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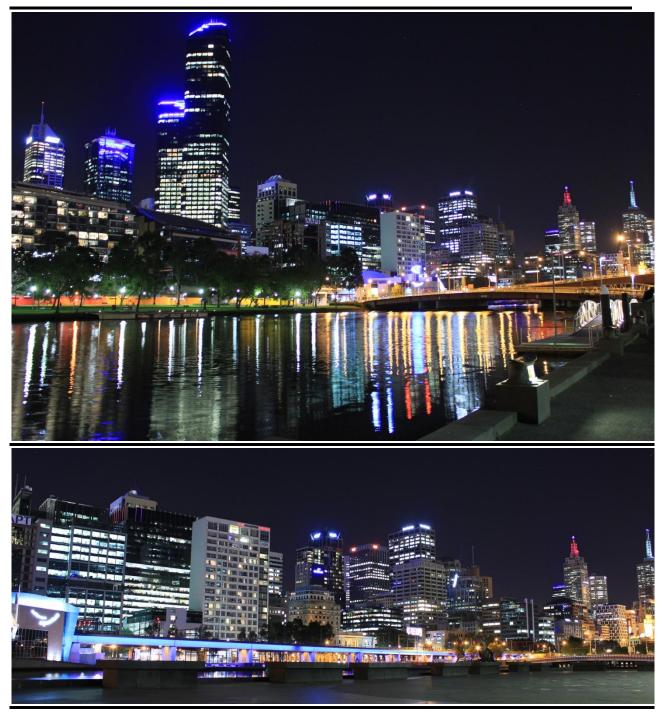
CSIRO Materials Science and Engineering 343 Royal Parade, Parkville VIC 3052, Australia



CSIRO Cutting Edge 2012 Symposium

Welcome to the Symposium on Biological and Chemical Conversion of Renewables to fuels and chemicals

On behalf of the organising committee I have great pleasure in inviting you to the OCE Cutting Edge Symposium to be held at the CSIRO Conference Room, 343 Royal Parade, Parkville Victoria on 13-15th November 2012.



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CSIRO Cutting Edge Symposium Program:

Tuesday 13 November 2012

1230 -1330 Lunch and Registration

Session A

Chair: Matt Wilding

Time	Speaker		Title
13:30 – 13:50	Victoria Haritos	A1	Welcome and scene setting
13:50 – 14:20	David Alonso	A2	Strategies for Catalytic Conversion of Lignocellulosic Biomass to Fuels and Chemicals
14:20 – 14:40	Colin Scott	A3	New opportunities in redox biocatalysis
14:40 – 15:00	Mamad Amin	Α4	The role of ytterbium as a promoter on nickel catalyst activity during dry reforming of biogas
15:00 – 15:30	Coffee Break		

Tuesday 13 November 2012 (cont)

Session B

Time	Speaker		Title
15:30 – 16:00	Doug MacFarlane#	B1	
16:00 – 16:20	Abhijit Shrotri	B2	Continuous process for catalytic conversion of cellulose to sorbitol: A step closer towards Industrial application
16:20 – 16:40	Andrew Smith	B3	Structural Requirements for Lignin Peroxidase Activity: Engineering New Enzymes for Lignin Degradation
16:40 – 17:00	Munish Puri	B4	Nanobiotechnology advances biofuel production: Nanomaterial functionalization for enzyme immobilization with potential application in cellulose hydrolysis

17:00 – 19:00 Poster Session (including drinks and snacks)

Wednesday 14 November 2012

Session C

Chair: Victoria Haritos

Time	Speaker		Title
09:00 – 09:30	Thomas Maschmeyer	C1	From Biomass to Fuels and Chemicals with Hydrothermal Treatments
09:30 – 09:50	Andrew Warden	C2	New computational methods for modelling cellulase activity on cellulose
09:50 – 10:10	Kirk Torr	C3	Improving Enzymatic Saccharification of Pretreated Radiata pine in the New Zealand Lignocellulosic Biofuel Initiative
10:10 – 10:30	Florian Graichen	C4	Sustainable polymer modifiers derived from woody biomass and fatty acids
10.20			

10:30 – 11:00 **Coffee Break**

Session D

Chair: Matthew Hill

Time	Speaker		Title
11:00 – 11:30	Richard Noble	D1	Novel Materials for Gas and Liquid Separations Associated with Bioprocessing
11:30 – 12:00	Robin Rogers	D2	Unique Roles for Ionic Liquids in a Biorefinery: Extraction, Separation, and Processing of Lignin, Cellulose, Hemicellulose, and Chitin
12:00 – 12:20	John Kavanagh	D3	Membranes for Biorefineries
12:20 – 13:30	Lunch		

Wednesday 14 November 2012 (cont)

Session E

Chair: Colin Scott

Time	Speaker		Title
13:30 – 14:00	Atsushi Fukuoka	E1	Depolymerization of cellulose by heterogeneous catalysis
14:00 – 14:30	Jorge Beltramini	E2	Conversion of Lignocellulosic Biomass to Fuels and Chemicals over Heterogeneous Catalysts
14:30 – 14:50	Geoff Dumsday	E3	Microbial biotransformation of 1,8- cineole
14:50 – 15:30	Coffee Break		

Session F

Chair: Akshat Tanksale

Time	Speaker		Title
15:30 – 16:00	Chris Easton	F1	
16:00 – 16:20	Asyraf Kassim	F2	Biodiesel Production from Algae Biomass Cultivated with Carbon Dioxide (CO2) Via Biocatalytic Process
16:20 – 16:40	Matt Hill	F3	Separation, Storage, and Triggered Release of CO ₂
16:40 – 17:00	Aidyn Mouradov	F4	Innovative strategies to convert waste material into biofuel

18:30 – 22:30

Conference Dinner – Melbourne Aquarium



Thursday 15 November 2012

Session G

Chair: Lizhong He

Time	Speaker		Title
09:00 – 09:30	Brian Haynes	G1	Pilot-Plant and Bench-Scale Studies of Hydrothermal Treatment of Biomass
09:30 – 09:50	Warwick Raverty	G2	The Lignocellulose-Based Biorefinery - An Australian Approach
09:50 – 10:10	Craig Wood	G3	New engineered crops for the large scale production of renewable oils
10:10 – 10:30	Kapil Lokare	G4	Valorization of Bio-ethanol: Conversion of Ethanol to Higher-Value Products Using Transition Metal Catalysis
10:30 -	Coffee Dreek		

10:50 Coffee Break

Session H

Chair: Andrew Warden

Time	Speaker		Title
11:00 – 11:30	Michael Crowley	H1	Computational modelling and experimental data showing detailed mechanisms and the barriers to fast conversion of cellulose to sugars
11:30 – 12:00	Milton Hearn	H2	
12:00 – 12:20	Xiao Liang	H3	Thermodynamically Consistent Modelling of the Decomposition of Cellobiose
12:20 – 13:30	Lunch		
13:30	Optional tours of CS	IRO Cla	yton Research Facilities

Oral Abstracts:

A2: Strategies for Catalytic Conversion of Lignocellulosic Biomass to Fuels and Chemicals

David Martin Alonso, James A. Dumesic

Department of Chemical and Biological Engineering, University of Wisconsin – Madison

Diminishing fossil fuel resources and the increasing impact of global climate change have driven research for utilization of lignocellulosic biomass resources as renewable feedstocks of carbon. To become a sustainable alternative, from an economic and technical point of view, the conversion of lignocellulosic biomass to fuels and chemicals requires the effective utilization of the C5 and C6 sugars present in hemi-cellulose and cellulose. Here, we will give special attention to strategies based on the production of high value chemicals and liquid fuels within the same process. We will address how the cellulose fraction, via production of HMF, and the hemicellulose fraction, via production of furfural and furfuryl alcohol, of the lignocellulosic biomass can be converted into levulinic acid. We will consider the advantages and drawbacks of several strategies using different acid catalysts in monophasic and biphasic systems, and organic solvents. The effective management of the acid catalyst used in the process will be critical, as acid catalysts affect negatively downstream reactions. In addition, we will address the conversion of levulinic acid to gamma-valerolactone (GVL) in the presence of organic solvents to minimize separation steps. While a Ru/C catalyst shows initially high activity for hydrogenation of levulinic acid, this catalyst is also active for hydrogenation of the solvent, and it undergoes continuous deactivation versus time on stream. In contrast, a tin-promoted Ru/C catalysts displays stable activity versus time on stream and it shows 100% selectivity for hydrogenation of levulinic acid versus hydrogenation of the solvent. Finally, we will explore the possibility of convert GVL into liquid fuels in an integrated step. First, we will address the decarboxylation of GVL to produce butene over a SiO_2/Al_2O_3 catalyst. The reaction kinetics for this process can be described in terms of the reversible ringopening of GVL to pentenoic acid (PEA), coupled with direct GVL decarboxylation and PEA decarboxylation. Second, the butene produced is oligomerized over a second acid catalyst, Amberlyst-70 to increase the molecular weight of the final product, so it can be used as liquid fuel.

