Nanocatalysts from Ionic Liquid Precursors for the Direct Conversion of CO_2 to Hydrocarbons

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Introduction

The direct conversion of carbon dioxide (CO_2) into hydrocarbons in the gasoline range (C_5-C_{11}) is a highly desirable process as a sustainable production route and it provides a key solution to managing the current CO_2 waste emissions. The reaction proceeds *via* two main consecutive reactions: Reverse Water Gas Shift (RWGS) to produce CO followed by the further conversion of CO to hydrocarbons *via* the Fischer–Tropsch reaction[2]. This process is achieved by a multifunctional iron-based catalyst supported on zeolites providing three types of active sites (Fe_3O_4, Fe_5C_2 and acid sites), which cooperatively catalyse a tandem reaction[1].

To date, attempts at synthesising a suitable catalyst for the direct hydrogenation reaction follow a conventional precipitation procedure, whereby Iron Oxide Nanoparticles (*IONs*) are produced and then embedded within a zeolite structure by granule mixing. This method provides no control over the size and shape of the IONs formed; a characteristic of imperative importance due to its significant effect on the hydrocarbon product distribution obtained. In our novel approach, ionic liquids are utilised for the synthesis of the *IONs* resulting in better control over size and morphology of the nanostructured material, and therefore, better conversion and selectivity towards gasoline range hydrocarbons.

Materials and Methods

Fe_3O_4 nanocatalysts have been synthetized by a new ionic liquid-assisted synthesis by heating the reaction medium consisting of the ionic liquid 1-butyl-3-methyl imidazolium bistriflimide, [C_4mim][Tf_2N], oleic acid and iron pentacarbonyl under reflux (Method 1). The precursor iron pentacarbonyl decomposed in a controlled manner by heating the sample up; CO is produced and the iron reacts with residual H_2O in the ionic liquid mixture to result in Fe_3O_4. Following decomposition, the produced magnetite nanoparticles are separated from the reaction medium through application of a neodymium magnet.

Another ionic liquid-assisted synthesis method consisted of heating two iron precursors of Fe (II) and Fe (III) chloride hydrates in a reaction medium of [C_4mim][OAc] ionic liquid (Method 2) w/o ammonia. This method involves calcination of the iron oxide nanoparticles at 420°C under N_2 to prepare crystalline material. The Fe_3O_4 particles were then supported on zeolites by granule mixing Fe_3O_4 particles prepared with the methods above with zeolite HZSM-5 (SiO_2/Al_2O_3 = 300) in a ball mill at a mass ratio of the two components of 1:1.

CO_2 hydrogenation reactions were performed at 320 °C, 3 MPa H /CO 3,3 in a stainless steel fixed-bed reactor with an inner diameter of 15 mm. Typically,1 g of composite catalyst (20–40 meshes) with Fe_3O_4/Zeolite 1⁄4 1/1 (mass ratio) was used. Prior to reaction, the catalyst was in-situ reduced at 350 °C for 8 h in a pure H_2 flow at atmospheric pressure.

All of the products from the reactor were introduced in a gaseous state and analysed with an online gas chromatograph (GC).

Results and Discussion

The ionic-liquid assisted synthesis of a nanocrystalline magnetite precursor showed that ionic liquids provide a controlled precipitation method thanks to their dual functionality as solvent and templating agent. This level of control over the morphology of the produced IONs allows for the selectivity of the hydrocarbon distribution to be directly tailored. Characterization of the prepared catalysts by PXRD (Figure 1) shows the presence of high purity Fe_3O_4, small particle size and good dispersion with the zeolite component for both Method 1 and 2. This has also been confirmed by SEM and TEM analysis. Hydrogen temperature-programmed reduction (H_2-TPR) was used to determine the reducibility of the Fe_3O_4 particles and their hydrogen uptake. As shown in Figure 2, all the catalysts presented two peaks of H_2 consumption, which are assigned to the conversions Fe_3O_4—FeO and FeO—Fe, respectively. It is observed that Fe_3O_4/HZSM-5 (Method 1) is reduced at lower temperature compared to the catalysts prepared with Method 2. All catalysts prepared with ionic liquid-assisted synthesis showed high reducibility in the low temperature region (250-350°C), which correspond to the activation temperature typical of Fischer -Tropsch catalysts.

