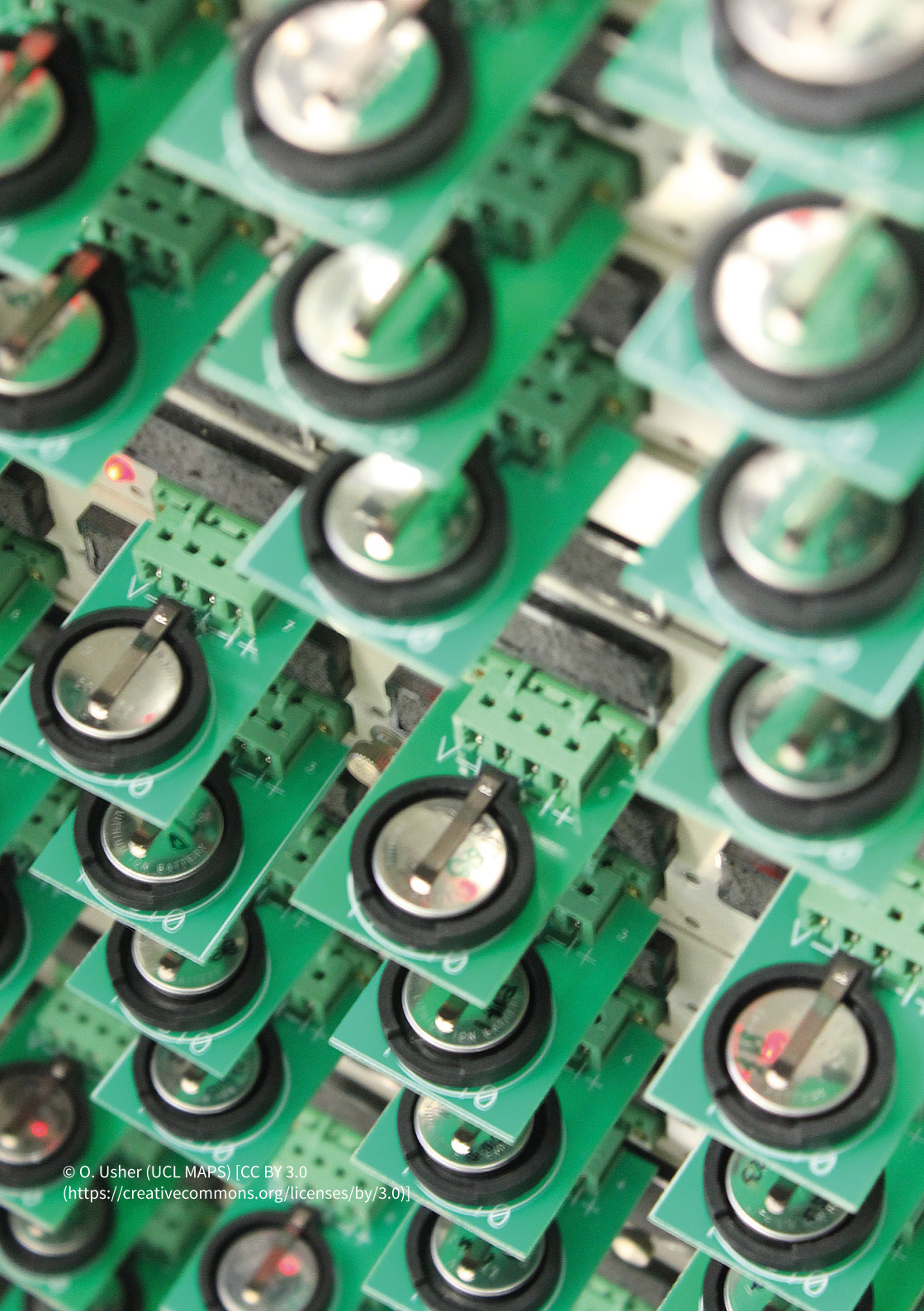
LIÈGE université
Interface Entreprises
Research & Innovation

Battery Technologies at University of Liège

Technology Offers & Research Teams

August 2019



The University of Liège (ULiège) is a Belgian research based university, hosting more than 3,500 researchers, scientists and technicians, and offering many advanced skills in science and technology, humanities and social sciences, and life sciences. For many years, open innovation is a key concept in our university, which is by the way involved in hundreds of collaborative projects at regional, national and international levels.

