

# Butler2020

RSC Scotland and North of  
England Regional  
Electrochemistry Symposium

April 2<sup>nd</sup> 2020  
Newcastle University

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**IKA**



## Welcome to Butler 2020

Welcome to the Royal Society of Chemistry Scotland and North of England Regional Electrochemistry Symposium, 2020! The following contains the abstracts and a running order for the symposium. If you arrive on the 1<sup>st</sup> of April, you can join in a lab/campus tour at Newcastle University that will start around 14:00. Please contact Marloes ([marloes.peeters@newcastle.ac.uk](mailto:marloes.peeters@newcastle.ac.uk)) if you are interested in attending.

This meeting would not be happening without the hugely generous support of our sponsors, so please do visit them in the exhibition area and show your gratitude for keeping the Butler meetings free to attend. Many thanks also to my research group (Alex Hudson & Oliver Jamieson) for their helping in organising this meeting. I hope you enjoy Butler2020.

Marloes & Eileen

### Meeting Information

**Registration** will be in the foyer of the Old Library building, 2<sup>nd</sup> floor. Keynote talks in the morning and afternoon will be held in room 2.29 in the Old Library building, while plenary sessions are in break out rooms 2.20 and 2.21. Breaks, lunch and the poster session will take place at foyer area. We would be grateful if poster exhibitors could put up their posters as soon as they can in order to allow people more time to read them during breaks. In the book of abstracts, you can find the number of your poster.

### Prizes

We have a number of generous prizes available, including:

Radleys - £100 prize for best poster presentation (general electrochemistry)

IKA - £100 prize for best oral presentation (general electrochemistry)

Alvatek - £100 prize for best oral presentation (bioelectrochemistry)

SCI - £100 prize for best poster presentation (bioelectrochemistry)

RSC Electrochemistry Interst group (overall winner): free RSC early bird registration for Electrochem 2020

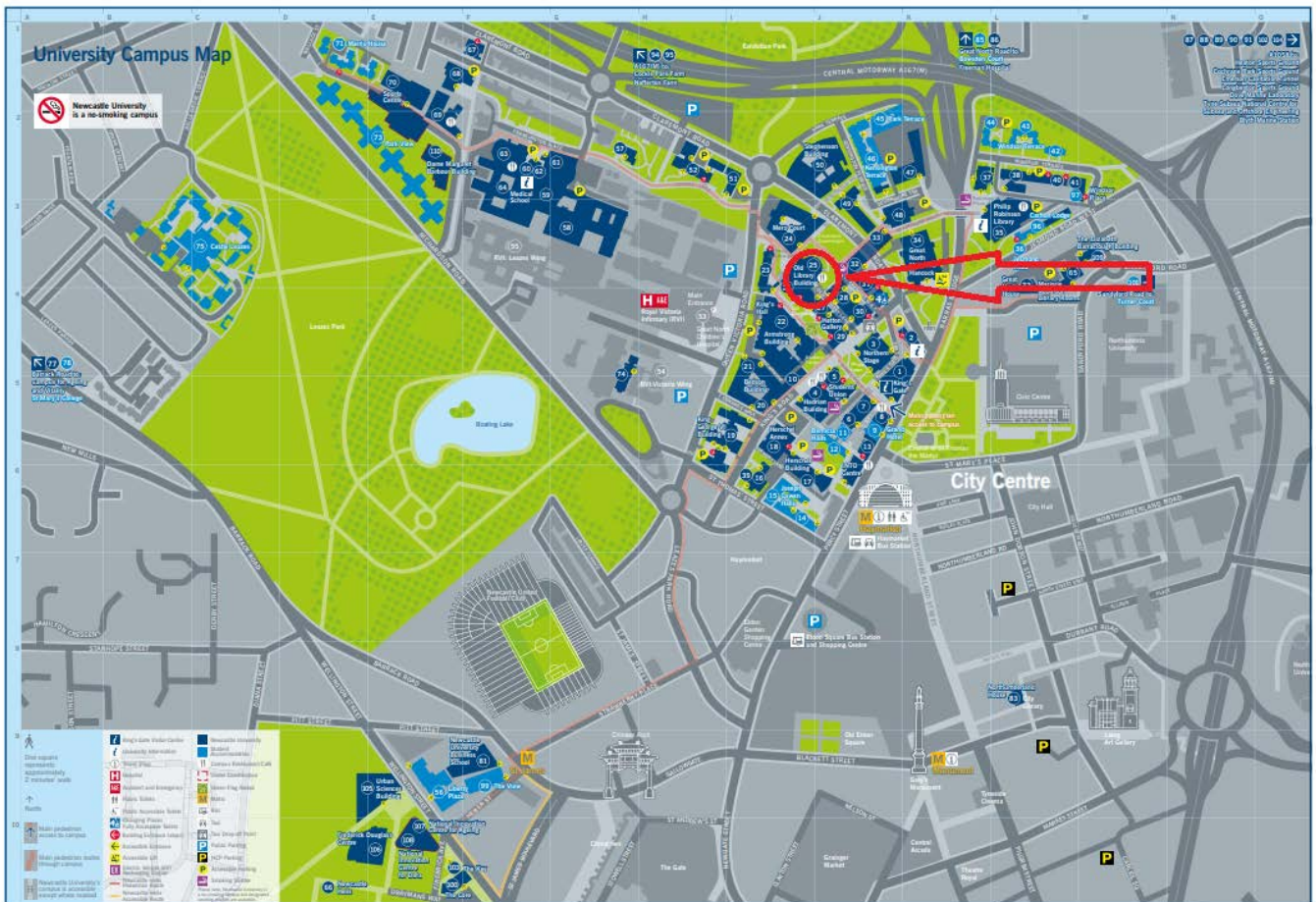
### Internet Access

UK delegates should use the Eduroam wireless internet service. For details on using Eduroam please see:

<http://www.its.qmul.ac.uk/network-services/Eduroam.html>

Please note that although we will attempt to resolve any technical issues people have with the wireless networks we may need to refer you back to your home institutions for technical support in the case of the Eduroam network.

Map – Red circle indicates location of Old Library building



## How to get to Newcastle University

### By train

**Newcastle Central Station** is on the **East Coast Mainline** and just a 15 minute walk to campus. Reach us from London in under 3 hours, and regular direct rail services run from many UK cities.

### By Metro

Newcastle University is connected to the city's suburbs, airport, the coast, and a range of museums and sight-seeing opportunities by the **Tyne and Wear Metro** light rail service.

**Haymarket** is the closest station to the University campus and trains run frequently during the day, starting at around 5.45 and ending at around 23.30. Travel is quick, inexpensive, and even cheaper with a Student Metro pass.

Visit the Nexus website to find [maps of the Metro system](#) and [Metro timetables](#).

### By car

It's easy to reach us by **car**, particularly if you're travelling along the **A1 (M)**.

Our postcode is **NE1 7RU** if you're using a sat-nav or route planner. **Park and ride** is the best option for drivers, as the University doesn't offer parking on campus.

### By bus

Reach us by **Megabus** or **National Express** and enjoy a scenic journey that's fuss-free and wallet-friendly. Reach us from London or Glasgow for under £10.

Newcastle is well-connected to all the culture, countryside, and nightlife the north east has to offer via our local Tyne and Wear bus service. For more information visit the [Nexus bus website](#) to find [bus timetables](#) and [bus ticket fares](#).

### By plane

**Newcastle Airport** is located about seven miles north west of University campus and hosts regular flights to domestic and international airports. Domestic flights are quick and affordable.

The University is connected to the airport by **Metro** line, and it's just a cheap, quick, and fuss-free 25 minute journey from the airport to campus.

**Buses from the airport entrance run to Newcastle city centre** regularly, and you can plan this in advance through the [Traveline website](#).

### Parking

We are **unable to offer parking on campus**. However, we do have a limited number of spaces for visitors with mobility issues.

If you have reserved on-campus parking (via a member of staff) or are arriving by taxi, use the entrance on Claremont Road. The taxi drop-off point is on King's Road.

### City centre car parks

There are **many car parks in walking distance** of campus. The nearest [NCP car parks](#) are:

- John Dobson Street (NE1 8HL)
- New Bridge Street (NE1 8AB)
- Carliol Square (NE1 6UL)

## Programme of Butler meeting 2020

9:30 – 10:20	Registration and coffee	
10:20 – 10:30	Opening conference (Dr Marloes Peeters and Eileen Yu)	
10:30 – 11:15	Plenary 1: Prof Jin Xuan (Chair in Low Carbon Processes at Loughborough University)	
11:20 – 13:00	Parallel session 1 General electrochemistry and catalysis	Parallel session 2: Bioelectrochemistry
11:20 – 11:55	Plenary lecture Dr Mohamed Mamlouk (Newcastle University)	Plenary lecture Dr Damion Corrigan (University of Strathclyde)
11:55 – 12:10	Wenchuan Zhang (Newcastle University)	Dahlia Eldosoky (University of Edinburgh)
12:10 – 12:25	Xingxing Gu (Northumbria University)	Oluwatosin Obata (Newcastle University)
12:25 – 12:40	Franziska Boessl (University of Edinburgh)	Courtenay Patterson (University of Glasgow)
12:40 – 12:55	Athanasios Stergiou (University of Glasgow)	Perrine Lasserre (University of Strathclyde)
12:55 – 14:00	Lunch and poster session	
14:00 – 15:00	Parallel session 1: General electrochemistry and catalysis	Parallel session 2: Bioelectrochemistry
14:00 – 14:15	Yuchen Wang (Northumbria University)	Fiona Moore (University of Edinburgh)
14:15 – 14:30	Zhiming Feng (Newcastle University)	Paul Williamson (University of Strathclyde)
14:30 – 14:45	Matt Leech (University of Greenwich)	Yanan Wu (Newcastle University)
14:45 – 15:00	Da Li (Newcastle University)	Stuart Hannah (University of Strathclyde)
15:00 – 15:20	Coffee break	
15:20 – 16:05	Plenary 2: Dr Alison Parkin (University of York)	
16:05 – 16:20	Awards and closing	

## **Keynote 1: Towards digital transformation of electrochemical processes**

*Prof Jin Xuan – Loughborough University, Department of Chemical Engineering*

**Bio:** Professor Jin Xuan is a Personal Chair in Low Carbon Processes at Loughborough University since September 2019. He has published 100+ journal papers (including 4 Invited Articles, 2 ‘ESI 1% Highly Cited Papers’, 2 ‘Top 25 Hottest Article’ and 1 ‘Most Cited Article’), 50+ conference papers and granted 9 patents. Dr Xuan has led/co-led £3M+ research grants, as the PI for the EPSRC First Grant on solar energy (EP/R012164/1), the PI for the ETP grant for Energy Storage Optimisation (PJ0179ES), the PI of Royal Society Research Grant (RSG\R1\180162) and Newton Advanced Fellowship (NAF\R1\180146) and the work package leader for the £1.8M EPSRC low carbon fuel project (EP/N009924/1). He also leads the UK team in the Global Innovation Initiative (GII) project for CO<sub>2</sub>-to-fuel microreactors with Yale University, Shell, and Chinese Academy of Sciences. He is the winner of 2015 Scottish Energy News Researcher of The Year Award (Energy Storage & Materials) and held the Royal Society Kan Tong Po International Fellowship.

**Abstract:** The future development of novel energy generation and materials manufacturing relies on radical innovations in electrochemical processes with highly embedded functionality, integration and multi-physics interactions. Conventional model, design and control strategies are unlikely to meet the ever increasing complexity and uncertainty associated with future electrochemical processes. There is a high desire to develop digital twin solutions, i.e., the creation of living cyber platforms that update and evolve as the physical processes change, in order to unlock possibilities for maximising the resources utilisation, value creation, and lifetime sustainability.

