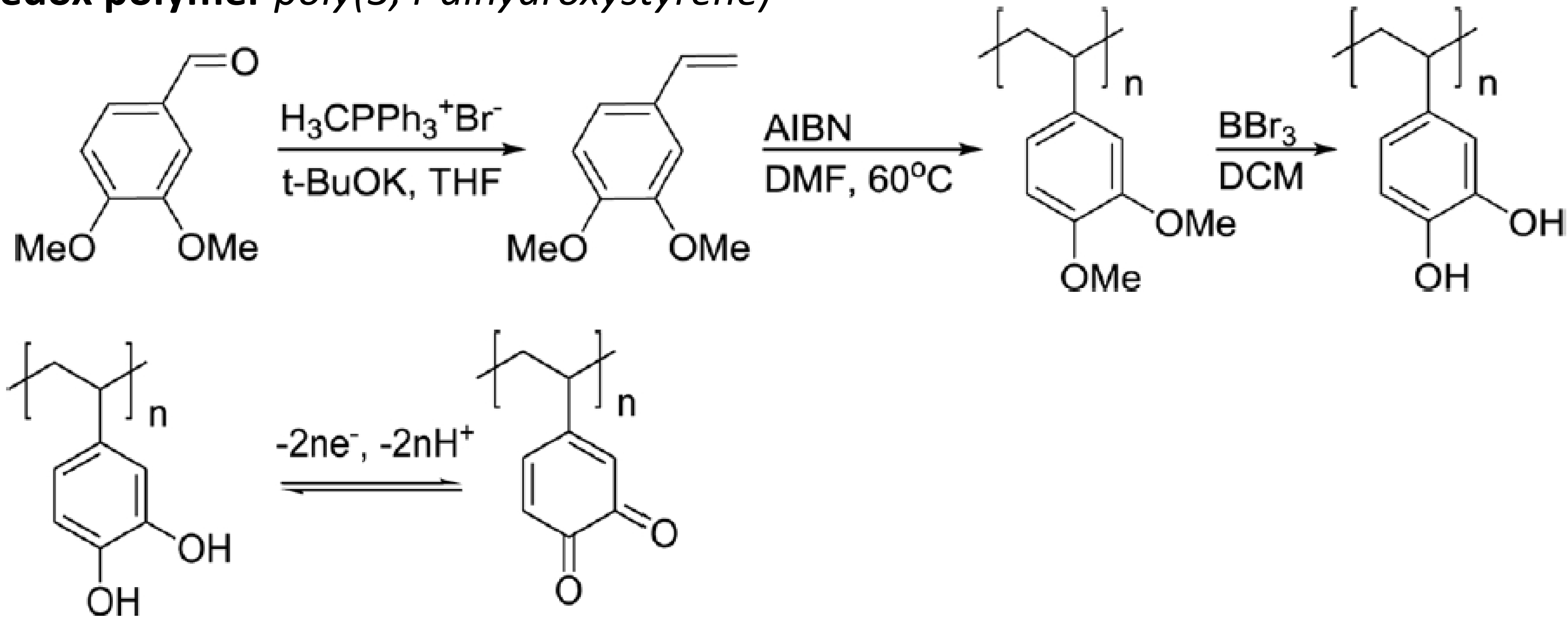


Electrolyte 1:1 EC:EMC + 1M $LiPF_6$.

Pratik. R. Das, Lidiya Komsiyska, Oliver Osters and Gunther Wittstock / ECS Transactions, 68 (2) 45-58 (2015)

Modification of PEDOT

Redox polymer *poly(3,4-dihydroxystyrene)*



D.A. Lukyanov, R.V. Apraksin, A.N. Yankin, P.S. Vlasov, O.V. Levin, E.G. Tolstopjatova, V.V. Kondratiev / Synthetic Metals 256 (2019) 116151

Modification of PEDOT

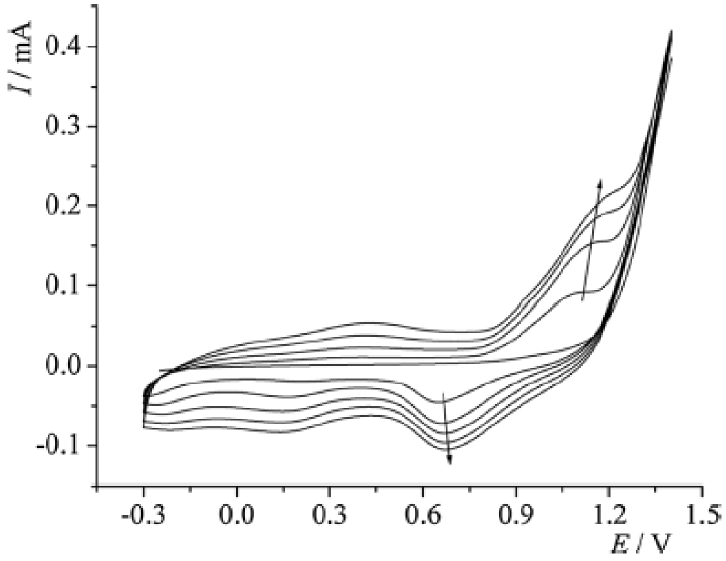
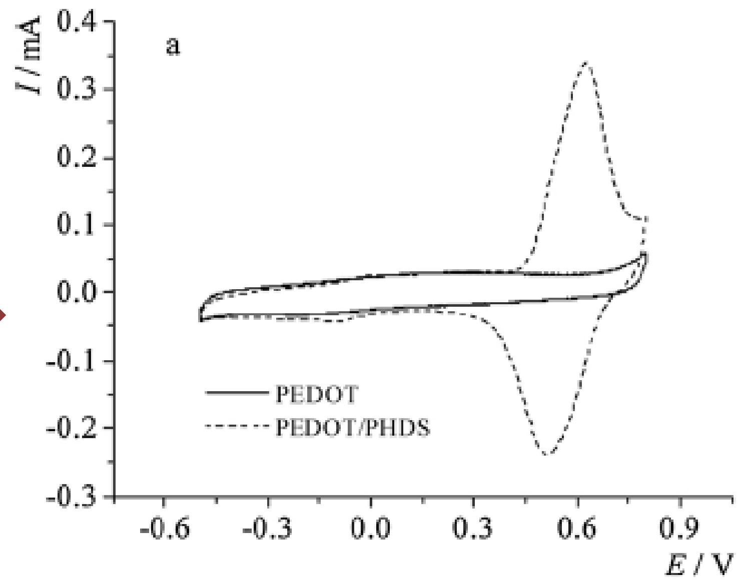
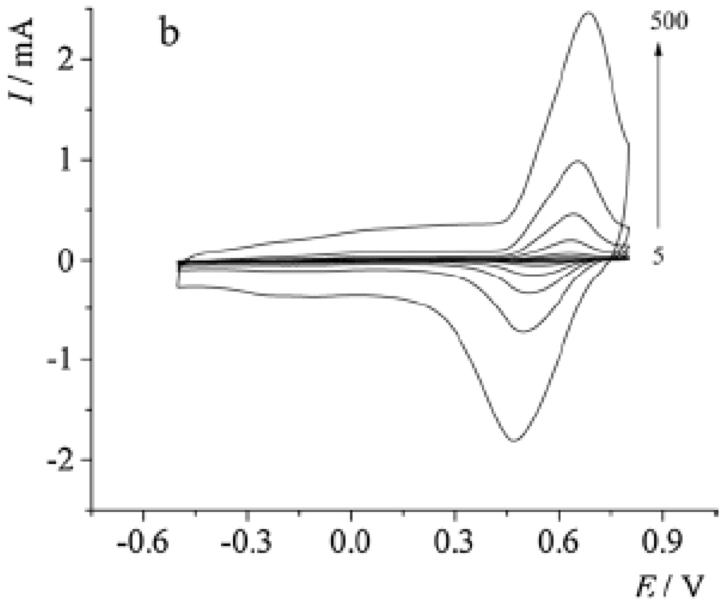


Fig. 3. Electropolymerization of PEDOT/PDHS on a GC electrode from the solution of 0.05 M EDOT + 0.5 M $LiClO_4$ + 0.3 M PDHS in PC (ν = 50 mV s^{-1}).