Research in the field of batteries at ULiège is encompassing several topics of major interest such as raw and processed materials, cells and components manufacturing, energy management systems, recycling, ... ULiège has notably built up a strong expertise in the development and processing of materials for the manufacturing of Li-ion battery electrodes.

This brochure aims to give you a non-exhaustive insight into ULiège's skills in the battery sector, and gathers:

- Technology offers benefiting from a patent protection;
- Research teams active in the field.

ULiège is actively looking for companies ready to take part in their next development stages through specific research and/or commercial agreements.

ULiège-Interface Entreprises, the Knowledge Transfer Office of the University, plays a central role in bringing scientists and companies together on projects, in supporting the maturation process of technologies based on results from research, in licensing and company creation. By developing partnerships with the appropriate companies, ULiège-Interface Entreprises is committed to operate a fruitful collaboration and technology transfer for the benefit of the actors and the society. We are convinced that ULiège's opportunities represent valuable assets for economic growth and innovation solutions, especially in the field of energy.

We look forward to exploring with you any strategic partnership to help you to grow your business.

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BATTERY
TECHNOLOGIES

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Technology Offers

Silicon-carbon composite anode material

In order to increase the performances of Li-ion batteries, silicon-carbon composites (Si/C) are nowadays considered as a promising alternative to the conventional graphite based anodes. This is mainly due to the very large theoretical capacity of silicon (+/- 4000 mAh/g). However, a major drawback of silicon is its high volume expansion during the charge-discharge process of a battery which can reach up to 300% and damage prematurely the electrode.

University of Liège has developed new active silicon-carbon (Si/C) composites for anodes with a specific powder shape design that buffers the volume expansion of silicon leading to anodes with high capacity values and high capacity retention over long cycling time.

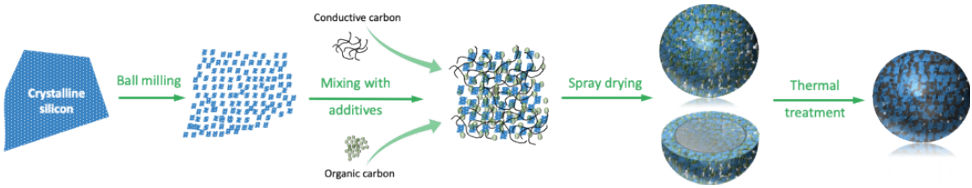

Fig. 1: Preparation of silicon-carbon composites.

Description

This technology relates to Si/C anode active compositions obtained by spray-drying starting from silicon recycled from photovoltaic (PV) panels. The first step is focused on the preparation of Si nanoparticles from silicon wafers by ball-milling in a solvent. A carbon source is then added to the nano-Si suspension and mixed to obtain a homogenous and stable suspension. The carbon source is a mixture of a conductive source (such as carbon black, carbon nanotubes, etc.) and an organic additive. This suspension in then spray-dried to obtain composite particles with sizes of 1 to 50 µm. These particles are subjected to a thermal treatment process under a reductive atmosphere to obtain the silicon-carbon composite anode material.

Potential Applications

The technology is developed towards the manufacture of Li-ion batteries for various applications, ranging from consumer electronic devices to electric vehicles (EV) and grid energy storage systems.

IP status

Patent application 2018 pending - unpublished

Opportunities

Research collaboration and/or license agreement(s)

Key Advantages

- High silicon loading (30 wt%)
- Excellent homogeneity of Si/C composites
- High capacity values
  (e.g. 1200 mAh/g at 1C when limited to 1200 mAh/g)
- High capacity retention
  (e.g. almost 100 % over 1500 cycles at 1200 mAh/g at 1C)
- Possibility to use recycled silicon from PV panels

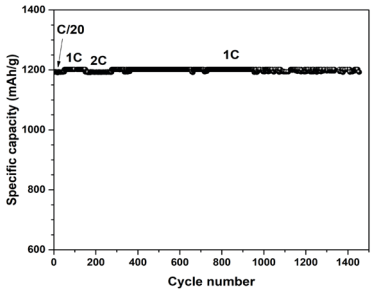

Fig. 2: Long cycle life with 100% efficiency over 1500 cycles.

