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Editorial:

Welcome to the CoLaBATS newsletter

The project is approaching its one year anniversary achieving: development of a system process design (discussed in the project update), specified ionic liquids and eutectic solvents selected and created.

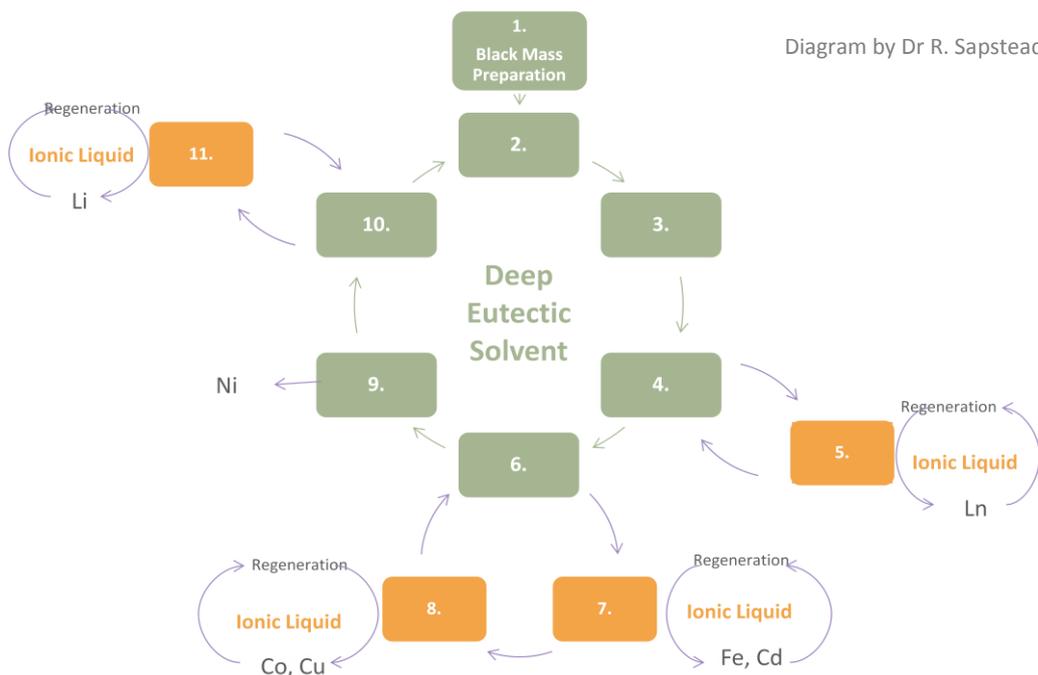
The projects month 12 consortium meeting was hosted in San Sebastian by Tecnalía.

From this point onwards the consortium plan to deliver the specifications of the chosen task specific ionic liquids and materials for recovering metals from the black mass, and the validation of metal recyclability (striving for a 95% metal recovery yield).

Over the coming year focus will be maintained on the up-scale design, construction and operation of a prototype unit. The main objective is the integration of unit operations, achieving an entire system process validation including products and yields (WP3).

Project Update:

Chalmers University, Gothenburg hosted a technical meeting in May 2014, to gather a focus consortium group aimed at delivering a process design. The aim was to determine the optimal use of task specific ionic liquids (TSILs) and deep eutectic solvents (DES) to recover metals from battery black mass. The diagram below demonstrates the proposed 11 step design. The primary concept is a four cycle process, with each cycle having targeted metal recovery.



CoLaBATS Consortium Members



News from the Batteries Directive

The Directive currently limits the permitted heavy metal content of batteries.



More stringent criteria are to be placed on button cells by October 2015, banning 100% mercury from the cells.

Additionally the use of cadmium in batteries for cordless power tools will be banned by the Directive by end of 2016.

The UK will adapt to these EU Directive changes by updating regulations in July 2015.



Ionic Liquids Dr Rachel Sapstead, University of Leicester

Metal recovery and separation from recycled waste streams are currently heavily reliant upon hydrometallurgical processes. Such methods use strong acids and bases both to dissolve metal complexes/oxides and to solubilise metals. In solution, metal salts predominantly form positively charged species (cations), and therefore have a greater solubility in polar or ionic solvents containing high concentrations of negatively charged ions (anions).

The 3 stages of hydrometallurgy are:

Leaching – where the desired metal components are dissolved into solution using acid/base;

Solution concentration/purification – which involves precipitation, cementation, solvent extraction and ion exchange to remove impurities and concentrate the desired metals ready for

Recovery – through electrolysis or precipitation.