In this talk I will introduce our recent development and deployment of AI-based computer simulation program which allows car manufacturers to virtually test hydrogen fuel cell engines instead of building expensive prototypes. The virtual digital platform is based on comprehensive 3D multiphase multiscale simulations, being further enhanced by an AI-enabled data-driven framework. Since developed, the digital platform has been used by several automotive and fuel cell companies to help optimise their fuel cell engines so that the final fuel cell product will be close to design specs and with maximum performance without expensive in-process modification or trial and error. It has significantly sped up the research and development process and reduced costs.

## Keynote 2: Performance and stability of radiation grafted based anion exchange membrane electrolyzers

Dr Mohamed Mamlouk , G. Gupta, K. Scott, Newcastle University

**Bio:** Dr Mohamed Mamlouk is a Senior Lecturer for the School of Engineering at University of Newcastle with an area of expertise in Electrochemical Engineering Science. He has over 16 years of experience in the field of electrochemical engineering, membrane science, and mathematical modelling of electrochemical systems. He holds a MSc in chemical engineering and process control from Newcastle University (2003) and a PhD in Electrochemical Engineering (2008, Newcastle University). In recent years, he has published 50+ papers in the field of energy conversion and storage, fuel cells, electrolyzers and batteries, with over 1700 citations and a h-index of 28. He has been PI and co-I on a number of large EU/UK research projects, worth over >£3M. Conferences he has organised include CARISMA 2017, Electrochem UK 2015, and UKES2019. He has chaired several sessions in (inter)national conferences including 6th International Conference on Fundamentals and Development of Fuel Cells (Toulouse, 2015), Electrochemistry 2015 (Durham), and North East Centre for Energy and Materials Conference (Newcastle, 2019). Furthermore, he is a member of the SCI Electrochemical Technology group of which he is the honorary treasurer. He collaborates closely with industry and has done consultancy work for energy conversion and storage industry, including recent work with NewCell Technologies and Enocell Ltd.

**Abstract:** Hydrogen is a promising renewable fuel and energy storage solution, due to its highly efficient conversion with electricity and good energy density in comparison to most batteries. However, 95 % of the produced hydrogen globally is generated from non-renewable sources and only 5% is generated from electrolysis due to high systems cost [1,2]. Around 50% of the cost of the electrolyser system comes from the precious metal catalyst used in proton exchange membrane system [3,4]. Alkaline anion exchange membrane water electrolyser AAEM-WE allow the use of non-precious metals as: effective electro-catalyst and affordable flow fields and bipolar plates, reducing the cost significantly. It is estimated that replacement of PEM electrolyzers with AAEM electrolyzers could offer a 43% reduction in stack cost [5]. We look here at advantages and limitations of AAEM-WE and their ideal operating range.

Higher performances can be achieved using high alkaline feed concentration but this in turn reduces the lifetime of the system due to degradation of AEM membrane/ionomers. Thus, in order to reduce the degradation and improve the lifetime, low alkaline concentration should be used as feed solution for AEMWE i.e. deionised water. We have shown previously [4] that an electrolyser current density of 100 mA cm<sup>-2</sup> at 1.65V using NiCo<sub>2</sub>O<sub>4</sub> for Oxygen Evolution Reaction (OER) could be achieved at 60 °C [4]. We present here a binary-catalyst based on manganese oxides MnO<sub>x</sub> as very efficient electro-catalyst for OER with Tafel slope of 30 mV dec<sup>-1</sup>. Using low cost LDPE based membranes radiation grafted AEM, a very promising performance electrolyser was obtained of 1.59 V at 100 mA cm<sup>-2</sup> and a current density of 1 A cm<sup>-2</sup> at a potential of 1.78 V in 0.01 M NaOH at 60 °C. The current highest performance reported for Ni based catalysts (2.7 mg cm<sup>-2</sup>) in the AEM system is 500 mA cm<sup>-2</sup> at a potential of 1.9 V in 1% K<sub>2</sub>CO<sub>3</sub> solution (pH 10-11) at 60 °C [6,7] which is ca. a third of the current density reported here. This performance is comparable to PEM electrolyzers performances with the reported operating voltage is in the range of 1.6-1.7 V [8] at 1 A cm<sup>-2</sup>. However, the long-term stability of these systems remains a challenge.

**Keywords:** AEM, water electrolysis, alkaline, non-precious metal catalyst,

### References:

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### Keynote 3: The use of microelectrode sensors for the sensitive and selective measurement of nucleic acid and protein biomarkers

Dr Damion K Corrigan, University of Strathclyde, Department of Biomedical Engineering

**Bio:** Dr Corrigan's areas of particular interest include: electrochemical impedance spectroscopy (EIS), microfabricated sensor system and microelectrode sensor arrays for multi target diagnostic assays. He has been awarded a Longitude Prize Discovery Award in recognition of his "Microplate" project, which is a rapid antibiotic susceptibility testing device he is currently working on. He joined Strathclyde in 2016 as a Lecturer and Chancellor's Fellow in Health Technologies. His background is in sensing and analytical chemistry, having obtained a PhD in Bioanalytical Chemistry from Cranfield University followed by periods of postdoctoral research at Southampton and Edinburgh universities. He has expertise in electrochemical sensing in harsh and extreme environments. His work is often highly interdisciplinary and involves multiple industrial partners, including GSK, Astra Zeneca, and the National Nuclear Laboratory.

**Abstract:** Compared to macroelectrodes, microelectrodes have well known analytical advantages which include: reduced  $iR$  drop, enhanced signal to noise, relative insensitivity to convection and simple quantitation of response. A microelectrode can be defined as an electrode with at least one dimension similar in scale to the diffusion length. In practice this critical dimension is around 50  $\mu\text{m}$ . Whilst many studies report biosensor development with "micro" or small scale electrodes, very few operate at or under the scale where true microelectrode performance is observed. In our lab, we have been investigating the use of microelectrodes ( $<50 \mu\text{m}$ ) for the detection of clinically relevant biomarkers. We have discovered that microelectrode surface functionalisation has additional considerations which need to be made compared to macroelectrode functionalisation [1]. We have also developed microelectrode based assays for MRSA [2], the inflammatory cytokine IL-6 [3] and RNA sequences from TB [4]. This presentation will explore the nature of the microelectrode response when functionalised with thiolated self-assembled monolayers and how this differs from the well-established macroelectrode response. Understanding the behaviour of biologically functionalised microelectrodes is critical to developing sensors which exploit their inherent electroanalytical advantages.

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#### **Keynote 4: Title to be confirmed**

*Dr Alison Parkin, University of York*

**Bio:** Alison grew up in a small village in East Yorkshire, and went to South Hunsley School, which is to the west of Hull. In 2000 she went to the University of Oxford, studying for an MChem degree. In her fourth year project she worked for Prof Fraser Armstrong and became interested in how electricity can be harnessed to drive chemical reactions, particularly in Biology, as will be shown by this talk. Alison stayed in the Armstrong research group to carry out a PhD from 2004-2008. Alison was then awarded a 4-year Merton College Junior Research Fellowship in Chemistry at Oxford. In 2012, Alison moved to the University of York as the Anniversary Research Lecturer in Chemistry and was made a Senior Lecturer by the University in 2017. She has been very happy setting up her research group and teaching in the Department. Alison was awarded a 2013 Biochemistry Society Young Investigator award, and this year was awarded two Royal Society of Chemistry prizes, the Parsons Medal in Electrochemistry and the Edward Frankland Fellowship in Inorganic Chemistry.

## Plenary session 1: General electrochemistry and catalysis (morning programme)

### O1: Magnesium Borohydride based Coordination Solid electrolytes for Mg ion battery

Wenchuan Zhang, Paul Christensen and Mohamed Mamlouk

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**Keywords:** Magnesium Borohydride, Diamine, Solid Electrolyte

Magnesium ion batteries (MIBs), a metal ion battery, have received increased attention recently, due to their great potential to replace the current state-of-the Lithium ion batteries (LIBs) in the current battery market and apply in the field of electric vehicles, portable devices and grid energy storage. However, there is currently no suitable electrolyte for  $\text{Mg}^{2+}$  electrolyte, due to instability of solvents during charging and discharging. This research explores solvent-free solid electrolyte to overcome solvent stability or passivation issues. To achieve a good ionic conductivity at room temperature when using solid electrolyte, however, remains a challenge.

$\text{Mg}(\text{en})_1(\text{BH}_4)_2$  (en: Ethylenediamine) has been reported to have good conductivity of  $6 \times 10^{-2} \text{ mS cm}^{-1}$  at the temperature of  $70^\circ\text{C}$  and conductivity of  $\text{Mg}^{2+}$  [1]. In this study,  $\text{Mg}(\text{en})_1(\text{BH}_4)_2$  has been shown to be promising electrolyte with coulombic efficiency  $>80\%$  for Mg stripping and plating and good reversibility at  $50^\circ\text{C}$  for 50 cycles.

Additionally, two other ligands namely: the dimethylethylenediamine (DMEDA) and diethylethylenediamine (DEEDA) were tested.  $\text{Mg}(\text{DMEDA})_1(\text{BH}_4)_2$  has shown good ionic conductivity by  $6.1 \times 10^{-1} \text{ mS cm}^{-1}$  at  $70^\circ\text{C}$  which is higher than  $\text{Mg}(\text{en})_1(\text{BH}_4)_2$ . However, the reversibility of Mg stripping and plating in  $\text{Mg}(\text{DMEDA})_1(\text{BH}_4)_2$  was poor with coulombic efficiency of ca. 30%. Similarly,  $\text{Mg}(\text{DEEDA})_1(\text{BH}_4)_2$  did showed poorer ionic conductivity than two other studied electrolytes with very poor reversibility for Mg stripping and plating.

### O2: Porous, conductive carbon materials contributes to high-performance lithium-sulphur batteries

Xingxing Gu, Xiaoteng Liu

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**Abstract:** Lithium–sulphur (Li–S) batteries are receiving much attention as a potential replacement for Li-ion batteries due to their high energy density ( $2600 \text{ Wh}\cdot\text{kg}^{-1}$ ), and also their abundant and environmentally benign active materials<sup>1-2</sup>. However, the commercialization of Li–S batteries has been hampered by numerous barriers (shown in Fig. 1), including the severe dissolution and shuttle effect of the lithium polysulphides, large volume expansion (up to 80%) of the sulphur cathode as well as the insulating nature of sulphur and its discharge product  $\text{Li}_2\text{S}_2/\text{Li}_2\text{S}_2$ <sup>3-5</sup>.

In order to overcome these obstacles, various strategies such as modifying the cathode, protecting the lithium anode and developing new electrolytes and separators have been proposed. Among them, the conductive confinement of sulphur and polysulphides in a conductive porous carbon host is considered to be the most promising strategy. Therefore, in recent years, Dr. Gu has developed various kind of porous and conductive carbon materials to fabricate the high-performance Li-S batteries.

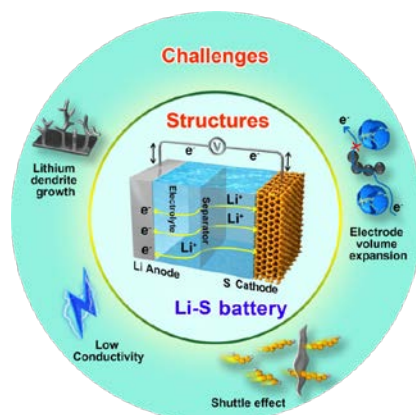


Fig. 1 The challenges of the Li-S batteries

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### O3: Piezo-electrocatalysis – is that a thing?