Research Team

GREENMAT Research Team

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Flexible thin-films for battery electrodes

With the emergence of portable and flexible electronics there is an increasing need for high-performance flexible batteries which can be implemented in products such as flexible mobile phones & tablets, smart textiles, smart watches or health monitoring systems.

University of Liège has developed new active film compositions suitable for the preparation of flexible battery electrodes.

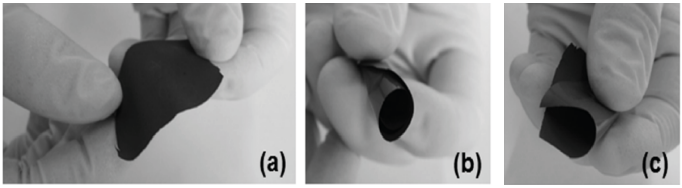

Fig. 1: Flexible film made of $Li_4Ti_5O_{12}$, carbon black, and polymer additives.

Description

This technology relates to anode and cathode active compositions containing a mixture of partially hydrolysed polyvinyl acetate and a polyalkylene glycol as the binder. Figure 1 shows some pictures illustrating the flexibility of a film suitable for the preparation of Li-ion battery anodes.

Such compositions can be formulated with water or with alcoholic solvents (ethanol, propanol, etc.) and represent thereby an environmental friendly alternative to current manufacturing processes in which N-methylpyrrolidinone is generally used. Additionally, formulations with active material, carbon source and binder, can be dried to obtain a ready-to-use powder (Figure 2) that is easily re-dispersible before application.

Key Advantages

- Highly flexible thin films (10-25 µm)
- Fluorine-free binder
- Aqueous (or alcoholic) formulation
- Good electrochemical performances (150 mAh/g-1C)
- Applicable by tape casting, roller coating, spray coating
- Easy reclycling of electrode bases
- High security : no (nano)powder get out of the film when manipulated

Potential Applications

The technology is developed towards the manufacture of flexible electrodes for flexible electrochemical storage devices such as Li-ion, Na-ion and Mg-ion batteries.

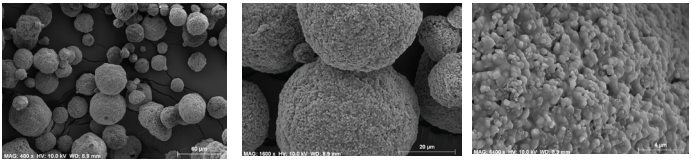

Fig. 2: Dry composite powder obtained from slurry after drying.

IP status

Patent application pending (WO2018/141659A1)

Research Team

GREENMAT Research Team

Opportunities

Research collaboration and/or license agreement(s)

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Sprayable water-based compositions for Li-ion battery electrodes

Electrodes for Li-ion batteries are commonly manufactured by casting a liquid composition onto a metallic current collector. This liquid composition contains an active material, conductive carbon and usually a fluorinated binder dissolved in an organic solvent. Such an approach is not optimal in terms of cost and environmental impact because of the fluorine-based compound itself but also because the latter needs the use of toxic and expensive organic solvents when processed during manufacturing and recycling steps.

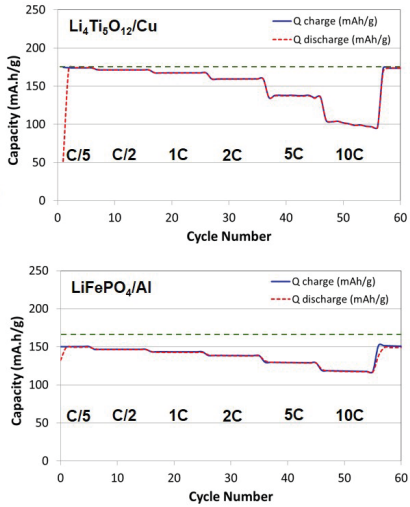
University of Liège has developed water-based compositions for the preparation of Li-ion battery electrodes avoiding the use of fluorine-based compounds and organic solvents.

Description

This technology relates to a process for preparing electrodes by spraying an aqueous slurry composition using a water-compatible polysaccharide (xanthan gum) in substitution of usual fluorine-based binders.

