Butler Meeting 2019
Scotland And North of England Electrochemistry Symposium
9th April 2019
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ON Organising Committee 2019

On behalf of the Organising Committee and sponsors - welcome to the Scotland and North of England Electrochemistry Symposium for 2019, hosted by the University of Edinburgh.

We would like to thank all the presenters for their diverse contributions that showcase the exciting electrochemistry research happening across our region. This year we are pleased to be running two parallel oral presentation tracks, focusing on electrochemistry and bioelectrochemistry. We are also honoured to be hosting an opening plenary lecture by Prof Kevin Sivula, Head of the Laboratory for Molecular Engineering of Optoelectronic Nanomaterials (LIMNO) at École Polytechnique Fédérale de Lausanne (EPFL), and a closing plenary lecture by Dr Eileen Yu, Head of the Bioelectrochemical Systems and Engineering Research Group at Newcastle University.

This meeting would not be possible without the generous support of our sponsors and exhibitors: the Royal Society of Chemistry, the Royal Society of Edinburgh, IJ Cambria Scientific, Alvatek, Metrohm, IKA, and ScotCHEM. Please do take time to visit them in the exhibition area and find out about how they can help with your research.

On behalf of the Organising Committee, we wish you an enjoyable and stimulating day!

Ignacio Tudela-Montes
School of Engineering

Ilka Schmueser
School of Chemistry

Francisco R Garcia-Garcia
School of Engineering

Stewart Smith
School of Engineering

Jamie Marland
School of Engineering

Jonathan Terry
School of Engineering

Norbert Radacsi
School of Engineering
MEETING INFORMATION

Locations

The meeting will take place within the School of Engineering at the University of Edinburgh King’s Buildings campus. **Registration** is in Sanderson Building Classroom 2. **Talks** will be held in Sanderson Building Lecture Theatre 1 (Electrochemistry Track) and Hudson Beare Building Lecture Theatre 2 (Bioelectrochemistry Track). **Posters** and the **Exhibition** will be in Sanderson Building Classroom 2, and **lunch** will be available adjacent in Classroom 3. All meeting spaces are accessible without steps. **Toilets** are in the Sanderson Building corridor behind Classroom 2, and behind the EngInn café in the Hudson Beare Building.

Catering

Lunch is provided for all registered delegates, and refreshments will available during registration and the afternoon coffee break. The EngInn café in the Hudson Beare Building will also be available throughout the day.

Fire and Security

In case of a fire alarm in any building, please leave in an orderly fashion and assemble outside. In case of emergency on campus, dial 2222 from an internal phone to contact Security.

Internet Access

Academic delegates should use the eduroam wireless internet service. For details see: https://www.ed.ac.uk/information-services/computing/desktop-personal/wifi-networking/jrs-eduroam/jrs-eduroam-vistors

Travel

The School of Engineering is located on the eastern side of the University of Edinburgh King’s Buildings Campus, approximately 2.2 miles south of the city centre. If arriving into Waverly railway station, King’s Buildings can be accessed by bus (Route 42/67, stopping at Rankin Drive, Route 41, stopping at King’s Buildings, or Route 3/7/8/31/37 stopping at Cameron Toll), by taxi (£10-£15), or on foot (45 min). Car parking is not available on the campus, but may be found on surrounding residential roads. For more information on getting to King’s Buildings, see: https://www.ed.ac.uk/transport/travelling-here/kings-buildings
School of Engineering
Sanderson Building
The King’s Buildings
Edinburgh
EH9 3FB

https://www.ed.ac.uk/maps/maps?building=sanderson-building
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The 2019 Butler Meeting has been made possible by the support of the following organisations & sponsors:
PLENARY ABSTRACTS
Direct solar-to-fuel energy conversion using semiconductor photoelectrochemistry

Kevin Sivula
Molecular Engineering of Optoelectronic Nanomaterials Lab – LIMNO, Institute of Chemical Sciences and Engineering, Ecole Polytechnique Fédérale de Lausanne, Switzerland

Short biography: Originally from the United States, Prof. Sivula studied at the University of Minnesota, obtaining a Bachelor degree in Chemical Engineering in 2002, and at the University of California, Berkeley, completing a doctorate in 2007 under the direction of Prof. Jean Fréchet. He then joined Prof. Michael Grätzel’s group at EPFL as a postdoc, and in 2011 he began an independent research program in the Institute of Chemical Sciences and Engineering at EPFL, where he was promoted to Associate Professor of Chemical Engineering in 2018. He directs the Laboratory for molecular engineering of optoelectronic nanomaterials (LIMNO), and teaches courses in transport phenomena, chemical product design, and solar energy conversion.

Abstract: The development of robust and inexpensive semiconducting materials that operate at high efficiency are needed to make the direct solar-to-fuel energy conversion by photoelectrochemical (PEC) cells economically viable. In this presentation the strategy of PEC solar fuel production is introduced and our laboratory’s progress in the development new light absorbing materials and co-catalysts will be discussed along with the application toward overall solar water splitting tandem cells for H2 production. Specifically, this talk will highlight recent results with the ternary oxides (CuFeO2 and ZnFe2O4) 2D transition metal dichalcogenides, and organic (π-conjugated) semiconductors as solution-processed photoelectrodes. With respect to ternary oxides, in our recent work [1,2], we demonstrate state-of-the-art photocurrent with optimized nanostructuring and addressing interfacial recombination by the electrochemical characterization of the surface states and attached co-catalysts. In addition, we report an advance in the performance of solution processed two-dimensional (2-D) WSe2 for high-efficiency solar water reduction by gaining insight into charge transport and recombination by varying the 2D flake size [3] and passivating defect sites [4]. Finally, with respect to π-conjugated organic semiconductors, in our recent work [5] we demonstrate a π-conjugated organic semiconductor for the sustained direct solar water oxidation reaction. Aspects of catalysis and charge-carrier separation/transport are discussed.

References
Microbes and electrons, a bio-electrochemical engineering route towards circular economy

Eileen Yu
School of Engineering, Newcastle University, UK

Short biography: Dr Eileen Yu is a Senior Lecturer (Associate Professor) in the School of Engineering at Newcastle University. Her PhD study worked on the development of direct methanol alkaline fuel cells, which was one of the first studies in the area. After obtaining her PhD from Newcastle University, she worked as a research fellow at Max Planck Institute for Dynamics of Complex Technical Systems, Germany, before returned to Newcastle to take a prestigious EPSRC Research Fellowship (Life Science Interface) in 2006.

This fellowship enabled her to extend her research into the biosciences, from which she has developed a multidisciplinary research programme. This gives her the solid ground to further develop her career in the emerging field of bioelectrochemical engineering. She has established her research group with focuses on novel bioelectrochemical systems for healthcare, CO2 utilization, energy and environmental applications. She is the initiator and Co-PI of £1.2m NERC project of Bioelectrochemical systems for resource recovery from wastewater (MeteoRR). She is also currently the PI leading £2m EPSRC funded “Liquid fuel and bioenergy supply from CO2 Reduction” (LifesCO2R) project. She is Editorial member of Fuel Cells, and Associate Editor for Frontiers of Energy Research. With development of global economy and increase of populations, demands for resources.

Abstract: With development of global economy and increase of populations, demands for resources, energy and water increase drastically in the recent decades. In the meantime, pollution and greenhouse gas emission from industrial processes enhance deterioration of natural environment and climate change. Innovative technologies tackling the challenges are needed.

Bio-electrochemical system (BES) is an emerging technology combining wastewater treatment, resource recovery and energy generation and storage. It uses microorganisms as biocatalysts and electrons from oxidation of organic pollutants in wastewater or from renewable energy for reduction reactions to recover metals, nutrients and synthesis high value chemicals from CO2. Research has been carried out on developing microbial electrochemical systems to recover Cu from distillery wastewater, producing hydrogen and acetate and alcohols with microbial electrosynthesis.