A3: New opportunities in redox biocatalysis

Colin Scott

CSIRO Ecosystem Sciences, Canberra, ACT Australia

Stereospecific redox chemistry is a cornerstone of modern biocatalysis, with applications in fine chemical, pharmaceutical, flavor and odorant synthesis. The enzymes used as catalyst for these chemistries are most often dependent upon nicotinamide (NADH and NADPH) and flavin (FMN and FAD) cofactors. Here, we describe the discovery and characterization of a new family of redox biocatalysts that natively use the unusual deazoflavin co-factor F_{420} . The prospect for these enzymes as biocatalysts is discussed, including the advantages that may be gained from an unusually promiscuous cofactor specificity.

A4: The role of ytterbium as a promoter on nickel catalyst activity during dry reforming of biogas

Mohamad Hassan Amin, James Tardio, Suresh K. Bhargava

Advanced Materials & Industrial Chemistry Group, School of Applied Sciences, RMIT University, Melbourne, VIC 3001, Australia

Recently, considerable attention has been paid to the catalytic dry reforming of biogas in view of the environmental protection as well as potential industrial application aspects. This reaction converts biogas into valuable synthesis gases, which is the basic building block for many valuable chemicals. In comparison to conventional steam reforming or partial oxidation of biogas, dry reforming is more appropriate in remote areas, where water supplies are limited and its results in "CO-richer" synthesis gas with a relative low H_2/CO ratio, which is more desirable for the direct use as feedstock for carbonylation, hydroformylation, Fischer–Tropsch synthesis of long chain hydrocarbons, direct production of dimethyl ether and so on.

In this study, the effect of Yb on Ni catalysts supported on different ceramic oxides (γ -Al₂O₃, KIT-6, SBA-15) was investigated using Synchrotron powder diffraction, XPS, TEM, TPR, BET and thermo gravimetric analysis. Results show that the introduction of low quantities of ytterbium as promoter to the Ni catalyst supported on γ -Al₂O₃ via sol-gel method has a superior effect on catalytic activities and stabilities. The most important reason for this is due to the increment in number of Ni crystallites along with a decrease in metal particle size which leads to the better metal dispersion and more active sites. Higher levels of Yb doping (3-4 wt %) has a significant effect on the frame work which play an important role in declining the long term stability of the catalyst.

B2: Continuous process for catalytic conversion of cellulose to sorbitol: A step closer towards Industrial application

Abhijit Shrotri¹, Akshat Tanksale², Jorge Beltramini¹

¹ARC Centre of Excellence for Functional Nanomaterials, University of Queendland, St Lucia, QLD 4072; ²Department of Chemical Engineering, Monash University, Clayton, VIC 3168

Cellulosic is the most abundant organic raw material available on our planet. Various platform chemicals can be produced from cellulose which can then be used to produce sustainable alternative fuels. All conversions methods used today are inefficient and uneconomical due to severe conditions required in conversion of celluloses. Insolubility of cellulose in conventional solvents under normal conditions also presents many engineering challenges in designing a continuous process. One step conversion of cellulose to sugar alcohols via sequential hydrolysis and hydrogenation presents an attractive opportunity to undertake large scale production of a key platform chemical. However the progress is hindered due to slow hydrolysis of cellulose which is the rate limiting step in this process. Reaction rates can be increased by dissolving cellulose in a suitable solvent. In this paper we present a method to dissolve cellulose in water at ambient conditions. Cellulose was impregnated with small amount of mineral acid and milled in a ball mill. The resulting solid was amorphous and completely soluble in water at room temperature. Soluble cellulose composed of small oligosaccharides of glucopyranose units linked primarily via β 1-4 bonds. Milling in presence of acid resulted in formation of additional bonds including α 1-4 and α 1-6 glycoside bonds. Dissolved cellulose was converted to sugar alcohols in presence of bimetallic Ni-Pt catalyst supported on alumina. Sugar alcohol yield of 90% was obtained in 1hr of reaction. Ni-Pt bimetallic catalyst used is relatively inexpensive due to the low amount of Pt used to promote activity of Ni. A lab scale plug flow continuous process for the reaction is currently under development.

B3: Structural Requirements for Lignin Peroxidase Activity: Engineering New Enzymes for Lignin Degradation

Andrew T. Smith^{+,} Anabella Ivancich^{*}, Klaus Piontek[#], & Wendy A. Doyle^{\$}

⁺School of Applied Sciences, RMIT University, Melbourne, [&]Dept of Chemistry and Biochemistry, School of Life Sciences, University of Sussex. UK. *Centre d'Etudes de Saclay, Service de Bioénergétique, Gif-sur-Yvette, France. [#]University of Freiburg, Germany

Lignin peroxidase (LiP) is one of three enzymes produced by wood degrading fungi that play a key role in the degradation of lignin. The enzyme is uniquely able to oxidise extremely stable methoxy benzenes with high (>1.4V) redox potential that play a role in potentiating the destruction of lignin. This involves a free radical mediated reaction that eventually cleaves the C-C bonds in lignin. The ability to do this depends on a specialised substrate interaction site containing a unique redox active Trp residue located in a highly acidic microenvironment [1-3].

Mutational work on Lignin peroxidase and *Coprinus cinereus* peroxidase (CiP) has allowed us to successfully engineer veratryl alcohol oxidation activity (lignin peroxidase activity) into a commercial peroxidase, which normally lacks this ability. Only three mutations, the redox active Trp itself and two adjacent charge replacements on the surface of the enzyme were necessary to confer this activity at low level [4]. The engineered enzyme is kinetically competent in the oxidation of veratryl alcohol and gives rise to a trappable Trp radical on treatment with stoichiometric peroxide [5]. Evidence is presented that the Trp radical is specifically reduced by veratryl alcohol and that the enzyme is effective in other lignin model reactions.

[1] Doyle, W.A., (1998) Biochem **37**, 15097.

- [2] Blodig, W., (1999) Arch. Biochem Biophy. **370**, 86.
- [3] Blodig, W., (2001) J. Mol. Biol. 305, 851.
- [4] Smith, A.T,. WO/2006/114616, PCT/GB2006/001515.
- [5] Smith, A.T., (2009) PNAS 106, 1684.

B4: Nanobiotechnology advances biofuel production: Nanomaterial functionalization for enzyme immobilization with potential application in cellulose hydrolysis

Munish Puri

Centre for Biotechnology (BioDeakin), Geelong Technology Precinct, Waurn Ponds, Deakin University, Geelong, Victoria 3217, Australia

Nanobiotechnology is emerging as a new frontier to advance biofuel production. Robust nanomaterials support possessing nanoscale dimension has surpassed the conventional support for immobilizing enzyme due to inherent features like higher surface area, greater enzyme loading, lower mass transfer resistance, selective, nonchemical separation from the reaction mixture and cost effectiveness. To economise the bioenergy (an energy produced from agriculture/forest residues rich in cellulose polymer or organic matter by employing enzymatic hydrolysis) production¹, enzyme plays an important role in loosening/ hydrolysing cellulosic structure to release monomeric sugars suitable for biofuel production. It is opined that such enzymes should be immobilized to a support which provides thermostability, easier recovery and ensures reusability².

The present study was undertaken to develop a model for immobilizing enzymes such as β -glucosidase (an enzyme involved in cellulose hydrolysis) onto functionalized nanomaterial (10-20 nm). The functionalization and binding of enzyme on nanoparticles was further characterised by spectroscopic studies³. Immobilized nanoparticle-enzyme conjugate can be reused thus economising the process of energy generation. The results of the study encompassing use of various novel nanomaterials for immobilizing enzymes involved in biomass hydrolysis shall be discussed during presentation.

References:

- 1. Puri M, Abraham RE, Barrow CJ. 2012. Biofuel production: prospects, challenges and feedstock in Australia. Renewable and Sustainable Energy Reviews 16, 6022-6031.
- 2. **Kim J, Grate JW and Wang P,** Nanobiocatalysis and its potential applications. Trends Biotechnol 26 (2008) 639-646.
- 3. Verma M, Barrow CJ, Kennedy JF, Puri M. 2012. Immobilization of B-D-glactosidase from *Kluyveromyces lactis* on functionalised silicon dioxide nanoparticles: characterization and lactose hydrolysis. International Journal of Biological Macromolecules 50, 432-437.

C1: From Biomass to Fuels and Chemicals with Hydrothermal Treatments

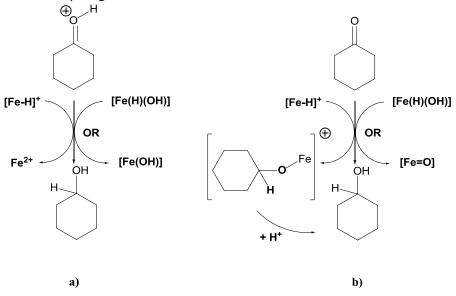
Thomas Maschmeyer

Laboratory of Advanced Catalysis for Sustainability, School of Chemistry, F11, The University of Sydney NSW 2006

A brief general discussion of some of the big picture issues related to our carbon-based economy (availability and security of resource supply, GHG emissions, big ticket infrastructure) will be presented.

This is to be followed by an introduction to the new semi-commercial Catalytic Hydrothermal Reactor (Cat-HTR) Technology¹ that can convert biomass (e.g. pulp & paper waste or macro-algae) into high quality syncrude oils, which are suitable for refining in conventional or dedicated oil upgrading facilities. In fact, the syncrudes that are produced have energy contents of around 36 MJ/kg and can be used as part of the Vacuum-Gas-Oil (VGO) processing stream.