Anodes made of $Li_4Ti_5O_{12}$ and cathodes made of $LiFePO_4$ or $LiCoO_2$ prepared by this process show good and similar electrochemical properties to those prepared by a bar-coating process using polyvinylidene fluoride (PVDF) as binder and N-methyl-2-pyrrolidone (NMP) as solvent. Full Li-ion cells including such electrodes work well and display good cycling stability.

Figures on the right show the (dis-)charge capacities in half-cell configuration at several rates for a $Li_4Ti_5O_{12}$/Cu electrode (top) and a $LiFePO_4$/Al electrode (bottom).



Key Advantages

- Fluorine-free binder
- Aqueous formulation
- Free of dispersant additives
- Improved coating adhesion (no prior surface treatment needed)
- Applicable by spray
- Excellent electrochemical properties of the electrodes
- No need of costly solvent recovery systems
- Easy recycling of electrode bases

Opportunities

Research collaboration and/or license agreement(s)

Potential Applications

The technology is developed towards the manufacture of electrodes for electrochemical storage devices: Li-ion and Na-ion batteries as well as supercapacitors.

IP status

Patent application pending (WO2017/211555A1)

Research Team

NCE Research Team

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Water-based coating process to obtain $LiCoO_2$-microelectrodes free of conductive and binder additives

The manufacture of electrodes for Li-ion microbatteries involves the deposition of metallic oxides thin films, usually applied by expensive, slow and high temperature processes such as physical vapor deposition or sputtering. ULiège and Prayon have developed in partnership a simple, low-cost, and environmentally friendly water-based spray coating process to obtain mechanically stable microelectrodes of $LiCoO_2$ and $LiCoO_2/TiO_2$.

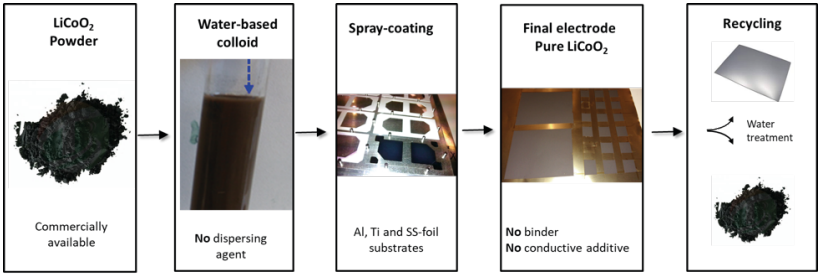

Fig. 1: Illustration of the wet deposition process.

Description

The technology relates to a simple electrode manufacturing process which involves first the preparation of a stable water(ethanol)-based colloidal suspension of $LiCoO_2$. The suspension, free of any dispersant, is then spray- coated onto current collectors and subsequently post-treated at 350 °C. This process produces electrode films up to 10 mg cm^{-1} of pure $LiCoO_2$ (without any conductive nor binder additives).

Electrochemical tests on coin cells showed a remarkable charge/discharge capacity and cyclability at the first cycle (136 mAh g^{-1}), which is maintained around 80 mAh g^{-1} after 1000 cycles. This corresponds to 0.056 % decay per cycle only (charge at constant voltage mode at 4,2 V during 15 min and discharge at 10 C-rate).

Research Team

NCE Research Team in partnership with Prayon - a chemical company leader in phosphate industry.

PRAYON

IP status

Patent application pending (WO2016/097396A1)

Key Advantages

Eco-designed coating process:

- **Green-manufacturing:** free of dispersing, binder and electric conductive additives; use of environmentally friendly solvents; low-temperature post-treatments;
- **Product:** flexible and stable microelectrodes of pure $LiCoO_2$ and pure $LiCoO_2/TiO_2$; good high-rate charge/discharge capacities and good cyclability;
- **End-of-Life:** Easy recycling of active materials and current collectors.

Potential Applications

The technology is developed towards the manufacture of electrodes for Li-ion micro- and/or flexible batteries for portable electronic devices.

Opportunities

Research collaboration and/or license agreement(s)

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Metal oxide wet deposition process

ULiège and Prayon have developed in partnership a low-cost process for the deposition of metal oxide powders onto metallic substrates. The final coatings are made of pure and stable metal oxide avoiding the use of any binder additives. This process represents therefore an alternative to other expensive, slow and high temperature coating processes, such as physical vapor deposition or sputtering, and is particularly suitable for the manufacturing of Li-ion battery cathodes.