Electrochemical processes offer good reaction selectivity and reduced cost because of the possibility of direct control of electrode surface free energy through the electrode potential. Converting CO2 to valuable fuels and chemicals using electrochemical catalytic reduction (eCO2R) with renewable electricity has attracted growing interest aiming to reduce CO2 emission and energy security. High selectivity of > 90% Faraday’s efficiency, for C1 compounds (formate or CO) was achieved with optimised catalyst structure, electrolyte and reactor configurations. Proof of concept of converting CO2 to formate, and using formate as energy storage medium has been conducted showing promising technology of storing renewable energy in chemical energy carrier.
ORAL ABSTRACTS
**Tough Ionogel Electrolytes for All-solid-state Supercapacitors**

**Xinhua Liu**, Oluwadamilola O. Taiwo, Mengzheng Ouyang, Ridwanur Chowdhury, Huizhi Wang, Nigel P. Brandon, Billy Wu, Samuel J. Cooper, Qigang Wang

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**Abstract:** Ionogels are a new class of promising materials for use in all-solid-state energy storage devices in which they can function as an integrated separator and electrolyte. To improve the mechanical properties but compromises their ionic conductivity, ionogels with ordered structures have recently received attention due to their enhanced ionic transport properties [1]. However, their applications have been limited by poor thermal and mechanical stabilities resulting in decomposition at high temperatures [2]. Therefore, there is a need to develop ionogels with improved thermal and mechanical stability, whilst maintaining good electrochemical performance through structural alignment. Here, we use directional freezing followed by a solvent replacement method to prepare aligned nanocomposite ionogels which exhibit enhanced ionic conductivity, good mechanical strength and thermal stability simultaneously. The aligned ionogel based supercapacitor achieves a 29% higher specific capacitance (176 F.g⁻¹ at 25 °C and 1 A.g⁻¹) than an equivalent non-aligned form. Notably, this thermally stable aligned ionogel has a high ionic conductivity of 22.1 mS.cm⁻¹ and achieves a high specific capacitance of 167 F.g⁻¹ at 10 A.g⁻¹ and 200 °C. Furthermore, the diffusion simulations conducted on 3D reconstructed tomography images are employed to explain the improved conductivity in the relevant direction of the aligned structure compared to the non-aligned. This work demonstrates the synthesis, analysis and use of aligned ionogels as supercapacitor separators and electrolytes, representing a promising direction for the development of wearable electronics coupled with image based process and simulations.

**Fig. 1** Reconstructed images from the XCT and simulation results highlighting the projected flux density resulting from the diffusion simulation, where the bright regions represent higher flux.

**References**

**Effect of bipolar plate materials for proton exchange membrane fuel cell**

Oluwatosin S. Ijaodola¹, Tabbi Wilberforce¹, Emmanuel Ogungbemi¹, Zaki El Hassan¹, James Thompson¹, Abdul Ghani Olabi²,³

¹Institute of Engineering and Energy Technologies, University of the West of Scotland, Paisley, PA1 2BE, UK.
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**Abstract:** The University of the West of Scotland (UWS), Institute of Engineering and Energy Technologies (IEET), is currently embarking on a series of innovative strategies to improve the performance of a Proton Exchange Membrane (PEM) fuel cell both numerically and experimentally. In this work, computational fluid dynamics (CFD) is used to investigate the performance of bipolar plates made of different materials that are used as flow field plates. This is simulated using a single fuel cell stack as test geometry to determine the impact of the material type on the performance of the fuel cell. Serpentine flow field channel design was used for the bipolar plate to investigate the effect of different materials on the functioning of the fuel cell. The polarization curves were used to compare the performance of the different materials tested and to determine the maximum power output from the said material. The bipolar plate materials were simulated under the same operating conditions to facilitate comparison.

**References**

Optimizing Submicron Solid Oxide Fuel Cells Operating on Waste Carbon via Fuzzy and Electrochemical Modelling

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Abstract: Nickel-Gadolinium Doped Ceria (Ni-GDC) cermet anode films were deposited on one side of 50~300µm thick ($T$) zirconia electrolyte supports, by radio frequency (RF) sputtering. The sputtering was done at room temperature and the RF power ($P$) was varied between 50~200 W. Lanthanum Strontium Manganite (LSM/YSZ) cathodes were screen-printed on the other side of the supports. The solid oxide fuel cells (SOFCs) were tested experimentally for maximum power densities under the product fuel of CO$_2$ electro-reduced via industrial waste carbon (IWC) [1] at a temperature ($C$) range of 600 °C ~ 800 °C by voltage ($V$)-current ($I$)-power curves. The resistances of various cell components were measured by Nyquist plots. From the results of these electrochemical measurements, a fuzzy model was built with $T$, $P$, $C$, & $V$ as inputs and $I$ as output. Then, the particle swarm optimization (PSO) algorithm was applied to obtain the optimal parameters of the SOFC, which maximizes its power density. Furthermore, high resolution scanning and transmission electron 2D images were used to build an electrochemical model to simulate the reaction kinetics of these optimized IWC-SOFCs. The results showed that the IWC-SOFC’s power density can be increased by 45% when using the PSO algorithm as compared to experimental results.

Fig. 1 Schematic diagram showing the optimization of lipid recover process.

References
**Session 2: B1.1**

**Electrochemical Diagnostics: Antibiotic resistant Tuberculosis**

_Ewen O. Blair, Damion K. Corrigan_
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**Abstract:** Electrochemical sensors combined with anchored DNA strands on the electrode surface are a powerful diagnostic tool, with the capability to detect the presence of any genetic sequence in a clinical sample. These sensors are a timely development given the increase of antibiotic resistant infections, especially in low-income countries. The MycoChip project at the University of Strathclyde, in collaboration with Yıldırım Beyazıt University in Ankara, is focussed on developing a full point of care diagnostic system for detecting the rising cases of antibiotic resistance in tuberculosis.

Few electrochemical DNA sensors take advantage of the enhanced benefits of micro-scale electrodes. The MycoChip system will therefore use microfabricated microelectrodes (an example is shown in figure 1), decorated with single stranded DNA probe molecules which will bind to the target sequence when present in the sample (an example measurement is shown in figure 2). The advantage of the microfabricated system enables many identical electrodes to be produced, with features on the micrometre scale. These will be combined with microfluidics for processing complex samples, giving a full and miniaturised clinical testing system. This presentation will outline the project, describe the microfabricated gold microelectrodes, and demonstrate their capability of detecting Tuberculosis DNA.

![Fig. 1](image1.png) **Fig. 1** Microfabricated gold electrode, with a 30 μm microdisc.

![Fig. 2](image2.png) **Fig. 2** Example TB DNA detection measurement. Current before and after adding a DNA detection layer and after incubating with TB DNA, recorded on a polycrystalline gold macroelectrode.
Promoting the Remineralisation of Subsurface Caries Lesions

D. Eldosoky, J. P. Elliott, I. Schmüser, F. Nudelman

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The World Health Organisation (WHO) have reported that dental caries – also known as cavity formation - is a major health problem amongst the majority of adults and school children [1]. Dental caries that have progressed onto a later stage tend to be treated through restorative means - mainly fillings which can be metal or composite resins [2]. These are usually invasive and predominantly cause patients’ discomfort, with the effectiveness of these fillings being only temporary [3,4].

A new technology under development by Reminova Ltd sets out to electrochemically promote remineralisation of subsurface caries lesions. The concept behind this is to utilise iontophoresis as a method of delivering calcium and phosphate ions to enhance remineralisation and regrowth of enamel in a way that does not cause pain towards a patient. As this technique is still in its early stages of development, the aims of this research are to characterise how it promotes the nucleation and growth of calcium phosphate – apatite - crystals. In order to achieve this, we are studying calcium phosphate crystallisation inside track-etch membranes placed inside a double-diffusion set-up. We are using an electrical potential to promote the migration of calcium and phosphate ions and create a local supersaturation inside the pores of the membranes.

Preliminary results from in situ monitoring of impedance – using Electrical Impedance Spectroscopy (EIS) - suggest that upon application of a potential, the increase in resistance hints to faster migration of ions to the membrane pores when an electric field is applied. Furthermore, scanning electron microscopy analysis of the membranes showed that the pores were filled with calcium phosphate crystals, which is consistent with the increase in resistance shown by the EIS measurements. Taken together, these results demonstrate that an applied electrical potential may promote ion migration and crystallisation of apatite inside the membrane pores.

References


**Session 2: B1.3**

**Electrochemical detection of Sepsis causing bacteria**

V. Vezza¹, D. Corrigan¹, P. Hoskisson², D. Alcorn³

¹Department of Biomedical Engineering, University of Strathclyde, Glasgow, G1 1RD, UK.
²Institute of Pharmacy and Biomedical Sciences, University of Strathclyde, Glasgow, G4 0RE, UK.
³Royal Alexandra Hospital, Paisley, PA2 9PN, UK.