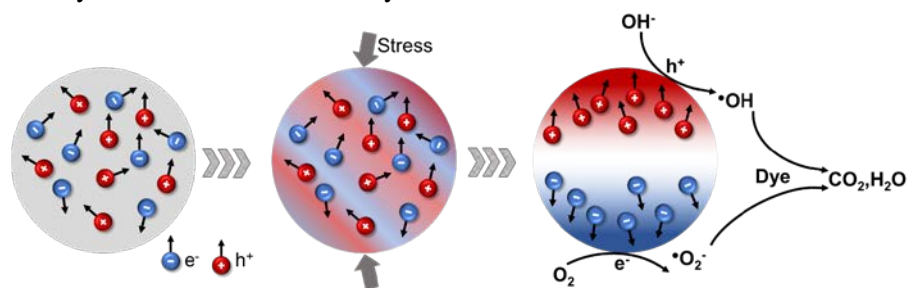
Franziska Boessl, Ignacio Tudela

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**Abstract:** Piezo-electrocatalysis is an emerging new research area that has the potential to convert redundant mechanical vibrations in industrial systems into useful electrocatalytic processes<sup>1-6</sup>. Charges within the piezo-electrocatalyst are polarised due to mechanical stress, leading to enhanced redox reactions on the piezo-electrocatalyst surface. Due to the conversion of mechanical into chemical energy, this new area of research has the potential to create new opportunities to design miniaturised electrochemical devices.

So far, piezo-electrocatalysis has been mainly investigated as a new green technology for dye wastewater treatment<sup>1-5</sup>. However, its promising potential has not been fully exploited as several factors influencing the piezo-electrocatalytic process are still not clear. In this work, we discuss some of the existing issues in the area of piezo-electrocatalysis and how research could benefit from existing knowledge and common practice from other research areas such as general piezoelectricity, sonochemistry and sonoelectrochemistry.



**Fig. 1** Example of redox mechanisms in piezo-electrocatalysis.

#### References

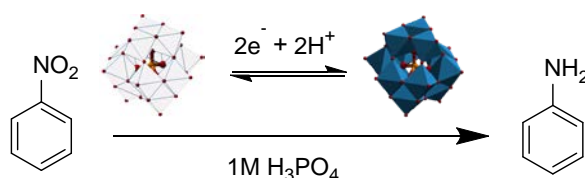
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### O4: Catalytic electroreduction of Nitroarenes

Athanasios D. Stergiou, Mark D. Symes

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**Abstract:** Nitroarenes are key ingredients in the synthesis of many industrial fine chemicals including pharmaceuticals and agrochemicals.<sup>1</sup> Aniline is one of the most important nitroarenes in industry and is currently synthesized by hydrogenation of nitrobenzene.<sup>2</sup> Direct electro-reduction of nitrobenzene to aniline leads to a mixture of products which has been previously demonstrated.<sup>3</sup> The utilization of mediators for the reduction of nitroarenes is not a new research area. MacDonald et al. used polyoxometalate (POM) mediators to effectively reduce a variety of nitroarenes in stoichiometric amounts where the POM proved to be recyclable.<sup>1</sup> Herein, applying the same principles and using another POM, we effectively reduced nitrobenzene for the first time using the mediator in only catalytic amounts (Scheme 1). We assess the performance using bulk electrolysis in a custom made, typical electrochemical cell. Characterization of the reaction product using NMR, shows 100% conversion to aniline together with excellent yields.



*Scheme 1: Reduction of Nitrobenzene to Aniline.*

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## Plenary session 2: Bioelectrochemistry (morning programme)

### 01: Electrochemically driven synthesis of amorphous calcium phosphate films

***D. Eldosoky,<sup>a</sup> J. P. Elliott,<sup>a</sup> I. Schmäser,<sup>a,b</sup> A. R. Mount<sup>a</sup> and F. Nudelman<sup>a</sup>***

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b) School of Engineering, Scottish Microelectronics Centre, King's Buildings, University of Edinburgh, Edinburgh, EH9 3JJ

Bio-composite materials, such as bone and dentin, are highly organised architectures comprised of an organic and inorganic component that vary in ratio depending on the material.<sup>1-4</sup> These scaffolds have remained challenging to mimic synthetically, considering that in nature, the production involves intricate biological pathways.<sup>4,5</sup> Regardless, these materials share the trait of hierarchical organisation, and an interesting feature that also remains common amongst these bio-materials is the high mineral content contained in both. In our study, we utilise chronoamperometry to control mineral deposition, such that this technique could be used for target remineralisation of bone and dentin.

To investigate how chronoamperometry controls the formation of calcium phosphate films, we are studying how the applied potential promotes the migration of calcium and phosphate ions using track-etch membranes that are placed inside a double-diffusion set-up. Through this, an enhanced local supersaturation is created to enable calcium phosphate crystallisation. This talk will present preliminary results, from *in situ* monitoring via Electrochemical Impedance Spectroscopy, to characterise the crystallisation process resulting from the effects of a potential. The increased rate of crystallisation hints to faster migration of ions through the membrane pores, which is consistent with subsequent Scanning Electron Microscopy and Transmission Electron Microscopy analysis showing the membrane pores and surfaces being filled with calcium phosphate crystals.

#### References

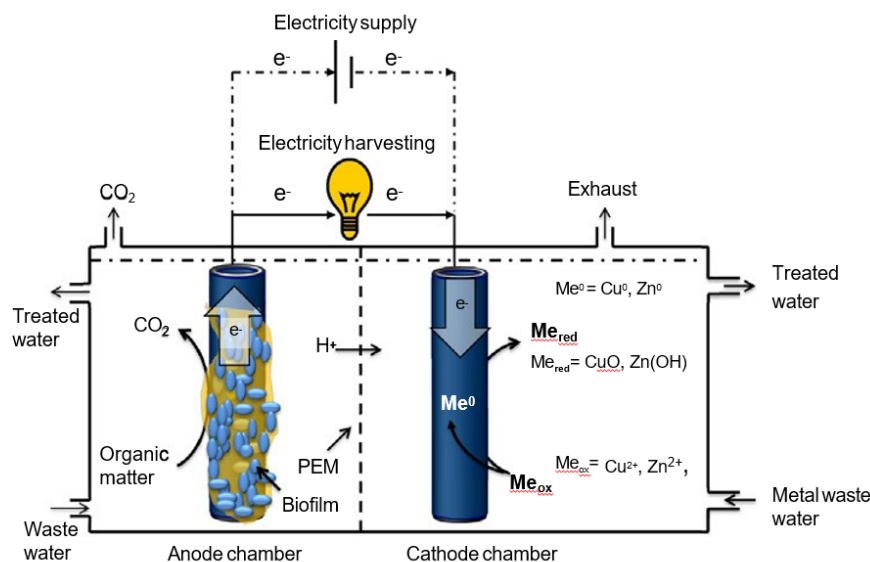
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## O2: Two stage metal recovery using bioelectrochemical systems.

*Oluwatosin Obata and Eileen Yu*

Department of Chemical Engineering, Newcastle University, Newcastle.

**Abstract:** Contamination of natural resources by metals constitute significant health risks to the environment as well as human health. However, waste streams containing substantial amounts of metals could be an important resource for the recovery of valuable metallic elements. Recovery of metallic ions via bioelectrochemical systems have been shown as an inexpensive and sustainable way for metals recovery<sup>1</sup>. However, in some industrial processes such as steel manufacturing, metal contaminations occur in the solid state where metal like iron is contaminated with zinc, therefore rendering the iron unusable. In this case it is important to separate the metals prior to recovery. As such a 2-stage recovery process involving bioleaching (using suitable iron oxidizers) and bioelectrochemical recovery would be applied. In our current study, we utilised iron oxidizers (obtained from environmental sample) as the inoculum for bioleaching of blast furnace slurry (as received) from steel making process. The experimental procedure of pH 1.8, temp 30°C, 10% pulp density and 250 rpm<sup>2</sup> were adopted resulting in efficient leaching of the metals over 10 days. Leached metallic solution obtained was fed into the cathode chamber of an MFC while coke oven effluent (obtained from steel manufacturing) was used as the anolyte. Power density obtained by the MFC reached 8 W/m<sup>3</sup> while metal recovery of up to 90% was achieved within 10 days. These results highlight the possibility of inexpensive solid metal separation and recovery from industrial processes, with possible re-use of the recovered metals.



**Fig. 1** Bioelectrochemical systems for metal recovery and removal adapted from<sup>3</sup>.

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<sup>2</sup> Haragobinda Srichandan and others, 'Bioleaching Approach for Extraction of Metal Values from Secondary Solid Wastes: A Critical Review', *Hydrometallurgy*, 189 (2019), 105122 <<https://doi.org/10.1016/j.hydromet.2019.105122>>.

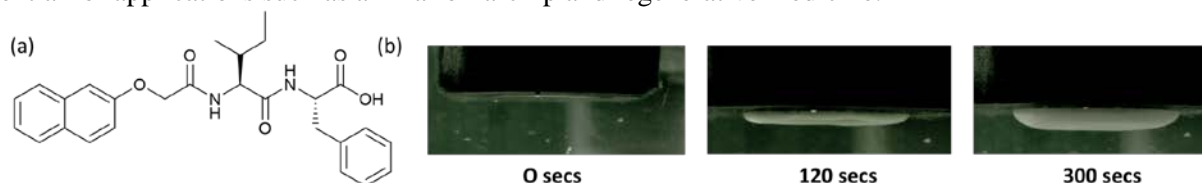
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### O3: Fabrication of gels by electrochemical methods

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**Abstract:** Hydrogels are a class of materials that can be fabricated by imposing electrical inputs to gelator solutions.<sup>1,2</sup> 2NapIF (Figure 1a) is a pH responsive gelator that self-assembles to form a self-supporting hydrogel at low pH. To induce gelation, we employ a three-electrode system to locally reduce the pH of the medium at the surface of the working electrode. This allows spatiotemporal control over the gelation process by regulating the applied current imposed on the system. Using this method, we create hydrogel films of varying thickness deposited on the surface of the working electrode (Figure 1b). In order to locally reduce the pH at the working electrode surface, hydroquinone was added to the medium as it undergoes electrochemical oxidation to benzoquinone at certain potentials, liberating hydrogen atoms.<sup>3</sup> This local reduction in pH at the electrode surface causes a change in the gelators structure from the deprotonated form to the protonated form, inducing self-assembly and gelation specifically at the working electrode surface. In order to monitor gel growth a camera was set up in view of the working electrode and recorded using the software imageJ. Bulk rheology was used to analyse the mechanical properties of the resulting hydrogel films. The electrodeposition of hydrogels is a versatile and highly useful technique for the fabrication of soft materials and has shown potential for applications such as animal-on-a-chip and regenerative medicine.<sup>2</sup>



**Fig. 1** (a) Chemical structure of 2NapIF (b) Gel growth on the surface of the working electrode with time [hydroquinone] = 10 mg/mL. In all cases [2NapIF] = 7 mg/mL and [NaCl] = 0.1 M.

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### O4: Enhanced antibiotic stewardship through development of new point of care technologies

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Lily Riordan, Katherine Duncan, R David Dunphy, Paul Murray, Christopher McCormick and Damion Corrigan.