Description

The process involves the formation of a stable metal oxide (MO_2) colloidal suspension in an aqueous and/or alcoholic solvent (S_N) through a MO_2 pre-functionalization with aromatic/aliphatic acids ligands (L_X) (A), and/or a pre-dilution of the ligands L_X in the solvent S_N (B) (see Figure 1). The colloidal suspension is then applied onto metallic substrates before a final thermal post-treatment.

This process can be applied to any type of nano/micro-structured (< 2 µm particle size) metal oxide powders (TiO_2, MnO_2, $LiCoO_2$, $MiCoO_2/TiO_2$, $Li_7La_3Zr_2O_{12}$, $LiNi_xMn_yCo_zO_2$, etc.). Experiments showed that this wet-deposition methodology can be used to manufacture flexible and mechanically stable electrodes for battery applications. For instance, cathodic microelectrodes made of $LiCoO_2$ on stainless steel have been obtained and display good reversible charge/discharge capacities (see Figure 2).

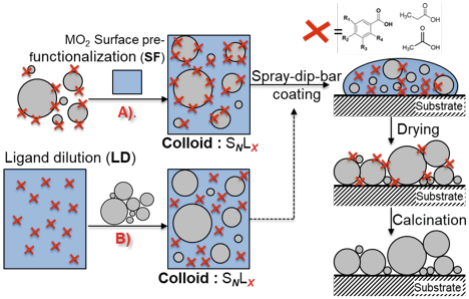

Fig. 1: Deposition process.

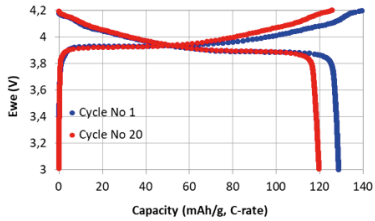

Fig. 2: Charge and discharge curves of a coin cell with an $LiCoO_2$ cathode on stainless steel.

Key Advantages

- **Versatily**: low-cost process suitable for the deposition of various types of MO_2 powders;
- **Green-manufacturing**: use of environmentally friendly solvents; no binder additives; low-temperature post-treatments;
- **Product**: suitable for flexible and stable microelectrodes (e.g. $LiCoO_2$ cathodes);
- **End-of-Life**: Easy recycling of MO_2 powders and substrates.

Research Team

NCE Research Team in partnership with Prayon - a chemical company leader in phosphate industry.

PRAYON

Potential Applications

Specific applications are the wet-depositions of cathodic powders ($LiCoO_2$, $LiNi_xMn_yCo_zO_2$, NMC) and of inorganic separator/electrolyte powders such as $Li_7La_3Zr_2O_{12}$.

Opportunities

Research collaboration and/or license agreement(s)

IP status

Patent application pending (WO2013/171297A2)
Granted in CN (CN104302808B) & in JP (JP6346605B)

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Metal oxide thin fims by Sol-Gel deposition

ULiège and Prayon have developed in partnership a Sol-gel deposition process suitable for the deposition of thin lithiated transition metal oxide films for the manufacturing of micro-electrodes. This process represents a cost-effective alternative to the traditional deposition techniques used in the microbattery industry.

Description

The process involves the preparation of a percursor solution containing precusors of the desired metal oxide. For instance, in the specific case of the cathodic $LiCoO_2$ active material, the precursors may be lithium acetate (LiAc) in association with cobalt acetate ($Co(Ac)_2$) or cobalt nitrate ($Co(NO_3)_2$). The precursor solution is then sprayed onto a substrate; spraying is followed by a thermal treatment and a final calcination step which allows for the successful conversion of the precursors into a metal oxide thin fim. The thickness of the film depends on the number of successive layers. Typically, thin films of about 50 to 60 µm can be achieved with 100 layers.

Figure 2 presents the charge/discharge curves of a $LiCoO_2$ cathode obtained by the present sol-gel process from an ethanolic precursor solution made of $LiAc/Co(NO_3)_2$ The cathode is made of 72 layers (35 µm) calcined at 540°C for 6h.