Some mention will be made of model reactions that showcase the role of the reactor wall in the decomposition of small organic acids and transfer hydrogenations, re-examining the concept of 'nascent hydrogen'.²



Possible mechanisms for the reduction of cyclohexanone

Furthermore, a chemicals stream can also be isolated and used to supply a range of bulk and specialty chemicals – leading to renewables such as "green" plastics, flavours and fragrances, anti-oxidants, etc.

¹ <u>www.licella.com.au</u>

² (a) <u>Chemistry – An Asian Journal</u>, **2012**, 7(11) 2638–2643; (b) <u>Chemistry – An Asian Journal</u>, **2012**, 7(11) 2629–2637.

C2: New computational methods for modelling cellulase activity on cellulose

Andrew Warden

CSIRO Ecosystem Sciences, Canberra, Australia

Cellulases comprise a complex mixture of enzymes that work synergistically to break cellulose down into its component sugars. While there have been many efforts to model the cellulase system using traditional Michaelis-Menten kinetics, broad assumptions and approximations need to be made to fit these equations to the observed activities of cellulases owing to the fact that they are designed to describe solution phase systems, whereas cellulases act upon a solid surface. We have taken a different approach whereby each individual enzyme and cellulose component sugar is treated separately and programmed with a set of behaviours that mimic their reported motions, interactions and reactions as closely as possible. This is known as an 'agent-based' or 'cellular automata' approach to modelling. This presentation will cover the range of behaviours observed in cellulases and describe the way we have implemented them in the computer model. Some of the results of our simulations will also be discussed.

C3: Improving Enzymatic Saccharification of Pretreated Radiata pine in the New Zealand Lignocellulosic Biofuel Initiative

Kirk Torr, Michael Jack, John Lloyd, Karl Murton, Roger Newman, Lloyd Donaldson, Ian Suckling, Trevor Stuthridge and Alankar Vaidya

Scion, Private Bag 3020, Rotorua 3046, New Zealand

Plantation softwood forestry offers one of New Zealand's most promising biomass resources for the future production of renewable fuels, chemicals and biomaterials as replacements for petrochemicals. The New Zealand Lignocellulosic Biofuel Initiative has developed a cost-effective and environmentally sustainable process for the conversion of softwoods, specifically radiata pine, to monomeric sugars and functional lignin. The sugars can be subsequently processed into ethanol, butanol or bioplastics using other technologies. The LBI process involves an initial thermomechanical treatment of wood chips to give a pretreated substrate and a hemicelluloserich liquid stream. Enzymatic saccharification of the pretreated substrate produces a sugar syrup rich in hexose sugars with few fermentation inhibitors, and a lignin-rich solid residue. Improving the efficiency of the enzymatic saccharification has been a key area of research in targeting lower production costs. This presentation will report on progress toward improving saccharification yields and decreasing enzyme consumption through the use of new enzyme cocktails and saccharification additives. The use of confocal microscopy to probe the mechanism by which saccharification additives improve enzymatic digestibility in softwood substrates will be discussed.

C4: Sustainable polymer modifiers derived from woody biomass and fatty acids

Florian H.M. Graichen¹, Michael S. O'Shea¹, Benjamin A. Leita², Gary Peeters¹

¹CSIRO Materials Science and Engineering, Clayton, VIC, Australia; ²CSIRO Earth Science and Resource Engineering, Newcastle, NSW, Australia

There is significant global momentum around the (re)emergence of bio-derived feedstock for the chemicals and polymer industries. Many of the current renewable polymer modifiers are normally unsuitable due to reliability of supply, cost or both.

CSIRO's research in the areas of woody biomass, industrial oils and complex monomers is aimed at the production, evaluation and modification of novel oil, fatty acid and woody biomass building blocks for a range of industrial applications including:

- chemical intermediates
- oligomers
- cross-linkable polymers
- bioactive compounds
- fuel additives
- thermo/mechano-chromic devices.

CSIRO believes that there are significant opportunities in the production of woody biomass based polymer modifier and novel hydroxylated oils/fatty acids with novel hydroxy-group positions. We have investigated the synthetic and biological production of a range of structures and their utility in the chemicals and polymers industries.

Alpha-hydroxy fatty acids for example have a range of interesting properties, some of which are strongly influenced by the strong self assembly properties of the 2-hydroxy fatty acids. Uses for these novel fatty acids include: monomer for use on commodity polymer, modifiers and adhesives through to materials to produce optoelectronic devices.

Cineol diol – an example for a woody biomass derived polymer modifier – has the potential to improve the thermo and barrier properties of a range of polymers – regardless of bio or petrochemical derived.

This paper will cover some synthesis methods for the saturated and unsaturated alpha hydroxy fatty acids and cineol diol derivatives as well as applications ranging from optoelectonic polymers to adhesives and monomers and modifiers for polyurethanes, polyesters and polyamides.

D1: Novel Materials for Gas and Liquid Separations Associated with Bioprocessing

Richard D. Noble

Alfred T. & Betty E. Look Professor of Chemical Engineering. University of Colorado, Boulder, CO 80309, USA

The production of chemicals by biological routes has many elements in common with conventional chemical processing. This commonality includes chemical separation steps. The use of CO_2 as a carbon-based feedstock requires a gas separation step prior to introduction of the gas to the bioreactor. The production of organic chemicals in the bioreactor needs a separation step at the reactor exit to remove the products but keep the biomass and nutrients in the reactor. This presentation will discuss two novel membrane materials for these separations.

Room Temperature Ionic Liquids (RTILs) are solvents consisting entirely of ions resembling the ionic melts of metallic salts; however, RTILs are liquids at much lower temperatures. RTILs have high thermal stability, high ionic conductivity, negligible vapor pressure and are non-flammable. Unlike traditional organic media, RTIL's properties may be adjusted via chemical alteration of the cation or anion to produce application specific compounds. Many RTILs are liquids over a wide temperature range with some known melting points as low as -96 $^{\circ}$ C and some liquid ranges in excess of 300 $^{\circ}$ C.

This presentation provides an overview of our recent efforts and research directions in the design and synthesis of several new types of functionalized, imidazolium-based RTILs, poly(RTIL)s, and RTIL-based composite materials for use in the area of targeted gas separations. Polymeric versions of ILs have been synthesized and used as membranes for gas separations. In addition to a physical solvent, RTILs might also be incorporated into RTIL polymers or in supported ionic liquid membranes (SILMs) as the selective component. The addition of 20% RTIL into an RTIL polymer increases the permeability ten fold (100 vs. 10 barrer) while maintaining selectivity. Gelled versions exhibit large permeabilities (~ 1000 barrers) as well as CO_2/N_2 selectivities (~ 30) that outperform many polymer membranes in that application.

The development of polymers with ordered, interconnected, sub-1-nm size pores for molecular size separation will also be presented. These nanostructured polymers are made by cross-linking lyotropic liquid crystal (LLC) assemblies formed by polymerizable surfactants in the presence of water. Supported LLC polymer membranes possess a 3-D interconnected water pore system with a uniform pore size of ca. 0.75 nm. These LLC membranes are able to cleanly size-exclude hydrated salt ions and a variety of small organic solutes from water (i.e., water desalination) with good permeabilities. The water desalination/nanofiltration performance of this LLC polymer membrane is superior to that of conventional nanofiltration membranes, and close to that of commercial reverse-osmosis membranes.

D2: Unique Roles for Ionic Liquids in a Biorefinery: Extraction, Separation, and Processing of Lignin, Cellulose, Hemicellulose, and Chitin

Robin D. Rogers

The University of Alabama, USA

Clean separation of the three major components of lignocellulosic biomass is an important and challenging 'Grand Challenge' for the production of reproducible feedstocks for further chemical processing. However, while the current 'biorefinery' concepts do emphasize other chemicals besides fuel, it is typically the cellulose and hemicellulose which are utilized in producing paper, fibers, membranes, and other commodity materials and chemicals, while lignin is usually burned for energy. There seems to be relatively little emphasis on using natural biopolymers as polymers rather than feedstock for producing molecular chemical entities. Indeed chitin, the second most plentiful biopolymer on earth after cellulose, and the most abundant polymer in the marine environment, is often similarly ignored. This presentation will discuss how the components of both lignocellulosic and chitin biomass interact with ionic liquids (ILs) and how these interactions can be used to manipulate solution-properties to process and regenerate biopolymers into a desirable form for specialized applications. The differing solubilities of the biopolymer fractions also allow the exploitation of the IL medium to provide a ready separation mechanism. The unique properties of ILs can be used to provide a platform for biomass to chemicals strategies and yield endless possibilities for forming environmentally-friendly new and enhanced functional materials.