D.A. Lukyanov, R.V. Apraksin, A.N. Yankin, P.S. Vlasov, O.V. Levin, E.G. Tolstopjatova, V.V. Kondratiev / Synthetic Metals 256 (2019) 116151

Modification of PEDOT



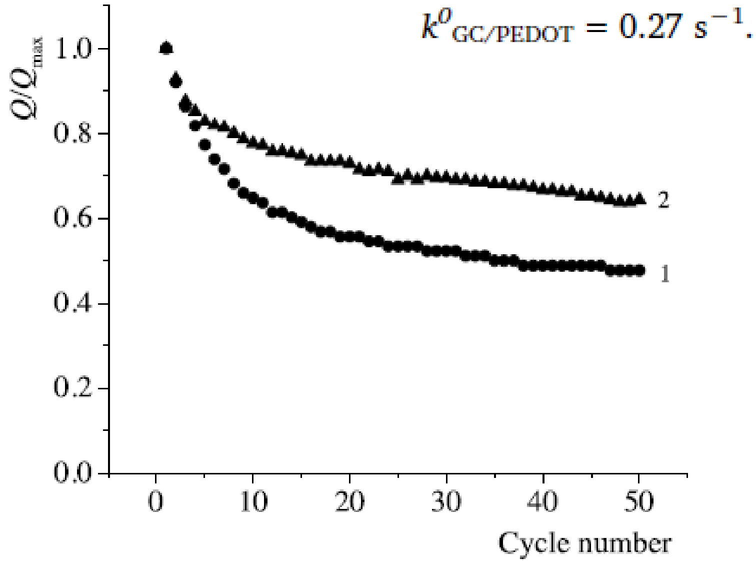
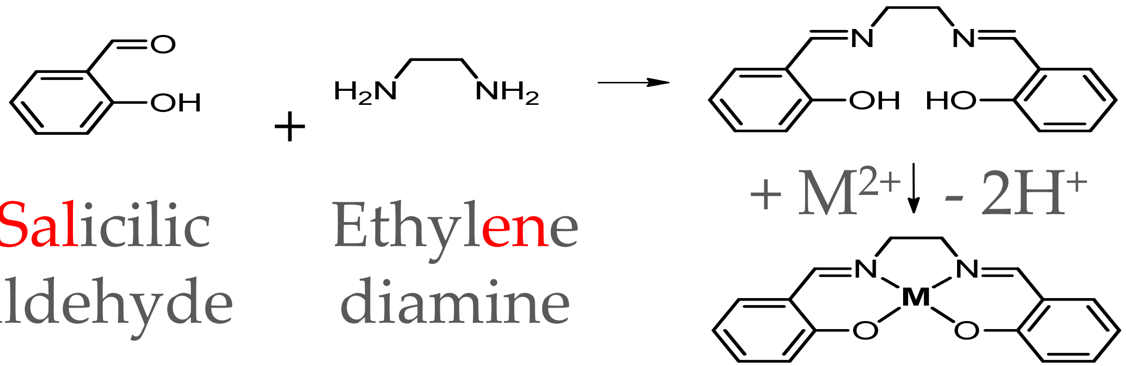


Fig. 5. Normalized capacities vs. number of cycle for GC/PDHS(1) and GC/PEDOT/PDHS(2) electrodes in 0.5 M H_2SO_4.

D.A. Lukyanov, R.V. Apraksin, A.N. Yankin, P.S. Vlasov, O.V. Levin, E.G. Tolstopjatova, V.V. Kondratiev / Synthetic Metals 256 (2019) 116151

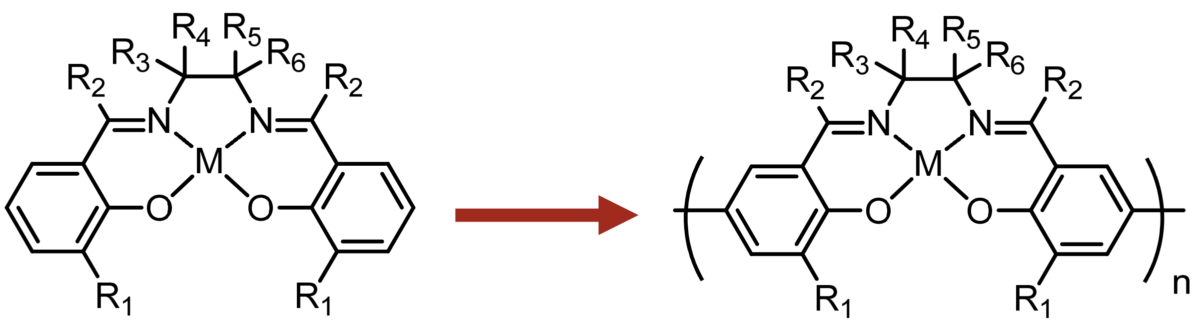
Salen-type complexes



Widely applied in catalysis:

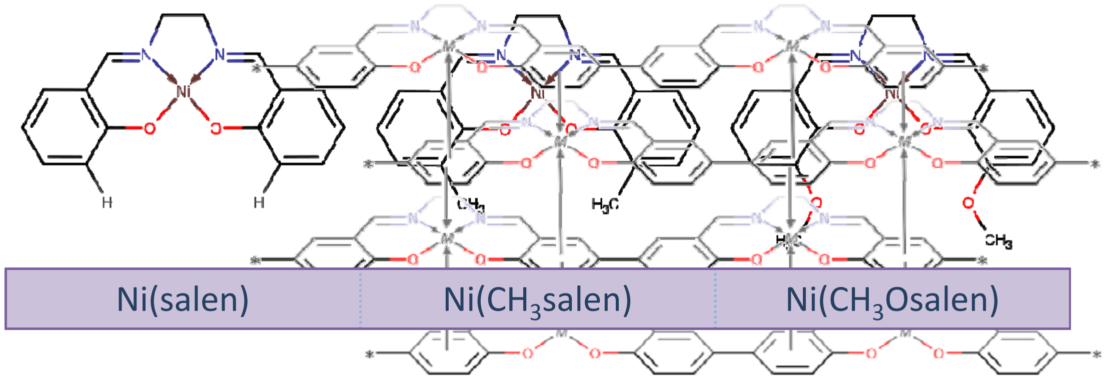
Oxidation,
Epoxydation,
Hydrolysis etc…

Electrochemical Oxidation

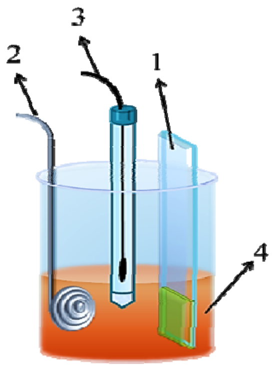


Polymerisation of **Salen-type complexes**

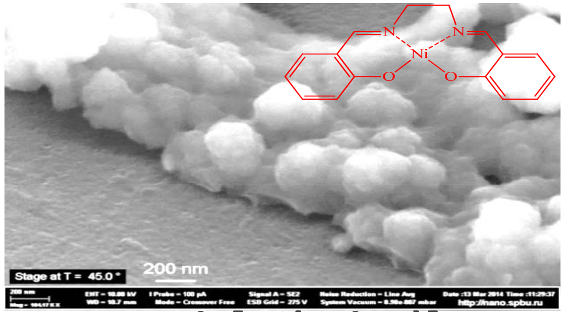
salen = (N,N’-ethylenebis(salicylimine))



