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Energimyndighetens titel på projektet – engelska Organic Batteries for sustainable and Increased Energy Efficiency in local energy storage	
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Foreword

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Sammanfattning

I och med ständigt ökande efterfrågan på elektrisk energilagring (EEL) ökar även behovet av att ta hänsyn till miljöpåverkan, geopolitisk säkerhet samt tillgång till råmaterial vid utveckling av nästa generation EEL-lösningar. Batterier baserade på organiska material kan tillverkas från biomassa, som finns lättillgänglig i de flesta delar av världen och kan extraheras utan energikrävande gruvdrift som ofta är förknippad med kraftig lokal miljöpåverkan. Organiska material kan också omvandlas vid låga temperaturer genom organisk syntes och erbjuder en enorm variationsrikedom vilken kan användas för att tillgodose specifika behov från slutanvändare. Till exempel kan batterier med variabel spänning utformas och användas för att reducera antalet elektroniska komponenter samt att öka energieffektiviteten vid användning i elektroniska produkter. Syftet med detta projekt var att utveckla tre olika batterityper baserade på organiska material i form av ledande redoxpolymerer (LRP). Vi benämner dessa batterityper 1) vattenbaserat-LRP-batteri, 2) proton-skyttel-batteri, respektive, 3) protoninfångningsbatteri. Och målet med projektet var att utveckla batterier som tillsammans täcker ett brett spektrum av batterispänningar, från 0,4 V till 3,5 V. För att åstadkomma detta har vi syntetiserat organiska batterimaterial som är kompatibla med olika cykelkemikalier, som t.ex litium-, natrium- och protoncykling. Mer än 25 nya elektrodmaterial har syntetiserats, karaktäriserats och presenterats i 14 vetenskapliga publikationer under projektet. Från karaktärisering av de olika materialen har vi identifierat molekylära designkrav för LRP material som är av central betydelse för att uppnå önskade materialegenskaper. De två viktigaste designkraven är 1) kravet på redox-

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matchning mellan polymerbas och den redox aktiva gruppen och 2) användningen av polythiofen som polymerbas eftersom LRP-material baserade på denna polymerbas uppvisar effektiv och icke-aktiverad elektrontransport, eller så kallad semi-metallisk ledningsförmåga. En patentansökan, som skyddar den materialfamilj som möjliggör användande av industriellt gångbara beläggningsmetoder för de LRP-material som används i projektet, har också lämnats till patentverket. Dessutom har elektrolyter för organiska batterimaterial utvecklats, karaktäriserats och presenterats i 3 vetenskapliga publikationer. Dessa har utvecklats dels utifrån behovet av elektrolyter som är specifikt anpassade för organiska batterimaterial och dels för att möjliggöra framställning av organiska batterier utan flytande elektrolyt. Genom att kombinera kompetens från Chalmers tekniska högskola, Lunds universitet och Uppsala universitet har samtliga batteriteknologier ovan realiserats och teknologi för organiska batterier som täcker spänningsområdet mellan 0,4 V och 3,5 V finns nu tillgängliga för vidare utveckling. Nästa steg mot kommersialisering av organiska batterier är en noggrann prestandautvärdering av de batteriteknologier som utvecklats inom projektet samt livscykelanalys och marknadsanalys för den mest lovande batteritekniken som vi i nuläget bedömer är batterier baserade på protoncyklingskemier.

Summary

The need for electrical energy storage (EES) is steadily increasing and with that the need for taking environmental and geopolitical concerns as well as raw material availability into account when developing the next generation of EES solutions. Organic batteries can be made from biomass, which is readily available in most parts of the world and can be extracted without energy greedy and environmentally costly mining. Organic matter can also be refined at low temperatures through organic synthesis and offers an enormous variability that can be used to meet specific end-user needs. For instance, batteries with variable voltage output can be designed and used in order to reduce the number of electronic components as well as to increase energy efficiency when used in electronic products. In this project we set out to develop three different organic battery types, The *all-Conducting Redox Polymer (CRP) water based battery, The proton-shuttle battery and The proton-trap All-CRP battery* which together cover a broad range of voltage outputs ranging from 0.4 V to 3.5 V. To that end organic battery materials compatible with different cycling chemistries, i.e. lithium, sodium and proton cycling chemistries, were developed in this project. More than 25 novel electrode materials have been synthesized, characterized and presented in 14 scientific publications during the project. From the combined results design strategies for CRP materials have been identified, the most important being 1) the requirement for redox matching between polymer and the redox active pendent group, and 2) the use of polythiophene backbones to achieve efficient and non-activated electron transport through the materials. A patent application has also been filed protecting a material family that enable industrially viable processing methods for the electrode materials used in the project. In addition electrolytes specifically designed for organic battery materials have been developed, characterized and presented in 3 scientific publications in order to enable efficient and environmentally benign EES technologies as well as batteries devoid of liquid electrolytes. By combining the competences at Chalmers University of Technology, Lund University and Uppsala University all targeted battery technologies have been realized and organic battery devices are now available that cover the voltage output range between 0.4 V to 3.5 V. The next step towards