D3: Membranes for Biorefineries

John Kavanagh^a, Tim Handelsman^a, Geoff Barton^a, Hans Coster^a, Felicity Roddick^b, Thang Nguyen^b

^aThe University of Sydney School of Chemical and Biomolecular Engineering, ^bCivil, Environmental & Chemical Engineering RMIT

Wastewater treatment is looming as a major challenge for the development of a cellulosic ethanol industry. Recent studies estimate the cost of the wastewater treatment to be between 25% and 33% the value of the ethanol produced. The challenges of treating this waste include a very dark colour (40,000 PtCo), high COD (40,000 -150,000 mg/L) and high levels of sulfur and ammonia (2,500 and 3,000 mg/L respectively). Ultrafiltration membranes have the potential to preferentially separate the COD from the dissolved inorganic species and thus allow lower cost waste treatment strategies to be considered.

This study presents a systematic survey of the performance of a range of ultrafiltration membranes with nominal molecular weight cut offs (nMWCO) ranging from 1,000 to 20,000 on a cellulosic effluent originating from a pilot scale acid digestion process.

Results from this study include that membrane surface properties have a far greater effect on effluent COD and colour than the nMWCO. Hydrophobic membranes were found to give significantly greater COD and colour removals than hydrophilic membranes. However within classes of membranes a strong linear relationship was found between colour removal and nMWCO. The relationship between colour and COD was not strong, indicating that the majority of the COD is colourless.

The results of the study provide direction for future work in tailored surface modified membranes, as well as wastewater system design.

E1: Depolymerization of cellulose by heterogeneous catalysis

Atsushi Fukuoka

Catalysis Research Centre, Hokkaido University, Japan

Conversion of cellulose has attracted significant attention as a key issue in the utilization of nonfood biomass. Heterogeneous catalysis would be promising for the conversion of lignocellulose, because solid catalysts can be used under a wide range of conditions, easily separated from the reaction mixture, and reused in repeated reactions. In this talk, I will report our recent work on the depolymerization of cellulose by heterogeneous catalysis, in which modified activated carbon shows high and durable activity in the hydrolysis of cellulose.

E2: Conversion of Lignocellulosic Biomass to Fuels and Chemicals over Heterogeneous Catalysts

Jorge Beltramini

ARC Centre of Excellence for Functional Nanomaterials – AIBN, The University of Queensland, Brisbane, Queensland, 4072, AUSTRALIA

Biomass is an important feedstock for the renewable production of fuels, chemicals and energy. With the depletion of fossil fuels, the fraction of energy and chemicals supplied by renewable resources such as biomass can be expected to increase in the foreseeable future. Indeed, several government around the world have recently passed legislation mandating increases in the gross domestic energy and chemicals production from renewable resources, especially biomass. Unfortunately, tapping this resource directly to obtain fuels and chemicals is not possible, but instead new catalytic processes will be required to facilitate and control conversion and desirable product selectivities. Most of the current chemical processes are not suitable for converting biomass as the potential energy to harvest is stored in complex molecule such as carbohydrates, lignins, proteins, glycerides, terpenes and others, where each carbon atom is connected to an oxygen atom. An efficient transformation of biomass should then reduce its oxygen content through efficient catalytic processes that provide the alternative pathways for the production of chemicals and fuels. It is also well known that solid catalysts are always the preferred option for most processes and dominate the spectrum of the chemical industry. Then it would be expected that an increase in the use of biomass would lead to an increase in the share of homogeneous and enzymatic catalysis, mainly for two reasons. Firstly homogeneous catalysis and enzymes are dissolved in the reaction medium, facilitating the interaction and having a high selectivity. But despite the high selectivity achieved, many catalytic systems have not been commercialized because of the difficulties encountered when trying to separate from the reaction media. The main reason to consider heterogeneous catalysis for industrial processes is the case for catalyst separation after reaction. Separation processes represent more than half of the total investment in equipment for the chemical and fuel industry. Then the ease of separation of solid catalysts can be a crucial advantage. However these catalysts should also be highly selective to guarantee the cost effectiveness of the process. In this presentation we will focus on the challenges facing heterogeneous catalysis in biomass processing. Traditionally, cellulose has been converted to glucose by dilute or concentrated acid hydrolysis. However, this process suffers from acid corrosion and glucose degradation under high acid concentration. Various types of supported metal precious catalysts have been tested for this reaction, however, there is a need for a low cost catalyst, which can effectively replace or reduce the precious metal catalysts. In this regard Nibased catalysts, which are widely used in existing industries due to their low cost and availability are known to be a good hydrogenation catalyst, therefore, it is expected that Ni may perform well as a catalyst for glucose hydrogenation to sorbitol, as glucose is an intermediate product in this reaction. It is the intention of this presentation to provide a practical view of the challenges met in the development of solid catalysts for production of fuel and chemicals from biomass. Different strategies for biomass pretreatment such as degradation/separation techniques, involving temperatures, pressures, solvents, pH, etc as well catalytic cracking, hydrolysis, reductions and oxidations reactions are discussed in view of the whole biomass feed composition and desired products. A detail study of the mechanism on structural and chemical changes that increase the total solubility of cellulose in water by optimizing the pre-treatment step is fully presented. Finally the catalytic behaviour of dissolved oligomers to sorbitol, fuels, chemicals and hydrogen using a continuous plug flow reactor system and with bimetallic catalysts based on Ni promoted with

metal precious (Pt, Pd, Re, Ru, etc) supported on alumina nano-fibre, mesoporous alumina, mesoporous zeolites and mesoporous carbon at different level of acidity is also investigated. As a comparison we tested non-functionalised supported catalyst to test the role of metal/acidity ratio in the biomass reaction.

F2: Biodiesel Production from Algae Biomass Cultivated with Carbon Dioxide (CO₂) Via Biocatalytic Process

Mohd Asyraf Kassim^{1,2}, Ravichanda Potumarthi¹, Akshat Tansale¹, Sankar Bhattacharya¹

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Production of biofuel from algae biomass is a promising approach for replacing part of the fossil fuel sources for energy production. Production of algae biomass in sustainable and environmentally friendly is important to ensure the competitiveness of the fuel produced. This study reports the production of algae biomass and algae lipid cultivated in medium using two types of algae namely *Chlorella* sp. and *Tetraselmis* sp. supplied with 14% CO₂. In this study, the growth rate and lipid profile produced in the system has been determined. The maximum biomass and carbon fixation rate of 0.42 gL-1 and 0.41gL⁻¹day⁻¹ was observed for *Chlorella* sp. and 0.42 gL⁻¹ and 0.47 gL⁻¹day⁻¹ for *Tetraselmis* sp. . The lipid productivity of both algae species was up to 27% and 40% for *Chlorella* sp and *Tetraselmis* sp. respectively during the experiments. The main fatty acid compositions of these two algae were determined. In this current work, production of biodiesel by enzymatically trans-esterification of algae lipid produced in 14% CO₂ has been carried out. The present results carried out using algae grown in small laboratory set-up are being tested in larger rig open to sunlight. Subsequently these will be used for an assessment of the viability of the production of these two species for CO₂ capture and their further use for the production of chemicals.

F3: Separation, Storage, and Triggered Release of CO₂

Matthew R Hill¹, Kristina Konstas¹, Richelle Lyndon^{1,2}, Constantinos Dimitrakakis^{1,2}, Bradley P Ladewig², Anita J Hill¹, Aaron W Thornton¹

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Given that the ongoing usage of carbon based fuels is likely for several decades to come, means to capture produced carbon dioxide as efficiently as possible are crucial. Presently, liquid amine solutions are employed to separate the carbon dioxide emitted from coal-based power generation sources. However, the storage capacity is limited in these materials, and they require significant heating to release the captured carbon dioxide. This heating places a large parasitic energy demand on the power generator.

Metal organic frameworks (MOFs) are ultraporous materials that can act as solid adsorbents for carbon dioxide. Consisting of metal atoms or clusters linked periodically by bridging organic groups, there internal pores can be tailored to act as high capacity sites for carbon dioxide capture. Furthermore, organic groups may be introduced to allow the triggered release of the captured gas, removing the need for heating during regeneration, lowering the parasitic energy demand of the technology upon the power generator.

This presentation will describe recent work at CSIRO that has resulted in the development of an adsorbent with a CO_2 capture capacity over 300% that of commercial materials, and a strategy to allow the release of captured gas by using concentrated sunlight.

F4: Innovative strategies to convert waste material into biofuel

Cristina Prada, Mohamed Taha Elfouly, Andy Ball, Trevor Stevenson, Aidyn Mouradov

Plant and Algal Biotechnology Group, School of Applied Sciences, RMIT University, Melbourne, Australia

Today, the danger of an energy shortage coincides with an increasing level of atmospheric CO_2 and an approaching global food shortage. This all dictates urgency for the development of a new generation of feedstock that will not only produce biofuels or their various components, but that can also be used for feeding animals and humans and at the same time will reduce the levels of nitrogen and phosphorus from waste biomass and reduce atmospheric CO_2 . These organisms have to be grown on marginal lands not used for food crops and require low inputs of fresh water and fertilizer. In our group we've become interested in aquatic plants and algae (fastest growing photosynthetic species on Earth) based on their ability to utilize efficiently waste biomass and CO_2 and produce biofuels: bio-alcohols and bio-diesel. Some algal and duckweed species can double biomass every 8 hrs and 30 hrs, respectively. Potential biomass yield of duckweed representatives is 80 dry metric tons/hectare/year. Starch content in both duckweed and algae can reach up to 45% of dry weight with average annual starch production 28 tons/he/year (corn produces 5 tons/he/year). Some algal strains can produce up to 75% of oil/dry weight. Our research is focused on optimization of different stages of biofuel production from duckweed and algae growing on different waste biomasses as sources of carbon, nitrogen and phosphorus.