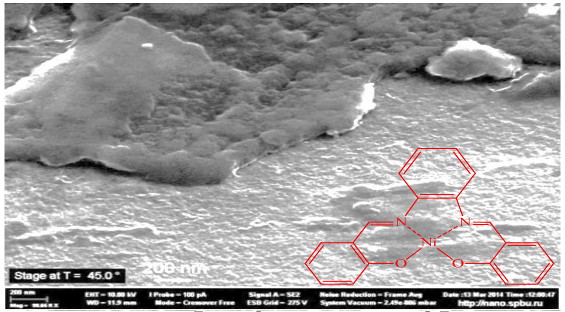
poly[Ni(**R**salen)]



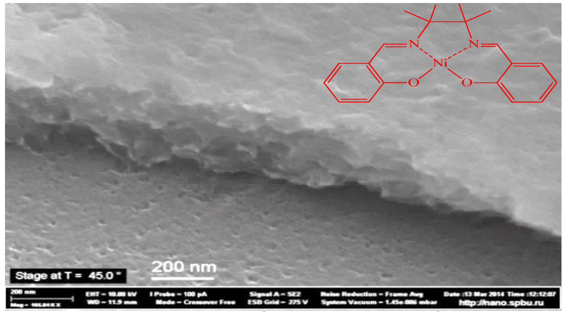
Morphology of poly[Ni(Schiff)] polymeric complexes