**Abstract:** Antimicrobial resistance (AMR) is currently responsible for a patient death every 45 seconds. One of the major drivers for its emergence and spread is the overuse and misuse of antibiotics. Without any change to current practices, AMR could cost the life of one person every 3 seconds by 2050 (AMR-Review.org). Every antibiotic prescription represents an opportunity for AMR to emerge. In addition, each new antimicrobial drug has an approximate cost of \$200 million from early discovery to commercialisation and these medicines are likely to generate very low returns on investment for the pharmaceutical sector. Current antibiotic susceptibility testing (AST) methods were first described in the 1970s. They were suitable then since the rate of antibiotic discovery was outpacing emergence of resistance. However, these approaches no longer meet contemporary needs, with a long time-to-result, low sensitivity and expensive procedures. Efficient and reliable point-of-care diagnostics are now a key priority for appropriately treating infections. Point-of-care biosensors allow diagnosis to take place on a more suitable time scale and to move closer to the patient. Such tests will provide efficiency savings to already overburdened healthcare providers, give better clinical outcomes for patients and critically, enhance antibiotic stewardship to help mitigate the spread of AMR. We have been working on a low cost electrochemical approach to point of care antibiotic susceptibility testing<sup>1</sup>. Our approach involves producing electrodes modified with gel-antibiotic deposits and recording electrochemical bacterial growth curves in order to assess antibiotic susceptibility. Our aim in this project is to further develop the format of the test and enlarge the pool of pathogen strains tested to encompass all or most of the ESKAPE pathogens. This poster will describe our AST methodology and show details of recent engineering advancements we have made to produce a more generic and reliable version of the test, show results with *E.coli* and drug resistant *E.coli* strains and as part of a larger combined research effort (Strathclyde Chancellors Fellows CDT on AMR), showcase advances we have made with colleagues from microbiology and electrical engineering aimed at combining optical and electrochemical modalities for real time monitoring of biofilm formation.

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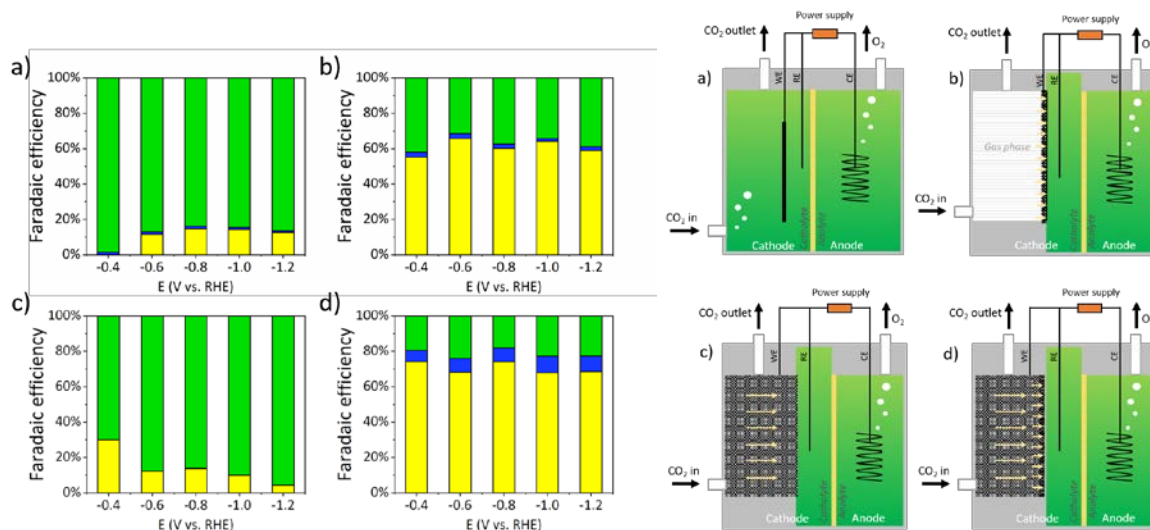
O5: Mass transfer effect to CO<sub>2</sub> electrochemical reduction reaction through different gas diffusion mechanism

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**Abstract:** Electrochemical reduction of CO<sub>2</sub> to low carbon organic compounds has been considered as a promising method to mitigate the greenhouse effect and produce useful energy carrying chemicals. However, some hurdles still existing which prohibit this concept become commercialized, including poor catalytic properties, high overpotential and low mass transfer of reactants. Enhancing mass transfer behaviour is one of the solutions to enhance CO<sub>2</sub> reduction reaction properties. Therefore, in this work, we introduced fuel cell type gas diffusion electrode device (GDE) for CO<sub>2</sub> reduction reaction to solve the CO<sub>2</sub> mass transfer problem. We assembled catalysts based on carbon paper and graphene aerogel as gas diffusion layer (GDL) with CO<sub>2</sub> gas diffused to catalyst layer (CL) which can significantly enhance the CO<sub>2</sub> supply. To realise our idea and design, 3D-printer was introduced in this project to achieve the device print and assembly. The electrochemical results present the newly designed GDE device can solve the mass transfer problem of reactants (CO<sub>2</sub>) and enhanced the Faradaic efficiency (FE) of formic 3-folds higher than conventional two chamber cell. With potential of -1.2V (vs. RHE) in 1M KOH, formic FE of 80% and current density of 125 mA/cm<sup>2</sup>.



**Fig. 1** Faradaic efficiency profiles of a) H-type b) CP GDE c) GA GDE d) GACP GDE device using Cu<sub>2</sub>O catalyst in 1M KOH electrolyte of CO<sub>2</sub>RR with products including CO (yellow, bottom), Formic (blue, middle), H<sub>2</sub> (green, top).

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## O6: Fundamental studies of poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) based anion exchange membranes prepared via Friedel-Crafts reaction with high ionic conductivity

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**Abstract:** Poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) based membranes for application in anion exchange membrane water electrolysis [1], were prepared via Friedel-Crafts reaction by using  $\text{SnCl}_4$  as catalyst, as well as 1,3,5 trioxane and chlorotrimethylsilane as environmentally friendly chloromethylating reagents, to replace toxic and carcinogenic chloromethyl methyl ether [2]. The effect of chloromethylation conditions were fully investigated and it was found that the medium degree of substitution, e.g. 40%, and low degree of crosslinking, e.g. 1.8%, provided promising chloromethylated polymer. The optimum reaction condition were when  $n_{\text{PPO}}:n_{\text{Tri}}:n_{\text{SnCl}_4}$  equalled 1:0.5:0.2, reacted at 35 °C for 5 h, and the concentration of PPO was 1.7 wt%. The crosslinking structure was newly proposed and analysed, as well as revised formulas for degree of chloromethylation and crosslinking. Homogeneous animation method demonstrated better amination results than the heterogamous one. Besides, the intrinsic properties of PPO based membrane were tested. The stress of break and elongation of quaternized PPO (QPPO) is above 12 MPa, and the ionic conductivity can reach over  $0.133 \text{ S cm}^{-1}$  at 80 °C and 100% relative humidity. PPO was also employed as both membranes and ionomers in electrolyser test and showed an area specific resistance of  $104 \text{ m}\Omega \text{ cm}^{-2}$ . The current density was  $814 \text{ mA cm}^{-2}$  when the cell voltage was 1.75 V in 0.1M NaOH solution at 40 °C.

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## O7: Electrosynthesis: A Modern Methodology with Unlimited Potential

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Organic Electrosynthesis has long been a useful synthetic tool, with examples as early as 1832.<sup>1,2</sup> However, the need for specialist equipment, alongside issues with reproducibility have hindered method development. With the introduction of commercially available standardised equipment such as the IKA Electrasyn 2.0 and its accessories, electrosynthesis has experienced a resurgence in interest,<sup>3</sup> and has proven to be a valuable and versatile tool in both small- and industrial-scale chemical synthesis.<sup>3</sup>

Previously, we reported the use of electrogenerated aryloxy radicals, formed *via* anodic oxidation of aromatic carboxylic acids, to synthesise a library of functionalised phthalides under mild and green conditions.<sup>4</sup> We have now extended this methodology towards the use of aliphatic carboxylic co-acids as a cheap and readily available source of alkyl radicals, which readily undergo an unusual metal-free  $sp^3$ - $sp^3$  cross coupling in solution.<sup>5</sup>

The exceptional versatility of our new methodology is exemplified through our successful synthesis of functionalised lactones, which are of great interest due to their fungicidal, antibiotic, and anti-cancer properties, under mild and ecologically friendly conditions.<sup>5</sup> Moreover, using similar conditions, it has been possible to synthesise a library of functionalised orthoesters, which are highly versatile and reactive compounds with uses in medicinal and material chemistry, thus circumventing the use of traditional methods which rely on dangerous and toxic reagents, and have no scope for derivatisation.<sup>6</sup> A summary of our recent results will be presented.



**Fig. 1** Electrosynthesis of pharmaceutically desirable small molecules using the IKA Electrasyn

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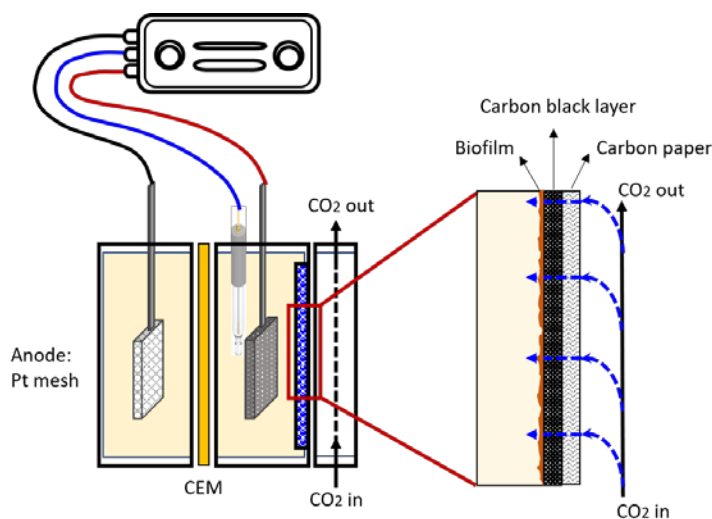
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**Abstract:** Microbial electrosynthesis cells (MES) in which microorganisms used electrons from electrode to reduce carbon dioxide to multi-carbon products, was regarded as a promising technology for carbon capture with production of value-added chemicals and fuels when renewable energies like solar or wind was used <sup>1,2</sup>. To improve performance, gas diffusion electrode (GDE) electrode coated with polyaniline (PANI) was adopted with previously acetogens enriched effluent as inoculum. The MES was operated in batch mode at a poised potential of -1000 mV vs Ag/AgCl for over 60 days. Quick start of bioproduction in reactors equipped with PANI-modified GDEs was found in 10 days, and the maximum concentration of acetate reached 8 g L<sup>-1</sup> with highest production rate of 850 mg L<sup>-1</sup> d<sup>-1</sup> which were 2.7 and 1.9 times higher than that without PANI coating. Besides, butyrate was also produced, reaching 245 mg L<sup>-1</sup> d<sup>-1</sup>. Electrochemical analysis indicated PANI coating electrode had a lower charge transfer resistance that benefited to interfacial electron transfer. In addition, PANI modified GDE promoted hydrogen evolution reaction due to the acid dopants which also played an important role for acetate synthesis. PANI modification also increased the hydrophilicity of GDE which would favour microorganism attachment and growth. Taken together, these results demonstrated the PANI modified GDE is a promising strategy to increase bioproductivity of CO<sub>2</sub> conversion.



**Fig. 1** Schematic diagrams of MES system with GED electrode.

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## Plenary session 2: Bioelectrochemistry (afternoon) programme)

### O5: Development of a High Sensitivity Electrochemical Glucose Sensor

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#### **Abstract:**

Treating diabetes costs the UK approximately £14 billion per year and a major proportion of this is from complications, including cardiovascular disease and renal damage. The health outcomes and severity of these complications are considerably worse if the disease is poorly managed. Monitoring is primarily performed by measuring glucose concentration in blood via finger-prick tests, which doesn't provide continuous monitoring and can have low patient compliance. Monitoring glucose levels non-invasively and continuously would be hugely beneficial to patients' health outcomes.