G1: Pilot-Plant and Bench-Scale Studies of Hydrothermal Treatment of Biomass

Chris Jazrawi, Xiao Liang, Alejandro Montoya and Brian S. Haynes

School of Chemical and Biomolecular Engineering, The University of Sydney, Australia

We describe the design, construction and operation of a continuous hydrothermal slurry reactor. The plant can process up to 10 kg dry biomass per hour at temperatures up to 350 °C and pressures to 250 bar.

Results will be presented for the processing of various biomass samples, with the focus on microalgae. The yield of organics from microalgae greatly exceeds what could be derived from the lipids because, under hydrothermal conditions, not only the lipids but also the carbohydrates are converted to "oil".

The presentation will conclude with an overview of theoretical and experimental studies of reaction kinetics of biomass-related compounds under hydrothermal conditions.

G2: The Lignocellulose-Based Biorefinery - An Australian Approach

Gregory C. Court, Tony Duncan, Stephen G. R. Lawrence and Warwick D. Raverty

Circa Group Pty Ltd., Melbourne, Australia

The US Department of Energy's National Renewable Energy Laboratory has published a list of the 8 most promising renewable platform chemicals that it considers the most likely candidates that can be produced from lignocellulose to compete with current petro-based platform chemicals. Of these 8 compounds, only 3 can be produced by thermochemical means and only one of those (sorbitol) can be produced by commercially proven technology. The remaining 5 compounds are derived from glucose by fermentation, a process that requires huge volumes of fresh water that are simply not available in most of Australia. The Melbourne-based chemical manufacturer, Circa Group has developed the first thermochemical process to produce the chiral compound (-)levoglucosenone from plant-derived biomass continuously on a commercial scale. The new process has been designed specifically to be operable in rural areas where agricultural and forestry wastes are available. The process is modular and scalable, has low capital cost, produces fresh water, rather than consuming it, and is carbon-neutral. Arguably (-)-levoglucosenone is the most valuable low molecular weight compound that can be produced from cellulose as it preserves two of the chiral centres present in glucose and its rigid molecular shape and α , β -unsaturated ketone functionality permits complete regio-specificity and stereo-specificity in the majority of its chemical reactions. The development of the new process used to produce levoglucosenone from lignocellulose is described as are a number of chemical transformations of the molecule that lead to commercially valuable fine and bulk chemical products.

G3: New engineered crops for the large scale production of renewable oils

Craig Wood

Oils and fatty acids are valuable commodities and crops offer scalable solutions for supplying feedstocks into the oleochemical industries. Here I outline two CSIRO activities addressing the need for renewable sources of oils, via developments in oilseeds and radical re-engineering of leaves to produce oil.

Pure monounsaturated oils (PMO) are long-sought after bulk oleochemical feedstocks due to their potential uses in oxidatively-stable lubricants and valuable derivatives. Although oilseeds can attain high levels of monounsaturated oils via conventional and GM-based breeding that are suitable for nutritional uses, the remaining polyunsaturated and saturated oils preclude their feasible use as industrial feedstocks. Here we report broadacre safflower containing ~95% monounsaturated oils suitable as a PMO. Seed-specific silencing of safflower FAD2 and FATB activities using hairpin RNAi generated PMO-safflower with no apparent adverse phenotypes. This renewable source of bulk PMO meets all criteria as an engineered industrial oil.

Leaves are a large biomass yet produce only low levels of oils. We have previously found that leaves engineered to express oil accumulating genes, normally found in oilseeds, can force the synthesis of oils in leaves. These findings have been advanced significantly by multigene combinations such that leaves were engineered to overexpress combinations of seed-specific transcription factors and oil accumulation genes. The best combination of genes raised the percentage of leaf oil from 0.5% to over 13 (on a dry weight basis) and also modified the fatty acid profile of these oils. At these concentrations such engineered leaf oils can exceed canola in terms of production per area.

G4: Valorization of *Bio*-ethanol: Conversion of Ethanol to Higher-Value Products Using Transition Metal Catalysis

Kapil S. Lokare

Laboratory of Advanced Catalysis for Sustainability, School of Chemistry, The University of Sydney, New South Wales 2006, Sydney, Australia

Interest in renewable fuels has been encouraged in recent years by a number of environmental, economic and geopolitical consideration, including global warming, dwindling petroleum deposits, rising crude oil prices and a desire for energy independence. Against this background, biofuels - fuels derived from biomass - have been proposed as a renewable, carbonneutral alternative to fossil fuels. Over the years, a number of biofuels have been proposed e.g. *n*-butanol from *bio*-ethanol¹. To date relatively few catalyst systems have been developed that



allow the conversion of ethanol to *n*-butanol and higher hydrocarbons in low yields, and there are still large gaps in our fundamental knowledge of how to design such systems². A direct fermentation process from sugars to *n*-butanol instead of ethanol is under study, but until date proceeds with limited efficiency³.

This talk will highlight how the utility of imidazole ligand backbone allows the isolation of a range of transition metal catalysts for probing the valorization of *bio*-ethanol in the presence of strong bases on the bench scale. Importantly, imidazole-based ligands offer reversible *de*-protonation/protonation by strongly basic solvents and thus homogeneity in the reaction media. These and specific inventions will have broad applicability to the valorization of alcohols and the development of improved processes for chemical synthesis.

¹Mitsubishi Chemical Corporation, Tokya (JP), Tanaka, Y.; Utsunomiya, M. US 2010/0298613 A1, **2010**

²Kunkes, E. L.; Simonetti, D. A.; West, R. M.; Serrano-Ruiz, J. C.; Gärtner, C. A.; Dumesic, J. A. *Science* **2008**, *322*, 417

³Festel, G. W. Chem. Eng Technol. **2008**, *31*(5), 715-720

H1: Computational modelling and experimental data showing detailed mechanisms and the barriers to fast conversion of cellulose to sugars

Michael Crowley

National Renewable Energy Laboratory, Colorado, USA

At NREL, Michael and his team have published many studies of the components of biomass and the industrial bottlenecks to its conversion to fuels. They have gained insight into the structure and dynamics of cellulose, the major component of biomass and a particularly difficult sugar polymer to convert to fuels. They have determined many aspects of the mechanisms of cellulase enzymes which convert cellulose to sugars and have suggested multiple modifications to the enzymes and cellulose to increase the efficiency of commercial biomass conversion. They are working on cell wall structure and synthesis to help design plants that are both highly productive and improve industrial conversion to fungible fuels. All their studies are performed on High Performance Computers (HPC) and use the most current theoretical techniques and are in close collaboration with the most current experimental efforts in biofuels.

H3: Thermodynamically Consistent Modelling of the Decomposition of Cellobiose

Xiao Liang, Alejandro Montoya, Brian S. Haynes

School of Chemical and Biomolecular Engineering, The University of Sydney, Australia

Kinetic modelling of glycosidic polymer decomposition under hydrothermal conditions has been investigated for more than 50 years. However, most of available models lack thermodynamic consistency, largely because aqueous phase thermodynamic properties of the species formed in these processes have been unknown.

In this work, cellobiose is selected as a model compound of a glycosidic polymer. A detailed thermodynamically-consistent kinetic model of cellobiose decomposition under hydrothermal conditions is developed and validated against experimental results. The thermodynamic properties of the species considered in the model are determined using high level *ab initio* molecular computations. Experimental data used for the validation are obtained using a plug-flow microreactor system. Local sensitivity analysis and reaction flux analysis (state of balance and rate of progress) are carried out to interpret model results and identify important reaction channels of cellobiose decomposition under hydrothermal conditions.

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Posters

P1 Based Catalysts

Conversion of Cellulose to Tar Fan Liang Chan and Akshat Tanksale Free Synthesis Gas over Nickel Department of Chemical Engineering, Monash University, Clayton, VIC 3800 Nickel based catalyst is widely used in various modern chemical processes. Our interest is particularly in biomass gasification. Nickel catalyst is proven as one of the most effective transition metal catalysts in tar cracking and reforming, which provides a significant upgrade on the quality of the final gaseous products. Commercial nickel based catalysts are generally employed in the biomass gasification in two ways: primary catalyst or secondary catalyst. Primary catalyst is the catalyst used directly in the gasifier whereas secondary catalyst is used in the post gasification reactors. The primary catalysts suffer from rapid deactivation due to the coke deposition. This research project aims to develop stable nickel based catalysts for cellulose reactive flash volatilization reaction, where powder cellulose is converted into tar-free synthesis gas in a single reactor using a combination of pyrolysis, gasification, steam-reforming and water gas shift reactions, in a short residence time reactor (~0.3s). Our approach includes synthesis of catalysts with different support materials, metal promoters and synthesis methods. We will also investigate nanoparticle catalysts to increase the active metal surface area which can enhance the nickel catalyst performances on product selectivity, reaction efficiency and resistivity to coking in biomass gasification. Catalyst characterization showed that the specific surface area of the catalyst was not affected by the addition of Ni-Ru but reduced by the addition of Ni-Pt and Ni-Re. TPR results demonstrated that reducibility of the Ni catalyst can be significantly improved with the addition of Pt and Re. Preliminary catalytic studies with the 10:1 Ni-Pt/Alumina catalyst showed that production of tar free synthesis gas can be achieved at the temperature of 700°C. Hydrogen to carbon-monoxide in the synthesis gas produced was 1:1 which is ideal for dimethyl ether (DME) production. DME can replace diesel as a transportation fuel.