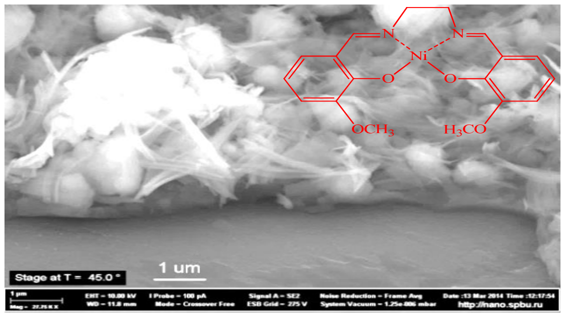
poly[Ni(Salen)]
ρ=2,4 ± 0,2 g/cm^3



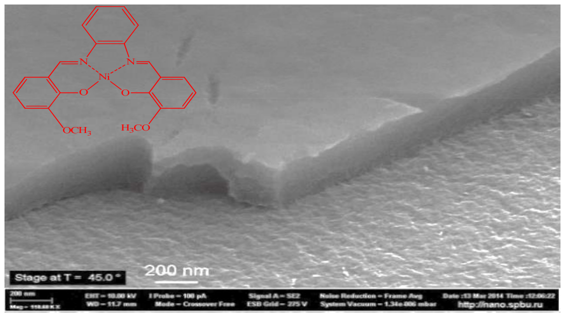
poly[Ni(Salphen)]
ρ=2,4 ± 0,1 g/cm^3



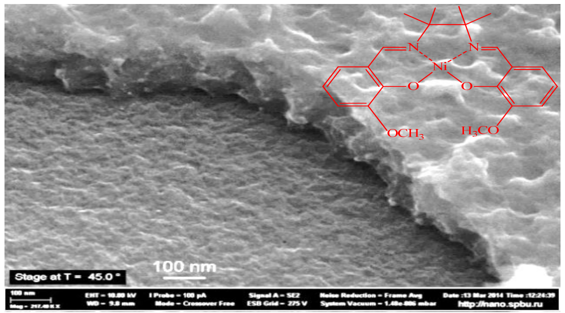
poly[Ni(Saltmen)]
ρ=1,9 ± 0,1 g/cm^3



poly[Ni(CH_3OSalen)]
ρ=1,9 ± 0,1 g/cm^3

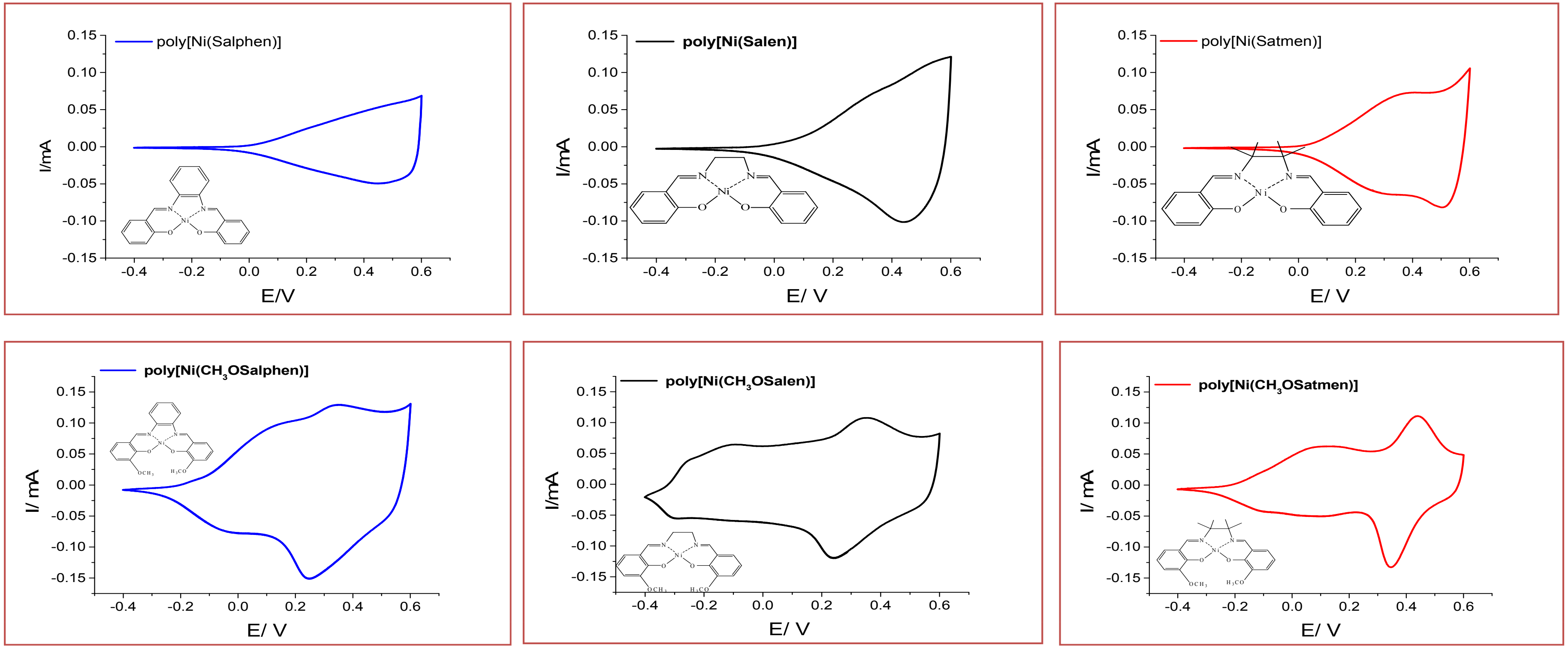


poly[Ni(CH_3OSalphen)]
ρ=1,8 ± 0,1 g/cm^3



poly[Ni(CH_3OSaltmen)]
ρ=1,9 ± 0,1 g/cm^3

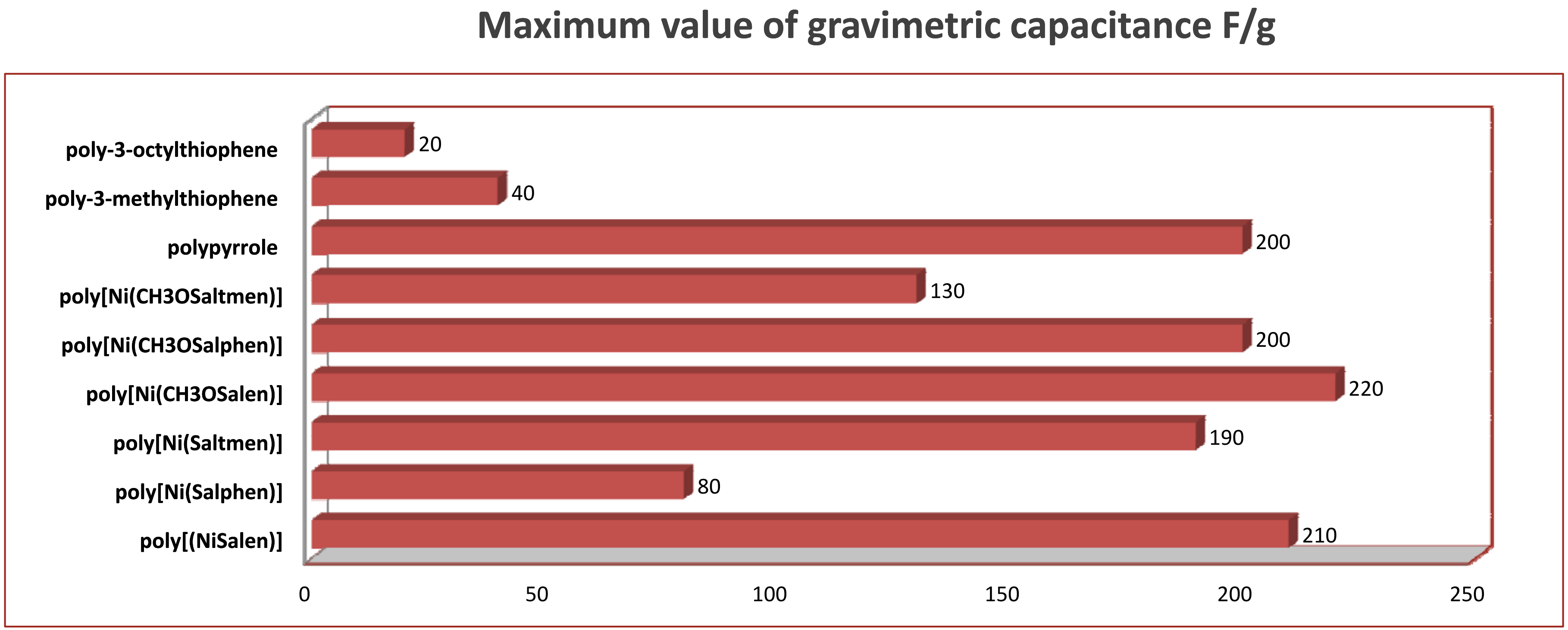
Cyclic voltammograms of poly[Ni(Schiff)]



E.V. Alekseeva et al. / Electrochimica Acta 225 (2017) 378–391

17:22

Charge transfer parameters of poly[Ni(Schiff)] complexes



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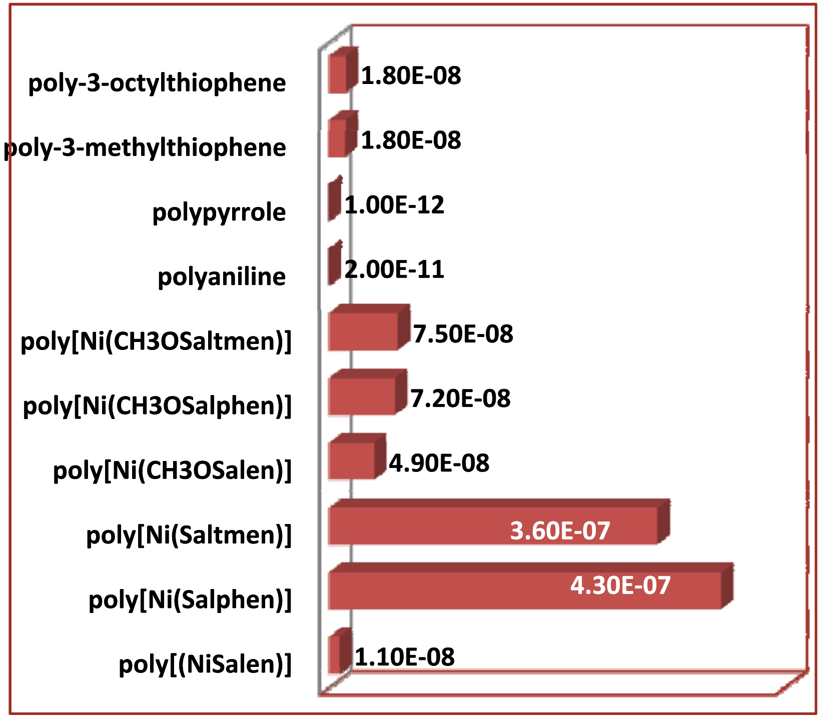
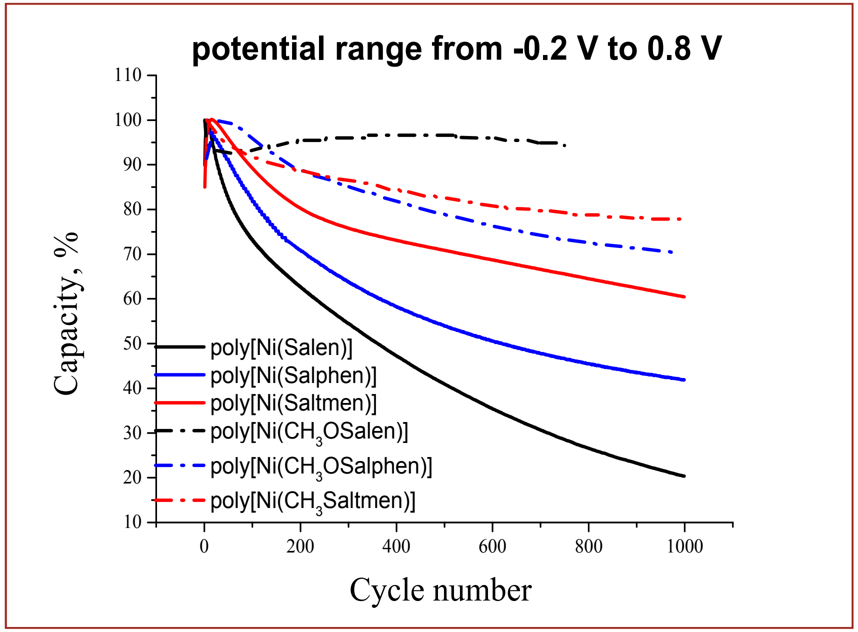
Charge transfer parameters of poly[Ni(Salen)] complexes