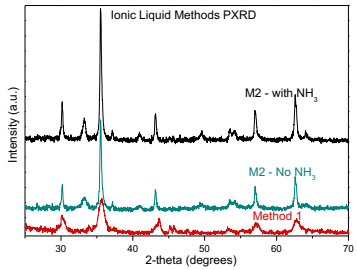

Figure 1. XRD patterns of ionic liquid methods.

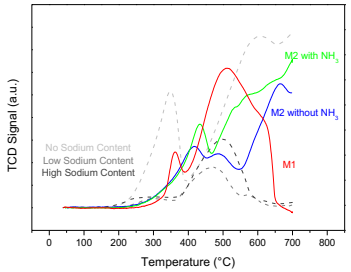

Figure 2. TPR profile of 5%H_2 in Ar from RT to700ºC, 10ºC/min

Significance

We report here on a novel methodology for the controlled synthesis of a Fe_3O_4/HZSM-5 multifunctional catalyst for the direct hydrogenation of CO_2 to gasoline. The catalyst morphology can be tuned by the choice of ionic liquid in the synthetic method and this also affect the selectivity of the reaction. The catalytic testing under industrially relevant conditions resulted in improved selectivity to C5–C11 as well as low CH_4 and CO selectivity

This study provides a new pathway for the synthesis of nanocatalysts to produce liquid fuels by utilising CO_2 and H_2, which may in the future lead to alternative approaches to overcome issues with the intermittency of storing and/or utilising energy from renewable sources (photovoltaics, wind energy).

References

[1] Y. Yuan, S. Huang, H. Wang, Y. Wang, J. Wang, J. Lv, Z. Li, and X. Ma, ChemCatChem 2017, 9, 3144 – 3152

[2] J. Wei, Q. Ge, R. Yao, Z. Wen, C. Fang, L. Guo, H. Xu, J. Sun, Nat Comm, DOI: 10.1038/ncomms15174

UKCC 2020

6th UK Catalysis Conference, 7-9 January 2020
Loughborough, UK

Tuesday, 7th January			
11:00	Registration desk opens at Burleigh Court Hotel		
12:30	Lunch at Holywell Park		
13.50	Welcome – Conference commences at Holywell Park		
	Chair - Catlow		
14.00	Duncan Wass *(Turing Lecture Theatre)*		
14.45	Coffee		
	Session A *(Turing Lecture Theatre)*	*Session B* *(Brunel/Murdoch Lecture Theatre)* CatalysisHub session	*Session C* *(Stephenson Lecture Theatre)*
Chair/IT	Garforth/Deshmukh	Lennon/Shiels	Diez-Gonzalez/Keogh
15.15	K1	O22	O47
15.35		O23	O48
15.55	O1	O24	O49
16.15	O2	O25	K9
16.35	O3	O26	
16.55	Coffee		
Chair/IT	Taylor/Keogh	Kondrat/McDermott	Marr/Isah
17.25	O4	K6	O50
17.45	O5		O51
	Chair - Hardacre		
18.10	Johannes Lercher *(Turing Lecture Theatre)*		
20.00	Dinner		