Caitlin S Byrt, Ghazwan Karem, Marilyn Henderson, Natalie S Betts, Geoffrey B Fincher, *Don Chambers, Konstanze Beck-Oldach and Rachel A Burton ARC Centre of Excellence in Plant Cell Walls, Waite Campus, University of Adelaide, Urrbrae, SA 5064. *AusAgave, PO Box 32, Aldgate, South Australia, 5154

We are interested in hardy crops that can generate high yields of sugar and biomass on marginal lands with minimal requirement for water and fertilizer. *Agave* is efficient at using water, since it employs the crassulacean acid metabolism (CAM) photosynthetic adaptation where carbon dioxide is fixed at night. *Agave* also has the potential to generate lots of sugar in semi-arid environments; however, little is known of the composition of the biomass. We have investigated sugar and cell wall composition in the leaves of two agave varieties *A. americana* and *A. tequilana*.

Sugarcane waste (i.e. bagasse) is an abundant and relatively low cost carbohydrate resource that can be utilized to produce platform chemicals such as levulinic acid and furanics through acid-catalyzed reactions. These chemicals can be easily upgraded to commodity and specialty chemicals and biofuels by high yielding and well established technologies. Commercial production of levulinic acid and furfural suffers from the use of corrosive mineral acid catalysts such as sulfuric acid (H_2SO_4) which increase equipment and operating costs and can lead to waste disposal issues associated with non-recovery of the catalyst.

The poster reports on research that is currently being undertaken with green acids (e.g. methanesulfonic acid) and co-solvents such as glycols in an effort to improve the overall process of producing the compounds. While catalyst activity was shown to be linked to acid strength, the selectivity between H_2SO_4 and the sulfonic acid catalysts was not significantly different. The use of glycol as co-solvent reduced side reactions and also produced esters that allowed overall product carbon yield to be improved by up to ~20%. As high yields could be achieved under relatively mild conditions, the process could readily be integrated with the existing infrastructure and utilities of sugar factories. This would allow sugar factories to be converted into biorefineries permitting greater utilisation of the sugarcane resource.

Further trials are being conducted to examine strategies for product recovery and recycling of the catalyst and solvent mixtures to fully evaluate process viability.

Agave biomass composition and sugar yields

P2

Conversion of Sugarcane Bagasse into Organic Acids and Furanics D.W. Rackemann, J.P. Bartley and **W.O.S. Doherty** Centre for Tropical Crops and Biocommodities, Queensland University of Technology, Brisbane Lignocellulosic biomass hydrolysis for biofuel production using nanoscafold bound enzyme

Reinu E Abraham, Madan L Verma, Colin J Barrow and Munish Puri[#] Centre for Biotechnology (BioDeakin), Geelong Technology Precinct, Waurn Ponds, Deakin University, Victoria 3216, Australia

The second generation¹ biofuel production technology depends on processing lignocellulosic biomass (LCB) which is available in abundance in the form of agricultural and forestry residues, bagasse and various other feedstocks². Due to the pretreatment steps required for commercial applications, LCB remains a severely underutilised polymer despite its abundance. Pretreatment facilitates the loosening of cross linkages that give lignocellulose its robust and compact structure. Production of cellulosic ethanol is commonly carried out using different enzymes (such as glucosidases and cellobiohydrolase) to hydrolyse cellulose into glucose. The addition of more enzymes in turn leads to better saccharification thus leading to biofuel production; however, this process increases downstream cost. This study primarily investigated the effect of an immobilized enzyme on pretreated biomass under different conditions to make this process economical. Alkaline pretreated biomass yielded maximum conversion of holocellulose into monomeric sugars. The resulting morphological changes in the biomass structure were evidenced by microscopy (SEM images) and spectroscopic techniques (ATR-FTIR and XRD). The LCB conversion rate was improved by immobilizing enzyme on a nanoscaffold³. The immobilization process was achieved via covalent linkage between aldehyde group of activated surface and amino group of enzyme. HPLC analysis further validated the presence of reducing sugars in the LCB hydrolysate (as a result of enzymatic hydrolysis) that are suitable for biofuel production. References:

- 1. Subbaraman N. 2010. Ethanol blend hike to jump start cellulosic investment. Nature Biotechnology 28, 1229.
- 2. Puri M, Abraham RE, Barrow CJ. 2012. Biofuel production: prospects, challenges and feedstock in Australia. Renewable and Sustainable Energy Reviews 16, 6022-6031.
- 3. Verma M, Barrow CJ, Kennedy JF, Puri M. 2012. Immobilization of B-Dglactosidase from Kluyveromyces lactis on functionalised silicon dioxide nanoparticles: characterization and lactose hydrolysis. International Journal of Biological Macromolecules 50, 432-437.

There is significant global momentum around the (re)emergence of bio-derived feedstock for the chemicals and polymer industries. The catalytic conversion of 1,8-cineole, sourced from the steam distillation of eucalyptus waste, to *p*-cymene has been studied. *p*-cymene is then converted to terephthalic acid (TPA), the main monomer used in the production of PET.

The main chemical component in Eucalyptus oils is 1,8-cineole (cineole) and is produced from the leaves of the Eucalyptus.¹ Eucalyptus is a diverse genus of trees and shrubs which dominate the tree flora of Australia. They are very hardy plants and in many cases can be grown on marginal land due to their drought and salt resistance. Species of Eucalyptus are cultivated throughout the world, including the Americas, Europe, Africa, the Mediterranean and China.¹

Cineole is an attractive renewable feedstock for the production of C_{10} chemicals. *p*-cymene is used in the production of *p*-cresol, a key intermediate in the synthesis of many industrial chemicals.³ Here we further convert the *p*-cymene into TPA, the key monomer for the production of PET.

The catalytic conversion of cineole into to p-cymene has been achieved at very high conversion and selectivity rates producing easily separated products of p-cymene (oil layer) water and hydrogen gas.^{2,3}

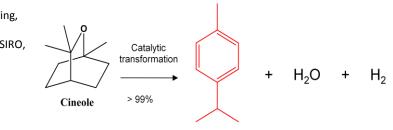
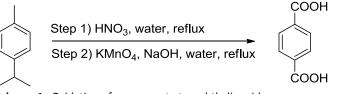




Figure 1. First step, conversion of cineole into *p*-cymene, water and hydrogen. The next step involves the oxidation of *p*-cymene into terephthalic acid. This oxidation is achived using the litreture method published by Pulkkinen *et. al.*⁴ This method converts *p*-cymene to terephthalic acid using HNO₃ and KMnO₄ as oxidizing agents.



Scheme 1. Oxidation of *p*-cymene to terephthalic acid

The green bio-terephthalic acid is then polymerisied with bio ethylene glycol, to produce a PET polymer that is made from 100% renewable feedstocks.

Renewable Plastics from Terpenes - Eucalyptus oil to BioPET

Graichen², Nick Burke¹ ¹Earth Science and Resource Engineering, CSIRO, Clayton, VIC, Australia ²Materials Science and Engineering, CSIRO, Clayton, VIC, Australia

Ben Leita^{*1}, Mike O'Shea², Florian

Organic carbonates for the functionalization of bio based chemicals Alessio Caretto^a, Alvise Perosa^b, Thomas Maschmeyer^a ^aThe University of Sydney, School of Chemistry, Building F1, NSW, 2006, Australia ^bUniversità Ca' Foscari Venezia, Dipartimento di Scienze Molecolari e Nanosistemi,Dorsoduro, 2137 - 30123 Venezia, Italy Studies on biomass transformation into chemicals have demonstrated that some of them can be efficiently produced, such as levulinic acid and C_4 - C_5 lactones.^{1,2} In order to obtain new synthetic routes from these platforms to more functionalized compounds, their reactivity with some organic carbonates has been studied; moreover the latter permit performing reactions with green chemistry protocols. This approach is in agreement with the idea of studying new broad-based technologies to produce multiple compounds (*divergent approach*),³ combining the production of chemicals alongside biofuels as an incentive for the biorefinery.

For example, levulinic acid reacts with dimethylcarbonate in the presence of a base, to yield methyl levulinate. Then at higher temperature (180-240 °C) the reaction proceeds to dimethyl succinate through a formal oxidation mechanism.⁴ In another example, D-valerolactone, under similar conditions undergoes D-methylation to afford D-methyl-D-valerolactone. Using other carbonates (*e.g.* diethyl- and dibenzylcarbonate) and other lactones (D-butyro-, and D-valerolactone) an interesting array of derivatives can be obtained.