bringing All-Organic Batteries to market is a thorough performance testing of developed battery technologies together with life-cycle assessment including market analysis for the most promising battery technologies.

Introduction/Background

The current need for sustainable materials in batteries for energy storage applications is enormous. Today's lithium ion batteries (LIBs) are to a large extent relying on inorganic materials (e.g., $LiCoO_2$, $LiFePO_4$, $LiMn_2O_4$, $Li_4Ti_5O_{12}$ ...) obtained from limited mineral resources which are expected to become increasingly more energetically expensive to fabricate as the battery materials become scarcer and the need for batteries becomes larger. High expectations are put on organic matter based batteries to be able to meet future requirements for energy storage technologies to manage the intermittent renewable energy sources and ensure the global safety of the future "smart grid". Our project has been a focused effort to develop novel organic matter based materials assembled in energy storage devices ***having long cycling life and eco-friendly end-of-life handling, and that can be produced in an up-scalable process with a low CO_2 footprint***.

There are several challenges hampering the development of organic materials for batteries that need to be addressed on a fundamental level, the most important being *low energy densities* (gravimetric and volumetric) due to low cell voltages and/or specific capacities, *limited stability* (cyclability) and *low conductivity*. These key questions are addressed in the project.

Four different groups of organic redox-active functional groups (reacting at both high and low potentials) have to date been identified. These include specific quinone-type structures, [1-5] stabilized nitroxide radicals, conjugated carboxyl moieties and di-sulphur containing molecules and they all have benefits and drawbacks. The nitroxide radicals and the conjugated carboxyles have potentials that are well suited for positive and negative electrode materials, respectively, while the quinone- and di-sulphur-type materials show higher specific capacities. Common to all these materials are that they require efforts to reduce solubility of the active material in common battery electrolyte and to overcome the limited electronic conductivity that these materials possess.

Five methods have been suggested in order to reduce solubility of organic matter based energy storage materials: 1) anchoring of soluble electroactive organic molecules onto the surface of an insoluble substrate, [6] 2) increasing the molecular weights of the compounds (polymers), 3) increasing the molecular charge (lithium salts), 4) using polymer electrolytes and/or ILs, and 5) employing appropriate cell designs.

The limited electronic conductivity of organic matter based electrode materials is in general overcome by the addition of conducting additives. Such additives, however, increase the weight of the electrode thereby reducing the effective capacity and alternative methods to overcome limited conductivities are desirable.

Conducting polymers (CPs) constitute a fifth class of organic matter based energy storage materials that has been intensely studied and that has a central role in the current proposal. CPs are electronically conducting and they can be oxidized and reduced at relatively high rates indicating that these materials are well-suited for high power applications. The main problems with CP based batteries and supercapacitors are generally considered to be their high self-discharge rates [7, 8]

and low capacities due to the limited inherent doping capacity. [9, 10] In recent years several research groups have anticipated that combining CPs with high capacity redox functional groups could provide a high capacity battery material free of both resistance- and dissolution problems.

In this project we have brought together the competences necessary for creating a cluster for organic matter based electrical energy storage (EES) research and development. Such clusters did already exist for inorganic matter based EES but for the organic counterparts the research has until the start of our project been carried out in separate smaller projects. By bringing together expertise from Uppsala, Lund and Chalmers our aim was to create a strong fundament for next generation of sustainable EES and also a platform for young researchers to expand from and to attract international and industrial collaborators to.