Wednesday, 8th January			
	Chair - Hutchings		
9.00	Angelika Brückner *(Turing Lecture Theatre)*		
	Session A (Turing Lecture Theatre)	*Session B (Brunel/Murdoch Lecture Theatre)*	*Session C (Stephenson Lecture Theatre)*
Chair/IT	McGregor/Sun RSC INTEREST GROUP SURFACE REACTIVITY & CATALYSIS session	Fan/McDermott	Wood/Tanvir
9.50	K2	O27	O52
10.10		O28	O53
10.30	O6	O29	O54
10.50	Coffee		
Chair/IT	Thompson/Akor	Wu/Keogh	Reina/Hao
11.20	K3	O30	O55
11.40		O31	O56
12.00	O7	O32	O57
12.20	O8	K7	O58
12.40	O9		O59
13.00	Lunch		
	Chair - Manyar		
14.00	José Odriozola *(Turing Lecture Theatre)*		
14.45	Coffee		
	Session A (Turing Lecture Theatre)	*Session B (Brunel/Murdoch Lecture Theatre)*	*Session C (Stephenson Lecture Theatre)*
Chair/IT	Paterson/Yue RSC INTEREST GROUP APPLIED CATALYSIS session	Moody/McDermott	Whiston/Deshmukh
15.15	K4	K8	O60
15.35			O61
15.55	O10	O33	O62
16.15	O11	O34	K10
16.35	O12	O35	
16.55	Coffee		

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Chair/IT	Kroner/Shiels	Berlier/Sun	Raveendran/Akor
17.25	O13	O36	O63
17.45	O14	O37	O64
18.05	O15	O38	O65
18.30	**BP Poster session**		
20.00	Conference Dinner		
Thursday, 9th January			
	Session A (Turing Lecture Theatre)	*Session B (Brunel/Murdoch Lecture Theatre)*	*Session C (Stephenson Lecture Theatre)*
Chair/IT	Beale/Keogh	Artioli/Deshmukh	Upadhyayula/Tanvir
9.00	O16	O39	O66
9.20	O17	O40	K11
9.40	O18	O41	
10.00	K5	O42	O67
10.20		O43	O68
10.40	Coffee		
Chair/IT	Minova/Shiels	Mitchell/Deshmukh	Hintermair/McDermott
11.10	O19	O44	K12
11.30	O20	O45	
11.50	O21	O46	O69
	Chair - Davidson		
12.20	Stewart Parker *(Turing Lecture Theatre)*		
13.05	Closing remarks		

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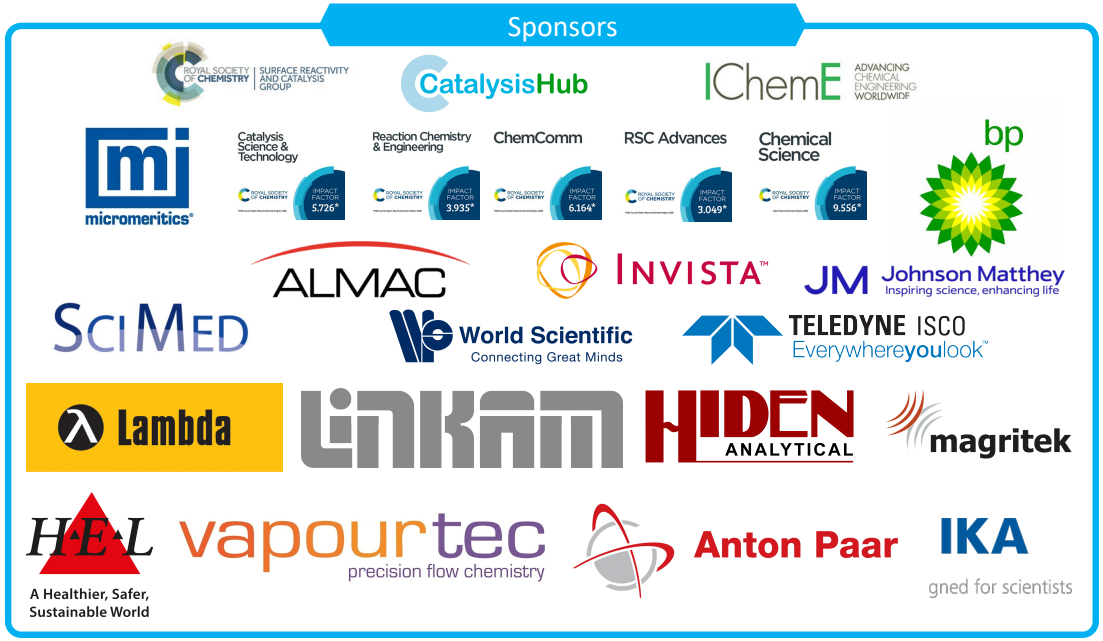