References

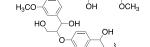
- 1. Corma, A.; Iborra, S.; Velty, A. Chem. Rev. 2007, 107, 2411-2502.
- 2. Werpy, T.; Petersen, G. *Top Value Added Chemicals from Biomass, U.S. D.O.E.* **2004**.
- 3. Bozell, J. J.; Petersen, G. R. Green Chem. 2010, 12, 539-554.
- 4. Selva, M.; Marquez, C. A.; Tundo, P. Gaz. Chim. It. 1993, 123, 515-518.

Hemicellulose is the primary source for the commercial production of furfural and valueadded furans and the potential exists for much greater utilisation of such products as fuel additives and chemical feedstocks. Under hydrothermal conditions, hemicellulose breaks down into oligomers, sugars, furans and acids, but the detailed product distribution is not well understood, especially considering the effects of temperature, pH, and residence time.

The objective of this study is to understand hemicellulose depolymerization under hydrothermal conditions (200-300°C, 120-150bar). Xylan, the primary constituent of hemicellulose, has been used as the primary reactant and its decomposition products have been characterised as oligomers of different molecular weight (123,000-1000 g/mol), xylotetrose, xylobiose, xylose, organic acids, and furans. The effects of temperature and reaction time on xylan decomposition and product distribution have been established. A detailed kinetic model based on parallel and consecutive reactions has been developed, to understand the disappearance of xylan, providing insight into the factors determining selectivity to oligomer production, monomer formation from oligomers and direct oligomer degradation to organic acids. Kinetic modelling reveals that all the oligomers disappear at a higher rate compared to xylose; with increasing temperature, xylotetrose and xylobiose directly form organic acids, thus reducing the xylose and furanic product yield. The proposed model provides a basis for optimising operational conditions for xylan depolymerisation.

Interest in bio-based compounds has been encouraged in recent years by a number of environmental, economic and geopolitical consideration, including global warming, dwindling petroleum deposits, rising crude oil prices and a desire for self sustainable society. Against this background, bio-based chemicals have been proposed as a renewable, carbon-neutral alternative to the more traditional petroleum based products.1

phenols, acids, diacids, aldehydes, catechols, cresols, polyhydroxy aromatics, keto acids, etc.



Lignin

-0



P7

Asanka Rahubadda, Alejandro Montoya and Brian S. Haynes

Hemicellulose **Depolymerization under** Hydrothermal Conditions School of Chemical and Biomolecular Engineering, The University of Sydney, Sydney, NSW 2006, Australia

Product

Identification/Development in the Bio-based Chemicals Arena: Phenols from Lignin

Maschmeyer Laboratory of Advanced Catalysis for Sustainability, School of Chemistry The University of Sydney, New South Wales 2006, Sydney, Australia

Dr. Kapil S. Lokare, Professor Thomas

The main components of biomass resources typically are lignin, cellulose, hemicellulose and minerals, with C-O bonds forming the basis of their polymeric backbone.² Several technologies have been developed to convert complex polymeric biomass into a more accessible subset of compounds, such as gasification, pyrolysis and hydrothermal upgrading (HTU). A general method for the separation/identification of individual functionalities and the conversion of these functionalities to produce compounds that are entirely fungible with those derived from a more traditional fossil fuels is thus of paramount importance.³ In view of this, we have shifted our attention to the analysis and identification of compounds obtained from the hydrothermal treatment of lignin and their potential uses in the chemical industry. This poster will highlight some of these potential applications.

¹Goodall, B. L. in *Thermochemical Conversion of Biomass to Liquid Fuels and Chemicals*, Ed. Crocker M. RSC Publishing, Cambridge, pp v-vii, **2010**

²Kunkes, E. L.; Simonetti, D. A.; West, R. M.; Serrano-Ruiz, J. C.; Gartner, C. A.; Dumesic, J. A. *Science* **2008**, *322*, 417

³Sergeev, A. G.; Hartwig, J. F. *Science*, **2011**, *332*, 439, b) Huber, G. W.; Iborra, S.; Corma, A. *Chem. Rev.* **2006**, *106*, 4044.

Kim Jye Lee Chang^{1,2,5}, Susan I.

Blackburn^{1,2}, Geoff Dumsday³, Graeme A. Dunstan^{1,2}, David Batten¹, Peter D. Nichols^{2,4} and Anthony Koutoulis⁵ ¹Energy Transformed National Research Flagship, CSIRO GPO Box 1538, Hobart TAS 7001 ²CSIRO Marine and Atmospheric Research,

GPO Box 1538, Hobart TAS 7001 ³CSIRO Materials Science and Engineering, Private Bag 33, Clayton Sth MDC VIC 3169 ⁴Food Futures National Research Flagship, CSIRO GPO Box 1538, Hobart TAS 7001 ⁵School of Plant Science, University of Tasmania, Private Bag 55, Hobart TAS 7001 The potential of biofuel production from microalgae is of intense interest globally owing to growing concern with rising crude oil availability and prices. Heterotrophic growth of thraustochytrids has potential in co-producing biodiesel to supply the large dependence on liquid fuels for transportation, as well as producing a feedstock of omega-3 long-chain polyunsaturated fatty acids (LC-PUFA), especially docosahexaenoic acid (DHA, 22:6 ω 3) and eicosapentaenoic acid (EPA, 20:5 ω 3), for use in the nutraceutical and other industries.

An alternate way forward -

Heterotrophic cultivation of

thraustochytrids for biofuel

new Australian

and co-products

Enzyme-based bioprocess for production of bioactive peptides from milk proteins

P10

Dominic Agyei and Lizhong He Department of Chemical Engineering, Monash University, Clayton, Victoria 3800, Australia Bioactive peptides are important components of functional foods that can improve human nutrition and well-being. Their health benefits include enhancement of digestive functions, antimicrobial activity combating gastrointestinal diseases, and reduction of risks of cardiovascular disease and obesity [1, 2]. Although milk proteins contain amino acid sequence of bioactive peptides, they are inactive unless being liberated from the proteins.

Production of bioactive peptides from milk can be based on gastrointestinal digestion, milk fermentation, or hydrolysis by proteolytic enzymes [3]. Enzyme-based process is particularly attractive because it has the potential to produce products with a high quality. Such process can either utilize whole cells or purified proteolytic enzymes. However, both whole cells and proteolytic enzymes have poor stability, and enzymebased process is not yet economically feasible.

This study aims to prepare stable biocatalysts for bioactive peptide production using microbial enzymes. Proteolytic enzymes were expressed using *Lactobacillus delbrueckii* subsp. *lactis* 313. We have explored different strategies to make stable biocatalysts using the expressed enzyme. These methods include crosslinking wholes cells, crosslinking enzyme aggregates, and immobilization of enzymes onto different carriers. Activity, stability and reusability of prepared biocatalysts have been compared, and we found that enzyme immobilization may have the potential to economically produce bioactive peptides at a large scale.

Reference

- 1. Muro Urista, C., et al., Review: Production and functionality of active peptides from milk. Food Science and Technology International, 2011. **17**: 293-317.
- 2. Phelan, M. and D. Kerins, The potential role of milk-derived peptides in cardiovascular disease. Food & Function, 2011. **2**: 153-167.
- 3. Meisel, H., Bioactive peptides from milk proteins: a perspective for consumers and producers. Australian Journal of Dairy Technology, 2001. **56**: 83-92.

P11 Biochemical Conversion of Pine Wood

Kirk Torr¹, Karen Love¹, Stefan Hill¹, Blake Simmons² ¹Scion, Rotorua, New Zealand, ²Joint BioEnergy Institute, Emeryville, CA, USA

Feedstock pretreatment to improve enzyme accessibility is a key requirement for efficient biochemical conversion of softwoods to fermentable sugars and lignin. Certain types of ionic liquids (ILs) have been shown to be effective solvents for pretreating lignocellulosic materials to enhance cellulose saccharification yields. These ILs can disrupt the hydrogen bonding network between cellulose molecules and open up the cell wall structure, thereby rendering the pretreated biomass more susceptible to enzymatic hydrolysis. In this study the impact of pretreating Pinus radiata wood with 1-ethyl-3-methylimidazolium acetate/chloride ([EMIM][OAc] and [EMIM][CI]) at temperatures ranging from 80 °C to 120 °C was investigated. The rate and extent of cellulose hydrolysis was monitored by measuring glucose released upon saccharification with commercial enzymes. Increasing the pretreatment temperature led to improvements in enzymatic digestibility with [EMIM][OAc] performing better than [EMIM][CI]. Glucan conversions were improved from 6% for untreated wood, to 50% and 84% after [EMIM][CI] and [EMIM][OAc] pretreatment at 120 °C, respectively. Changes in chemical composition and cellulose crystallinity were assessed using wet chemistry and solid-state ¹³C NMR spectroscopy. Both ILs caused only minor changes in the chemical composition of the wood. [EMIM][OAc] disrupted cellulose crystallinity whereas [EMIM][CI] did not. A Simons' staining method was used as a semi-quantitative measure of overall accessible surface area. The amount of dye adsorbed by the pretreated samples correlated with the efficiency of glucan conversion. Our results suggest accessible surface area, rather than cellulose crystallinity or lignin removal, is a key factor in enzymatic digestibility of IL pretreated pine wood.