Email: vincent.vezza@strath.ac.uk.

**Abstract:** Sepsis is a medical emergency with a high mortality rate. It is a result of the body’s immune and inflammatory systems dysregulated response to bacterial pathogens [1]. Currently, bacterial detection times can take up to 72 hours and every hour sepsis goes without targeted antibiotic treatment, mortality raises by 10%. Sepsis affects 260,000 people per year in the UK with 44,000 fatalities [2]. Current bacterial detection methods are expensive, lengthy and complex. Electrochemical sensors allow for cheap, quick and simple detection of sepsis-causing bacteria.

The aim of this project is to develop a low cost, multiplexed sensor capable of detecting the genetic sequences for identification of sepsis causing bacteria. My work has so far examined microelectrodes (because they offer high sensitivity and potential for multisensor arrays) and printed circuit boards (PCBs) (because they are low cost and easily fabricated) as possible options for a point of care system. I have also investigated the importance of the attachment chemistry for immobilising single stranded probe DNA onto the electrode surface and providing a stable baseline with predictable changes in the electrochemical signal upon target DNA binding. The three bacterial species I am developing assays for are *Streptococcus pneumoniae*, *Neisseria meningitidis* and *Haemophilus influenzae*. Infections with these organisms can trigger sepsis. This talk will cover electrode characterisation work, findings on self-assembled DNA monolayer formation and showcase some example electrochemical detection data with the *lytA* gene from *Streptococcus pneumoniae* [3].

![Fig. 1 Example of microelectrode on the left and PCB sensor array (eight working electrodes around counter and reference electrodes) on the right.](image)

**References**

Studies on π-A/Cu pairs in solid-state dye-sensitised solar cells

Ellie Tanaka,1 Hannes Michaels,2 Marina Freitag,2 Neil Robertson1
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2Ångström laboratoriet, Uppsala University, Lägerhyddsvägen 1, Uppsala 751 20, Sweden.
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Abstract: It has been over 25 years since the photovoltaic research field went viral with the emergence of dye-sensitised solar cells (DSSCs) presented by O’Regan and Grätzel [1]. A DSSC is unique in its architecture, employing separate materials for light absorption and charge separation roles (Fig. 1). This concept has inspired materials scientists to test various candidates for the light absorber and charge-conductors, with a goal to achieve better power output and stability in the final devices. There have been successful achievements in the past few years to replace the volatile liquid electrolyte, used in a conventional DSSC, with a solid hole-conductor. The state-of-the-art solid-state DSSCs (ssDSSCs) can be constructed using a method invented by Freitag et al. [2], where the highest efficiency of 11.7% has been reported in 2018 [3]. Here, we would like to share our latest work on ssDSSCs employing a family of donor-free (π-A) thiophene-based dyes (Fig. 2) paired with Cu-based hole-conductors. The importance of finding a better match between light-harvest and charge-transport will be discussed based on some device studies.

Fig. 1 Energy diagram of a DSSC.

Fig. 2 Molecular structure of 5T (π-A dye, left) and standard Y123 (D-π-A dye, right) used in this study.

References
Anodic Reaction and the Corrosion of Cu from Water-containing Deep Eutectic Solvents

Priscila Valverde, Todd Green, Sudipta Roy
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Abstract: An analysis of the anodic reaction occurring at soluble copper anodes during the electrodeposition of copper from an ethaline-based deep eutectic solvent (DES) has been performed. It was shown by UV-Vis spectroscopy and electrochemical measurements that the dominant anodic species produced is the \( [CuCl_2]^- \) complex. In pure ethaline the current efficiency of the anodic process is 100% and the dissolution valency is one. However, in the presence of Cu(II) species the apparent dissolution valency measured gravimetrically was typically less than unity, corresponding to an observed mass loss greater than that expected from Faraday’s law. Moreover, the apparent dissolution valency showed a marked dependence on the electrode rotation rate, Cu(II) concentration and the water content of the deep eutectic solvent. These observations were consistent with a corrosion reaction occurring in parallel with anodic dissolution [1]. The most likely corrosion process is the comproportionation reaction: \( [CuCl_4]^{2-} + Cu \rightarrow 2[CuCl_2]^- \), which is shown in Fig. 1. Voltammetric data indicate that the rate of this process is controlled by the mass transport of the \( [CuCl_4]^{2-} \) complex to the surface and can readily explain the observed dissolution valency dependencies.

Fig. 1 Schematic of the overall dissolution processes showing contributions from anodic dissolution and the corrosion reaction.

References

Investigation into the effect of humidification and external cooling of PEM fuel cell

Emmanuel Ogungbemi1, Oluwatosin S. Ijaodola1, Tabbi Wilberforce1, Zaki El Hassan1, James Thompson1, Abdul Ghani Olabi2,3

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2Department of Sustainable and Renewable Energy Engineering, University of Sharjah, P.O. Box 27272, Sharjah, UAE.
3Mechanical Engineering and Design, Aston University, School of Engineering and Applied Science, Aston Triangle, Birmingham, B4 7ET, UK.
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Abstract: For the PEM fuel cell to perform at optimum condition, there is a need for effective humidification and water management [1-4]. In this work, an investigation into the effect of humidity and cooling of the PEM fuel cell is done using a fuel cell experimental set up at the University of the West of Scotland (UWS) fuel cell laboratory. A PEM fuel cell with a Nafion 212 membrane was subjected to different condition. The following scenarios were tested; a non-humidified hydrogen gas (H2) is used without cooling with fan, a non-humidified hydrogen gas (H2) is used cooling with fan, a humidified hydrogen gas (H2) is used without cooling with fan, and finally, a humidified hydrogen gas (H2) is used, cooling with fan. The experiments were carried out at room temperature and subjected to atmospheric pressure.

The experimental data was collated, and the results were analysed using GAMRY and Microsoft excel software. As a result of the analysis, it was shown that the best polarisation curve was established when the hydrogen was humidified and cooled with the supply of air by external fan. The worst case when the hydrogen gas was not humidified, and the cell was not cooled with a fan. From the result, it can be concluded that hydrogen used for PEM fuel cell should be humidified when possible and external air like fan, used to ensure proper cooling of the system.

References
Session 3: E2.4

Using an Ultrasonic Input for Ammonium Persulfate Electrosynthesis and Advanced Oxidation Processes

A. G. Wallace, P. J. McHugh, M. Symes
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Abstract: Persulfate as an anion is a powerful oxidant that has been demonstrated to have applications in persistent organic pollutant degradation. In this work, it is shown that low-power ultrasonic irradiation does not adversely affect Faradaic yields of persulfate electrosynthesis from ammonium sulfate solutions, and that sonoelectrochemical treatment of sulfate solutions containing exemplar pollutant Amido Blue-black 10B provides swift and effective degradation of dye solutions as compared to treatments where the sonochemical and electrochemical steps are performed separately. The implications for the degradation of persistent organic pollutants are discussed.
Session 3: E2.5

**Electroless Metallisation of Non-Conductive Textiles using a Copper Nanoparticle Catalyst**

_Daryl Fox, G. Taghavi Pourian Azar, L. Krishnan, S. Begum, Andrew J. Cobley_

Functional Materials Group, Coventry University, Alison Gingell Building, Whitefriars St, Coventry, CV1 2DS, UK.
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**Abstract:** Maturolife (‘Metallisation of textiles to make urban living for older people more independent fashionable’) is a three year, European Union (Horizon 2020) funded project aimed to develop assistive technology for older people using selectively metallised textiles as an enabling technology in items such as clothing, furniture and footwear. This approach allows electronics to be truly integrated into textiles which brings the desired functionality with more discretion and without a loss of comfort.

The advantage of using electroless deposition over electroplating is the ability to coat non-conductive materials including natural and synthetic fibres. Electroless plating is achieved by first coating the fabric in a nanoparticle (NP) catalyst before the copper is subsequently plated. The catalysed textile is then placed into a strongly basic solution of electrolyte, HCHO, copper ions and a redox reaction takes place between the formaldehyde and copper ions in solution. This process converts Cu\(^{2+}\) to Cu\(^0\) directly on the catalysed areas of the textile without any electromotive force applied. In the electroless copper plating of textiles various catalysts have been used including as Pd and Ag however these are relatively expensive and Pd is classified as a critical raw material. Previous work at Coventry has established the feasibility of using Cu nanoparticles (CuNP) as an electroless plating catalyst [1,2] which offers advantages in terms of sustainability (Cu is earth abundant) and it is low cost. The copper nanoparticle catalyst (CuNP) needs to be functionalised by an organic molecule and the effect of different organic molecule can have on Cu deposition will be shown in this work.