The project, we hope, has offered a unique opportunity to place Sweden in a favorable position to be the leader in developing the basic and applied research aiming at creating next generation of rechargeable batteries that are foreseen to meet the requirements on eco-friendliness and sustainability and that are powerful enough to provide an alternative to presently available, inorganic secondary batteries. Hence, the steadily increasing demand of inorganic materials used in today's batteries with concomitant cost and energy consumption, due to the diminishing mineral resources, can be reduced.

Implementation

The present project has been based on collaboration between researchers at Uppsala University, Lund University and Chalmers University of Technology. The participants are the NFM (Nanotechnology and Functional Materials) group within the Dept. of Engineering Sciences, Uppsala University (Prof. Strømme and Prof. Sjödin); Lund University (Prof. Patric Jannasch) and the Condensed Matter Physics group, Dept. of Physics, Chalmers University of Technology (Prof. Jan Swenson).

In the project we have developed 3 different Organic matter based batteries with tunable voltage:

1) *The all-Conducting Redox Polymer (CRP) water based battery - A safe and environmentally benign secondary battery:* This battery type has been developed in collaboration between Uppsala University and Lund University where Uppsala University has been responsible for the development of electrode materials as well as battery assembly and testing while Lund University has developed proton conducting membranes for this battery type. Both this battery-type and the proton-shuttle battery (see below) rely on the ability to tune quinone potentials as this is the means by which a cell voltage is achieved. An initial study was therefore performed where the effect of substitution in various electrolytes was performed and we could conclude that protic ionic liquids developed within the project allowed for an increased susceptibility to potential tuning of the quinone potentials and hence higher output voltages. Synthetic methods to attach substituted quinones onto conducting polymer backbones were then developed along with the development of specific proton conducting membranes for this battery type. Finally complete all-CRP water based batteries were assembled and tested. For this cell-type the battery performance has been investigated using a non-traditional charging (potentiostatic charging) as well as traditional galvanostatic charging. The

former method is inherently simpler than the galvanostatic method and allows the battery material to be charged at maximum rate at all timed during charging which, for traditional battery technologies, is not possible due to material limitations, i.e. cracking of active materials at high current densities.

2) *The proton-shuttle battery - A novel concept:* Also this battery type has been developed in collaboration between Uppsala University and Lund University where Uppsala University has been responsible for the development of electrode materials as well as battery assembly and testing while Lund University has developed protic ionic liquids for this battery type. Successful development of this battery type relied on the development of electrolytes specifically designed for the quinone redox chemistry allowing efficient proton transport. To that end 7 different electrolytes were developed and characterized with respect to proton conducting properties during the project and two were chosen for continuous use for proton shuttle batteries. In order to verify the feasibility of using the developed electrolytes in batteries the electrochemical behavior of quinone CRPs in the electrolytes were investigated. Finally complete proton shuttle batteries were assembled and tested using traditional battery characterization techniques.

3) *The proton-trap All-CRP battery*: This battery type has been developed in collaboration between Uppsala University and Chalmers University where Uppsala University has been responsible for the development of electrode materials as well as battery assembly and testing while polymer electrolytes for this battery type has been developed at Chalmers University. Material synthesis has been focused on the development of proton-trap materials as well as on high potential quinone CRPs for use as active cathode material and their characterization in various electrolytes while polymer electrolyte development has been focused on the combination of polymer electrolytes and ionic liquids. Both proton-trap materials and high potential quinone CRPs have been assembled into complete battery cells and the battery characteristics been investigated using ionic liquid electrolytes as well as traditional electrolytes for lithium ion batteries and sodium batteries.

Results

For the development of active CRP-battery-materials two general platforms were targeted, one with polypyrrole as conducting polymer backbone and one with thiophene-based backbones. Organic synthetic procedures were summarized in H. Huang et al. 2016 and H. Huang et al. 2017 for the polypyrrole platform and in X. Huang et al. 2016 for the polythiophene platform. Fundamental studies on developed materials (L. Yang et al. 2016, H. Huang et al. 2017, H. Huang et al. 2017, L. Yang et al. 2017 and M. Sterby et al. 2019) showed unambiguously that the thiophene based materials provided superior properties. Most importantly polythiophene based CRPs exhibit non-activated (semi-metallic) electron transport, fast redox conversion rates as well as stable redox chemistry (M. Sterby et al. 2017, L. Yang et al. 2018 and M. Sterby et al. 2019). In order to enable judicious choice of pendant groups a computational screening study was performed (R. B. Araujo et al. 2017) as well as a combined experimental-computational study focusing on substituted quinones (internal report H. Wang et al. 2019). From the screening study pendant groups for the three battery

technologies outlined above were identified and the corresponding CRP-precursors were synthesized using developed synthetic procedures.