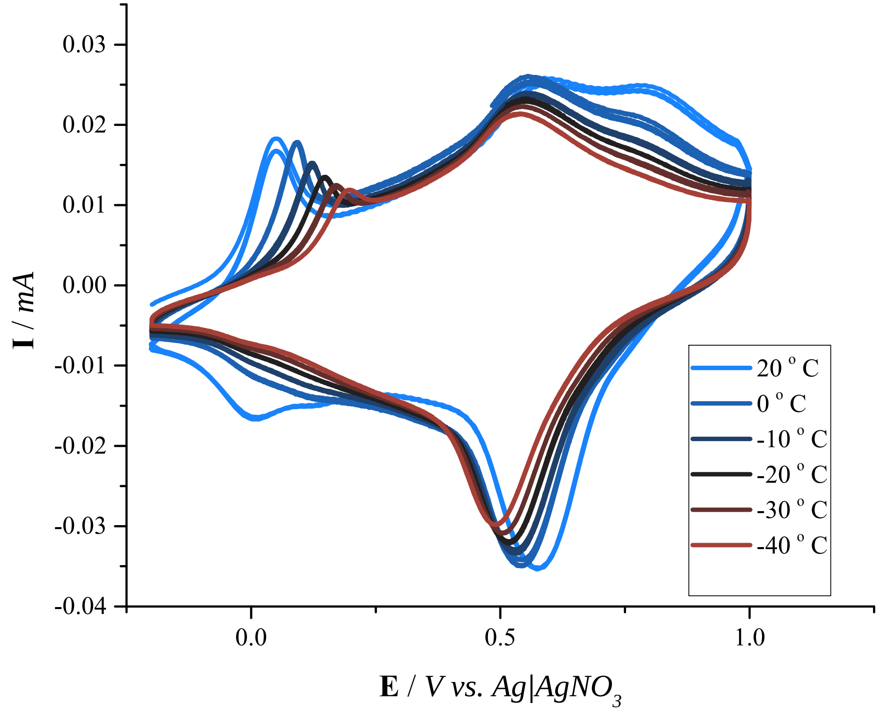
Maximum value of Def, cm²/s

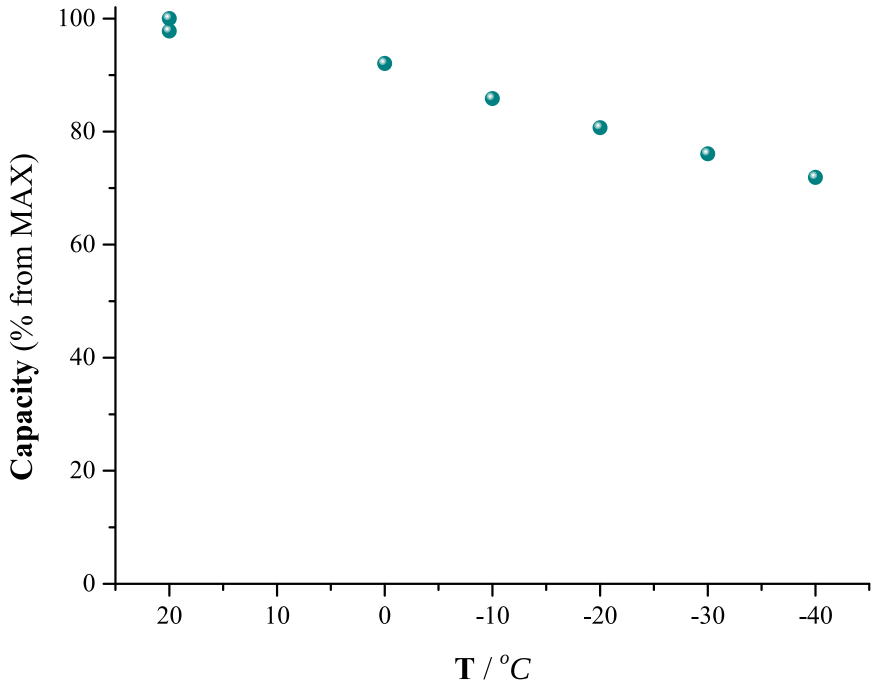


Relative capacity drop in dry electrolyte

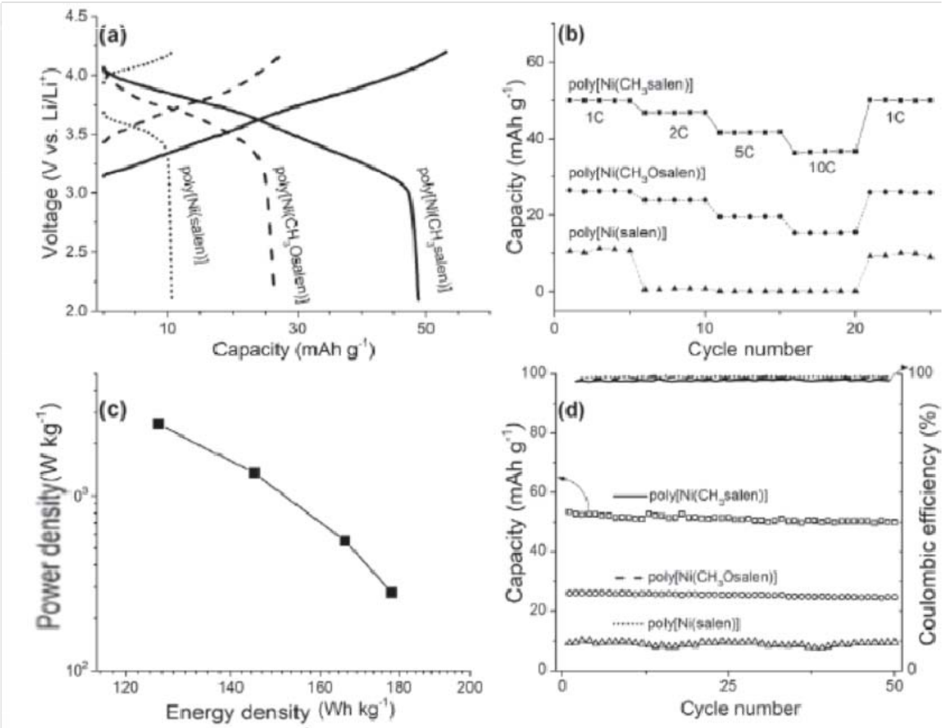


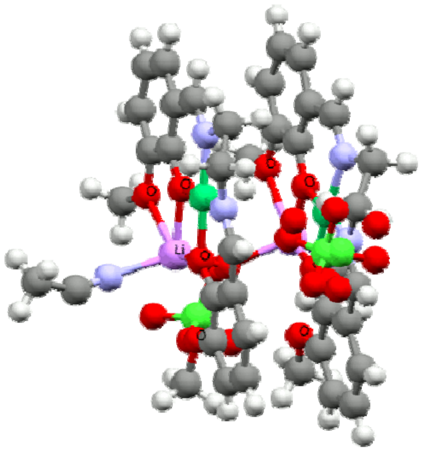
poly[Ni(Schiff)] complexes are suitable for extra low operating temperatures