Sweat is a potential alternative biological fluid to blood, which is easily accessible and has already shown its potential for detecting a range of analytes. Glucose concentrations in sweat are lower than in blood and sweat volume is limited, therefore sensitive measuring techniques are important to provide meaningful results. Electrochemical measurements using on-chip nanoelectrode arrays provide many applicable benefits including higher sensitivities, lower limits of detection and less analyte depletion.

Reference electrodes are an essential component in an electrochemical sensing 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 need for a defined electrolyte solution. As a result, the stability and lifetime of such electrodes can be significantly affected, representing a major obstacle in device development.

This presentation will focus on the development of a high sensitivity electrochemical method for glucose sensing, incorporating platinum nanoelectrodes with miniaturised silver/silver chloride reference electrodes fabricated at the University of Edinburgh's microfabrication facilities.

### O6: Enhancing electrochemical biosensor performance for improved sensitivity, stability and combined sensing and release

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**Abstract:** Electrochemical biosensors have tremendous potential in the diagnosis and treatment of infectious and chronic diseases. These technologies offer promise of low cost, sensitive and specific testing for nucleic acid sequences and protein biomarkers in serum and whole blood. Many of the current approaches rely on the use of a self-assembled monolayer (SAM) to functionalise the electrode surface with a biological receptor. Disadvantages with the SAM approach to biosensing include, signal instability with the layer decomposing over time, difficulties with label-free measurements because of interfacial changes in the SAM itself affecting the signal and finally, biofouling due to the surface adsorption of proteins and lipids from e.g. the blood sample.

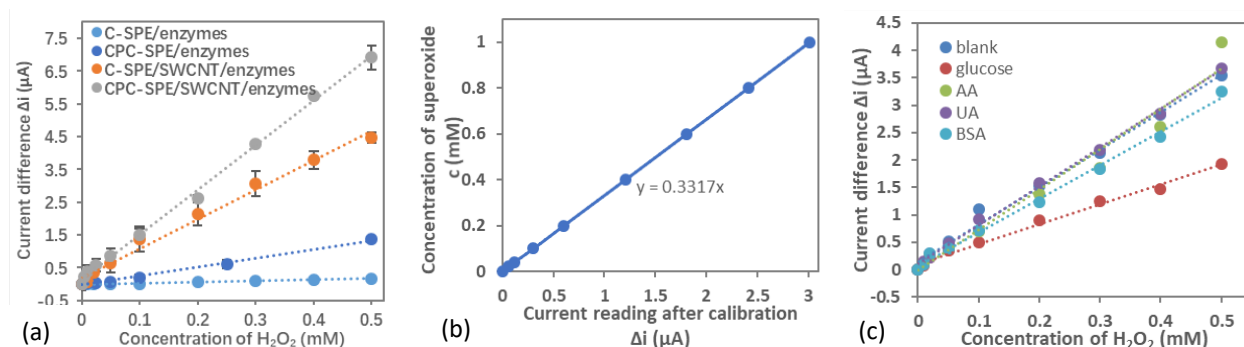
This project is looking to improve the sensitivity and stability of SAM layers. To achieve this, we are focusing on DNA sequence detection and designing and testing a range of novel DNA probe structures, including linear redox tagged probes, hairpin probes and tetrahedron inspired probes. These DNA nanotechnologies are currently being evaluated for sensitivity, specificity and stability for detection of a common antibiotic resistance gene and compared with established approaches. In addition, with collaborators from Aalto University, we are currently testing a "DNA zipper" which is a structure which once immobilised to a surface can be made to open and close based on changes to the local pH. This is our first attempt at a structure which can be used to combine on chip sensing with release. The talk will report our latest findings in these areas of enhanced DNA sensing and selective open and closing of DNA origami structures bound to an electrode surface.

## O7: Development of electrochemical biosensor using for superoxide detection

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The incidence of aging diseases caused by the high-level concentration of reactive oxygen species, especially superoxide in the human body increased by years and reached its highest level nowadays, and the biosensor, as an analytical device, is one of the most common ways to detect superoxide.<sup>1</sup> However, the existing biosensor is lack of easy-operation, high-sensitivity, and quick-response. This project is aimed to develop the sensitivity and the stability of the biosensor and apply to clinic tests. In this work, the biosensor based on the oxidation reaction of hydrogen peroxidase produced by superoxide was established by immobilizing superoxide dismutase layer-by-layer,<sup>2,3</sup> and four systems, carbon screen printed electrode (C-SPE)/enzymes, Cobalt-Phthalocyanine (CPC)-SPE/enzymes, C-SPE/single-wall carbon nanotube (SWCNT)/enzymes and CPC-SPE/SWCNT/enzymes were compared (Figure 1a). The result shows the system C-SPE/SWCNT/enzymes has the highest accuracy, high sensitivity 8.5 uA/mM and low limit of detection at 23 uM. Figure 1b shows the calibration curve for superoxide detection after current calibration of the biosensor. The interferences test (Figure 1c) shows that uric acid (UA) and ascorbic acid (AA) have ignorable effect on the detection, the effect of bovine serum albumin (BSA) is a few negative towards oxidation signal and the effect of glucose is more significant.



**Fig. 1** (a) The relationship between concentration of  $H_2O_2$  and current of four systems; (b) Calibration curve for biosensor based on oxidation reaction of  $H_2O_2$ ; (c) Interferences test results for biosensor based on oxidation reaction.  $\Delta i$  is the current difference between detection reading in sample and the solution without  $H_2O_2$  or superoxide.

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## O8: Rapid antibiotic susceptibility testing using low-cost, commercially available screen-printed electrodes

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Antimicrobial resistance (AMR) is an issue of worldwide concern and utmost importance, requiring urgent, international action. Better diagnostic tests must be developed as part of that action in order to tackle AMR effectively; ultimately improving antibiotic stewardship and reducing the emergence of antibiotic resistant organisms.

This talk presents the development of a rapid, antibiotic susceptibility test using a low-cost, commercially available screen-printed electrode sensor modified with an agarose-based hydrogel deposit<sup>1</sup>. The sensor is able to electrochemically monitor bacterial growth using techniques including electrochemical impedance spectroscopy (EIS) and differential pulse voltammetry (DPV), providing a novel method of measuring susceptibility. To test the device, susceptible and drug resistant *Staphylococcus aureus* strains were deposited onto gel-modified electrodes containing clinically relevant antibiotics to establish growth profiles for each bacterial strain and ascertain the influence of the antibiotic on bacterial growth. Using EIS, we found that the susceptible *S. aureus* strain is able to grow on electrodes featuring no antibiotic, but is hindered when antibiotic is present. Conversely, methicillin-resistant *S. aureus* (MRSA – the drug resistant strain) still displays growth in the presence of antibiotics at clinically relevant concentrations. Electrochemical growth profiles were supported by traditional microbiological streak-plate techniques to confirm either growth or lack of growth using different bacterial strains/antibiotic combinations. Results show rapid growth profiles for antibiotic susceptibility, with differences between susceptible and drug-resistant strains of *S. aureus* being produced in < 45 minutes, a significant improvement on the current gold standard of at least ~ 1-2 days for a positive result.

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## Poster session 1: General electrochemistry and catalysis

*P G1: Effect of Temperature on Corrosion of Carbon Steel under Mineral Wool Insulation*

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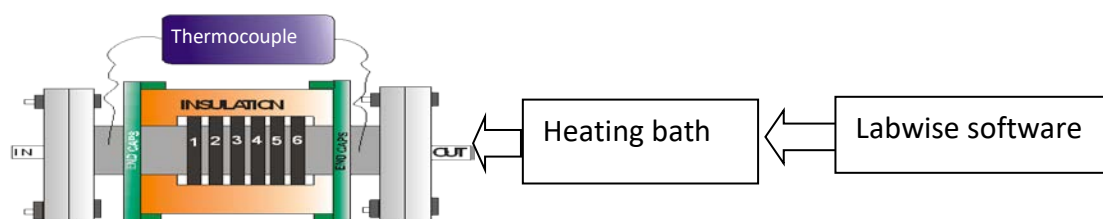
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**Abstract:** Corrosion under insulation is the degradation of metals due to ingress of water and contaminants such as chlorides through the insulation to the surface of the metal, resulting in anodic dissolution of the metal. Due to the fact that the insulation covers the underlying metal and hides it from view, degradation may proceed unnoticed till final consequences are severe<sup>1</sup>.

Although the effect of temperature on corrosion of carbon steel has been studied for uninsulated cases<sup>2</sup>, studies quantifying the effect of inner pipe temperatures and external contaminants such as chlorides in insulated materials are quite scarce. In industry, the high temperature of fluids flowing through insulated metals as well as contaminants may have significant impact on the corrosion rate. Therefore, it is important to assess and quantify these factors to better understand their contribution to corrosion of insulated assets. This study is targeted at designing a corrosion under insulation test system to study the effect of temperature on the corrosion of carbon steel rings under mineral wool insulated environment.

The experimental set up is shown in figure 1. This consists of carbon steel rings numbered 1-6, separated by polytetrafluoroethylene spacers, held together in a stack and kept in position by two end flanges made of stainless steel. Mineral wool pre-soaked in 1% sodium chloride for about 48 hours is wrapped around the rings and held in place by aluminium tape. Hot silicone oil from a temperature programmed bath is circulated through the internal sections of the pipe at different temperatures of 60 - 130 °C.

Results indicated an increase in corrosion rate and reaches a peak at 80 °C, which decreases on further increase in temperature. This may be attributed to rapid drying out of the insulation as the temperature is increased. Also, significant variability among replicate trials was observed, which may be attributed to the non-uniform distribution of the test solution across the insulation or inconsistent contact of the rings with the insulation. This provides a basis for failure analysis and technical guidance for material selection.



**Fig. 1** Corrosion under insulation set up

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Carbon materials, including single-walled carbon nanotubes (SWNT) and highly oriented pyrolytic graphite (HOPG), have gained great interest in electrochemistry. The advantages of carbon electrodes include chemical stability, biocompatibility, low background currents and good electrical conductivity. With the increasing importance of carbon electrode materials for biological and energy applications, the systematic investigation is mandatory.

SWNT networks, with different densities, are firstly investigated using microscale capillary electrochemical (EC) methods. Pristine high density (HD) SWNT networks are shown to exhibit more facile electron transfer (ET) for dopamine (DA) electro-oxidation and are less susceptible to blocking by reaction products when compared to low density (LD) SWNT networks. Acid treatment of SWNT networks results in an enhancement in electrode kinetics and a reduction in their susceptibility to surface fouling.[1]

A comparison is then made between SWNT electrodes, with different densities, and a commercial screen-printed carbon electrode (SPCE) for trace voltammetric analysis in complex aqueous media (ferrocenecarboxylic acid (FcCOOH) in polyethylene glycol (PEG) or albumin) encountered in electrochemical based diagnostic devices. We find SWNT electrodes outperform SPCEs, with both cyclic voltammetry and differential pulse voltammetry (DPV) providing a lower detection limit (1 nM FcCOOH) by up to three orders of magnitude. Toward practical devices, a patterned SWNT network 3-electrode chip has been designed and demonstrated to have impressive detection sensitivity (nM) in a challenging media (PEG). The simplicity and practicality of the design widens the potential applications of these ultra-sensitive diagnostic tools.