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Dr. Chris Mitchell, Sabic UK
Dr. Paul Collier, Johnson Matthey

List of Talks UKCC 2020

#	Title	Authors
Pl 01	Catalytic Conversion of Renewable Feedstocks to Advanced Synthetic Fuels	Duncan Wass
Pl 02	New strategies for enhancing catalytic rates	Johannes Lercher
Pl 03	Identifying active sites and mechanisms: Opportunities and limitations of in situ and operando spectroscopy in catalysis	Angelika Brückner
Pl 04	From electrons to reactors: The WGS revisited	José Odriozola
Pl 05	What's on your catalyst? Characterization of surface species on Pd and Pt catalysts	Stewart Parker
K 01	Elementary Steps in the Formation of Olefins from Surface Methoxy Groups in ZSM-5 and SAPO-34 Seen by Operando Infrared Microspectroscopy (OIMS)	Ivalina Minova, Santhosh Matam, Alex Greenaway, Richard Catlow, Mark Frogley, Gianfelice Cinque, Paul Wright and Russell Howe
K 02	Simultaneous removal of NO_x and soot particulate from diesel exhaust by in-situ catalytic generation and utilisation of N_2O	Anna Cooper, Catherine Davies, Kate Thompson, Stuart Taylor, Stan Golunski, Maria Bogarra Macias, Omid Doustdar and Athanasios Tsolakis
K 03	Cu-CHA for NO_x Selective Catalytic Reduction: insights into Cu speciation and reaction mechanisms by in situ spectroscopic techniques	Gloria Berlier
K 04	Insights into the CO_2 formation pathways over bimetallic Fischer-Tropsch catalyst for selective production of synthetic diesel: A theoretical & experimental study	Shashank Bahri and Sreedevi Upadhyayula
K 05	Catalytic scissoring of lignin C–C and C–O bonds	Wang, Luo, Liu and Li
K 06	Complex Transition Metal Oxides in Zeolitic Crystalline Forms As Selective Oxidation Catalyst	Wataru Ueda
K 07	Recent Strategies for the Application of Molecular Catalysts to Aqueous Substrates	Andrew Marr
K 08	(Trans)Forming C-N and C-O Bonds with Copper Catalysis	Silvia Díez-González
K 09	Renewable Furan building Block for Biorefinery Applications	Chandrashekhar Rode