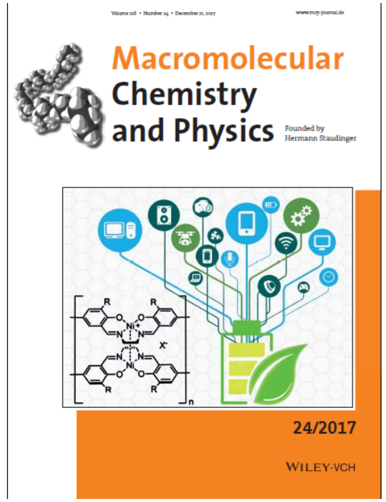


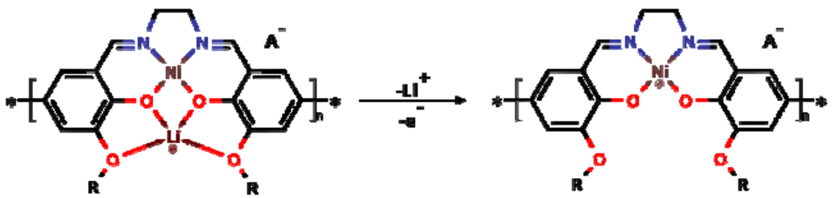


poly[Ni(Schiff)] complexes in Li-ion batteries









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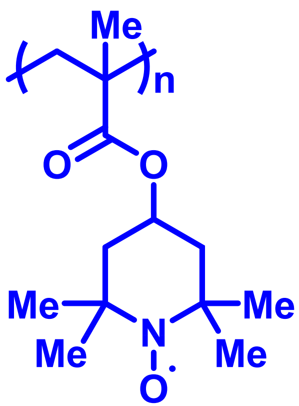
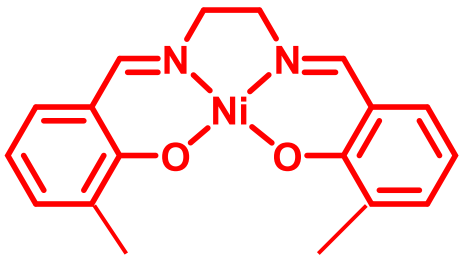
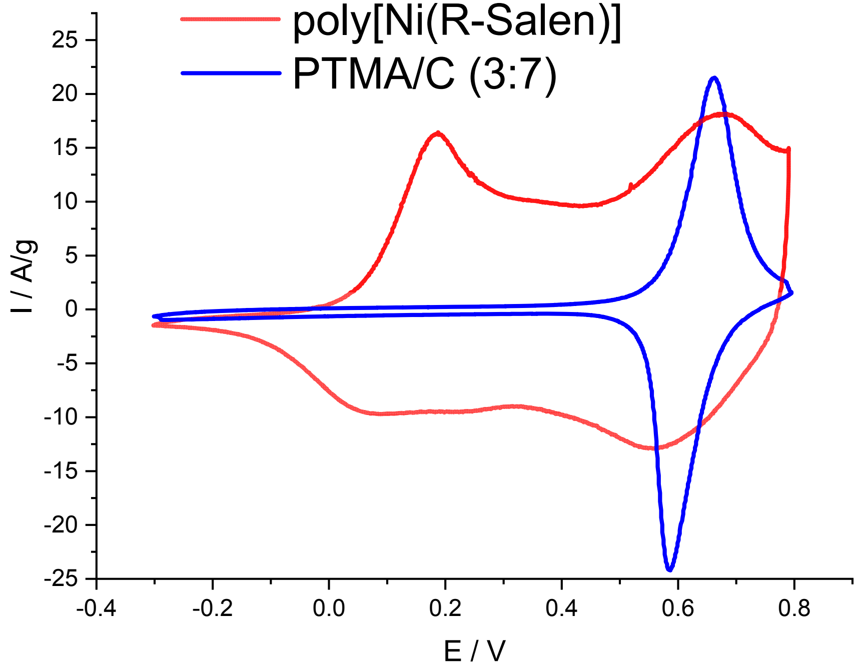
Modification of polymeric Salen-type complexes







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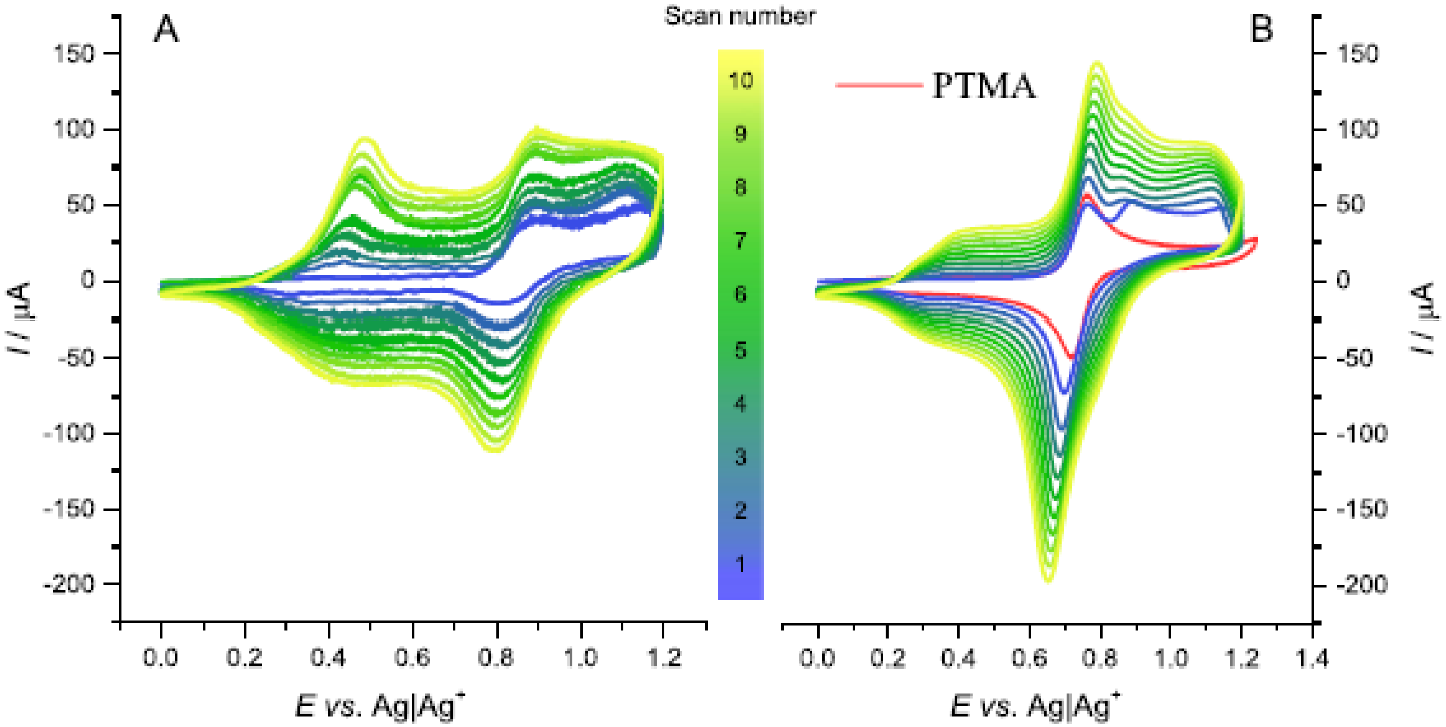
Modification of polymeric Salen-type complexes