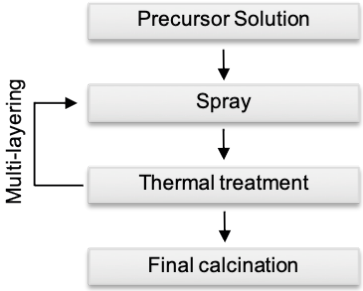

Fig. 1: Deposition process.

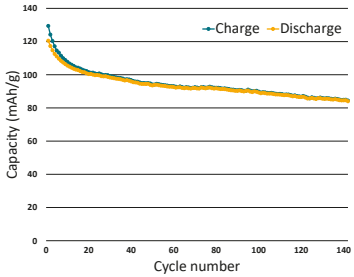

Fig. 2: Charge and discharge curves of a $LiCoO_2$ cathode (72 layers) on stainless steell.

Key Advantages

- Low cost process compared to PVD, PLD, CV and sputtering deposition techniques
- Spray deposition suitable for micro-electrodes
- Optimal adhesion
- Fast production process
- Homogeneous coating distribution

Potential Applications

The process is dedicated to the deposion of lithiated transition metal oxides such as $LiCoO_2$ or $Li_4Mn_5O_{12}$ and is suitable to the formation of any transition metal oxide films of formula $A_aM_bO_c$ or $A_aM_bZ_cO_d$ (in which A is an alkali metal, M is a metal, Z a chemical element and O is oxygen).

IP status

- Patent application pending (WO2015/063264A1) Granted in EP (EP3063809B)
- Patent application pending (WO2015/063266A1)

Research Team

NCE Research Team in partnership with Prayon - a chemical company leader in phosphate industry.

PRAYON

Opportunities

Research collaboration and/or license agreement(s)

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Research Teams

Nanomaterials, Catalysis, Electrochemistry NCE Research Team

www.chemeng.uliege.be/cms/c_2275228/en/chemical-engineering-nanomaterials-catalysis-electrochemistry-nce

The Nanomaterials, Catalysis, Electrochemistry (NCE) laboratory, which is part of the Research Unit in Chemical Engineering of ULiège, has developed a wide expertise in the conception and set-up of devices linked to chemical engineering based on an integrated approach, from the preparation of materials and their characterization to trials in prototype (continuous) reactors. The first part of the activities concerns the design and preparation of new materials with controlled pore texture at the nanoscale. The mastering of the synthesis process enables to perform scale-up studies. Activities about materials also deal with shaping or assembly for industrial applications. The second part of the activities concerns the building of test benches dedicated to the determination of the materials properties (activity of catalysts, performances of fuel cell assemblies or batteries) and to the set-up of a pilot device for the production of a given material. These materials and methodologies have been developed and extended to various research domains in order to solve specific problems. Applications are thus numerous: rational synthesis of nanostructured materials, catalysis and electrocatalysis, purification of liquid or gas effluents, heat and electricity conducting materials, materials for electrochemical energy conversion and storage (fuel cells, batteries, supercapacitors, etc.), multifunctional films, etc.

Since 2009, the electrochemical engineering applications constitute a major axis of the research at NCE. Several large research projects allowed us to acquire state-of-the-art equipment and develop a good expertise in the domain of Li-ion batteries. The laboratory also includes remarkable equipment for the manufacture and in situ characterization of Proton Exchange Membrane fuel cell components, among which fully automated house-built monocell and stack test benches. The objective of the laboratory is to be able to cover the complete manufacture chain, from basic materials to the complete device. In particular, the laboratory has developed a strong expertise in carbon materials for electrochemical applications, but the methods are extended to the processing and characterization of many others.

Key activities in the field of batteries

- Integration of nanostructured materials in composite electrodes (made of active material and, in most cases, binder, conductive additive and electrolyte);
- Physico-chemical and electrochemical characterization of electrode materials and composite electrodes;
- Design of innovative electrode assembly processes (without binder and/or without toxic components);
- Several direct collaborations with companies active in the production of battery materials.

NCE
NANOMATERIALS CATALYSIS
ELECTROCHEMISTRY

NCE

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Group of Research in Energy and ENvironment from MATerials – GREENMAT research team

www.greenmat.uliege.be/cms/c_4468424/en/battery-research-at-greenmat

The GREENMAT group is a research laboratory specialized in the development and characterization of inorganic materials. The group develops powders, bulk materials and coatings for applications related to energy, environment, health or protective coatings. In most projects, it pays special attention to the control of microstructure, porosity and interfaces in order to optimize the material/device properties.