Pretreatment of sugarcane bagasse using acidified aqueous ionic liquid solutions **Zhanying Zhang**, Ian O'Hara, William Doherty Syngenta Centre for Sugarcane Biofuels Development, Centre for Tropical Crops

Development, Centre for Tropical Crops and Biocommodities, Queensland University of Technology, Australia Ionic liquids (ILs) are promising solvents for processing lignocellulosic biomass to produce fermentable sugars. However, the cost related to IL process is high, which limits their industrial application. One of the major costs is from recovery and recycle of ILs, which require the reduction of water content in ILs to < 1% in order to maintain ILs' processing ability. Our recent study shows that sugarcane bagasse can be pretreated effectively at 130 °C for 30 min with acidified 1-butyl-3-methylimidazolium chloride (BMIMCI) solutions in the presence of significant quantities (10 - 30%) of water. The subsequent glucan enzymatic digestibilities of pretreated sugarcane bagasse samples are $\ge 94\%$. The high glucan digestibilities are attributed to delignification, xylan removal, defibrillating and disintegrating the bagasse fibre bundles. The addition of water to ILs for pretreatment of lignocellulosic biomass could significantly reduce IL solvent costs and allow for increased biomass loadings, making the pretreatment by ILs a more economic proposition.

P12

Processing Microalgal Suspensions through High Pressure Homogenization **Benjamin Yap**¹, Geoff Dumsday², Peter Scales¹, Gregory Martin¹ ¹ Particulate Fluid Processing Centre, Department of Chemical and Biomolecular Engineering, The University of Melbourne ² CSIRO Materials Science and Engineering

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The mass culture and processing of microalgae for biofuel production is as much a chemical engineering problem as it is a biological challenge. Innovations in process engineering guided by a consideration of the overall energy balance are essential to facilitate this process from the lab-bench to the marketplace. Of the many downstream processes involved, cell disruption is of particular importance as it is the first unit operation post-biomass harvest and dewatering. The objective of cell disruption is to make intracellular products accessible for recovery, separation and purification. To date, the application of process-scale cellular lipid recovery with minimal energy input remains a major processing challenge. This project investigates the use of high pressure homogenization as the cell disruption unit operation of choice in the recovery of intracellular lipids from microalgae. This process has been widely used in the field of microbial biotechnology due to its scalability and many processing advantages. However, it has mostly been limited to systems involving common protein expression hosts such as yeast and E. coli. In this project, the relationship between physical properties and rupture behaviour of selected microalgal species are examined as a function of nitrogen availability during growth. Nitrogen depletion has been proposed as a key strategy to accumulate neutral lipids, mainly triacylglycerols (TAGs) which are fuel precursors. However, changes in cell mechanical properties as a result of nitrogen depletion are still poorly characterized. The processing challenges surrounding homogenization and the physical implications of nitrogen depletion will be better characterized through this project.

Lignocellulosic biomass is likely to be a major source of the carbon needed to synthesize future chemical fuels. Biomass consists predominantly of a heterogeneous matrix of polysaccharides, which are not readily available for bioconversion due to the recalcitrant nature of the cell wall (Burton et al, 2010). Existing methods for processing biomass to increase the availability of cell wall polysaccharides are summarized here. Pre-treatment techniques alter the biomass by using physical, chemical (such as dilute sulphuric acid) and/or thermal means. Next, an enzymatic cocktail is used to increase the liberated monosaccharides for fermentation. Sugar composition is dependent not only on the biomass used, but also on the methods in which it was conditioned. The final fermentation step converts the monosaccharides into ethanol. The feedstocks we are working with include monocot and dicot plants encompassing great diversity in cell wall composition. We are testing and optimizing pre-treatment and fermentation methods that are applicable to our biomass feedstocks of interest. Fermenting microorganisms are being investigated that are able to ferment both hexose and pentose sugars liberated from the feedstock.

Burton, R.A., Gidley, M.J. and Fincher, G.B. (2010) Heterogeneity in the chemistry, structure and function of plant cell walls. *Nature Chemical Biology*, 6, 724-732.

Field to Fuel: Conversion of
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P14

P16

Hydrothermal Liquefaction of Bagasse-Derived Black Liquor for Fuels and Chemicals

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Enhancement of enzyme Depart

stability by encapsulation with silica yolk-shell spheres

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Fractionation of sugarcane bagasse followed by the separate thermochemical treatment of the pulp and black liquor (i.e. lignin and hemicellulose-rich) components is currently being investigated as a means of obtaining high yields of biofuels and chemicals. This paper reports on the base-catalysed hydrothermal liquefaction (termed here as liquefaction) of the black liquor component. The effects of liquefaction conditions on the resulting bio-oil product have been analysed in terms of yield, elemental composition, heating value, functional groups, chemical groups and chemical constituents. Liquefaction of black liquor produced oil yields of up to 100% by mass on lignin content, significantly higher than the 20-25% reported in the literature for liquefaction of purified lignin. This apparently high yield is due to the contributing effects of hemicellulose and other organic components present in the black liquor. Liquefaction temperature was found to have a significant effect on the number of chemical constituents and oil stability. Liquefaction residence times of less than 15 minutes resulted in the highest measured proportions of monomeric species. Oils derived from the liquefaction of black liquor contain significantly higher concentrations of phenolic compounds with a reduction of aliphatic and methoxy structures compared with oils derived from the liquefaction of unfractionated bagasse.

Further optimisation trials are to be conducted in order to increase C/O ratio in the resultant oil and hence improve the heating value. The alternative use of these oils as chemical feedstocks is also being investigated.

Enzyme immobilization is essential to enhance stability and reusability of enzymes for their biocatalysis application. Among different approaches used for enzyme immobilization, enzyme encapsulation offers attractive advantages including separation of enzymes from external environment and retaining enzyme structure and activity [1-3].

In this study, a new two-step soft templating approach has been established to encapsulate enzyme lipase with a porous silica layer. Lipase was firstly immobilized onto an epoxy functionalized silica nanoparticle. A silica layer was then deposited onto lipaseimmobilized silica nanoparticle, followed by thermal treatment in alkaline condition to make pores within the silica layer. Essentially, the silica encapsulation layer is substratepermeable while isolating the enzyme from external hazard environment such as exposure to proteases. Activity and stability of encapsulated lipase were compared with those of free lipase and lipase immobilized on silica nanoparticle. We found that encapsulation with the porous silica layer has significantly enhanced enzyme stability against thermal denaturation and proteolysis, while retaining its activities for hydrolysis reaction. Compared to conventional sol-gel methods, the method described here can make uniform enzyme encapsulation with controllable particle size and defined pore structure of silica layer. This method may find application in industrial usage of enzymes where isolation of enzyme from external hazard environment is needed.

D17	Production of hydrogen from
P17	renewables using catalysis

Upgrade of Biomassderived Levulinic Acid via Ru/C catalyzed Hydrogenation to γ-Valerolactone in Aqueous-Organic-ILs Multiphase Systems. Marina Gottardo, Maurizio Selva, Alvise Perosa

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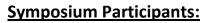
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Hydrogen, a clean fuel emitting only water when combusted, is in growing demand due to technological advancements made in the fuel cell industry. Unfortunately, at present 95% of hydrogen is produced from fossil fuels. In this work, a series of Pt catalysts supported on alumina that was doped with different amounts of CeO₂ were developed, characterized and tested in the aqueous-phase reforming (APR) of glycerol to H₂. Catalyst 3Pt/3CeAl, in which the support contained 3 wt% CeO₂ and the Pt loading was 3 wt%, showed the highest active surface area (4.3 m²/g) and metal dispersion (58%). It also showed the highest carbon conversion to gas (85%) and the highest hydrogen yield (80%) for a feedstock of 1 wt% glycerol in water at 240 °C and 40 bar. A ceria–alumina catalyst with only 1 wt% Pt, 1Pt/3CeAl, showed higher H₂ selectivity and carbon conversion to gas, as well as much lower CH₄ yield, than 3Pt/Al, clearly demonstrating that doping the support with 3 wt% CeO₂ improved the APR of glycerol. No CO was observed in the product gas, meaning that it could potentially be used directly in a PEM fuel cell. Thus including ceria in the alumina catalyst support enhanced both the activity and selectivity towards H₂ of a Pt catalyst for the APR of glycerol.

Levulinic acid (LA) can be cheaply produced from lignocellulosic materials via biological or chemical conversions, and thanks to its dual functionality, LA is a precursor for a variety of useful intermediates in both pharma and food sectors.(1) Not by chance, the US Department of Energy has recently classified LA among the twelve most attractive biobased-chemicals.(2)

In this study, the application of a liquid triphase system made by an aqueous phase, an organic phase, and an ionic liquid is reported for the catalytic hydrogenation/dehydration of levulinic acid to γ -valerolactone. The results demonstrate that by operating at 100-150 °C and 35 atm of H2, both in the presence of Ru/C or of a homogeneous Ru precursor, the use of the triphase system designed to match the investigated reaction allows: (1) to obtain up to quantitative conversions and 100% selectivity towards the desired product; (2) to recover the product by simple phase-separation, and (3) to preerve the catalyst activity for *in situ* recycles without loss of metal. The global sustainability of this chemical transformation has been enhanced by improving efficiency, product isolation, and catalyst recycle.

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