In this presentation, the Cu coated materials are primarily characterised by SEM imaging (Cu layer uniformity) and electrical conductivity (mΩ/sq) from various catalysts – Cu, Ag and Pd. Other characterisation methods will include particle size analysis, ASTM tape adhesion testing (D3359-17), Cu mass gain, XPS, cyclic voltammetry, and through the stability of the catalyst under storage.

The authors would like to thank the European Union and the Horizon 2020 research and development programme for funding this work via Grant Agreement number: 760789.

**References**

**Session 4: B2.1**

**Pt(IV) Prodrugs Activated By Implantable Microsystems**


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**Abstract:**

Conventionally, prodrugs are activated to their biologically active parent drugs by enzymatic processes. However, due to these enzymes having population-dependent variances or presence in both cancerous and healthy tissues, selectivity and efficacy can be problematic. In order to overcome this, we have developed a novel method of prodrug activation that uses an electrochemical stimulus to activate a Pt(IV) prodrug with only a low reduction potential required. This was achieved by immobilization of a redox mediator onto an electrode surface that reduces, and thus activates, a Pt(IV) prodrug. Since the oxidised mediator is regenerated by constant application of a low reductive potential, it provides an electro-catalytic prodrug activation system.

Here, discrete mediator–electrode devices were created using polymeric encapsulation of the mediator at the electrode surface. This system was optimised to enable efficient application and its ability to function in complex 2D and 3D cellular environments was characterized. Longer-term aspirations are an implantable device capable of activating cancer prodrugs with external control, affording unprecedented selectivity and control where, when and how much active drug is generated.

![Diagram showing ascorbic acid and glutathione activation](image)

**Fig. 1** Ascorbic acid and glutathione are bioreductants that activate Pt(IV) prodrugs in uncontrolled and non-selective manner. They are present in healthy and cancerous tissues and therefore lead to off-target toxicities. An electrochemical strategy was developed to allow temporal and spatial control over prodrug activation.

**References**

**Session 4: B2.2**

**Electrochemical Gels for Biological Applications**

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**Abstract:** Electrochemically grown low molecular weight gels (EGLMWG) have great potential to help improve performance for a variety of existing biological technologies such as membrane technology and drug delivery systems. EGLMWG form thin layers when grown in the presence of hydroquinone [1,2]. The oxidation of hydroquinone produces a localized pH gradient at the electrode surface which acts as the gelation trigger [1,2].

As hydroquinone is toxic it produces gels that are non-cell viable, this limits the gels direct biological applications. Currently EGLMWG are used only as a mould for bulk gelation [3,4]. Here, we show an alternative to hydroquinone that forms electrochemical hydrogels with similar rheological properties to gels formed from hydroquinone, that are cell viable for use in biological applications.

![Fig. 1 Schematic diagram showing (a) hydroquinone gels leading to cell death and (b) the hydroquinone substitute, dopamine, leading to cell viability.](image)

**References**

Development, Optimisation and Miniaturisation of Electrochemical Biosensors for Robust Protease Detection

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Abstract: A multi-disciplinary, EPSRC-funded project IMPACT (Implantable Microsystems for Personalised Anti-Cancer Therapy) aims to use miniaturised sensor chips to monitor real time status of an individual tumour, regarding highly-localised and transitory changes in hypoxia, pH and key biomarkers that influence a tumour’s local response to RT and chemotherapy. Proteolytic enzymes, also called proteases, are one of these key biomarkers that play a pivotal role in several physiological and pathological processes such as inflammation and cancer. In the light of this, there is a need for the generation of selective and sensitive tools able to detect protease activities in a biomedical setting. We have reported the development of peptide-based electrochemical biosensors for the detection of protease activity using self-assembled monolayers (SAMs) on gold surfaces, using the enzymes trypsin2 and human neutrophil elastase (HNE)3. The electrochemical detection of protease activity is obtained when the peptide substrate is cleaved causing the removal/loss of the electrochemical label that leads to a decrease in the electrochemical signal as measured by square wave voltammetry (Fig.1).

Fig. 1 Working Principle of the Electrochemical Protease Sensor

This talk will outline the development of these protease sensors, their optimisation (exploring effects such as employing spacers with different lengths, their labelling and their anchoring in the SAM structure) and their miniaturisation, characterisation and further optimisation to enable localised in vivo measurement.

References

Development of a DNA Biosensor to Detect Antibiotic Resistance

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Abstract: Antibiotic resistance is a growing challenge in the treatment of infectious diseases worldwide, and one which threatens to render our current antibiotics ineffective [1]. Recent surveys of antibiotic resistance in Europe have found extensive resistance in certain pathogenic Gram-negative organisms such as E. coli, P. aeruginosa, and A. baumannii, with multidrug resistant isolates accounting for up to 84% of the tested population in some cases [2]. Current diagnostic techniques typically require bacterial culture and susceptibility testing which can take several days. In order to provide effective treatment more quickly, early determination of the resistance or susceptibility profile of an infection is key. Rapid diagnosis of organism resistances will therefore improve patient outcomes and help limit the spread of antibiotic resistance.

Electrochemical DNA biosensors show promise for the rapid determination of antibiotic resistance due to their versatility, sensitivity and ability to be made compact for ease of use in a point-of-care setting. DNA biosensors with a carefully controlled interface layer (in this case an alkanethiol self-assembling monolayer (SAM)) can specifically identify a chosen genetic target from a complex mixture, making them ideal for detection of bacteria at the point-of-care. This presentation will describe the first stages of development of an electrochemical DNA biosensor for antibiotic resistance. Using physical and electrochemical techniques we evaluate a number of different electrode substrates and SAM layers which show promise as a DNA biosensor, culminating in the detection of antibiotic resistance genes in a population of E. coli [3].

We will also discuss the use of a PCB based portable potentiostat with integrated sensors for DNA biosensing [4]. This device is used to detect clinically relevant resistance genes extracted from a bacterial population and is compared to a benchtop potentiostat performing the same measurements. Such a PCB could provide a complete measurement system for point-of-care DNA biosensing with high sensitivity, ultra-low cost, ease of use, a simple on-board readout and internal controls.

References
1 J. Davies and D. Davies, Microbiol Mol Biol Rev 2010, 74, 417.
Biointegration of implanted biosensor

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Abstract: Implantable biosensors have the potential to transform the diagnosis, monitoring and consequently treatment of a whole class of diseases by providing much higher sampling rates and spatial resolution than is available with blood sampling. The interstitial fluid into which a sensor is placed is superior, in many regards, to excreted fluids such as sweat, tears, saliva that are the focus of wearable devices. One of the challenges to chronic implantation are the interactions between the biological milieu and the sensor materials. Adsorption of proteins and other biomolecules (biofouling) and the foreign body response (FBR) are intrinsically linked and can cause significant reductions in biosensor performance [1]. Implanted circuitry raises the possibility of creating ‘active’ biomaterials whose properties can be altered during and after implantation. By manipulating protein-surface interactions, we aim to modulate cell-material interactions and ultimately reduce the FBR. A number of electrochemical approaches have been described previously [2,3].

In my talk, I will first highlight the motivation by describing IMPACT, a project at Edinburgh to implant biosensors into tumours biology. I will then describe our approach of capacitively charging electrode surfaces and ongoing work in characterising the influence of charge on protein conformation along with in vitro and in vivo effects of this phenomenon.

Fig. 1 a) Image of our platform for electrical stimulation of mammalian cell culture. b) MDA-MB-231 cells excluded from a region near an Aluminium electrode by a ‘cellular electric fence’.

References
POSTER ABSTRACTS
The Solar Application of Few-Layer Molybdenum Disulfide (MoS₂) Films Produced at the Liquid-Liquid Interface

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Abstract: Transition metal dichalcogenides (TMDs) are comprised of natural abundant materials that can meet the future requirements of the global energy sector. Transparent thin film solar materials are an exciting alternative to traditional solar materials, offering nonintrusive energy solutions. Two-dimensional (2D) TMDs such as molybdenum disulfide (MoS₂) have received a lot of research interest for solar energy applications due to its direct band gap in the visible range (~1.9 eV) [1], chemical stability, transparency, flexibility and high charge carrier mobility (148 cm² V⁻¹ s⁻¹) [2]. However the performance of TMDs in solar energy applications has not been fully realised due to the difficulty in producing large area monocrystalline, defect-free films [3].