A Ni(OH)<sub>2</sub> nanoparticles (NPs) modified SWNT network is employed for methanol oxidation reaction (MOR) and ethanol oxidation reaction (EOR), showing improved ET processes with ~2.8 kA g<sup>-1</sup> for MOR and ~3.7 kA g<sup>-1</sup> for EOR, which are much higher than recent reports using other nanostructured catalysts. This study demonstrates the capability of electrochemistry for the tailored synthesis of Ni(OH)<sub>2</sub> nanostructures for electrocatalytic applications, and a powerful, but simple, combinatorial approach for quick activity screening.[2]

There is great interest in finding and developing new, efficient and more active electrocatalytic materials. A fundamental understanding of the structure-activity of “blistered” highly oriented pyrolytic graphite (HOPG), produced by electro-oxidation in HClO<sub>4</sub>, is obtained using a powerful tool of scanning EC cell microscopy (SECCM) coupled with multi-microscopy techniques. The disordered sp<sup>2</sup> carbon structure of the blister catalyzes the electro-oxidation of hydrazine compared to the basal surface. In this study, a potential sweep at each pixel of pre-defined scan area is recorded, providing potentiodynamic data with high resolution. The measurements reveal significantly enhanced activity at blisters compared to the basal planes, with a significant cathodic shift of the onset potential for hydrazine oxidation reaction. The improved EC activity of the hollow structure of blistered graphite could be explained by the increased adsorption of protonated hydrazine at oxygenated defect sites, the ease of ion-solvent intercalation/de-intercalation, and the susceptibility to N<sub>2</sub> nanobubble attachment (as a product of the reaction). This study highlights the capability of electrochemistry to tailor the surface structure of graphite and presents a new electrocatalyst for hydrazine electro-oxidation.[3]

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## P G3: Tailoring the electrochemical properties of 2D-hBN via physical linear defects: physicochemical, computational and electrochemical characterisation

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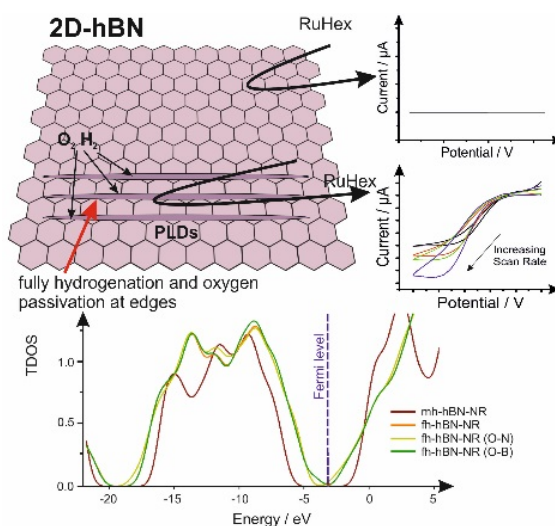
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### Abstract:

Monolayer hexagonal-boron nitride films (2D-hBN) are typically reported within the literature to be electrochemically inactive due to their considerable band gap (ca. 5.2–5.8 eV). It is demonstrated herein that introducing physical linear defects (PLDs) upon the basal plane surface of 2D-hBN gives rise to electrochemically useful signatures. The reason for this transformation from insulator to semiconductor (inferred from physicochemical and computational characterisation) is likely due to full hydrogenation and oxygen passivation of the boron and/or nitrogen at edge sites, resulting in a decrease in the band gap (from ca. 6.11 to 2.36/2.84 eV; theoretical calculated values, for the fully hydrogenated oxygen passivation at the N or B respectively). The 2D-hBN films are shown to be tailored through the introduction of PLDs, with the electrochemical behaviour dependent directly upon the surface coverage of edge plane-sites/defects, which is correlated with electrochemical performance towards electrochemical probes (RuHex and Fe<sup>2+/3+</sup>) and the Hydrogen Evolution Reaction. This manuscript de-convolutes, for the first time, the fundamental electron transfer properties of 2D-hBN, demonstrating that through implementation of PLDs, one can beneficially tailor the electrochemical properties of this nanomaterial.



**Fig. 1** CVD 2D-hBN is tailored through the introduction of PLDs showing the rise of electrochemically useful signatures towards RuHex, Fe<sup>2+/3+</sup> and the HER. Physicochemical/computational characterisation are explored, inferring that the reason is likely due to a full hydrogenation and oxygen passivation of the boron and/or nitrogen at the edge sites (band gap decreases from ca. 6.11 to 2.36/2.84 eV).

## P G4: Lignin Electrolysis in a Continuous Flow Anion Exchange Membrane Reactor

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Human development since the industrial age has been achieved based largely on our ability to exploit fossil fuels as an energy source. Beyond the issue of these fuels being finite, their consumption has had a devastating effect on the global climate<sup>[1-5]</sup>. Hydrogen has long been regarded as one of the most promising “carbon neutral” energy storage solutions. In theory hydrogen can be obtained in almost 100 % purity via electrolysis of water, which after combustion, reforms water.<sup>[6]</sup> The main disadvantage to water electrolysis is its high electrical energy requirement. Due to this, research into alternative feedstock solutions such as lignin have garnered interest. The research reported herein regards the optimisation and testing of an anion exchange membrane electrolyser, for lignin electrolysis. Two novel anion exchange membranes are assessed on their applicability through direct comparison with a commercial product.

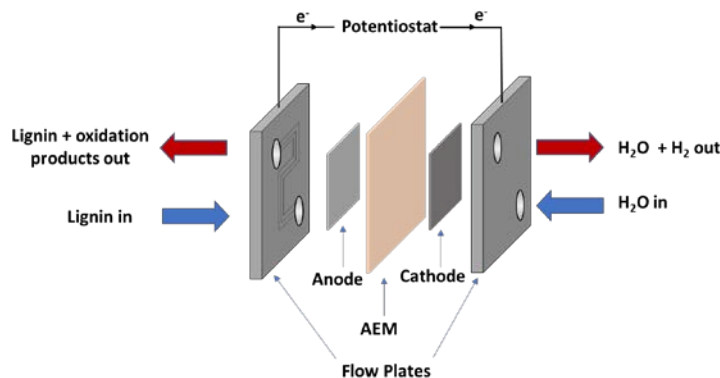


Fig. 1 Schematic of AEM electrolyser

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## P G5: The effect of temperature on nickel deposition from sulfamate baths

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**Abstract:** Nickel sulfamate electrolytes are widely used for industrial applications such as electroforming where low stress deposits are required. Due to the lack of adequate systematic data on the sulfamate electrolyte, knowledge about Ni deposition mechanism in this electrolyte has been gleaned from studies in the Watts bath. However, evidence has shown that the stress of deposits from the Watts bath is an order of a magnitude higher than those from a sulfamate bath, which would indicate different mechanisms.

In addition, there remains considerable confusion about the role of boric acid ( $\text{H}_3\text{BO}_3$ ) in the electrolyte. Boric acid was initially added as a buffer to stabilise surface pH but current efficiency data which show values approaching 100% indicates little hydrogen evolution occurs meaning there is little change in surface pH and boric acid acts under the guise of a buffer. In this regard, the role of boric acid is crucial and not fully understood. The aim of this work is to develop a deeper understanding of nickel deposition from sulfamate baths, which will allow for process optimisation and better process control.

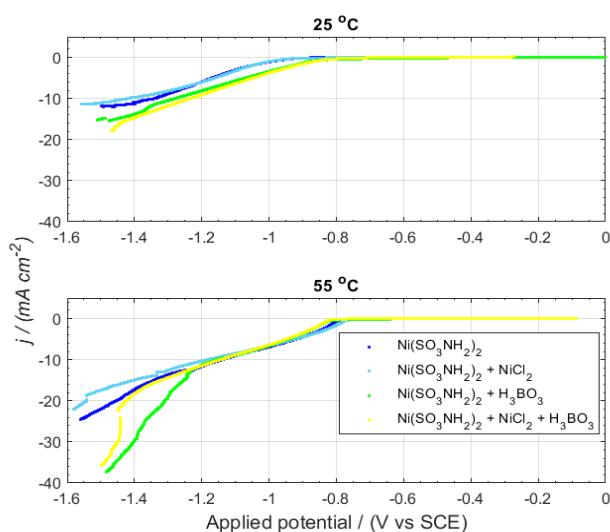


Figure 1: Linear sweep voltammograms at 25 and 55 °C

efficiency using gravimetric and electrochemical quartz microbalance (EQCM) techniques.

The temperature-dependent effects of  $\text{NiCl}_2$  and  $\text{H}_3\text{BO}_3$  on nickel electrodeposition in a sulfamate electrolyte have been systematically investigated by a combinatorial study with the use of linear sweep voltammetry on a stainless steel disc working electrode under quiescent conditions. The counter and reference electrodes used were platinum wire and saturated calomel electrode (SCE), respectively. Our results (Figure 1) show that  $\text{H}_3\text{BO}_3$  has a depolarising effect on the bath at low temperatures; and as temperature was increased, the effect is less pronounced.  $\text{NiCl}_2$  was observed to polarise the cathode, an effect which increases with current density and temperature. At high temperatures of 55 °C,  $\text{H}_3\text{BO}_3$  and  $\text{NiCl}_2$  appear to have little effect on the cathodic polarisation curves. Because nickel and proton reduction occur simultaneously, it cannot be concluded which reduction potential is shifted by the presence of  $\text{H}_3\text{BO}_3$ . Further information will be obtained from a systematic investigation of nickel current

## P G6: CAPACITIVE DEIONISATION FOR PHOSPHATE RECOVERY

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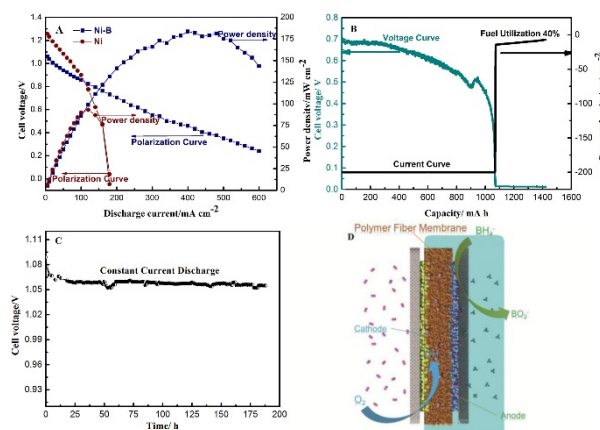
Phosphate run-off originating from agricultural sources has negative effects on crop yield and is often a contributor to eutrophication. Moreover, natural supplies of phosphate rich rocks-phosphorites- will eventually run out. Current methods for the recovery and recycling of phosphates rely heavily on chemical precipitation e.g. struvite formation ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ) and biological remediation e.g. EBPR (Enhanced Biological Phosphorus Removal). Electrochemical methods such as reverse osmosis and electrodialysis are primarily used for the desalination of brackish water. These techniques are highly energy intensive. Capacitive Deionisation (CDI) is one such electrochemical technique, that has a lower energy requirement, but its use on the industrial scale has yet to be realised. The research herein aims to successfully combine the recovery of phosphate from brackish water using a CDI cell. A diindolylurea based receptor has been shown to have strong affinity for phosphate over other anions. Following the successful synthesis and modification of the receptor. The receptor can be grafted to the electrode surface and tested for its suitability within a CDI system.