In R. Emanuelsson et al. 2017 we presented the first all-CRP based battery where we used an acidic water electrolyte and we show that CRPs constitute a promising class of materials for the development of organic matter based batteries with the potential to overcome the main limitations connected to this type of rechargeable battery systems including low conductivity and dissolution problems. In the device both anode and cathode are made from CRPs with different quinone pendant groups and good rate performance is achieved without any conductivity additives thus providing support for the hypothesized synergetic effect of a CP backbone and a covalently attached redox active pendant group. We show that the potential of quinones can be effectively tuned into the conducting region of the polymer backbone which is a prerequisite for profitable combination of the two units.

In Emanuelsson et al. 2016 a CPR based on pyrrole with a hydroquinone pendant group was investigated in organic solvent containing acid/base couples to enabling proton transport. We show that the formal potential in aqueous solution could be completely recovered in acetonitrile by utilizing pyridine bases with different donor−acceptor strengths. A decrease of 61 meV/pKa was found that corresponded exactly to the pH dependence of $E^{0\prime}$ in aqueous electrolyte thus enabling tuning of qunone potentials in non-aqueous media. The results form the basis for the development of the All-Organic Proton Battery presented in R. Emanuelsson et al. 2017. In this report we used conducting poly(3,4-ethylenedioxythiophene) (PEDOT), functionalized with anthraquinone (PEDOT-AQ) or benzonquinone (PEDOT-BQ) pendant groups as the negative and positive electrode materials, respectively, to make an All-Organic Proton Battery devoid of metals. The electrolyte consists of a proton donor and acceptor slurry containing substituted pyridinium triflates and the corresponding pyridine base. This slurry allows the $2e^-/2H^+$ quinone/hydroquinone redox reactions while suppressing proton reduction in the battery cell. By using strong (acidic) proton donors, the formal potential of the quinone redox reactions is tuned into the potential region in which the PEDOT backbone is conductive, thus eliminating the need for conducting additives. In this all-organic proton battery cell, PEDOT-AQ and PEDOTBQ deliver 103 and 120 mAh g^{-1}, which correspond to 78% and 75%, respectively, of the theoretical specific capacity of the materials at an average cell potential of 0.5 V. We show that PEDOT-BQ determines the cycling stability of the device while PEDOT-AQ provides excellent reversibility for at least 1000 cycles.

In order to further improve the presented All-Organic Proton Battery specifically designed protic ionic liquids were developed with several advantages compared to the original electrolyte including lower melting point, lower toxicity, and compatibility with plastic materials. For this electrolyte we use nonstoichiometric protic ionic liquids (NSPILs). NSPILs contain a mixture of proton donors and acceptors and are ideal for this purpose due to their high proton conductivity, high electrochemical stability, low cost, and ease of synthesis. However, the electrolyte proton activity must be controlled carefully in these devices since it greatly influences the kinetics and energetics of the electrode redox reactions and, hence, also impacts battery device performance. In this study (Karlsson et al. 2018), specific NSPILs were designed and evaluated as electrolytes for the All-Organic Proton Battery. The NSPILs were based on either 1,2,4-triazole or 1-methyl-1,2,4-triazole partially protonated with bis-(trifluoromethylsulfonyl)imide (TFSI) to

produce a range of NSPILs with different degrees of protonation. Both types of NSPIL investigated exhibited a maximum conductivity of 1.2 S/cm (at 120 and 70 °C, respectively), and the eutectic composition of 1-methyl-1,2,4-triazolium TFSI also had high conductivity at 25 °C (24.9 mS/cm), superior to, e.g., imidazolium TFSI NSPILs. We show that the quinone functionalized CRPs exhibited reversible, fast, and stable redox conversion in these electrolytes. The corresponding All-Organic Proton Battery cell using this electrolyte demonstrated fast and complete redox conversion with a cell potential of 0.45 V, even up to scan rates corresponding to 140 C. In addition to the NSPILs electrolyte polystyrene sulfonate rubber block copolymer membranes, Poly(dimethylsiloxane)-azole membranes as well as poly(vivylazole) membranes have been synthesized for use as proton conducting membraned for All-Organic Proton Batteries.