K 10	Insight into the mechanism of hybrid non-thermal plasma catalysis system	Xiaolei Fan, Huanhao Chen, Yibing Mu and Chris Hardacre
K 11	MAX Phases and MXenes as Efficient Heterogeneous Catalysts	Shiju Raveendran
K 12	The Effect of a bimetallic Pd/Pt species on Catalyst Activity for Methane Oxidation	Jillian Thompson, Tang Son Nguyen and Andrew Beale
O 01	Investigating Mass Transport in Hollow Mesoporous Zeolites Used for Fluid Catalytic Cracking (FCC)	Luke Forster, Carmine D'Agostino, Xiaolei Fan and Yilai Jiao
O 02	Unravelling mass transport in hierarchically porous catalysts	Carmine D'Agostino, Neil Robinson, Mark Isaacs, Chris Parlett, Karen Wilson and Adam Lee
O 03	Effect of Flue Gas Impurities on the Capture and Utilisation of CO_2 in Superbase Ionic Liquids	Rebecca Taylor, Adam Greer, Helen Daly, Chris Hardacre, Matthew Quesne, Richard Catlow and Johan Jacquemin
O 04	Elucidating the Significance of Nitrate Speciation in Small-pore Cu-containing Zeolitic Materials for the NH_3-SCR reaction	Leila Negahdar, Naomi Omori, Mark Frogley, Fernando Cacho-Nerin, Wilm Jones, Stephen Price and Andrew Beale
O 05	Understanding fluorescence emission dynamics from zeolite crystals to yield insight into framework-adsorbate interactions	Naomi Omori, Alex Greenaway, Paul Collier and Andrew Beale
O 06	Using high throughput experimentation technology to understand effects in large scale reactors	Chris Mitchell and Xander Nijhuis
O 07	Understanding the Mechanochemical Synthesis of Perovskite $LaMnO_3$ and its Catalytic Behavior	Blackmore Rachel, Maria Elena Rivas-Velazco and Peter Wells
O 08	Perovskites decorated with exsolved Ni nanoparticles; operando monitoring of phase and structural changes that dictate redox methane conversion to syngas	Leonidas Bekris, Kalliopi Kousi, Dragos Neagu, Evangelos I. Papaioannou and Ian S. Metcalfe
O 09	Introduction to the High-Resolution Benchtop NMR	Anna Gerdova
O 10	Hydrogen partitioning as a function of time-on-stream for an un-promoted iron-based Fischer-Tropsch synthesis catalyst applied to CO hydrogenation	Alisha Davidson, Paul Webb, Stewart Parker and David Lennon
O 11	Simultaneous In Situ Study of Fischer-Tropsch Catalyst Series by XRD-CT	Jay Pritchard, Andrew Beale and James Paterson
O 12	Observing the Effects of Mn-promotion in Co-based Fischer-Tropsch Catalysts using In-situ Gas Cell Scanning Transmission Electron Microscopy	Matt Lindley, Sarah Haigh and James Paterson
O 13	Tuneable transesterification of glycerol with dimethyl carbonate for synthesis of	Gunjan Deshmukh and Ganapati Yadav

	glycerol carbonate and glycidol on MnO_2 nanorods and efficacy of different polymorphs	
O 14	Modified Red Mud as an Efficient Catalyst for the Synthesis of Glycerol Carbonate by the Transesterification of Glycerol	Bikashbindu Das and Kaustubha Mohanty
O 15	Solar water remediation: efficient removal of ciprofloxacin from aqueous solution using WO_3/TiO_2 photoanodes	Natalia S. Sabatin, Jonas H. Costa, Caio R. Silva, Taicia F. Pacheco, Jose R Guimarães and Claudia Longo
O 16	Alkyl lactate formation from the depolymerization of polylactic acid by metal complex catalysts	Luis Antonio Roman Ramirez, Paul McKeown, Fabio Lamberti, Matthew D. Jones and Joseph Wood
O 17	Hydrocracking of Post-Consumer Polyolefins	Abdulrahman Bin Jumah and Arthur Garforth
O 18	Catalytic Cracking of Polymers over Zeolites in a Twin Screw Compounder	Isaac Campbell, Aleksander Tedstone and Arthur Garforth
O 19	Tetralin and Naphthalene as Exemplar of Poly-aromatic in Heavy Oil Upgrading using $NiMo/Al_2O_3$ Catalyst Heated with Steel Balls via Induction	Abarasi Hart, Mohamed Adam, John Robinson, Sean Rigby and Joseph Wood
O 20	Catalytic upgradation of bio-oil derived phenolic compounds to fuel precursors	Gul Afreen and Sreedevi Upadhyayula
O 21	One-pot transformation of glucose to HMF using a dual acidic catalyst	Firdaus Parveen, Shashank Bahri and Sreedevi Upadhyayula
O 22	Hierarchical Porosity in Zeolite Catalysts for Plastic Hydrocracking	Aleksander Tedstone
O 23	Treatment of high ionic strength wastewater	Xiaoxia Ou, Chris Hardacre, Simon Beaumont, Arthur Garforth, Xiaolei Fan and Helen Daly
O 24	Advances in sustainable catalysis: A computational perspective	Matthew Quesne, Fabrizio Silveri, Nora de Leeuw and Richard Catlow
O 25	Improvement of biocatalyst performance using continuous flow	Sebastian C. Cosgrove, Itziar Peñafiel, Ashley P. Mattey, Nigel S. Scrutton, Nicholas J. Turner
O 26	Management of data objects derived from computational chemistry research for catalysis	Abraham Nieva de la Hidalga, Nitya Ramanan, Brian Matthews
O 27	$CeFeO_x$ catalysts for the total oxidation of propane and naphthalene VOCs: Influence of cerium precursor and molar ratios	Kieran Aggett and Stuart Taylor
O 28	Synthesis and catalytic application of Titanium Silicoaluminophosphate Molecular Sieves	Rekha Yadav, Shashank Bahri, Kanthi Pusapati and Sreedevi Upadhyayula
O 29	Extracting structural information of Au colloids at ultra-dilute concentrations: identification of growth during nanoparticle immobilization	George Tierney, Paul Collier, Nikolaos Dimitratos and Peter Wells