Hence, we have developed a method of synthesising large area monocrystalline MoS₂ thin films at the liquid-liquid interface from the decomposition of a molecular precursor (tetrakis(N,N-diethyldithiocarbamato)molybdenum(IV)). The decomposition occurs over a few hours at room temperature without stirring or the addition of any surfactants and produces a film of MoS₂, which can be isolated onto substrates of choice. Currently thin film TMDs have achieved photocurrents of 4.0 mA cm⁻² [4], which is significantly lower than the 24.5 mA cm⁻² achieved for single crystal photoelectrodes [5]. We aim to close the gap in performance by generating photoelectrochemical cells from this production method, generating transparent and nonintrusive energy solutions.

References

Catalysts and Electrochemical Device for CO₂ Reduction

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Abstract: Electrochemical reduction of CO₂ to low carbon organic compounds has been considered as a promising method to mitigate the greenhouse effect and produce useful energy carrying chemicals. However, the development of catalyst with high activity, selectivity, and good stability is still the bottleneck to accomplish this goal. Cu based catalysts have been reported to meet such requirement. In this work, we prepared Cu particles with multiple shapes, including nano-flower, nano-needle shaped CuO/Graphene, micro-cube Cu₂O etc. The catalyst was examined using XRD, SEM/EDS, electrochemical testing, gas chromatography and ion chromatography. Results indicate that CuO/graphene nano-flower exhibits a catalytic activity for CO₂ conversion to CO and formic acid with a Faradic efficiency of 40% under -1.1 V vs. SHE. Reaction device plays an important role in CO₂ reduction reaction. In this case, graphene aerogel is utilized in our project for as the next generation electrode. We have synthesized graphene aerogel using Agar geltine method, and finished the properties testing including contact angle test, conductivity test and SEM test.

Fig. 1 a) CuO nano needle and b) CuO/graphene nano-flower catalysts

Fig. 2 a) Image of Graphene aerogel b) SEM of Graphene aerogel

References
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Electrochemistry Posters: E3

Electrochemical Separation and Removal of Neutron Poisons from Lithium Potassium Eutectic

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Abstract: Nuclear energy is great tool in the fight against climate change, as it produces vast amounts of uninterrupted energy without emitting carbon dioxide into the atmosphere. However, it does leave spent nuclear fuel. Currently, there are techniques being used to try and recycle spent nuclear fuel, one of these is pyrochemical reprocessing, which involves melting the fuel into a molten salt forming an ionic soup then electrochemically extracting the fissionable material. This requires very high extraction efficiency to be successful. A new technique is being developed, which is based on a molten salt based reactor system, then removing neutron poisons directly from the reactor system. Neutron poisons reduce the criticality and efficiency of the system, so if they are able to be electrochemically selectively removed from the system while leaving the fissionable material in the reactor, this will increase yield and efficiency. Removing the 4 biggest contributing neutron poisons from a system would remove 80% of the loss of criticality due to fission products [1]. This research project is based around the electrochemical removal and online monitoring of 2 key neutron poisons Samarium and Neodymium from within molten salt solutions using electrochemical separation and alloying techniques. The Samarium (II) to (III) couple has already been characterised on a macroelectrode within the molten salt and some of the key results are shown on this poster. Microelectrodes are also being manufactured for the online monitoring of these electrochemical separations due to their high signal to noise and more detailed response.

References

**Electrochemical conversion of higher alcohols to added value chemicals and energy**

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**Abstract:** Fuel cells convert chemical energy into electrical energy with high efficiency, resulting in lower or zero emission of pollutants. They can produce energy for as long as the fuel and oxidant supply to the electrodes is maintained.

Hydrogen is the best option as fuel, but high pressures are needed to achieve high (volume-related) energy densities [1]. For this reason, liquid alcohols with high volume energy density have been proposed as fuels. In addition to their high-volume energy density, higher alcohols can be obtained from many biological or industrial process. But breaking the C–C bonds is very difficult, and often leads to poisoning of the catalyst. An alternative is the in-principle easier oxidation of an alcohol to the corresponding aldehyde or carboxylic acid. This allows transforming higher alcohols to products with a higher value added at no energy cost or, in particularly favourable cases, coupled to the generation of energy, thereby obtaining a double benefit. We have attempted to oxidise glycerol, an important by-product of biodiesel production, without breaking its C–C bonds. Our target is to generate using a fuel cell Dihydroxyacetone, Tartronic acid or Mesoxalic acid, all of them with a considerably higher market price than glycerol, with high selectivity and the highest possible energy efficiency.

![Electric current diagram](image)

**Fig. 1** Example of the fuel cell.

**References**

**NiO/YSZ microstructure optimization for Solid Oxide Electrolysis Cell Durability Performance**

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**Abstract:** Hydrogen is touted to be a main future clean source of transportation fuel. However, the current process for \( \text{H}_2 \) production of natural gas steam reforming is a large \( \text{CO}_2 \) emission process when compared with hydrogen production via electrolysis from renewable energy [1]. One approach to product \( \text{H}_2 \) and minimize \( \text{CO}_2 \) emission level is \( \text{CO}_2 \) utilization by the process called co-electrolysis of (\( \text{CO}_2 \) and \( \text{H}_2\text{O} \)) via Solid Oxide Electrolysis Cell (SOEC) to form \( \text{CO} \) and \( \text{H}_2 \) at the fuel electrode and \( \text{O}_2 \) at “oxygen” electrode [2]. However, the main challenge for this technology is cell degradation owing to high temperature operation between (700-900 °C) which may impact on the long-term stability of the SOEC. [3-5] Accordingly, this work focused on stability of SOEC electrode where, Ni-YSZ was used as electrode material and characterised for its as crystal size and porosity by using XRD analysis, SEM and BET. The characteristics of electrode material was then used to describe how the synthesis methods and fabrication processes impacted on the cell performance. Preliminary results show that synthesis of a 40\% NiO-YSZ by wet-balling yields a narrow NiO crystal size of 50.01 nm and YSZ crystal size of 26.50 nm with a porosity 15.4\%. The results discussed will highlight for improvement in fabrication method of SOEC.

![Fig. 1 SEM cross section area images of 40\% NiO-YSZ](image)

**References**

**Dithiolene Complexes of First Row Transition Metals for Single-Species Non-Aqueous Redox Flow Batteries**

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**Abstract:** Redox flow batteries (RFBs) are energy storage devices where solutions of electroactive materials are pumped to/from external tanks to the electrode interface for charging/discharging. As energy is stored externally in solution, capacity can be increased independently of the battery power, thus making RFBs promising candidates for grid-scale energy storage [1,2]. Established RFBs utilise aqueous electrolyte solutions; however the voltage output is limited by the narrow (∼1.5 V) electrochemical window of water. Instead, the development of non-aqueous RFBs, which use organic solvents with wide electrochemical windows, is anticipated to improve the voltage outputs. Indeed non-aqueous RFBs containing metal coordination complexes have recently been reported with voltage outputs in excess of 2 V [3,4].

This work incorporates dithiolene ligands to coordination complexes of abundant transition metals for low-molecular weight, inexpensive non-aqueous RFB electrolytes. The dithiolene metal complexes exhibit multiple electron transfer processes and are therefore used as single-species electrolytes, i.e. as both the posolyte and negolyte (Fig. 1). The RFB charge/discharge cycling performance of these materials will be presented here.

![Fig. 1 Dithiolene metal complex (left) and single-species RFB schematic (right).](image)

**References**

The effect of strontium doping into Pr$_{1-x}$Sr$_x$FeO$_{3-\delta}$ perovskite oxides with regards to oxygen vacancy concentration, and the facilitation of the oxygen evolution reaction at electrolyser anodes

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Abstract: Hydrogen has one of the highest relative energy constants when compared to fuels such as natural gas, propane, gasoline, and ethanol. This means that the production of hydrogen through renewable, green technologies is one of the most important challenges faced to date. Production of H$_2$ via the electrochemical splitting of water is a promising example of such a future sustainable green energy technology, and is the focus of this research.