## P G7: Ultrafine amorphous transition metal boride with high catalytic activity for direct borohydride fuel cells *Sai Li, Eileen H. Yu*

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Direct borohydride fuel cells (DBFCs) is a special type of alkaline liquid fuel cell that uses easy-to-handle liquid borohydride as fuel for easy storage and transportation, and plays a role in portable applications. However, the borohydride reaction is very complicated. There are parallel side reactions, hydrogen evolution reactions, illegal Faraday reactions such as  $\text{BH}_4^-$  hydrolysis reaction, and the cost of the battery is also a factor restricting its commercialization.

we synthesized a series of amorphous transition metal borides (Co-B, Ni-B, Co-W-B), and used them for the first time in borohydride fuel cell anode catalysts, which have a very good catalytic effect on borohydride oxidation. At room temperature, Ni-B<sup>[1]</sup>, Co-B, and Co-W-B were used as DBFC anode catalysts, and their single cell power densities reached  $180 \text{ mW} \cdot \text{cm}^{-2}$ ,  $96 \text{ mW} \cdot \text{cm}^{-2}$  and  $101 \text{ mW} \cdot \text{cm}^{-2}$ , respectively. At the same time, PdNi<sub>x</sub>-B catalyst (PdNi<sub>x</sub>-B / CNT) supported by carbon nanotubes was synthesized. The catalyst showed good power performance ( $127.3 \text{ mW} \cdot \text{cm}^{-2}$ ) and a higher fuel efficiency of 55.7%<sup>[2]</sup>. We also synthesized an amorphous-wrapped crystal structured catalyst (Au @ Co-B), which discharge power density is reached  $85 \text{ mW} \cdot \text{cm}^{-2}$ . Hope this part of working readers will be interested.



**Fig. 1** Performances of the DBFC using Ni-B and pure Ni anode: (A) The discharge performances of Ni-B and pure Ni catalyzed DBFCs, (B) fuel utilization of the DBFC, (C) durability of the DBFC at ambient atmosphere, current density is  $20 \text{ mA cm}^{-2}$ , (D) the schematic diagram of DBFC-PFM.

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## P G8: Fluorine Substitution on Benzene Affects Rigidity, Conductivity, Electrochromic and Optical Properties of 1,4-Bis(2-(3,4-Ethyleneedioxythiophene)-Benzene)

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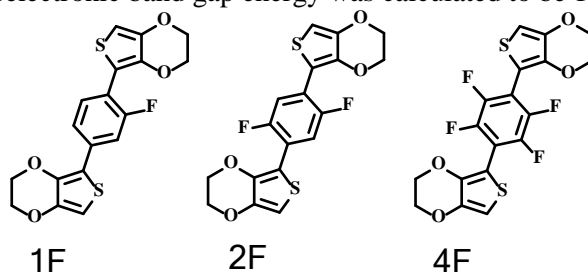
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**Abstract:** Fluorine substitution in organic semiconductors materials has led to significant enhancement in device performance for organic solar cell and field effect transistors. Due to intramolecular interactions that fluorine can have with hydrogen or other neighboring atoms, in addition to its electron withdrawing nature.<sup>1,2</sup> In this work,<sup>3</sup> we study the effect of different degree fluorination on the benzene ring of 1,4-bis(2-(3,4-ethylenedioxythiophene)-benzene (BEDOT-B)<sup>4</sup> on the monomers intermolecular interaction, polymers redox states, electrochromic and optical properties. The degree of fluorination was found to have a significant effect on the planarity of the monomeric structure. The most twisted monomer is with 4F atoms substituted on benzene. Poly 4F was found to have the lowest oxidation potential due to its twisted nature. The optoelectronic band gap energy was calculated to be 1.9 eV for 1F and 2F.



**Chart 1.** The chemical structure of the fluorine substituted monomers.

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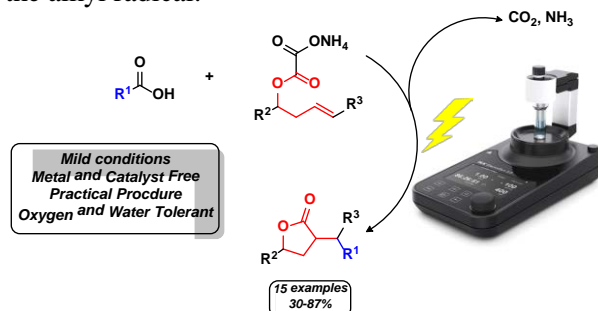
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## P G9: Electrosynthesis: A Green, Economical and Safe Method to Activate Small Organic Molecules

Alessia Petti and Kevin Lam

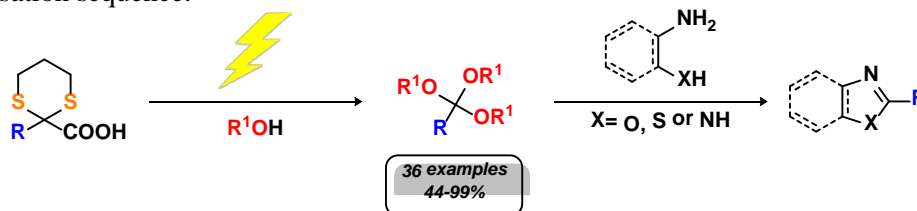
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**Abstract:** In this poster, we show how electrochemistry could activate carboxylic acids through a radical or cationic pathway for the synthesis of pharmaceutically relevant fragments. Platinum electrodes trigger the mono-electronic oxidation of carboxylic acids and lead to the formation of an unstable carboxyl radical that decomposes quickly into CO<sub>2</sub> and an alkyl radical. At the same time, a highly reactive oxycarbonyl radical is generated from an hemioxalate salt. Functionalised  $\gamma$ -butyrolactones were synthesized through *exo*-cyclisation of the oxycarbonyl radical with an alkene, followed by *sp*<sup>3</sup>-*sp*<sup>3</sup> capture of the alkyl radical.<sup>1</sup>



**Fig. 1.** Electrosynthesis of  $\gamma$ -butyrolactones

In contrast to platinum electrodes, carbon electrodes are promoting a dielectronic oxidation of carboxylic acids and generate an alkyl carbocation instead of a radical. We took advantage of this simple mechanistic switch to prepare functionalised orthoesters, a class of compounds that is known to be highly difficult to prepare using classical chemical methods. In our newly developed methodology, a dithiane carboxylic group is introduced as a new orthoester/ester synthon. Finally, we have shown that the electrolyses could be used to prepare bioactive heterocycles by a one-pot electrolysis-condensation sequence.<sup>2</sup>



**Fig. 2.** Electrosynthesis and condensation of orthoesters

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#### ***P G10: Nanostructured Tungsten-Tungsten Oxide Electrochemical Energy Storage***

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**Abstract:** Nanostructured tungsten oxides (WO<sub>3</sub>) are an interesting class of material due to the facile intercalation of protons within their crystal structure coupled to electronic reduction to form tungsten bronzes.<sup>1</sup> This pseudocapacitive behaviour is of particular interest for energy storage applications. Previous investigations aiming to exploit this material property have so far only been realised using active nanoparticles supported within a conductive matrix acting as the current collector, which decreases the specific capacity of the resulting device.<sup>2</sup> Growth of nanostructured WO<sub>3</sub> directly on a tungsten metal substrate has been investigated within the context of photocatalysis but has never been applied to energy storage.<sup>3-6</sup> This project aims to investigate tungsten foil-supported WO<sub>3</sub> materials for energy storage by screening various methods of producing nanostructured WO<sub>3</sub>/W electrodes. This configuration removes the necessity for a conductive support matrix, with tungsten foil acting as the current collector directly connected to the active material. This innovative design would simplify the manufacturing process leading to a cheaper, more efficient and easily scalable device.

A parametric study of metal substrate seed layer preparation, hydrothermal synthesis, and annealing conditions is presented together with electrochemical performance data, with a view to correlating synthesis conditions with microstructure and performance properties.

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## Poster session 2: Bioelectrochemistry

### P B1: Quantification of Chloride Inhibition of O<sub>2</sub> Reduction in Bilirubin Oxidases using Protein Film Electrochemistry

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**Abstract:** Bilirubin oxidases (BODs), a sub-group of multicopper oxidases (MCOs), have four Cu<sup>+</sup>/Cu<sup>2+</sup> ions responsible for O<sub>2</sub> reduction coupled with the oxidation of bilirubin to biliverdin.<sup>1</sup> BODs display a high activity and stability at neutral pH, a high tolerance towards chloride anions and other chelators, and for some species, a high thermal tolerance.<sup>2</sup> They have gained more attention in numerous biotechnological applications under mild pH conditions, especially biofuel cells, bilirubin sensors, depollution, decolorization of dyes and pulp bleaching.<sup>1,2</sup> The presence of halides in the environment inhibit the activity of MCOs and their halide tolerance varies enormously.<sup>3</sup> In this present study, the inhibition modes of chlorides in BODs from fungus *Magnaporthe oryzae* (*MoBOD*) and bacterium *Bacillus pumilus* (*BpBOD*) were determined by quantitative measurements of O<sub>2</sub> inhibition obtained using protein film electrochemistry at pH 7 and at different temperatures (4°C, 25°C, 50°C and 60°C). The data from the electrochemical assays were fit to four inhibition models: competitive, uncompetitive, mixed and hyperbolic. Chlorides inhibition of O<sub>2</sub>-reduction reaction catalysed by *MoBOD* and *BpBOD* were determined by the lowest reduced chi-squared values and an *F*-test analysis. Under continuous electrocatalysis, an electrode modified with *BpBOD* exhibits higher thermal stability and has a higher chloride tolerance than a *MoBOD* modified electrode, which is consistent with the literatures findings as bacterial MCOs generally have higher tolerance towards chlorides compared to fungal MCOs.<sup>4,5</sup>

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### P B2: Development of an implantable 'chemotrode' for localisation of seizure foci using in-vivo measurements of neurochemicals

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**Abstract:** When assessing epilepsy patients, localisation of seizure foci can be performed using a combination of patient observation, brain imaging, and implanted EEG electrodes. Neurochemical information is absent from these methods due to the complexity of making the required in-vivo measurements with sufficient temporal and spatial resolution. Enabling these measurements will allow a more accurate localisation of the seizure foci reducing unnecessary damage to the brain, enhanced surgical resection and provide further understanding of brain function during seizures.<sup>1</sup>

The fields of optogenetics and neuroelectrochemistry are active areas of research with work on implantable devices and neurotransmitter detection both being prominent.<sup>2,3</sup> This project ultimately seeks to develop an implantable device to assist with the localisation of epileptic seizures. To realise this aim it is necessary to first make progress with the development of glutamate biosensors and to this end we are developing enzymatic and non-enzymatic approaches. Secondly, development of in-vitro and in-vivo measurements will be necessary and in this area we are both modifying existing electrode arrays and developing post processing techniques for in house arrays. This poster will show our current findings on glutamate biosensor development alongside developments on sensor fabrication.

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### P B3: Electrochemical sensing for continuous monitoring of fetal health

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**Abstract:** During childbirth, oxygen supply to the baby can become restricted. This is known as fetal hypoxia, and can result in severe complications, including brain damage and death. The fetus is monitored during birth to determine whether medical intervention is required to ensure its safe delivery. Current fetal monitoring techniques are slow, inaccurate, and poor at predicting neonatal outcomes. They also require blood to be taken from the fetal scalp, which is an invasive and time-consuming process. Improved systems and techniques are required. Lactate is produced by the body when oxygen levels are low, so it can be used as a marker for fetal hypoxia during childbirth, where it may be better than current techniques at predicting health outcomes for the baby.

This poster will detail plans to develop a microelectrode-based enzymatic lactate sensor for use in a continuous fetal monitoring system. This work aims to exploit the benefits of microelectrode sensors in order to develop an accurate, rapid, and minimally-invasive system for monitoring fetal health using subcutaneous interstitial fluid. The research is at an early stage but the key objectives will be presented along with some preliminary findings.

### P B4: Electrochemical reports on substrate & inhibitor interactions with PceA dehalogenase

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**Abstract:** Tetrachloroethylene (PCE) and trichloroethylene (TCE) were used extensively as dry cleaning, degreasing and industrial solvents - when their toxic effects were realised, they were banned from being utilized further in industry. However, they continue to remain as major contaminants in groundwater.<sup>(1)</sup> Since the chemistry to deal with toxic halo-compounds are inherently difficult, it is envisaged that enzymes will provide specific dehalogenation but under mild conditions.