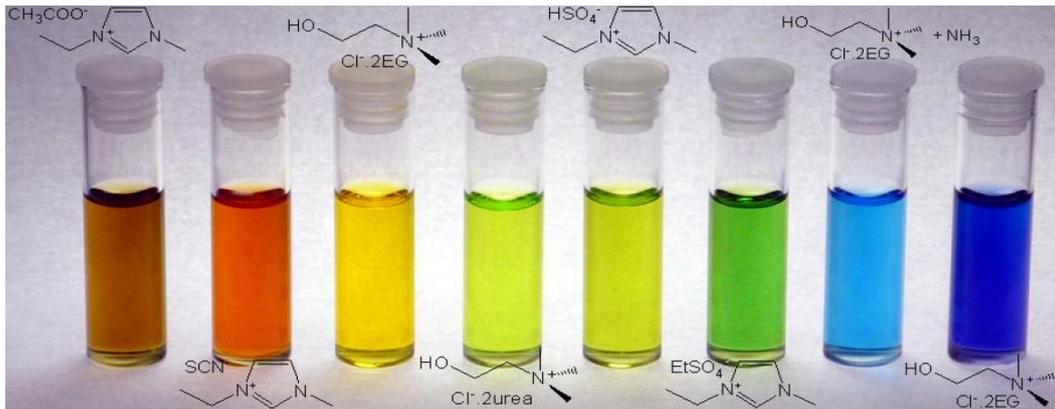
The main disadvantages of hydrometallurgy are the large amount of water used which not only has high potential for contamination but also means strong acids and bases are required for leaching. There are also difficulties in solid-liquid extraction. These factors make many of these processes inefficient, providing strong motivation for more energy efficient and environmentally sustainable chemistries.

Ionic liquids (ILs) are commonly defined as systems which are composed entirely of ions and are liquid below 100°C. As ILs are composed entirely of ions and contain no molecular solvent, such as water, the use of concentrated acids or alkalis conventionally used in hydrometallurgical processes is not necessary due to the high concentration of coordinating anions. As a result of this, ILs have a lot of potential as an environmentally sustainable alternative to traditional hydrometallurgical processing of metal waste streams such as recycled battery waste.

By modifying the structure of the cation and/or anion of the IL properties such as density, melting point, and viscosity can be easily controlled but importantly for metal recovery applications varying the choice of ions can be used to selectively leach a metal compound in media containing two or more different compounds. With regards to solution concentration and purification the miscibility with water can also be controlled in this manner which is an important factor for solvent extraction. ILs have high thermal stability so remain in the liquid phase over a wide temperature range.

Technical Meeting at Chalmers, May 2014

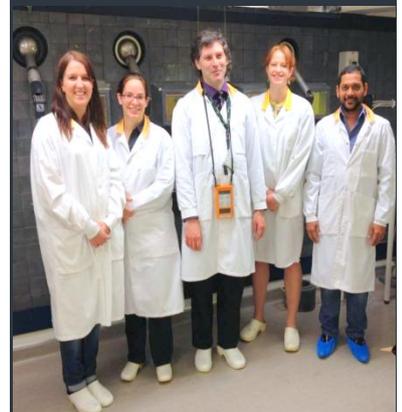
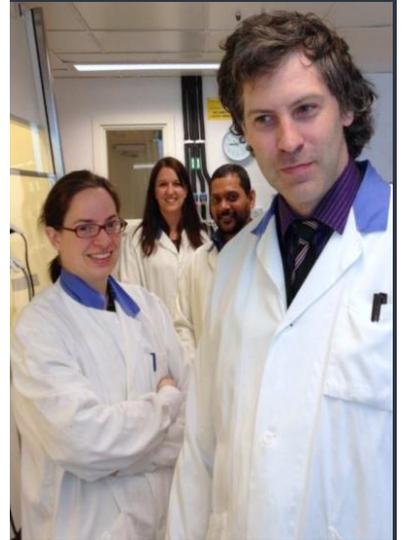
This allows a high degree of kinetic control for both chemical and electrochemical processing that cannot be achieved using conventional molecular solvent/electrolyte systems. This characteristic is also useful for temperature dependent separation techniques, such as extraction, precipitation or crystallisation. The figure below clearly shows that varying the components of the ILs plays a significant part in the coordination of metal ions in solution as the colour of the solutions change depending on the complexing agents present in the ionic liquids.



Solutions of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ dissolved in eight different ionic liquid and DES environment.

Deep eutectic solvents (DESs) are a type of ionic solvent with special properties composed of a mixture which forms a eutectic with a melting point much lower than either of the individual components. DESs offer several advantages over traditional ILs as many are non-toxic, non-flammable, and biodegradable and are inexpensive compared to traditional ILs. There are currently a significant amount of DES publications demonstrating their use in solid-liquid extraction. DESs have shown promising results for metal oxide solubility as they have similar characteristics to ILs. ULEIC has shown that DESs based on choline chloride mixed with various hydrogen bond donors can dissolve a range of metal oxides and can be used to selectively extract metals from complex mixtures using electrochemistry.