Key activities in the field of batteries

GREENMAT is specialized in the optimized synthesis of nano-/microsized powders (oxides, hybrid, ...) as cathode or anode materials, the development of formulations (suspensions and/or slurries) for the processing of layers (by spray or tape-casting) to be assembled into batteries (from coin to pouch cells).

GREENMAT facilities enable to produce and evaluate materials for Na- and Li-ion batteries from laboratory to pilot scale (grams to kilograms). The laboratory is equipped with several pilot units for the green synthesis of powders: a hydrothermal reactor (5.5 liters) and two spray-dryers (5 liters/h - aqueous or non-aqueous (ATEX) feed). GREENMAT has also equipment for the deposition of the electrode or electrolyte materials (Spray and Doctor Blade).

GREENMAT aims at preparing (new) materials with controlled morphologies and at reducing use of critical raw elements (using Fe, Mn, P, S, Si, ...) by green processes in order to maximize energy and power density and to improve the overall system cyclability.

GREENMAT has also an extensive expertise in material characterization and is equipped for:

- (i) the complete physico-chemical characterization of the designed materials (XRD, Mössbauer spectroscopy, scanning/transmission electron microscopes, TG/TDA, SEM, TEM, BET, Raman spectroscopy, etc.);
- (ii) the cell assembly and electrochemical characterization (galvanostatic cycling, cyclic voltammetry and impedance spectroscopy) of the materials in half-cell and full-cell configurations (coin cell and pouch cells).

GREENMAT is also experienced in the study of the reaction mechanisms operating during the discharge/charge processes of electrode materials by operando and in situ techniques (XRD, impedance and Raman spectroscopies).

Today, the laboratory is involved in several research projects related to the development of new materials for Alkali-ion batteries. Two projects are dedicated to recycling of silicon wafers from solar panels and to use extracted Si as anode material for Li-ion batteries.

Two projects are devoted to the synthesis of new phosphate materials by solvo/hydrothermal reactions. Another project is focusing on the development of polyanionic electrodes for Na-ion batteries. Other projects led to a patent on the design and manufacturing of flexible batteries. Recently, a new project just started on the development of a pouch cell line.

Greenmat
Group of Research in Energy and Environment from Materials

GREENMAT

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Center for Education and Research on Macromolecules CERM research team

www.cerm.uliege.be

CERM was founded in 1993 and, is constituted by 25-30 researchers. It has a worldwide recognition in polymer science, more particularly in:

- macromolecular engineering using controlled polymerization techniques, thus in the precision synthesis of polymers that fit the target application. These researches cover (i) the development of new (sustainable) polymerization processes, including monomers synthesis, catalyst design and mechanistic investigations, and (ii) the full structural and thermo-mechanical characterizations of the polymers by state-of-the-art analytical tools;
- the chemical transformation of carbon dioxide (CO_2) into novel sustainable CO_2-sourced functional polymers (polycarbonates, polyurethanes) and materials, and the use of CO_2 as a green medium for preparing or processing materials;
- the development of innovative applications for polymers and nanocomposites: biomaterials (implants, supports for cell culture, etc.), microcellular foams as electromagnetic interference (EMI) absorbers or for thermal insulation, multifunctional coatings (antibacterial, antifouling, indoor air purification, etc.), solid electrolytes for battery applications, organic batteries, etc.

Key activities in the field of batteries

- Preparation and characterization of new polymer based solid electrolytes with high ionic conductivity for room temperature all-solid based battery operation.
  See for instance: CO_2-sourced polycarbonates as solid electrolytes for room temperature operation lithium battery. J. Mater. Chem. A 2019, 7, 9844.
- Preparation and characterization of novel flexible and binder-free organic cathodes based on radical polymers or on catechol/o-quinone bearing polymers for the next generation of low cost organic batteries characterized by high rate capabilities (fast charge) and capacities. The performances of the cathode material are evaluated in half-cell configuration with lithium as anode.
  See for instance:
  Bioinspired redox-active catechol-bearing polymers as ultra-robust organic cathodes for lithium storage. Adv. Mater. 2017, 29, 1703373.
  Patil, N. et al. Integration of redox-active catechol pendants into poly(ionic liquid) for the design of high-performance lithium-ion battery cathodes. Chem. Mater. 2018, 30, 5831–5835.