*Sulfurospirillum multivorans* use organohalides such as PCE and TCE as electron acceptors in their respiratory systems because they possess PceA dehalogenase as a membrane-bound terminal reductase in their organohalide respiration chains.<sup>(2)</sup> PceA dehalogenase is O<sub>2</sub>-sensitive and since protein film electrochemistry (PFE) has proved to be very useful in the mechanistic studies of many O<sub>2</sub>-sensitive enzymes with redox-active centres,<sup>(3,4)</sup> we chose this technique to learn more about the dehalogenase. PceA dehalogenase has been studied with mediators while spotted onto carbon electrodes, but direct exchange with any electrode is yet to be reported. The inability to observe the direct exchange was attributed to the enzyme's large size.<sup>(5)</sup> Since PFE with pyrolytic graphite edge (PGE) electrodes has successfully been utilized to study enzymes which are considerably larger in size (*e.g.*, 91.3 kDa dimeric Hydrogenase-1 from *E. coli* (Hyd-1) and 110kDa dimeric CODH from *Carboxydotherrmus hydrogenoformans* (CODH<sub>Ch</sub>)), it was decided that characterization of PceA dehalogenase (~50.3 kDa) with this method is justified. Here we report the direct communication of the PceA dehalogenase with a PGE electrode in a three-electrode set-up. The suite of techniques under PFE (cyclic voltammetry and chronoamperometry) are used to report on substrate interactions as well as inhibitor interactions with the enzyme.

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## P B5: Bioremediation of Petroleum Hydrocarbons using Sediment Microbial Fuel Cells

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Removal of petroleum hydrocarbons by biological methods such as bioremediation require extensive treatment periods due to insufficient electron acceptors to drive the biodegradation process. Bioanodes of a microbial fuel cell (MFC) could speed up the removal of petroleum hydrocarbons by serving as electron acceptors<sup>1</sup>. Bioanode performance may be enhanced by enrichment using different sources of inoculum<sup>2</sup>. The aim of this research is to develop sediment microbial fuel cell (SMFC) for bioremediation of polycyclic aromatic hydrocarbons (PAHs) found in anaerobic strata of sediment contaminated by crude oil. Cyclic voltammetry was conducted to understand the electron transfer mechanism at the bioanode of the MFCs. Hydrocarbon removal was monitored by Total Organic Carbon (TOC) analysis whilst PAHs concentration was determined using Gas Chromatography-Mass Spectroscopy (GC-MS). TOC removal rates of  $52 \pm 0.2\%$  and  $74 \pm 0.3\%$  for effluent and sludge bioreactors respectively, as shown in Figure 1, highlighted the influence of source of inoculum and co-substrates in the environmental matrix on biofilm performance as well as the removal efficiency of petroleum hydrocarbons. Further results are expected for use of cost effective electrode materials and SMFC configurations for performance optimization.

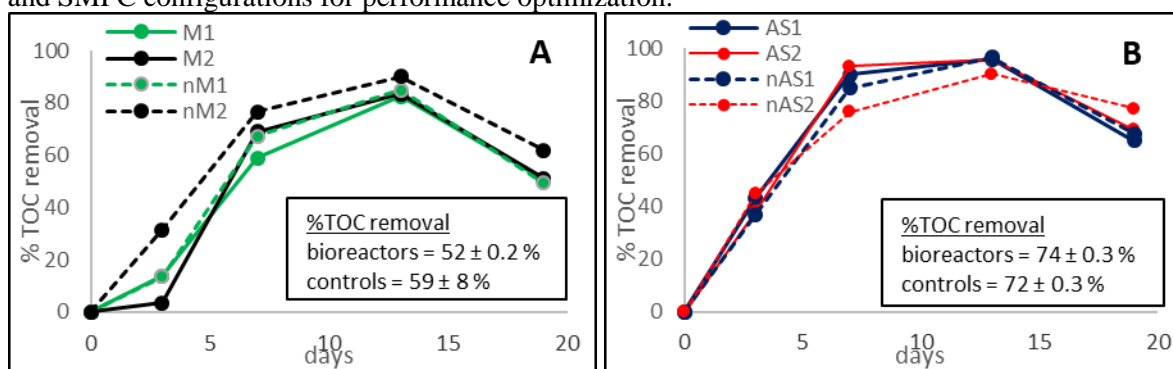


Figure 2. Average Total organic carbon (TOC) during hydrocarbons removal at 30°C in MFCs using inoculum from (A) effluent of pre-existing MFC and (B) activated sludge.

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## P B6: Tuneable Electrochemical Gels for Sensing Applications

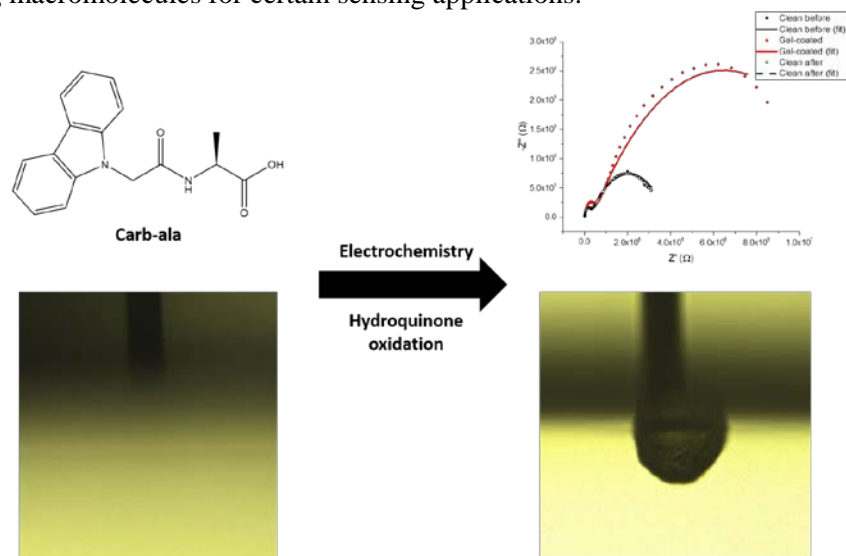
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**Abstract:** Microelectrodes offer enhanced mass transport and high signal-to-noise ratios in comparison to macroscale counterparts.<sup>1</sup> This makes them highly sensitive and ideal for biosensing applications. However, the negative effects of electrode-surface biofouling are also enhanced at this scale. Supramolecular hydrogels, grown directly onto an electrode surface through the electrochemical oxidation of hydroquinone,<sup>2</sup> have proven to be capable of protecting electrode sensors from biofouling materials.<sup>3</sup>

Here, chronoamperometry and chronopotentiometry are used to electrochemically-induce self-assembly of a particular gelator, Carb-Ala,<sup>3</sup> upon Pt disc microelectrodes. The transport of ions through the gel can then be understood using electrical impedance spectroscopy (EIS). With fine-tuned control over electrochemical-gelation, gels can be designed to block out biofouling macromolecules for certain sensing applications.



**Fig. 1** The oxidation of hydroquinone leads induces Carb-Ala self-assembly upon commercial Pt microelectrodes, which can be visualised under a microscope, and can be examined using EIS.

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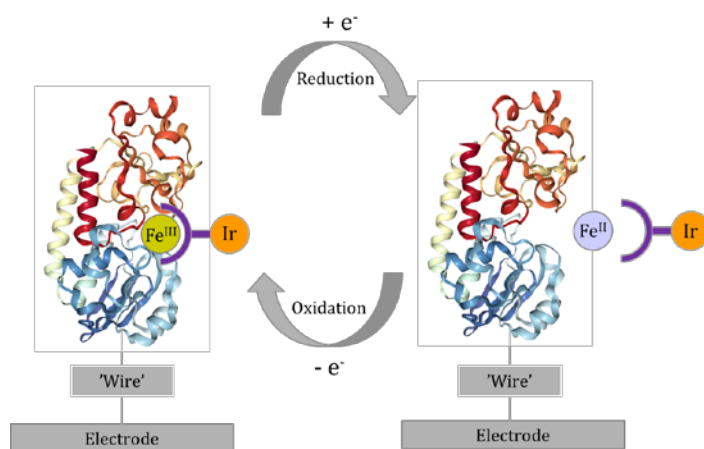
## P B7: Electrochemical switch for immobilised redox-reversible artificial metalloenzymes

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**Abstract:** Artificial metalloenzymes (ArMs) represent an exciting and rapidly expanding field of catalysis, particularly in the area of asymmetric synthesis. The integration of an organometallic catalyst into a protein scaffold, opens the opportunity for carrying out chemical transformations in a biocompatible environment and in an enantioselective manner, where previously racemic mixtures were obtained. An ArM based on a non-covalent interaction between the conjugate of a siderophore bound to an iridium based imine reduction catalyst and a periplasmic binding protein of *C. jejuni*, CeuE has been developed.<sup>1</sup> This has allowed to exploit the iron-siderophore binding mechanism for the recycling of the components through a reversible anchor which is facilitated through reduction of the iron(III) centre. We have identified scope for the immobilisation of the biocatalyst on a conducting surface; not only allowing the possibility for carrying out catalysis in flow but also for triggering the assembly and disassembly of the ArM using electrochemical means (Fig. 1). This will allow for a more efficient recovery of the components and eliminate the need for the use of harsh chemical reducing conditions and formation of associated by-products.



**Fig. 1** An artificial metalloenzyme consisting of a redox-reversible iron-siderophore anchor conjugated to an iridium-based catalyst, immobilised on a conducting surface through a bifunctional linker for electrochemically controlled disassembly and assembly.

Reference: D. J. Raines, J. E. Clarke, E. V. Blagova, E. J. Dodson, K. S. Wilson and A.-K. Duhme-Klair, *Nat. Catal.*, 2018, **1**, 680–688.

## P B8: Development of Fibre-optic pH sensor based on Silver Nanoparticle embedded in Silica Matrix for High temperature/pressure conditions

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**Abstract:** Quantifying hydrogen ion concentration of a solution (or pH) has important applications in different industries such as cosmetics, pharmaceuticals, textile, food and beverage, etc. However, it is challenging to maintain and detect accurately pH under conditions of high temperature and pressure (HTHP). At the moment, there is no reliable instrument that is capable of measuring pH at temperatures exceeding 100 °C in oil wells, or at elevated pressure and in the presence of aggressive chemical species.

Recently, surface plasmon resonance property of gold nanoparticles (Np) have been coated on optical fibres to determine the pH of solutions at different temperatures and pressures<sup>[1], [2]</sup>. The use of silver nanoparticles (AgNp) on fibre optic as a pH sensor in the HTHP environment, however, is yet to be reported. This study focuses on developing a coating of AgNp embedded in silica (SiO<sub>2</sub>) on optical fibres to be used as a pH sensor in the HTHP environment. In order for this system to be useful in harsh environments, it is expected to have excellent optical and adhesive properties, durability and sensitivity.

Silver nanoparticles embedded in silica was prepared by adding tetraethoxysilane (TEOS) to ethanol, water and silver nitrate, and the pH of the silver nitrate solution was adjusted to 2 using nitric acid. The solution was left to evaporate for gel formation. Glass slides were then dipped into the gel and dried in an oven at 300°C where silver ions reduced to form AgNp. Characterization of the coating using UV/Visible spectrophotometer indicated the formation of silver nanoparticles showing an absorbance peak around 400 nm, which is consistent with what is reported in the literature [3], [4].



**Fig. 1** Different layers of Silver nanoparticle/silica coating

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