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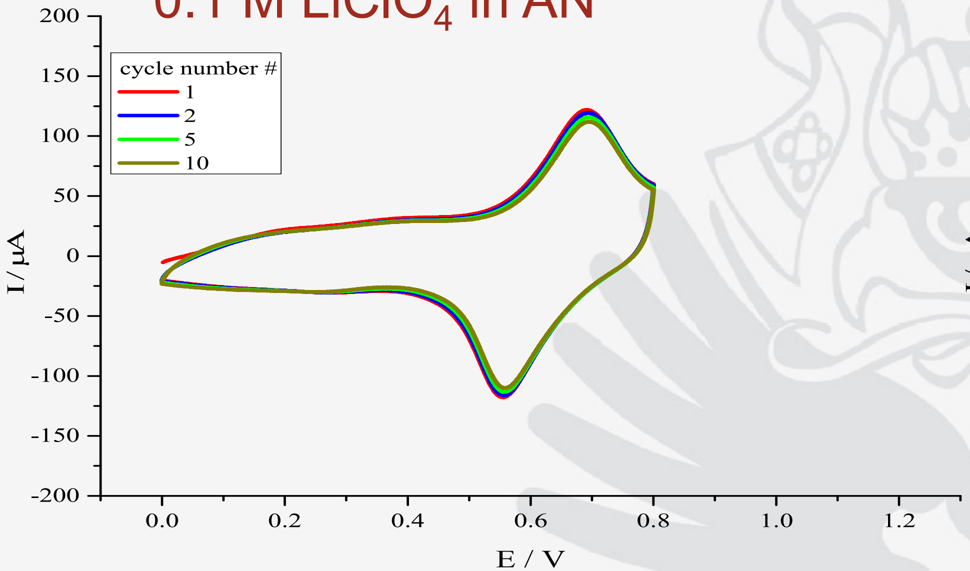
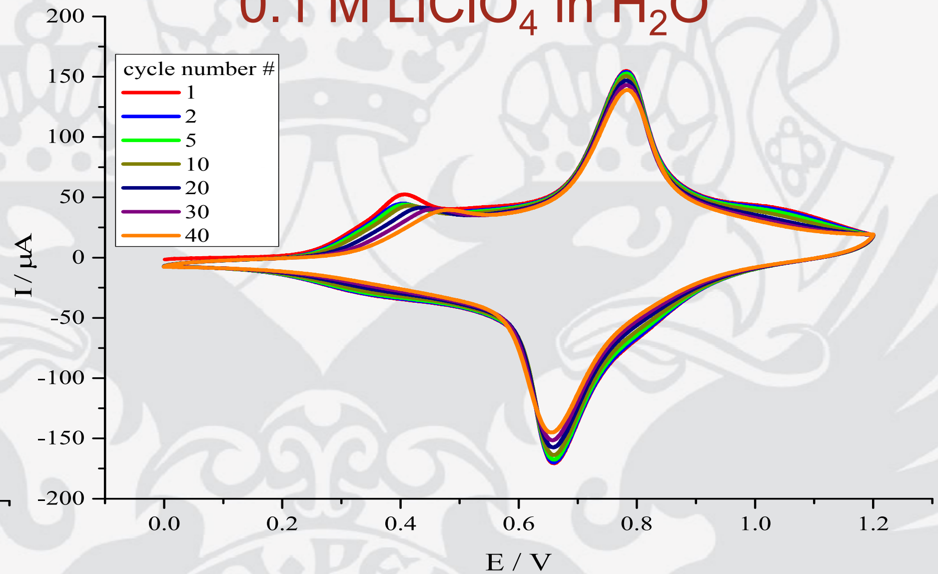
Test of copolymer

0.1 M $LiClO_4$ in AN



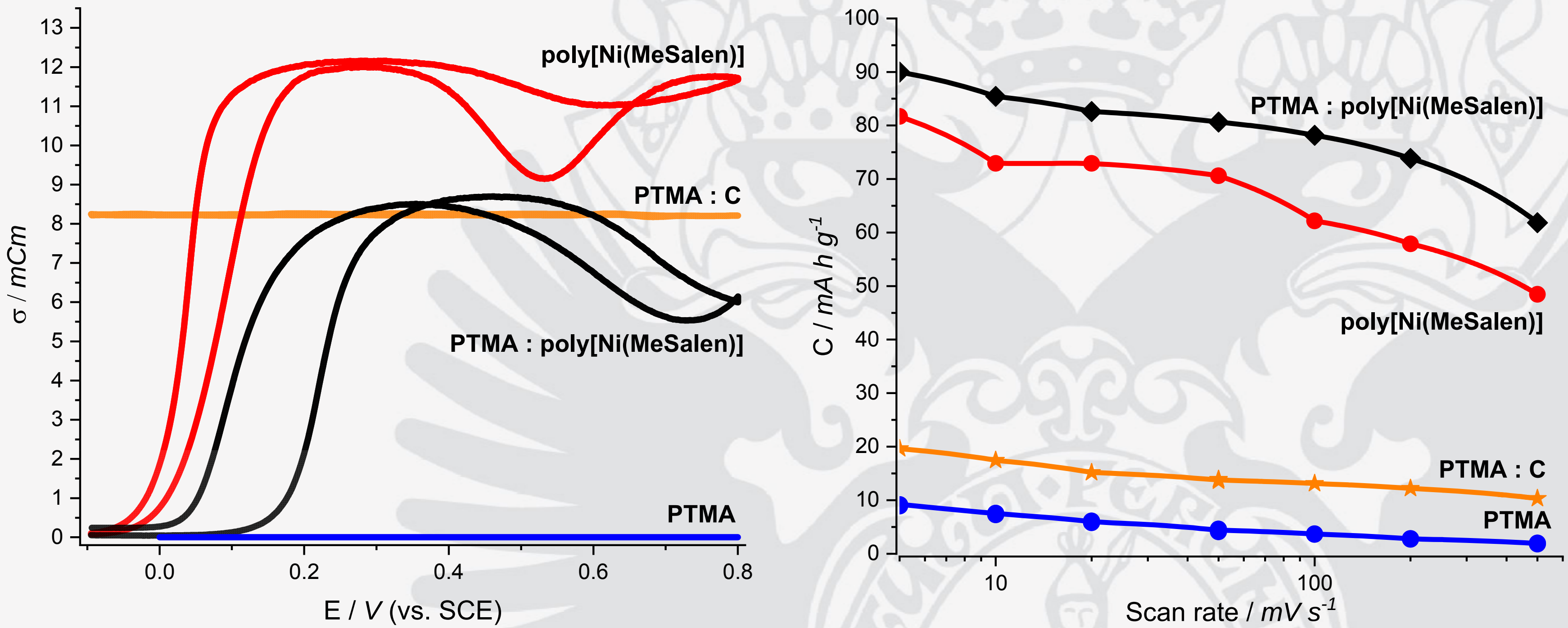
0.1 M $LiClO_4$ in H_2O



Composite is stable in aqueous and nonaqueous solutions

However, PTMA is soluble in AN → **PTMA is firmly bonded to the composite material**