CENTER FOR EDUCATION AND RESEARCH ON MACROMOLECULES

CERM

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GEoresources, Minerals engineering and extractive MEtallurgy – GEMME research team

www.gemme.ulg.ac.be

The GeMMe Research Groups develops processes for an efficient management of mineral and metallic resources with four key areas of expertise:

1. BIO – HYDROMETALLURGY (End-of-life products and complex raw materials efficiency)
2. GEOMETALLURGICAL CHARACTERIZATION (Process oriented "mineralogical" mapping)
3. SMART SORTING (Advanced 3D imaging and hyperspectral sorting)
4. PHYSICAL PRE-PROCESSING (Energy-Efficient fragmentation and conditioning)

Key activities in the field of batteries

The key innovation strength provided by GEMME, which is also a fundamental differentiator, is the capability to transfer and scale-up primary metal stream beneficiation techniques to secondary metal streams which is particularly useful in the context of battery development and is expressed as:

- Applied knowledge of process solution behaviour in the context of liquid-liquid and liquid-solid chemical and physical interactions;
- Broad expertise in a wide variety of battery metals (Ni, Co, Mn) and particularly in the field of cobalt hydrometallurgy;
- Know-how and state of the art laboratory installations for upscaling of hydrometallurgy processes with all key steps : Leaching, Precipitation, Solid/Liquid Separations, Cementation, Solvent Extraction, Ion Exchange and Electrowinning;
- Upscaling from laboratory to semi-industrial pilot : Batch (<200g pd*), Mini Pilot (200g pd to 1kg pd*) to Semi-Industrial Pilot (up to 20kg pd*);
- Analytical equipment, including ICP, AA and XRF as well as expertise in particle characterization through optical ore microscopy and quantitative image analysis, SEM-based mineral liberation analyzer (Zeiss Mineralogic SEM-EDX) and microanalytical tools (micro-XRF and EDX 60 mm^2).

With such a background, GEMME can provide the missing links to strengthen the business case for a truly circular battery value chain at two strategic levels:

- Upstream, as partner involved in the production and supply of "recycled" (or secondary) metal stream;
- Downstream, with the characterization and processing of spent battery active metallic compounds.

**weight of pure metal recovered (example for a Cu circuit)*

GEMME

GEMME

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Montefiore Research Unit

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Within the Montefiore research unit there are about 10 faculty members and 50 researchers with diversified and interdisciplinary research interests spanning

- Systems and control;
- Machine learning;
- Optimization;
- Power systems.

Key activities in the field of batteries

- Optimal control of battery storage systems alone or within microgrids accounting for real-time condition of the storage system and its environment;
- Test and characterization of battery storage systems (e.g. following PNNL protocols) for various power systems applications;
- Development of pilot projects.

INSTITUT MONTEFIORE
Département d'Electricité
Electronique & Informatique

Montefiore Institute

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Thermodynamics Laboratory

http://www.labothap.ulg.ac.be/cmsms

For more than 30 years, the Thermodynamics Laboratory of the University of Liège has been carrying out numerical and experimental research activities in the field of thermal and energy systems. The main fields of research are refrigeration, positive displacement machines (compressors and expanders), building physics and HVAC systems, combustion, internal combustion engines, heat exchangers and small and medium scale Organic Rankine Cycles.

Key activity in the field of batteries

- Thermal management of batteries in vehicles (battery cooling systems).

LABORATOIRE DE THERMO DYNAMIQUE UNIVERSITE DE LIEGE

THERMODYNAMICS LABORATORY

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Established in Liège, Gembloux and Arlon, the University of Liège is ideally placed at the heart of Europe. In Belgium, ULiège is the only complete public university of the Wallonia-Brussels Federation.

Its excellence is underpinned by two centuries of teaching and University research, in tandem with an intellectual tradition that has placed Liège firmly on the map since the Middle Ages.

A core priority of the University of Liège is openness to society and the world at large. Every year, the university develops collaboration projects with hundreds of international institutions.

The University of Liège has consolidated its local presence through the diversity of its various campuses. It is actively developing quality management in addition to a responsible environmental and energy policy.

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