O 30	Metagenomic enzyme discovery to commercial bioprocessing	Thomas Moody, Megan Smyth and Scott Wharry
O 31	Application of Ru-tethered catalyst to generate optically active value added chiral alcohols	Vijyesh Vyas, Richard Knighton, Bhalchandra Bhanage and Martin Wills
O 32	A heterogeneous platform for biocatalytic asymmetric deuteration	Jack Rowbotham, Miguel Ramirez Hernandez, Oliver Lenz, Holly Reeve and Kylie Vincent
O 33	Catalytic and biophysical investigation of rhodium hydroformylase	Hasan Tanvir Imam, Amanda G. Jarvis, Veronica Celorrio, Irshad Baig, Christopher C. R. Allen, Andrew C Marr and Paul C. J. Kamer
O 34	Highly selective reduction of α,β-unsaturated aldehydes, ketones and carboxylic acids under ambient conditions using tetraalkylphosphonium ionic liquids	Stephen Mc Dermott, Kathryn Ralphs, Eadaoin McCourt, Christopher Ormandy, Thiago A. Carneiro de souza, Peter Nockemann, Johan Jacquemin and Haresh Manyar
O 35	Catalytic Hydrogenolysis of 5-hydroxymethylfurfural via Polyphenylene Supported Ruthenium Catalyst	Xuze Guan, Ryan Wang and Qiming Wang
O 36	Electrochemical oxidation of dibenzothiophene and 4,6-dimethyldibenzothiophene on a silver/polyaniline modified electrode	Adeniyi Ogunlaja
O 37	Isolated Pd sites as selective catalysts for electrochemical and direct hydrogen peroxide synthesis	Simon Freakley
O 38	Pt-Pd Single Atom Alloys supported on carbon for the Oxygen Reduction Reaction in combination with XAFS	Sushila Marlow, Ruoyu Xu and Feng Ryan Wang
O 39	A DFT Study of SO_x (x = 0 – 3) and H_2O Reactivity on Pt (111) surface	Nora H. de Leeuw, Cornelia G.C.E. van Sittert, Marietjie J. Ungerer and David Santos-Carballal
O 40	Application of Transient Absorption (TA) techniques for Elucidating Charge Carrier Dynamics in Photocatalysis	Tina Miao, Qiushi Ruan, Paul Donaldson, Richard Catlow and Junwang Tang
O 41	Computational studies on poisoning of Ni catalyst in Methane Steam Reforming	Sai Sharath Yadavalli, Glenn Jones and Michail Stamatakis
O 42	Comparison of DFT Methods for the Description of the Bulk Properties of ZrO_2	Maicon Delarmelina, Matthew Quesne and Richard Catlow
O 43	Product Scale-Up Consideration of the Liquid Phase Hydrogenation of Mandelonitrile	Mairi McAllister, Cedric Boulho, Colin Brennan and David Lennon
O 44	Combined Neutron Scattering and NMR Spectroscopy Studies with EPSR - Examining Benzene Hydrogenation in Porous Media	Terri-Louise Hughes, Chris Hardacre, Marta Falkowska, Daniel Bowron, Tristan Youngs, Markus Leutzsch, Mick Mantle and Andrew Sederman