One of the main challenges of the electrochemical splitting of water is overcoming the sluggishness of the kinetics of the oxygen evolution reaction (OER). In particular, the slow proton coupled 4 electron transfer at the surface of the electrode to the oxygen intermediates [1]. Current gold standard anodes for facilitating this process in alkaline electrolytes are primarily based around IrO$_2$. However, these materials are not sustainable due to their low abundance, high cost, and poor ionic conductivity.

The perovskite oxide family of strontium doped praseodymium ferrites (Pr$_{1-x}$Sr$_x$FeO$_{3-\delta}$) are demonstrated to perform well as mixed ion electron conductors without the need for precious metals. It has been reported that transition metals are suitable catalysts for OER half-cell reactions for various reasons, including the fulfilment of the OER activity descriptor involving the near unit occupancy of the e$_g$ orbital of surface transition metal, and high covalency in bonding with the oxygen species$^1$, and the intrinsic nature of transition metals showing stability across multiple oxidation states.

This work aims to understand how perovskite oxides can be viable alternatives to precious metal electrolyser anodes, through an investigation of how the OER onset potential in alkaline solutions is effected by creating an increasingly higher concentration of oxygen anion vacancies within the perovskite crystal lattice, through means of A site substitution of rare earth Pr$^{3+}$ ions by alkaline earth Sr$^{2+}$ ions. The characterisation of these materials primarily comes from in depth powder X-Ray Diffraction, and electrochemical performance is primarily determined by cyclic voltammetry.

The project is fully funded by the Engineering and Physical Research Council.

References

Electrochemistry Posters: E8

CMOS Electrochemical Imaging Arrays

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Abstract: Energy conversion devices make use of thin films and functional materials that exhibit microscopic spatial heterogeneity in their efficiency. The relationship between the distribution of such irregularities and their impact on device performance is not well understood. Hence there is a requirement to map the electrochemical activity in a range of thin films and functional materials. This is termed “electrochemical imaging” [1]. This need is presently addressed by high resolution electrochemical current mapping techniques. One such approach is the use of scanning electrochemical microscopy (SECM) [2]. However high-resolution mapping techniques (example: SECM) are slow (order of minutes) over a wider area (~cm² scale). Hence there is a need to do 2D spatial electrochemical activity mapping at a faster rate (~µs/ms) than those obtained from the conventional techniques. A potential solution is the CMOS active-matrix electrochemical imager – an integrated circuit whose high-level architecture is similar to that of an CMOS optical imager but whose optically sensitive element (photodiode) is replaced by an electrochemically sensitive element (a working electrode (WE)). This is an interdisciplinary project between Schools of Chemistry and Engineering, in collaboration with NPL to develop an electrochemical array imaging sensor. The main aim is to produce an integrated sensor system with active matrix read-out capability for 2D electrochemical imaging. This has been achieved by the design and fabrication of micro-electrode array with active CMOS circuits on a single silicon CMOS chip. The device would potentially be used as an imager, functionally similar to the existing scanning electrochemical microscopy (SECM) technology. As a result, the device will be a lab-on-a-chip, allowing to get images of samples of interest within milli-seconds/micro-seconds rather than much longer times required for typical SECM images.

Fig. 1 Illustrative example: CMOS chip integrated with arrays of electrodes placed over a surface to be imaged [1]

Fig. 2 A sample current map from a conventional SECM [3]

References

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Alkaline zinc-air batteries are greener and cheaper than state-of-the-art lithium ion batteries, but the corrosive nature of aqueous electrolytes leads to long term performance losses, as current collector materials corrode chemically and electrochemically [1]. To investigate the corrosion resistance of current collector candidates, an electrochemical on-line inductively coupled plasma optical emission spectrometry (ICP-OES) setup was created. Dissolved ions from electrochemical experiments are measured downstream to obtain a transient dissolution profile for each material from the partial dissolution current $j_{\text{Diss}}$, and compared to the total current density $j$. With this setup, the most inert current collectors for the negative and positive electrodes were found.

**Fig. 1** Example of a CV of zinc in 1M KOH electrolyte obtained with on-line ICP-OES. Total current $j$ and partial dissolution current $j_{\text{Zn}}$. The oxide growth current is found by subtracting $j_{\text{Zn}}$ from $j$.

**References**

**Back CO\textsubscript{2} the Future: The Electrochemical Reduction of CO\textsubscript{2}**

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**Abstract:** The use of fossil fuels has resulted in great eras for human advancement, however this has come at an environmental cost: CO\textsubscript{2} emissions. CO\textsubscript{2} is recognised as one of the major contributors to greenhouse gasses, and is said to be the most common. It plays a key role in the growth of plants, facilitating wildlife and bio systems, however the balance has been offset due to the rise in industrial activity. Annually, approximately 35 gigatons of CO\textsubscript{2} is produced from just the use of fossil fuels. Due to this, there has been a global CO\textsubscript{2} increase by ca. 12 ppm [1-5].

Carbon capture and storage methods are being implemented, as well as ways to reduce emissions; however, the current area of interest is the conversion of CO\textsubscript{2} to useful chemicals. The electrochemical reduction of CO\textsubscript{2} is one of these methods. Also known as the electrocatalytic conversion, electoreduction, or simply CO\textsubscript{2}R, it is one of the most common methods alongside photocatalytic conversion. The electoreduction of CO\textsubscript{2} can yield a wide variety of products, which include but are not limited to methane, ethane, ethanol, propanol, formic acid and acetic acid. Around 16 different products have been reported in previous work conducted over the years with the use of a copper catalyst [6].

Due to this variety of products, an array of analysis methods are required. This work focusses on establishing different methods of product analysis that are suited to the products. Previous work established the use of High-Performance Ion Chromatography (HPIC) and NMR for qualification and quantification of CO\textsubscript{2}R products at Lancaster University. Current work includes establishing methods both ex- and in-situ for GC-BID (Barrier Discharge Ionisation Detector), -FID, -MS, and DEMS (Differential Electrochemical Mass Spectrometry).

Establishment of an array of product analysis methods enables testing of new, novel catalysts, which can be more selective and efficient. Therefore, further to product analysis work, this work also focusses on novel catalysts and reactor cell design with the use of CAD.

**References**

Molten Carbonate Fuel Cell: Process modelling & simulation

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Abstract: Molten carbonate fuel cells (MCFCs) can simultaneously generate clean energy and segregate CO2 from exhaust gases due to the electrochemical gradient between the electrodes, acting as an active membrane [1].

Aspen Custom Modeler, an equation oriented process modelling software was used in this master thesis to create a custom model of a carbonate fuel cell unit operation, which was validated over a broad range of operating conditions based on experimental data.[2] It was then exported into the Aspen Plus flowsheet simulator environment for process simulations. The objective was to optimize a novel post-combustion CO2 capture process in natural gas power plants,[3] presented in figure 1.

References

**Capital cost analysis and simulation of RFBs**

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**Abstract:** A model for determining characteristics of redox flow batteries (RFBs) based on fundamental electrochemical equations has been developed. Specific values for the equation parameters are based on literature data, and subsequent calculations lead to the determination of stack size and electrolyte volume necessary to satisfy pre-defined energy and power requirements.

The open circuit potential (OCP) at any given instance during operation is calculated from standard potentials and instantaneous concentrations. Overpotentials for a specified current density are calculated based on the individual contributions from the membrane resistance, contact resistance, and charge transfer resistance.

The specific resistance contributions vary depending on parameters such as electrolyte concentration, current density, and degree of component degradation.

Once the characteristics of the battery have been established the parameters can be altered independently to determine their influence on performance (Energy density, power density, resistance, capital cost etc.)

The model can therefore be used as a tool to determine whether optimisation of a specific component will have the desired effect on performance.

Simulations of battery operation can also be conducted – an attempt to model specific component degradation with time is made. For example, the amount of electrolyte crossover, specific electrode resistance, and hydrogen evolution can be modelled to varying degrees of success.

If information about component degradation is available, then the model can be used to predict performance over a long period of time and can be programmed to counteract performance loss by e.g. remixing the electrolytes.