For high voltage batteries proton-trap materials were developed. The first example of a fully functional proton trap material was presented in Åkerlund et al. 2017. Here an organic cathode material based on a copolymer of PEDOT containing pyridine and hydroquinone functionalities was used. The quinone to hydroquinone redox conversion was utilized for charge storage and we show that the proton trap material is compatible with lithium and sodium cycling chemistries. These materials have high inherent potentials that in combination with lithium give a reversible output voltage of above 3.5 V (vs $Li^{0/+}$) without relying on lithiation of the material, something that was not showed for quinones previously. Key to success stems from coupling an intrapolymeric proton transfer realized by incorporated pyridine proton donor/acceptor functionality, with the hydroquinone redox reactions. Trapping of protons in the cathode material effectively decouples the quinone redox chemistry from the cycling chemistry of the anode, which makes the material insensitive to the nature of the electrolyte cation and hence compatible with several anode materials. Furthermore, the CP backbone allows assembly without any additives for electronic conductivity. The concept was demonstrated by electrochemical characterization in several electrolytes and finally by employing the proton traps material as the cathode in lithium and sodium batteries. These findings represent a new concept for enabling high potential organic materials for the next generation of energy storage systems. The materials were further developed in order to improve cycling stability and in Åkerlund et al. 2019 an improved proton trap material was presented. In this study, a stable and nonvolatile ionic liquid was also introduced as electrolyte media, leading to enhanced cycling stability of the proton trap which is attributed to a low basicity of the solvent. Battery devices based on the proton trap material in ionic liquid electrolyte as well as in commercial 1 M LiPF6 in EC/DEC electrolyte yielded voltage outputs of 3.3 V and a capacity close to the theoretical 67 mAh/g. In addition to the ionic liquids used in the proton trap batteries specific polymer membrane separators have been developed and characterized for this battery type (K. Elamin 2019 and M. Shojaatalhosseini 2017).

To conclude we have within this project developed three battery-types, an *All-CRP Water Based Battery, the Proton-Shuttle Battery and the Proton-Trap Battery* providing organic batteries covering a broad voltage range from 0.4 V to 3.5 V. In addition specific electrolytes, both liquid and polymeric, for each battery-type have been developed and characterized. In addition, characterization of developed CRP materials have allowed us to identify important design principles for CRPs, the most important being 1) the requirement for redox matching between polymer and the redox active pendent group 2) the use of

polythiophene backbones to achieve efficient and non-activated electron transport through the materials.

Discussion

To find sustainable ways of providing and administrating the power our present lifestyle demands as well as the power needed to allow for sustainable growth in all parts of the world is one of the main research challenges for the coming decades. An important addition to the flora of energy-storage technologies would be the realization of EES systems based solely on renewable materials that could be produces in energy efficient processes and research to this end is indeed expanding. Decisive innovations in the field of organic matter based EES systems do however require research with full focus on these particular systems as well as a new set of established standards devoid of preconceptions related to cycling chemistries suitable for inorganic electrode materials. With this project, we have initiated the work towards providing such standards, (for active cathode materials, anode materials and electrolytes for well-defined cycling chemistries suitable for organic materials) to for the foundation for efficient and sustainable local energy production and storage solutions.

The project has been challenging and has taken into account all aspects necessary for forming the scientific basis of production of secondary batteries that are made of materials stemming from renewable resources based on high energy density organic redox molecules with CP matrix. The project has taken on a system perspective in which the developed electrode materials and electrolytes have been evaluated in full battery systems.

The set of available standards for cathode and anode materials and electrolytes for cycling chemistries suitable for organic electrode materials provided through this project hopefully sets the basis for new industrialization based on the technologies we have started to develop. The patent application we have filed during the project is currently being used in discussions with industrial players to start such a process.

Publications

The publication outcome of the project has been very high. In addition to the below listed journal publications the project has been presented both orally and as posters at numerous international conferences. One patent application has also been filed: (M. Sjödin, C. Strietzel and R. Emanuelsson, *Conducting redox oligomers*, Patentansökan nr 1950142-8 - Sverige)

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ACS Appl. Energy Mater., (2018), 1, 6451-6462

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