Efficiency of the approach



Practical loading of copolymer

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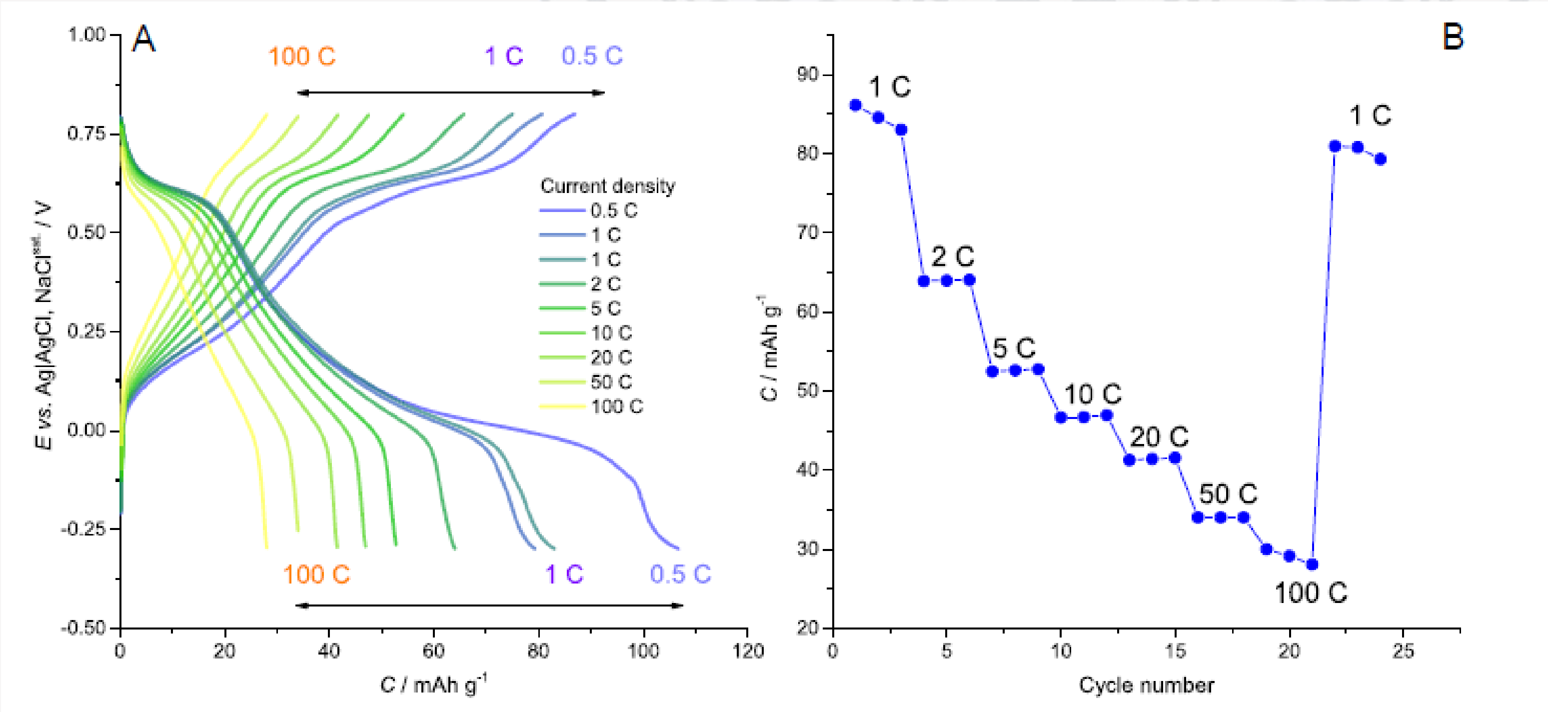
0.1 M $LiClO_4$ in H_2O, 1 mg cm^{-2}



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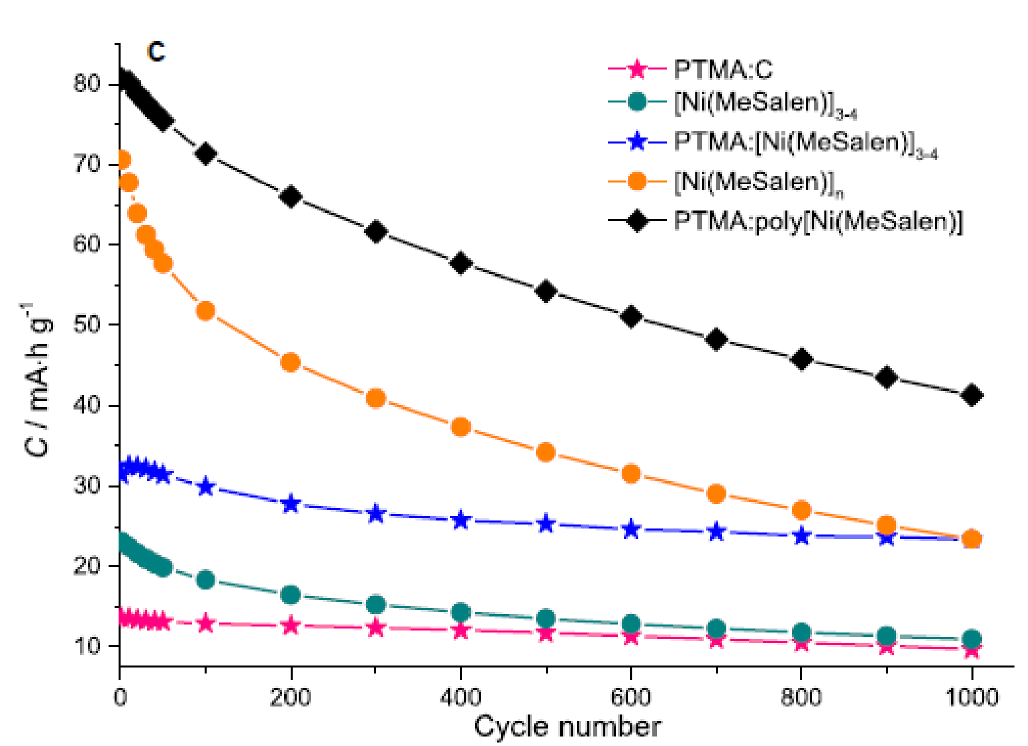
Stability of copolymer



0.1 M $LiClO_4$ in H_2O

[Ni(MeSalen)] based PTMA composites showed higher durability than [Ni(MeSalen)] materials without PTMA additive

RESEARCH GROUP OF MATERIALS FOR ELECTROCHEMICAL ENERGY BASED ON METAL-ORGANIC POLYMERS

•Supported bu Russian Science Foundation (grant #16-13-0038)

•With help of St. Petersburg State University Research Park

Thank you!