By performing simulations of various battery chemistries, it is possible to generate an estimation of the performance over time, the capital cost, and the lifetime cost/return on investment of any RFB.
Investigations on the Electro-Optic Tuning Characteristics of Liquid Crystal PCF for Sensing Applications

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Abstract: Liquid Crystal Photonic Crystal Fibres (LCPCFs) are gaining significant attention in recent years, owing to their electro-optic and thermo-optic properties. The propagation and polarization properties of PCF can be modified by infiltrating its air holes with the directionally dependent liquid crystal materials [1]. As a result, they find numerous applications in the field of optical telecommunication, all-optical switching and optical sensing. LCPCFs works on the principle of Photonic Bandgap (PBG) effect, wherein light is strongly confined within the low index core region. PBG guidance increases the amount of guided light and reduces the core-cladding mode overlap causing leakage losses.

The near infrared (NIR) and mid infrared (MIR) bands of the optical spectrum are of particular interest for fibre-optic sensing applications. This owes to their improved sensitivity and accuracy compared to other spectral regimes. The transmission properties of PCF can be manipulated by varying the liquid crystal materials infiltrated into the cladding holes, enabling the sensor to operate within the photonic bands having peak sensitivities. Hence, there arises a need to closely examine the electro-optic tuning capabilities of liquid crystal materials on various parameters of the PCF sensor.

Liquid crystal refractive index is susceptible to electrical and thermal changes. Hence, simulations were conducted on a four-ring hexagonal PCF designed using COMSOL MULTIPHYSICS software to analyze the effect of liquid crystal refractive index on fibre parameters such as bandwidth, effective area and confinement losses. By varying the liquid crystal material (refractive index) infused into the cladding air holes, a shift and increase in the photonic bandgap was observed. The bandwidth of the bandgap increases with increasing liquid crystal refractive index value. It was also observed that the spectral positions of the photonic bandgaps can be tuned by changing the liquid crystal infiltrations. Also, within the bandgap, confinement losses were found smaller compared to other wavelengths. Furthermore, the effective mode area graph experiences a downward shift in the bandgap region compared to other spectral regions. These trend attributes to the anisotropic nature of the liquid crystal materials. The simulations conducted revealed that the refractive index tunability (either electrically or thermally) of LCPCF can be exploited to customize the spectral bands to suit any specific application and also opens the possibility for electric field and temperature sensing applications.

References

Electrochemistry Posters: E14

**Electrochemical generation of Zn₂SnO₄ photocatalyst for degradation of methylene blue effluent**

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**Abstract:** The treatment of water contaminated with traces of toxic organic compounds is a common problem throughout the world. Advanced oxidation technology based on photocatalysis using titanium dioxide (TiO₂) is one of the emerging approaches for the water treatment [1,2]. Titanium dioxide is a photocatalyst which, in the presence of near-UV light, is capable of breaking down organic pollutants in water. Degussa P25, which is a nanoparticulate powder of TiO₂, is the most efficient, commercially available photocatalyst. A search for a better photocatalysts and efficient ways of using them, is ongoing worldwide [3,4]. Here, a simple two-step hybrid electrochemical-thermal route was developed for the synthesis of cubic shaped Zn₂SnO₄ (ZTO) nanoparticles (photocatalyst) using aqueous sodium bicarbonate (NaHCO₃) and sodium stannate (Na₂SnO₃) as an electrolyte. The sacrificial zinc metal was used as both anode and cathode in an undivided cell under galvanostatic mode at room temperature. The bath concentration and current density were respectively varied from 30 to 120 mmol and 0.05 to 1.5 A/dm². The electrochemically generated precursor was calcined for an hour at different range of temperatures from 60 to 500 ºC. The crystallite sizes in the range of 24-53 nm were calculated based on the Debye-Scherrer equation. Scanning electron microscopy results reveal that all the particles have cubic morphology with diameter of 40-50 nm. The as-prepared ZTO nanoparticles showed higher catalytic activity towards the degradation of methylene blue (MB) dye, and the 90% degradation was found for the sample calcined at 500 ºC, which is greater than that of the commercial TiO₂-P25 photocatalyst. These results indicate that the ZTO nanoparticles may be employed to remove dyes from wastewater.

**References**

A Redox-Flow-Battery based on aqueous Polyoxometalate Electrolytes

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Abstract: In order to utilise intermittent renewable energy sources like wind and solar power more efficiently, flexible and inexpensive electricity storage capabilities are required. One particularly well-suited option for this task are Redox-Flow-Batteries (RFBs). RFBs are the only type of battery where power and capacity can be scaled independently, thus providing high flexibility and versatile applications.

However, the prevailing technology, the all vanadium system, comprises low energy and low power densities. We investigate two polyoxometalates (POMs), [SiW12O40]4− and [PV14O42]9−, as nano-sized electron shuttles [1]. We show that these POMs exhibit fast redox kinetics (electron transfer constant $k_0 \approx 10^{-2}$ cm s$^{-1}$ for [SiW12O40]4−), thereby enabling high power densities. Furthermore, we could show that these POMs exhibit some more favourable properties for their use in a battery, like high solubility, multiple redox-centres per molecule and a high (electro)chemical stability, which lead to high energy densities and long cycle lifetimes. Water as a solvent provides inherent safety to the system, as no flammable electrodes or electrolytes are used.

In collaboration with Siemens AG, we were also able to scale up the presented system from a 25 cm$^2$ lab cell to a cell with 1400 cm$^2$ [2]. Cycling over a period of nearly three months provided some very promising results, including a coulombic efficiency of nearly 100%. Post-cycling analysis of the electrolytes indicated no sign of degradation and the observed capacity loss of 0.011% per cycle could be attributed to air oxidation. The energy efficiency dropped from 86.1% to 85.1% over the course of 1400 cycles, again showing a highly stable performance.

References

Abstract: The continued integration of renewable energy sources into the power grid, as well as the development of decentralised energy systems, requires energy storage in order to capture and store the energy produced intermittently from renewable sources. One energy storage option is the Redox Flow Battery (RFB) which has several advantages over other secondary batteries. As the active species of an RFB remain in solution during charge and discharge, degradation of the electrodes is minimised. Further, due to external storage of the electrolytes, the energy and power of an RFB can be adjusted independently by varying the volume of stored electrolyte or size of the cell stack. The most mature RFB is the all-vanadium RFB but this also has drawbacks, such as slow electron transfer kinetics of the vanadium ions [1] and a requirement for the use of acidic electrolytes.

Polyoxometalates (POMs) are anionic metal-oxygen clusters of early transition metals, commonly vanadium, molybdenum and tungsten, which can undergo redox reactions in which multiple electrons are transferred. The electrochemical properties of POMs prompted their investigation by our group and the development of a POM RFB which was based on the reactions of [PV$_{14}$O$_{42}$]$^{9-}$ at the cathode and [SiW$_{12}$O$_{40}$]$^{4-}$ at the anode [2]. This work demonstrated that an RFB cell with multi-electron redox reactions occurring in each half-cell could be created by using POMs and that the charge transfer reactions of POMs are relatively fast. [2] The [PV$_{14}$O$_{42}$]$^{9-}$/[SiW$_{12}$O$_{40}$]$^{4-}$ RFB was classified as an asymmetric RFB due to the use of different POM active species in each half-cell. Alternatively, symmetric RFBs can be designed which utilise the same active material in both half-cells.

Our group has also investigated the approach of using ‘bifunctional’ molecules, which can undergo redox reactions at positive and negative potentials, as the active species in symmetric RFBs. This includes the use of fullerene species functionalised with ferrocene units [3]. POMs can also be bifunctional species when composed of two different redox-active metals. In this poster, we outline the results of work in our group on the use of bifunctional species in symmetric RFBs, including the study of a POM for a symmetric RFB which has shown good stability in charge-discharge testing so far in conditions that are only mildly acidic.

References

Graphene Ring Nanoelectrodes (GRiNs): Electrochemical Characterisation and Application as Electroanalytical Sensor

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Abstract: A graphene nanoring electrode design [1], along with its electrochemical and physical characterisation, and examples of application will be presented. Specifically, further advances in nanoring electrode fabrication and electrochemical characterisation of such electrodes will be shown, including the reliability, reproducibility and electron transfer rate constant ($k^0$) measurements using an inner and outer-sphere redox systems. The application in the fields of environmental monitoring and energy storage will also be introduced.