Previous work done by ULEIC has shown that DESs can be used for both the selective leaching and electrolysis of metals from both ores and waste streams. For example Reline (1 choline chloride : 2 urea) can be used to process the mixed metal oxide matrix from an electric arc furnace. Zinc and lead can be selectively removed and subsequently recovered from the liquid by electrolysis (electrowinning). Similarly, ethaline (1 choline chloride : 2 ethylene glycol) enables metal dissolution during the electropolishing of steels. The recovery of precious metals (Pt, Re, Pd) from spent auto catalysts and Re from superalloy casting waste can also be achieved using DESs.





CHALMERS

At Chalmers University of Technology in the mid-2000s a new section was established in the department of Chemical & Biological Engineering which is named "Industrial Materials Recycling" (IMR). This is a section which is devoted to the study of recycling, the primary aim of this section is to create recycling methods for materials which cannot be recycled. Another key aim is to produce recycling processes which produce products which are at least as valuable as the original material was before it was fabricated into the product which is being recycled.

Staff of IMR have a range of different backgrounds, working in IMR we have expertise which includes organic & inorganic chemistry, incineration / ashes, organic chemistry, statistics, nuclear chemistry & solvent extraction. We work closely with the Nuclear Chemistry section and are able to harness their expertise to work on a range of problems.

The two members of the IMR team who are working on CoLaBATS are Britt-Marie Steenari and Mark R.StJ. Foreman. Britt-Marie underwent training in organic / food chemistry before becoming a specialist in the chemistry of incineration and ashes. Her interests include the characterization of ashes and other wastes, and the recovery of metals from ashes and other oxide materials.

Mark Foreman is a British chemist who originally trained as an organic chemist at Imperial College, after a PhD in organo-sulfur-phosphorus chemistry at Loughborough he worked as a postdoc in a range of areas which included coordination chemistry (new coordination polymers), asymmetric phosphorus compounds and organometallics. Mark moved to Reading where he worked in Mike Hudson's group on reagents for the solvent extraction of americium. The pinnacle of Mark and Mike's work together at Reading was the development of the CyMe₄BTBP. Using their experience of improving the nitric acid stability of the BTPs they created a nitric acid stable BTBP which has provoked considerable interest in the solvent extraction community.

As Mark matured as a chemist his role changed, during the end of his time at Reading he became an educator. Mark then moved to Chalmers in Sweden. Since arriving at Chalmers his interests have diversified. Mark's interests on the nuclear side now include the chemistry of serious nuclear accidents and the organic chemistry of radioactive wastes. Much of the nuclear accident chemistry revolves around volatile organic iodine compounds, one of his goals is to improve the mitigation of serious nuclear accidents. One of Mark's interests is improving the ability of scrubber technology at nuclear facilities to capture radioactive iodine which is in the form of organic compounds such as methyl and ethyl iodide.

Mark also has an interest in the chemistry of metal binding agents which can form from cellulose inside a radioactive waste store. The digestion of cellulose in calcium bearing alkaline media forms isosaccharinic acid (ISA). This is a compound which can form water soluble metal complexes; these could threaten the safety of future generations. A modern radioactive / nuclear waste store has a series of features which it is hoped will confine radioactivity inside the waste store. The first is the waste package, it can be assumed that waste packages will leak as a result of corrosion, when radioactive metals are leached out into the water in the store they will start a journey from the waste to the biosphere. By slowing down the transport of the metals they will be given more time to decay away thus protecting future generations. One of the important metal retarding processes which is studied at Chalmers is the absorption of metals onto mineral surfaces. It is well known that ISA and other small organic molecules are able to interfere with this process thus making the metals more mobile. Mark is working on a project funded by the Swedish Radiation Protection body (SSM) in which he is investigating the ability of different isomers of ISA to mobilise metals.

Mark's interests in recycling include the recycling of plastic waste, one of the problems in the plastic recycling industry is that the recycling of mixtures of polymers is not a simple undertaking. Many polymers are not miscible with each other, an attempt to melt and mix a random mixture of plastics will form a product with poor mechanical properties. Through a Chalmers interdisciplinary research initiative Mark and Antal Boldizar have projects working towards processes which can allow the recycling of mixtures of plastics from unwanted electrical and electronic equipment.