O 45	Low-cost Optical Sensors for Automation for Process Optimization in Selective Hydrogenation of Alkynes	Nikolay Cherkasov
O 46	Biocatalytic Process Design - Challenges and Solutions	Stefan Mix
O 47	Cascade Conversion of Bio-derived Platform Chemicals with Multifunctional Zeolitic Materials	Samuel Raynes and Russell Taylor
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O 49	The production of volatile fatty acids during hydrothermal conversion of biomass: Influence of feedstock composition and process variables	Jeanine Williams, James Hammerton, Aaron Brown, Gillian Finnerty, Kiran Parmar and Andrew Ross
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O 52	The effect of co-feeding methyl acetate on the H-ZSM-5 catalysed Methanol-to-Hydrocarbons reaction	Andrea Zachariou, Alex Hawkins, Russell Howe, Paul Collier, Iain Hitchcock, Stewart F. Parker and David Lennon
O 53	From starting molecules to steady-state: insights into the evolution of the hydrocarbon pool from methanol over ZSM-5 catalysts	Toyin Omojola
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O 56	Formate coupling revisited – a key step from CO_2 to polymers	Eric Schuler, Shiju Raveendran, Gert-Jan Gruter, Bernd Ensing and Alberto Pérez de Alba Ortíz
O 57	CO_2 valorisation via Reverse Water-Gas Shift reaction using Fe/CeO_2-$Al2O_3$ catalyst: Influence of Cu, Ni and Mo as second metal promoters	Liuqingqing Yang, Pastor Perez Laura and Ramirez Reina Tomas
O 58	Understanding the promoter effect of Cu and Cs over highly effective β-Mo_2C catalysts for the reverse water-gas shift reaction	Qi Zhang, Laura Pastor-Pérez and Wei Jin

O 59	Ce Mn mixed oxides for low temperature emission control catalysts	Nancy Artioli, Ruairi O'Donnell, Maxime Grolleau, Kevin Nawanalfred, Kathryn Ralphs and Haresh Manyar
O 60	Non-thermal Plasma (NTP) Catalysis for CO_2 Utilization	Shaojun Xu, Philip Martin and J. Christopher Whitehead
O 61	Kinetic Studies of Catalytic CO_2 Hydrogenation over Ni Catalyst Activated by Non-thermal Plasma (NTP)	Yibing Mu, Huanhao Chen, Christopher Hardacre and Xiaolei Fan
O 62	Non-Thermal Plasma Assisted CO_2 Hydrogenation over Ru Supported on MgAl Layered Double Hydroxide	Shanshan Xu, Sarayute Chansai, Huanhao Chen, Xiaolei Fan and Christopher Hardacre
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O 64	Opportunities for Catalysis in Methane Valorization - Catalyst Performance Evaluation Using Parallel Fixed Bed Reactor Systems and Data Driven Catalyst Development	Nicolas Popoff, Roel Moonen, Erik-Jan Ras, Math Lambalk and Carlos Ortega
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O 67	Ethanol Upgrading Catalysis for Advanced Biofuels – A Combined Computational and Experimental Study	Andres Richards Gonzales
O 68	Methane dehydro-aromatisation using a dual-phase high temperature hydrogen transport membrane	Matthew West and Danai Poulidi
O69	Effect of steam de-alumination on the interactions of propene with H ZSM-5 zeolites	Alex Hawkins, Andrea Zachariou, Stewart F. Parker, Paul Collier, Iain Hitchcock, Ian P. Silverwood, Russell Howe and David Lennon