References

Advanced Cation Exchange Membrane for Hydrogen Source of Water Splitting by Electro-Electrodialysis

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Abstract: Acid and oxidative resistant sulphonated copolymer cation exchange membrane (CEM) was prepared and electro-electrodialysis experiments were performed to improve HI molality in the HIx (HI + I2) mixture. Reported membrane was prepared by chemical grafting of 2-methyl-2-(N-(3-sulfopropyl)acrylamido)propane-1-sulfonic acid with dehydofluorinated poly(vinylidene fluoride-cohexafluoropropylene) in presence of radical initiator. Different CEMs were assessed by their morphology, physicochemical properties, and stabilities in compare to Nafion117 membrane. Most suitable PVSU-1.72 membrane showed superior proton conductivity (5.15×10−2 S/cm) and ion exchange capacity (1.72 m eq./g) values in comparison to other acid resistant membranes reported in the literature. Further, PVSU-1.72 membrane consumes low electrical power (65.5 kJ/mol) in comparison to Nafion 117 membrane (156.0 kJ/mol) during electro-electrodialysis. Also, in previous case, close to 100% current efficiency demonstrates membrane suitability. Further, PVSU-1.72 consumes about 290 kJ mol−1-H2 energy to produce 1 mol H2, while Nafion117 membrane consumes 280.80 kJ/mol-H2 energy. These observations are helpful for devising high performance electro-electrodialysis process to develop the HI concentration as H2 source.

References
Fabrication and development of electrode systems for label-free biosensing

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Abstract: Electrochemical detection technologies represent an exciting opportunity towards developing effective biosensors for a variety of medically-related conditions. Electroanalytical techniques present multiple advantages to label-free biosensing because they offer the potential to be low-cost, simple to use, with rapid time to result, and multiplexed onto ‘biochip’ like formats enabling multiple tests on a single device. Furthermore, through the use of electrode systems and the relevant measurement technique (e.g. voltammetric, amperometric, impedimetric), enhancements in sensitivity can be gained through the use of smaller electrodes. With the electroanalytical advantages of reduced $iR$ drop, improved signal to noise ratio, relative insensitivity to convection and steady state currents [1], microelectrodes represent an exciting candidate technology for the development of biosensors. Our group employs a broad range of electrodes including such microelectrodes amongst others to be able to monitor and diagnose complex medical conditions including sepsis [2], multi-drug resistant TB, as well as antimicrobial resistance.

This talk will cover two electrode systems currently under development. Firstly, an array of eight $\times$ 50 $\mu$m microelectrodes fabricated onto a needle shaped substrate will be discussed. The electrodes were used to detect the common inflammatory biomarker interleukin-6 (IL-6) and highly sensitive measurements were possible with a fast time to result, hence paving the way for a point of care test for sepsis [2]. Secondly, a low cost carbon electrode system fabricated on a flexible polymer substrate will be shown. This device was used to detect dopamine (a clinically important molecule, particularly amongst populations of dopaminergic neurons), again at low concentrations and also in the presence of ascorbic acid which is a common interfering species when trying to detect dopamine from clinical samples.

References

A Paper-based Electrochemical Aptamer Biosensor for Monitoring Water Quality

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Abstract: Chemical pollution of water systems has become a major global public problem in both urban and rural practices. Intensive agricultural practices, including prophylactic treatment of livestock with antibiotics often leads to significant does in run-off and-or soil – which may in future accelerate the emergence of antimicrobial resistance, both in natural bacterial flora or in pathogens. In this work, we report on a novel paper-based electrochemical aptamer biosensor for monitoring oxytetracycline (OTC) in water. The biosensor was fabricated using both hot wax and screen-printing, allowing for a low-cost paper-based electrochemical device (Fig. 1). Amino functional graphene/thionine/gold nanoparticles (NH2-G/Thi/AuNPs) nanocomposites were coated on printed electrodes and were used to immobilise a probe DNA aptamer (to capture OTC). Electrochemical measurements including cyclic voltammetry (CV), differential pulse voltammetry (DPV) and electrochemical impedance spectroscopy (EIS) were used analytically to detect OTC (with the biosensor showing a linear response ranging from 100 pg μL⁻¹ to 50 ng μL⁻¹, with a detection limit of 100 pg μL⁻¹). As a proof-of-principle, our work demonstrates sensitive and selective detection of antibiotics, with potential application for the rapid and low-cost monitoring of water quality.

Fig. 1 Preparation procedures of NH2-G/Thi/AuNPs nanocomposites and the assay protocol.
**Bioremediation of Petroleum Hydrocarbons using Sediment Microbial Fuel Cells**

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**Abstract:**

Removal of petroleum hydrocarbons by biological methods such as bioremediation require lengthy periods due to insufficient electron acceptors to drive the biodegradation process. In bioelectrochemical systems (BES), the bioanode serves as an additional electron acceptor and could speed up the bioremediation of petroleum hydrocarbons [1,2]. Different sources of inoculum can be used to enrich the biofilms (bioanodes) [3]. The aim of this research is to develop a BES (sediment microbial fuel cell, SMFC) for bioremediation of polycyclic aromatic hydrocarbons such as phenanthrene, pyrene and chrysene. These complex compounds can be found in anaerobic strata of soil/sediment contaminated by crude oil or petroleum products, and could be removed by anaerobic biodegradation. The research objectives are to cultivate and utilize enriched biofilms to improve hydrocarbon removal efficiency and to study the biofilm’s tolerance towards these compounds. In the first part of this project, a biodegradable surfactant will be used to enhance the hydrocarbon bioavailability and to increase the tolerance of the biofilm. Cost effective electrode material such as stainless steel would also be considered in SMFC configurations suitable for enhancing petroleum hydrocarbon removal rates. Cyclic voltammetry and polarization tests will be used to understand the electron transfer mechanism at the bioanode and power output of the SMFC respectively. Microbial community analysis will be performed to shed further insights on the mechanism and pathways of bacterial communities involved in hydrocarbon degradation in BES. The results of the research are also expected to elucidate the kinetics and dynamics of the biodegradation process, SMFC scale up with suitable materials, and SMFC configurations for performance optimization for biodegradation of petroleum hydrocarbons.

**References**

**Silver/Silver Chloride Reference Electrodes for On-Chip Sensing Systems**

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**Abstract:** Reference electrodes are an essential component in an electrochemical system and the silver/silver chloride reference electrode is one of the most common types. One challenge in developing on-chip electrochemical systems is the need for a miniaturised solid-state reference electrode. This type of reference electrode is limited by fabrication issues, which include limited layer thicknesses and the lack of a defined filling solution. As a result, the stability and lifetime of such electrodes can be significantly affected, representing a major obstacle in device development.

This poster will focus on the results of experiments to develop thin film on-chip silver/silver chloride reference electrodes fabricated in the Scottish Microelectronics Centre’s cleanroom facilities at the University of Edinburgh. The poster will also discuss the integration of on-chip reference electrodes for smart monitoring system applications such as wearable and implantable devices.
Low-Cost point of Care Graphene-Based Cell Flow Cytometry

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Abstract: The full blood cell count is a fundamental test for medical first response teams. However, to carry out this test generally requires counting of cells by bulky and costly optical instrumentation for fluorescent tagging of cells [1]. Microfluidic Impedance Cytometry (MIC), which is based on electrical detection, provides an advantage over optical readout because of the potential for developing devices much compact and smaller in size. MIC is an extensively used technique for single-cell electrical analysis [2] and discrimination where RF voltage is applied to a pair of electrodes that span a microfluidic flow. As a cell passes through the system the impedance is measured via differential current [3]. MIC has been used to analyse micro-organisms, erythrocytes, leukocytes, platelets, and animal and human cell lines [4].

There are three common configuration used in impedance based MIC: coplanar electrodes, parallel electrodes and constriction channel, all with their upsides and downsides. Each design is based on similar mechanism, with an excitation electrode and sensing electrode. Traditionally MIC devices use metal electrodes. In this project, CVD and Inkjet printed graphene is used to replace metal electrodes to improve MIC operation due to its flexibility, low electrical noise and low electrochemical reactivity [5]. We will show the progress towards implementation of several graphene based MIC configuration as presented in Fig. 1.

Fig. 1 (a) 3D model of co-planar electrode design with Metal/Graphene Pillar of few micro-meter. (b) Parallel electrode design. Where gold electrode is coated with graphene within Microfluidic channel.

References
4  D. Spencer et al., Lab on a Chip 2016, 8, 2467-2473.
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DELEGATE LIST

The following is a list of attendees who have chosen to appear on the Delegates List.

Mr Olatunde Akinbuja
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Ms Arlete Bambuwu
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