CEDRAT TECHNOLOGIES (CTEC) is a high tech SME based in the French Innovation Valley, close to Grenoble, which provides innovative solutions in the electrical and mechatronic fields, ranging from the development of software tools, to the study, design and manufacture of systems. CTEC extensive R&D activity is conducted by a multidisciplinary team of experts. Its laboratories are equipped with a complete library of engineering software and specialised measurement apparatus. CTEC focuses its expertise on industry needs for innovation and optimisation and is a member of EARTO (European Association of Research and Technology Organisations).

Areas of expertise include:

Smart Actuators (actuators & motors based on piezoelectric ceramics, electroactive polymers, ultrasonic effects, magnetostrictive alloys, magneto-rheological fluids (MRF) & magnetic effects);

Smart Sensors (magnetic, magneto resistive, magnetostrictive, piezoelectric sensors, transformers or generators; force, torque, position, speed, acceleration sensors, including contactless sensors & resonant sensors);

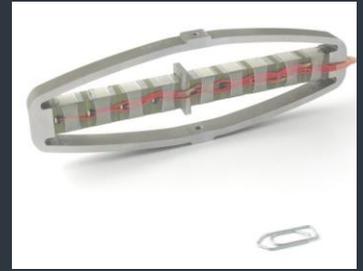
Mechatronic systems (Multi-degree-of-freedom mechanisms, micro robotics motion control, active damping of vibrations, vibration (ultrasonic or sonic) assistance, proportional valves, fast injectors);

Detection systems (Structural Health Monitoring (SHM), NDT using magnetic or acoustic effects, magnetic or acoustic localisation).

CTEC has worked for more than 10 years on ultrasonic system optimisation, based on ultrasonic or magnetostrictive materials. CTEC designs, manufactures and tests innovative sonic & ultrasonic transducers and associated electronics, as well as ultrasonic processes, high power acoustic emission, ultrasonic degassing, cleaning, non-destructive testing (NDT).

Previous Seventh Framework (FP7) activity includes the “SONO” project, where CTEC develops, manufactures, integrates and tests a pilot line based on a novel sonochemical reactor.

CTEC role in COLABATS deals with the ultrasonic (US) system to be used in the main reactor and final tank. CTEC will design, manufacture and test an appropriate US system, including the vibration generator, sonotrode, and electronics that will be adapted to the specific ionic liquid to produce homogeneous ultrasonic vibrations to promote breaking the battery electrode structure. CTEC will provide a US laboratory system to study the behaviour of US in ILs, and determine optimum US levels to achieve efficient break-up of the LiCoO₂ structure and minimise sonotrode material wear. In addition, as WP leader (WP4), CEDRAT will lead a simulation and optimisation task to find the best solution in terms of US system topology (shape, number of transducers, power, and tank position) for the main reactor and the secondary tank. A US system will be integrated in a prototype system (with CTEC and TECN), and scaled up to demonstrator level.



APA® Amplified Piezo Actuator



Piezo Stage



MUST Modular Ultrasonic Transducer



ISO5 & ISO7 rooms

Other UK & EU Battery Recycling

• Tesla Motors

<http://www.teslamotors.com>

Tesla has a closed loop battery recycling program developed for Europe, which is designed to achieve a circular economy for their lithium ion batteries. The loop is focused on reclaiming cobalt and nickel, as well as other metals, and selling on the cobalt as lithium cobalt oxide to other battery manufacturers.

• ABattReLife

<http://www.abattrelife.eu>

An EU project to develop a roadmap for the future of electric vehicle batteries and to establish possible technologies to ensure an optimal life cycle and circular economy.



Env-Aqua Solutions

Upcoming Milestones & Events:



CoLaBATS

Milestone 2	Identification of suitable TSIL	M15
Deliverable 2.2	Quantitative report on TSILs and DES	M15
Deliverable 2.3	Progress report for DES and TSIL selection	M15
Deliverable 4.1	Prototype Design	M15
Deliverable 7.6	Mid-Term Exploitation Workshop	M18

Dissemination Event

Research Project Dissemination Conference, Birmingham, UK. 9th Oct 2014

The Surface Engineering Association is hosting a one day conference to disseminate the results of a number of important UK and European collaborative research and development projects. This event is free, to register visit: <http://www.sea.org.uk/events/>

Conferences

Advanced Automotive Battery Conference Europe	Germany	January 2015
2015MMTA International Minor Metals Conference	USA	April 2015
7 th International Battery Expo & Recycling Conference	India	March 2015
World Advanced Automotive & Stationary Battery Conference	USA	June 2015

This conference features a technology-focused symposium on "large lithium-ion battery technology and applications".

Useful Links:

- CoLaBATS – <http://www.colabats.eu>
- Johnson Controls, Battery Recyclers www.recyclingmybattery.com
- European association for advanced rechargeable batteries www.rechargebatteries.org
- European Battery Recycling Association – EBRA www.ebra-recycling.org

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